ISIS@MACH ITALIA

Papers for MRF' Access Panel

Call Direct Access 24-2

7 & 8 October 2024

Important Note from the User Office

Dear Panel Member,

If you have not already done so, please complete your taxi and accommodation requirements online

Accommodation

Accommodation is arranged for you at *The Hotel degli Arcimboldi* (circle A on the map, page 4) in Viale Sarca 336, Milan. You can reach the IM@IT – University of Milano Bicocca in Piazza della Scienza 1 (circle B on the map, page 4):

- by taxi.
- with a 20-minute walk.
- by the underground line 5 starting from "Bignami "to "Bicocca" stop.
- by tram line 31 from "Bignami" to "Bicocca" stop.

Transport to and from Milan

We are not able to pre-book taxis from Milan airport or train Station to *The Hotel degli Arcimboldi,* so you will therefore need to take your own taxi. Please ensure you obtain a receipt from the driver if you wish to reclaim this cost.

If you do experience problems in finding a taxi you can book a taxi calling the following numbers: **+39 02 8585** or **+39 02 6969**.

MEDIUM-RANGE FACILITY 1 ACCESS PANEL
ROUND 24-2, 7 & 8 October 2024 https://isismachitalia.eu/about/

TIMETABLE

Sunday 6 October

19:00 *Meeting point at The Hotel degli Arcimboldi (see A in Bicocca MAP, page 4)*

19:30 *Dinner at Arcieri Restaurant (see C in Bicocca MAP, page 4)*

Monday 7 October

08:30 Meeting point: reception hall of *The Hotel degli Arcimboldi.* Transport to the Building U1 *(see B in Bicocca MAP, page 4)*.

09:00 – 10:00 MAP Chair Meeting Conference Room T010 Building U1

Refreshments will be available from 08:30.

10:00 – 12.00 MAP Meeting Conference Room T010 Building U1

12:00 – 13:30 *Lunch at "Tutto St'Orto" Restaurant (see D in Bicocca MAP, page 4) and group photo.*

13:30 – 18:30 MAP Meeting Conference Room T010 Building U1

Refreshments will be available at 15:30 and 17:00

19:15 Transport departs outside Building U1 for dropping off at *Primevo Restaurant (see E in Bicocca MAP, page 4).*

19:30 *Dinner at Primevo Restaurant (see E in Bicocca MAP, page 4).*

Tuesday 8 October

08:30 Meeting point: reception of *The Hotel degli Arcimboldi*. Transport to the building U1.

Refreshments will be available at 08:30 and 10:00.

09:00 – 12:30 MAP Meeting Conference Room T010 Building U1

12:30 – 14:00 *Lunch at "Tutto St'Orto" Restaurant (see D in Bicocca MAP, page 4)*

MRF1 Management Team

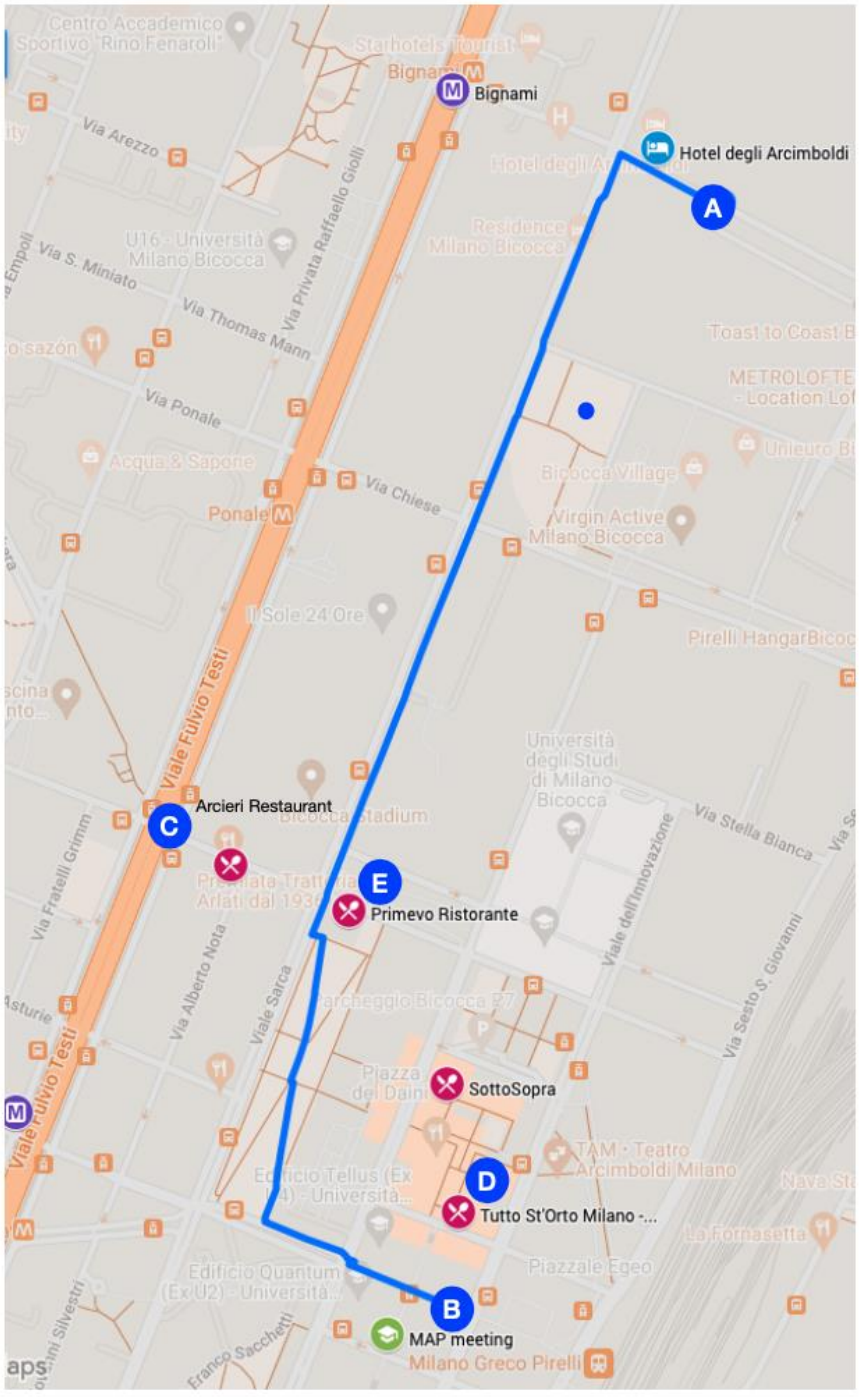
Andreani, Carla	Executive Director (Chair)
Parker, Stewart	MAP Chair
Albani, Giorgia	User Office
Cianchi, Alessandro	Science Director

MAP Members

Parker, Stewart	MAP Chair	ISIS-STFC	UK
Burgio, Lucia	Member	V&A Museum London	UK
Caciuffo, Roberto	Member	INFN - Genova	ITALY
Cazzaniga, Carlo	Member	ISIS-STFC	UK
Cianchi, Alessandro	Science Director	University of Rome Tor Vergata	ITALY
Ciesielski, Artur	Member	Fondation Jean-Marie Lehn	FR
Clifton, Luke	Member	ISIS-STFC	UK
Faraone, Antonio	Member	NIST	US
Frost, Christopher	Secretary	ISIS-STFC	UK
Hyde, Timothy	MAP Member	HRF University of Glasgow	UK
Luiselli, Donata	Member	University of Bologna	ITALY
Salzmann, Christoph	Member	University College London	UK
Scherillo, Antonella	Member	ISIS-STFC	UK
Telling, Mark	Member	ISIS-STFC	UK
Albani, Giorgia	User Office	University of Milano-Bicocca	ITALY

BICOCCA MAP

A: Hotel degli Arcimboldi (Viale Sarca, 336, Milan) - Accommodation
B: MAP Meeting (Piazza della Scienza 1, Building U1, Conference Room T010)
C: Arcieri Restaurant (Viale Fulvio Testi, 220, 20126 Milano MI) – Dinner 6th October
D: Tutto St'Orto (Piazza della Trivulziana, 2, 20126 Milano MI) – Lunch 7th and 8th October
E: Primevo Restaurant (Viale Sarca, 198, 20126 Milano MI) - Dinner 7th October



ISIS@MACH ITALIA
Hub for the analysis of composite materials

Guidelines for the Medium Range Facilities Access Panel (MAPs)
isismachitalia.eu

MAP is an external independent peer review panel responsible for the selection and scientific evaluation of the proposals submitted by potential users requesting access to the suite of MRFs. The panel is composed of 9-13 independent members with a collective scientific knowledge of complex materials and interphases (CMI) and atomic-to-micro analysis and technology, covering the science areas supported by the IM@IT Research Infrastructure.

List of the MRFs suite	*Technical details*
AFM	The Nanowizard II – JPK-Bruker
AFM BIO	AFM/SPM for topological images of biological samples
AFM Raman with Optical Profiler	Raman Spectrometer XploRA Plus
Confocal Microscope 1	Laser Scanning Confocal Microscope Leica TCS SP2
Confocal Microscope 2	Laser Scanning Confocal Microscope Leica TCS SP8
Confocal Microscope 3	Laser lines at 454, 488, 514, 635 nm
Cryogenic Electron Microscopy	CEM in Transmission, Thermo Scientific™ Glacios™
DNA Sequencing NGS	NextSeq 550
Dynamic Mechanical Analyzer	DMA Star Systems – Mettler Toledo
ESCALB QXi	X-ray photoelectron spectrometer XPS, UPS, REELS
FIB-SEM GAIA 3	FIB-SEM with simultaneous milling and EBSD
FT-IR Nexus	Nicolet Nexus 870
FT-IR Nicolet	Endowed with LightDrive Optical Engine components
Fluorescence Microscopy	BX51 microscope
Gas Chromatography - Ion Mobility Spectrometer	To separate & detect the components in sample mixtures
MONeutron	Prototype ground-level-neutron monitoring network
Mass Spectrometer 1	Rapiflex™ MALDI Tissuetyper™
Mass Spectrometer 2	Orbitrap Fusion Tribrid mass spectrometer
Multipurpose X-Ray diffractometer	With WAXS and SAXS
NMR 600 MHz	Bruker Avance III 600 MHz NMR
RETINA	2D/3D X-ray imaging techniques
Raman Confocal Microscope	Microscope inViaTM QontorTM model
SAXS GISAXS	Xenocs XEUSS 3.0
SAXS WAXD	Saxspace Anton-Paar
SEM FEI	SEM FEI QUANTA 200
SEM LEO SUPRA	SUPRA 35 Field Emission SEM
SEM ZEISS GEMINI	FEG-SEM with a nominal resolution of 1.2 nm
SEM ZEISS SIGMA	Scanning electron microscope - field-emission source
SEM&C-AFM & correlative AFM	SEM system with EDS-SPM
SOURIRE	A neutron source - Deuterium-Tritium (D-T) type
Spectrofluorimeter	Varian Eclipse Spectrofluorimeter
TEM FEI	LaB6 source (120 kV) and BF detector and FEI Eagle
TEM High Resolution	ThermoFisher Talos F200X
TEM JEOL	JEOL JEM 2100 Plus with a LaB6 emitter
TLM platform	Microscopy&time-lapse&lab-on-chip and organ-on-chip
UTEM & LUMINAD	The first national Ultrafast TEM
X-Ray diffractometer	Rigaku SmartLab SE
XRD TOMOGRAPHY	RIGAKU Nano3DX

The ***Remit*** of the MAP is:

- To recommend to the Executive Director of IM@IT a balanced science program based upon the criteria of ***scientific excellence and timeliness*** (all within the bounds of technical feasibility and safety implications) and, where appropriate, the potential economic impact and contribution to knowledge exchange and transfer.
- To comment on the appropriateness of the number of instrument days requested for the experiments proposed.
- To identify after each proposal round scientific trends and facility development issues (including software development) which are of relevance to the MRF instrument.

If a panel member is unable to attend, he is requested to notify the panel secretary as soon as possible in advance so that a substitute member can be found if necessary. Written comments are expected from non-attending members. Panel members who are unable to attend in person may attend by Zoom video conference.

Reasonable travel and subsistence costs are reimbursed to members when attending panel meetings.

Panel Working Method and Protocols

MAP members are provided with all the proposals for their panel in advance of the meeting. Each proposal will be assigned to two MAP members who act as primary speakers to give their assessment of the proposal at the MAP meeting. Proposals are then discussed by the MAP, considering any technical issues raised by IM@IT representatives. The MAP should arrive at a grade for each proposal (see ***Proposal Grading and Prioritisation*** in Table 1). The MAP will be notified of the number of days available to them for each instrument being considered, and panels should recommend, based on the days available, which proposals should be awarded instrument time and the number of days to be given.

Comments should be provided by panels to be fed back to proposers, particularly where instrument time is not awarded or significantly reduced.

Panel members should highlight any proposal where they consider there to be ethical issues. This may include unethical practice (*e.g.,* plagiarism), but also where additional protocols may be necessary before an experiment can be allowed to take place (*e.g.,* use of biological material, material from human subjects, genetic modification, etc).

Panel Code of Practice

Panel members should declare all conflicts of interest. Members are expected to leave the room during consideration of these proposals and if proposals from their own departments are being considered. The MAP Chair is responsible for deciding on potential conflicts of interest where these are raised. All papers relating to the proposal review are to be treated as ***confidential*** and should not be discussed outside the meeting; panel discussions and results of the peer review process should also be kept confidential.

Proposal grading and prioritisation

The MAP will peer review all the submitted proposals and agree on an overall grade for each proposal. The grades and an indication of the associated definitions and expected outcomes are given in the table below. Proposals which are scientifically or technically flawed should be rejected and marked X.

<table>
<tr><th>Grade</th><th>Expected Review Outcome</th><th>Definition – for guidance</th></tr>
<tr><td>10</td><td rowspan="2">Instrument time allocation is essential</td><td rowspan="2">Outstanding, World class</td></tr>
<tr><td>9</td></tr>
<tr><td>8</td><td rowspan="2">Instrument time allocation is recommended</td><td rowspan="2">Excellent</td></tr>
<tr><td>7</td></tr>
<tr><td>6</td><td rowspan="2">Instrument time allocation is possible</td><td rowspan="2">Good</td></tr>
<tr><td>5</td></tr>
<tr><td>4</td><td rowspan="4">Instrument time allocation should not be made</td><td rowspan="2">Fair</td></tr>
<tr><td>3</td></tr>
<tr><td>2</td><td>Uncompetitive</td></tr>
<tr><td>1</td><td>Unsatisfactory</td></tr>
<tr><td colspan="3"></td></tr>
<tr><td>R</td><td>Panel would like to see a resubmission with panel comments addressed</td><td>Resubmit</td></tr>
<tr><td>X</td><td>Panel do not want to see a resubmission</td><td>Reject</td></tr>
</table>

IM@IT Access Mechanisms

1. **Access to Medium Range Facilities**

Direct access is suitable for all service, training and instrument time using MRF' equipment. Proposals are submitted to two calls for proposals each year with deadlines in April and October each year. All direct access proposals are peer reviewed by the (MAP). Proposals which are allocated beamtime are scheduled by ISIS scientists normally between 2 months and 4 months after the proposal deadline.

2. **Industrial Collaborative Program**

An Industrial Collaborative program (ICP) is also offered. It is a fast-track route for industries based in Italy to use MRF' equipment for service, training, and instrument time. Requests of time using the ICP route can be submitted at any time. Requests are reviewed by a small panel with appropriate expertise, including the MAP chair, under strict confidentiality rules. Industrial users may also buy beamtime directly by contacting the IM@IT User Office (useroffice@isismachitalia.eu).

IM@IT User Office
Revised: May 18th, 2024

Suite of MRF’ Instrument

MRF' Instrument – AFM

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2024112	Palmioli Dr A.	ITALY	3	*Training on the use of AFM for morphological analysis of amyloids fibrils*
2024115	Minici Dr C.	ITALY	3	*Stiffness characterization of pancreatic ductal adenocarcinoma tissue*
2024122	Scielzo Dr C.	ITALY	3	*Nanomechanical characterization of healhy and pathological lymphoid tissue*
2024147	Lauta Miss F.	ITALY	6	*Exploring surface-induced mechanical forces and macrophage behaviour for microscale interactions with substrate topography*
2024151	Pellegrino Dr L.	ITALY	5	*Investigating microbial colonization and removal on bioinspired wrinkled surfaces*
5 Proposals	Total Requested Days:		20	

MRF' Instrument – AFM BIO

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2024070	Nigro Dr V.	ITALY	2	*Study of morphological changes of soft responsive microgels at solid interfaces through Atomic Force Microscopy*

1 Proposal Total Requested Days: 2

MRF' Instrument – AFM Raman with Optical Profiler

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2024045	Falletta Dr E.	ITALY	3	*Investigating the Corrosive Impact of Chrome-Tanned Semi aniline Calf Leather using RAMAN Spectroscopy and Profilometry*
2024088	Bouzin Dr M.	ITALY	2	*AFM/Raman & TERS Training for the Biophysics Research Group of UNIMIB*
2024089	Marini Dr M.	ITALY	1	*Optical profilometry experiments on two-photon polymerized microlenses*
2024095	Krzystyniak Dr M.	UNITED KINGDOM	2	*Raman determination of molecular composition of industrial-grade polymers for medical applications/*
2024161	Saliu Dr F.	ITALY	3	*UV Oxidation and Chemical Catalysis of PET Plastics for the Generation and Characterization of Uniform Nanoplastics*
2024168	Goletti Prof C.	ITALY	1	*Online training on multiple instrumentation for the Students of the GREENANO Erasmus Mundus Joint Master (EMJM) on Materials Science: event #1 organized by Tor Vergata Unit*

6 Proposals	Total Requested Days:	12

MRF' Instrument – Confocal Microscope 2

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2024148	Izzi Dr M.	ITALY	5	*Investigation of the early-stage interaction between bacterial cells and antimicrobial surfaces*

1 Proposal	Total Requested Days:	5	

MRF' Instrument - Confocal Microscope 3

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2024061	Perelli Cippo Dr E.	ITALY	1	*Study of the bonding of a Diamond detector matrix with the CONFOCAL MICROSCOPE*
2024170	Goletti Prof C.	ITALY	1	*Online training on multiple instrumentation for the Students of the GREENANO Erasmus Mundus Joint Master (EMJM) on Materials Science: event #3 organized by Milano Bicocca Unit*
2 Proposals	Total Requested Days:		2	

MRF' Instrument – DNA Sequencing NGS

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2024042	Lo Vetro Prof D.	ITALY	8	*Exploring environmental dynamics in ancient remains before and after the last glacial maximum using a DNA sequencing*
2024051	Fabbri Dr P.	ITALY	5	*Ancient DNA analysis of a Neolithic human tooth from eastern Sicily using NGS: insights and implications*
2024053	Bertoldi Prof F.	ITALY	5	*Ancient DNA analysis in a Late Bronze age human child from Doghlauri burial ground (Georgia), affected by tuberculosis*
2024078	Rubini Prof M.	ITALY	7	*A multidisciplinary aDNA study of the unique ancient Homo cepranensis petrous bone*
2024079	Piliposyan Prof A.	ARMENIA	5	*Reconstruction of the family relationships, kinship and lineage from Bronze Age collective burials from lake Sevan basin (Armenia)*
2024080	Bertoldi Prof F.	ITALY	5	*Inhumated graves from the eastern Venetic necropolis in Padua: a case study*
2024085	Gentile Prof G.	ITALY	5	*In-depth analysis of ancient DNA from sub-fossil bones of pink iguana from Galápagos islands using DNA sequencing*
2024091	Sonet Mr G	BELGIUM	5	*Exploring the presence of aurochs from the prehistoric period to medieval times using ancient DNA*
2024105	Capomaccio Prof S.	ITALY	5	*Genomic Analysis of an Iron Age Horse: Insights into Ancient Equine Genetics and Modern Conservation Implications*
2024113	Enei Dr F.	ITALY	5	*Genetic characterization of the Medieval community of Santa Severa*
2024146	Scorrano Dr G.	ITALY	5	*Biogeochemical Multi-Instrumental Study of Rocks and Microbial Biomass in Zechstein Salt Deposits of Boulby using DNA Sequencing NGS*
11 Proposals	Total Requested Days:		60	

MRF' Instrument - ESCALB QXi

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2024046	Falletta Dr E.	ITALY	4	*Investigating the Corrosive Impact of Chrome-Tanned Semi aniline Calf Leather using X-ray Photoelectron Spectroscopy (XPS) and Ion Scattering Spectroscopy (ISS)*
2024054	Falletta Dr E.	ITALY	1	*Training on X-ray Photoelectron Spectroscopy (XPS) with ESCALAB QXi X-Ray Photoelectron Spectrometer*
2024067	Falletta Dr E.	ITALY	3	*Advanced Analysis of Surface and Composition Variations in Treated Brass Samples with ESCALAB QXi (XPS)*
2024093	Falletta Dr E.	ITALY	2	*Steel alloys behaviours to corrosion testing for metal accessories quality control with ESCALAB QXi (XPS)*
2024128	Pavan Dr A.	ITALY	5	*Hydrothermal Sustainable Approaches for the Preparation of Carbon Quantum Dots: an XPS Insight into the Effect of Different Carbon Sources*
2024136	Tonelli Dr M.	ITALY	3	*Study of the interactions occurring between halloysite nanotubes and sodium hexametaphosphate in alginate biocomposites towards the preparation of anisotropic structures*
2024167	Tonelli Dr M.	ITALY	1	*Study of functionalized Halloysite nanotubes*

7 Proposals Total Requested Days: 19

MRF' Instrument - FIB-SEM GAIA 3

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2024123	Pierozzi Mr A.	IRELAND	4	*Ca-Mg-Fe carbonates alteration of basalts at Sverrejfellet Volcano (Svalbard, Norway)*
2024124	Capitani Prof G.	ITALY	3	*SEM imaging, FIB manipulation and TEM characterization of mineralized granules ("spherites") found in the honeybees' midgut*
2024145	Lenaz Prof D.	ITALY	3	*Thallium and As in sphalerite from Raibl mine (NE Italy): Nano-inclusions or Zn-vicariant?*

3 Proposals Total Requested Days: 10

MRF' Instrument - FT-IR Nexus

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2024047	Falletta Dr E.	ITALY	1	*Investigating the Corrosive Impact of Chrome-Tanned Semi aniline Calf Leather using FT-IR Spectroscopy*
2024084	Gentile Prof G.	ITALY	3	*In-depth analysis of ancient DNA from sub-fossil bones of pink iguana from Galápagos islands using FT-IR spectroscopy*
2024101	Falletta Dr E.	ITALY	1	*Stripping of surface treatments from ABS/metal composites: an FT-IR investigation*
2014109	Gentile Prof G.	ITALY	3	*Multi-instrumental characterization of Oniscidean isopods to establish the nature and function of their cuticle and tubercles: FTIR measurements*
2024157	Carosi Dr M.	ITALY	1	*Multi-instrumental characterization of primate baculum and baubellum bone tissues to support functional hypotheses: FTIR measurement case*

5 Proposals Total Requested Days: 9

MRF' Instrument - FT-IR Nicolet

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2024040	Lo Vetro Prof D.	ITALY	2	*Exploring environmental dynamics in ancient remains before and after the last glacial maximum using FT-IR measurements*
2024063	Pomarico Prof G.	ITALY	3	*IR spectroscopic analysis of electropolymerized Corroles*

2 Proposals Total Requested Days: 5

MRF' Instrument - Fluorescence Microscopy

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2024083	Prioriello Dr A	ITALY	1	*Fluorescence Microscopy investigation of Polyurethane-Single Wall Carbon Nanotubes composite*
2024130	Vercelli Dr B.	ITALY	5	*Hydrothermal Sustainable Approaches for the Preparation of Carbon Quantum Dots: a Photo- Physical Insight into the Effect of Different Carbon Sources*
2 Proposals		Total Requested Days:	6	

MRF' Instrument – Gas Chromatography - Ion Mobility

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2024142	Holtvoeth Dr J.	UNITED KINGDOM	2	*Biogeochemical Multi-Instrumental Study of Rocks and Microbial Biomass in Zechstein Salt Deposits of Boulby using Gas Chromatography – Ion Mobility Spectrometer*
1 Proposal		Total Requested Days:	2	

MRF' Instrument – MONeutron

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2024094	Pintacuda Dr F.	ITALY	90	*A power transistor monitor to record high energy neutrons*
1 Proposal		Total Requested Days:	90	

MRF' Instrument – Mass Spectrometer 2

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2024041	Lo Vetro Prof D.	ITALY	3	*Exploring environmental dynamics in ancient remains before and after the last glacial maximum using mass spectrometry measurements*
2024077	Rubini Prof M.	ITALY	2	*A multidisciplinary proteomic study of the unique ancient Homo cepranensis petrous bone using Mass Spectrometer instrument*
2024125	Brocca Prof S.	ITALY	10	*Assessing interaction of alpha-synuclein with copper*
2024126	Biasini Prof E.	ITALY	10	*Characterization of Protein Folding Intermediates of the Cellular Prion Protein by Native Mass Spectrometry*
2024127	Gelain Dr F.	ITALY	10	*Self-assembling peptidonucleic acids as novel biomaterials for tissue engineering*
2024144	Scorrano Dr G.	ITALY	2	*Biogeochemical Multi-Instrumental Study of Rocks and Microbial Biomass in Zechstein Salt Deposits of Boulby using the Mass Spectrometer 2*

6 Proposals Total Requested Days: 37

MRF' Instrument – Multipurpose X-Ray diffractometer

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2024038	Lo Vetro Prof D.	ITALY	3	*Exploring environmental dynamics in ancient remains before and after the last glacial maximum using X-ray Diffraction*
2024092	Taddei Prof M.	ITALY	5	*In situ powder X-ray diffraction of flexible metal-organic frameworks during gas adsorption*
2024110	Gentile Prof G.	ITALY	3	*Multi-instrumental characterization of Oniscidean isopods to establish the nature and function of their cuticle and tubercles: XRD measurements*
2024141	Minniti Dr T.	ITALY	2	*Biogeochemical Multi-Instrumental Study of Rocks and Microbial Biomass in Zechstein Salt Deposits of Boulby using the Multipurpose X-ray Diffractometer*
2024156	Carosi Dr M.	ITALY	3	*Multi-instrumental characterization of primate baculum and baubellum bone tissues to support functional hypotheses: XRD measurement case*

5 Proposals Total Requested Days: 16

MRF' Instrument - NMR 600 MHz

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2024131	Negri Dr I.	ITALY	3	*Study of the chemical and structural characteristics of biodegradable microplastics in contaminated honey bees through NMR spectroscopy*
2024159	Zappia Dr S.	ITALY	1	*Metal NP supported Over Microporous Polymelamine Framework for the CO2 absorption and reduction*
2 Proposals		Total Requested Days:	4	

MRF' Instrument – RETINA

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2024044	Cianchi Prof A.	ITALY	3	*Study of TArget Resistance to damaGE and contamination (TARGET) by means XCT and XRF measurements*
2024059	Falletta Dr E.	ITALY	2	*Investigating the Corrosive Impact of Chrome-Tanned Semi aniline Calf Leather using XRF*
2024064	Colombo Dr E.	ITALY	4	*Ion Migration Studies in Polymer Electrolyte Membranes Fuel Cells*
2024076	Rubini Prof M.	ITALY	1	*A multidisciplinary proteomic study of the unique ancient Homo cepranensis petrous bone using RETINA*
2024133	Rastelli Mr D.	ITALY	3	*Characterization of Silicon Avalanche Photon Detectors (APD) for X-ray Fluorescence analysis*
2024139	Minniti Dr T.	ITALY	4	*Biogeochemical Multi-Instrumental Study of Rocks and Microbial Biomass in Zechstein Salt Deposits of Boulby using RETINA*
2024154	Carosi Dr M.	ITALY	5	*Multi-instrumental characterization of primate baculum and baubellum bone tissues to support functional hypotheses: micrometric QCT-XRF measurement*

7 Proposals Total Requested Days: 22

MRF' Instrument - Raman Confocal Microscope

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2024086	Falletta Dr E.	ITALY	3	*Advanced Analysis of Surface and Composition Variations in Treated Brass Samples with Raman Confocal Microscope*
2024108	Gentile Prof G.	ITALY	3	*Multi-instrumental characterization of Oniscidean isopods to establish the nature and function of their cuticle and tubercles: Raman measurements*
2024119	Aglietto Dr I.	CZECH REPUBLIC	3	*RAMAN confocal microscopy characterization of innovative graphene-based inks for multifunctional applications*
2024132	Falletta Dr E.	ITALY	1	*Stripping of surface treatment from ABS articles investigation with confocal RAMAN microscopy*
2024158	Carosi Dr M.	ITALY	1	*Multi-instrumental characterization of primate baculum and baubellum bone tissues to support functional hypotheses: Raman measurement case*
2024164	Saliu Dr F.	ITALY	3	*"Dyes of Valor: Uncovering the Colorful History of Alpine Military Uniforms*
2024166	Minniti Dr T.	ITALY	4	*Characterisation of lipid multilayers mimicking the outer skin layers by Confocal Raman Microscopy*

7 Proposals Total Requested Days: 18

MRF' Instrument - SAXS GISAXS

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2024039	Lo Vetro Prof D.	ITALY	3	*Exploring environmental dynamics in ancient remains before and after the last glacial maximum using SAXS and WAXS*
2024050	Fabbri Dr P.	ITALY	1	*Ancient DNA analysis of a Neolithic human tooth from eastern Sicily with SAXS GISAXS: insights and implications*
2024052	Prioriello Dr A.	ITALY	2	*Training on the use of SAXS-GISAX to investigate Polyethylene-Single Wall Carbon Nanotubes composite*
2024071	Nigro Dr V.	ITALY	2	*Study of morphological changes of soft responsive microgels at solid interfaces using SAXS GISAXS*
2024072	Arduini Prof F.	ITALY	2	*SAXS characterisation of inks based on carbon black/Prussian Blue nanocomposites for paper- based electrochemical (bio)sensors*
2024138	Cockell Prof C.	UNITED KINGDOM	3	*Biogeochemical Multi-Instrumental Study of Rocks and Microbial Biomass in Zechstein Salt Deposits of Boulby using SAXS GISAXS*
2024155	Carosi Dr M.	ITALY	3	*Multi-instrumental characterization of primate baculum and baubellum bone tissues to support functional hypotheses: SAXS measurement case*
2024165	Naved Dr M.	INDIA	3	*Probing Structural Dynamics of Deep Eutectic Solvents Confined within the Pores of Bio-Metal- Organic Frameworks Using SAXS under Extreme Conditions*
8 Proposals		Total Requested Days:	19	

MRF' Instrument – SAXS WAXD

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2024069	Amazio Miss P.	ITALY	3	*SAXS/WAXD characterization of EoL fibers, and recycled non-wovens and compounds*
2024098	Chimenti Dr S.	SPAIN	3	*Structural characterization of hydrophobic/oleophobic coatings by SAXS WAXD*
2024118	Aglietto Dr I.	CZECH REPUBLIC	3	*SAXS WAXD characterization of innovative graphene-based inks for multifunctional applications*

3 Proposals	Total Requested Days:	9	

MRF' Instrument - SEM FEI

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2024068	Amazio Miss P.	ITALY	2	*SEM characterization of EoL fibers, and recycled non-wovens and compounds*
2024106	Gentile Prof G.	ITALY	4	*Multi-instrumental characterization of Oniscidean isopods for establish the nature and function of their cuticle and tubercles: SEM-EDX measurements*
2024017	Aglietto Dr I.	CZECH REPUBLIC	2	*SEM characterization of innovative graphene-based inks for multifunctional applications*
3 Proposals		Total Requested Days:	8	

MRF' Instrument – SEM ZEISS GEMINI

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2024056	Perelli Cippo Dr E.	ITALY	1	*Test of radiation hardness and material degradation of boron GEM foils using SEM ZEISS GEMINI*
2024121	Negri Dr I.	ITALY	3	*Identification and characterization of the size and shape of biodegradable microplastics in contaminated honey bees through SEM/EDX*
2024135	Colombo Prof A.	ITALY	2	*Fiber fractography of climbing ropes*
2024149	Limonta Dr M.	FRANCE	3	*Geochemical composition and microfeatures of single detrital biotite and carbonate grains in the provenance study of the Bengal Fan turbidites (IODP Expedition 354).*
2024160	Zappia Dr S.	ITALY	1	*SEM and SEM EDX analysis of Metal NP supported Over Microporous Polymelamine Framework for the CO2 absorption and reduction*

5 Proposals Total Requested Days: 10

MRF' Instrument - SEM ZEISS SIGMA

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2024065	Falletta Dr E.	ITALY	2	*Training on Scanning electron Microscopy with SEM ZEISS SIGMA*
2024074	Arduini Prof F.	ITALY	1	*FE-SEM characterisation of paper-based electrochemical (bio)sensors based on carbon black/Prussian Blue nanocomposites inks*
2024102	Falletta Dr E.	ITALY	2	*Steel alloys behaviours to corrosion testing for metal accessories quality control with SEM ZEISS SIGMA*
2024103	Falletta Dr E.	ITALY	2	*Stripping of surface treatment from ABS articles investigation with SEM ZEISS SIGMA*
2024104	Falletta Dr E.	ITALY	2	*Wire bending and adhesion investigation with SEM ZEISS (Scanning Electron Microscopy)*
2024169	Goletti Prof C.	ITALY	1	*Online training on multiple instrumentation for the Students of the GREENANO Erasmus Mundus Joint Master (EMJM) on Materials Science: event #2 organized by CSGI Unit*

6 Proposals Total Requested Days: 10

MRF' Instrument - SEM&C-AFM with Optical Profiler

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2024043	Cianchi Prof A.	ITALY	3	*Study of TArget Resistance to damaGE and contamination (TARGET) by means SEM and profilometry measurements*
2024048	Falletta Dr E.	ITALY	3	*Investigating the Corrosive Impact of Chrome-Tanned Semi aniline Calf Leather using SEM with correlative EDX*
2024055	Battisti Dr F.	ITALY	3	*Silver diffusion in Glydcop grain boundaries for ultra high vacuum applications*
2024058	Pietrosanti Miss V.	ITALY	2	*How optical and electron microscopies can help investigating radiation-damaged electronic devices*
2024060	Basoli Dr F.	ITALY	3	*Continuation of Morphological characterisation and compositional analysis of SiCa based Bioglasses using SEM-EDX*
2024066	Falletta Dr E.	ITALY	3	*Advanced Analysis of Surface and Composition Variations in Treated Brass Samples with Scanning Electron microscopy (SEM) and Profilometry*
2024087	Loreto Prof R.	ITALY	2	*Archaeometric study of iron ingots from the North Arabian oasis of Dumat al-Jandal using SEM- EDX*
2024090	Baldassarre Dr L.	ITALY	4	*SEM-EDX characterization of polymeric dosimeters for fast neutrons*
2024099	Chimenti Dr S.	SPAIN	3	*Morphological characterization of hydrophobic/oleophobic nanoparticles and coatings by SEM@C-AFM*
2024143	Scacco Dr V.	ITALY	2	*Surface characterization of amorphous GaN and GaP thin films deposited by magnetron sputtering*
10 Proposals		Total Requested Days:	28	

MRF' Instrument - SOURIRE

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2024057	Perelli Cippo Dr E.	ITALY	1	*Test of radiation hardness and material degradation of boron GEM foils at the SOURIRE Neutron Source*
2024062	Perelli Cippo Dr E.	ITALY	1	*Characterisation of a Diamond detector matrix at the SOURIRE Neutron Source*

2 Proposals Total Requested Days: 2

MRF' Instrument - Spectrofluorimeter

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2024129	Vercelli Dr B.	ITALY	5	*Hydrothermal Sustainable Approaches for the Preparation of Carbon Quantum Dots: an Insight into the Effect of Different Carbon Sources on the Photo-Emission Behavior*
1Proposal		Total Requested Days:	5	

MRF' Instrument - TEM FEI

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2024075	Arduini Prof F.	ITALY	2	*TEM characterisation of inks based on carbon black/Prussian Blue nanocomposites for paper- based electrochemical (bio)sensors*
2024096	Chimenti Dr S.	SPAIN	5	*TEM characterization of water dispersed fluorine-free polymer based nanoparticles*
2024111	Gentile Prof G.	ITALY	2	*Multi-instrumental characterization of Oniscidean isopods to establish the nature and function of their cuticle and tubercles: TEM measurements*
2024116	Aglietto Dr I.	CZECH REPUBLIC	3	*TEM characterization of innovative graphene-based inks for multifunctional applications*

4 Proposals Total Requested Days: 12

MRF' Instrument - TEM High Resolution

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2024073	Pasqual Dr U.	ITALY	2	*High resolution TEM characterization of metallic nanoparticles supported on perovskite oxides as electrocatalysts for water splitting*
2024114	Credi Dr C.	ITALY	4	*Advanced Nanoparticle Probes for Early Disease Detection in Biofluids*
2024120	Ferretti Dr A.	ITALY	3	*Morphological and microanalytical characterization of electronspun porous high-entropy spinel oxide nanofibers containing the following ions: Fe, Co, Ni, Zn, and Nd*
2024137	Migliorini Dr F.	ITALY	2	*Effect of laser irradiation on soot nanostructure*
2024140	Rizzo Dr F.	ITALY	4	*Innovative strategies for Improving the pinning efficiency of YBCO thin films*
2024152	Muhyuddin Dr M.	ITALY	3	*Influence of pyrolysis conditions on the Mn-incorporated Fe-N-Cs*
2024162	Gabbani Dr A.	ITALY	4	*High Resolution Transmission Electron Microscopy on Yb-doped CsPbX3 perovskite Nanocrystals*

7 Proposals Total Requested Days: 22

MRF' Instrument - TEM JEOL

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2024150	Ferretti Dr A.	ITALY	3	*HRTEM,STEM and STEM EDX Noble metal nanoparticles (NPs) supported on 3D metal/metal oxide framework Characterization*
2024163	Casetta Dr M.	ITALY	5	*Nanocrystals evolution in volcanic glasses*

2 Proposals Total Requested Days: 8

MRF' Instrument – X-Ray diffractometer

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2024100	Amendola Prof V.	ITALY	5	*Porous organic materials for CO2 separation from gaseous mixtures*
2024134	Mastronardi Mr G.	ITALY	5	*Powder X-ray Diffraction experiment on MOF*

2 Proposals Total Requested Days: 10

MRF' Instrument - XRD TOMOGRAPHY

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2024049	*Fabbri Dr P.*	ITALY	1	*Ancient DNA analysis of a Neolithic human tooth from eastern Sicily XRD TOMOGRAPHY: insights and implications*
2024081	Falletta Dr E.	ITALY	1	*Training on XRD TOMOGRAPHY (CNR-IPCB)*
2024082	Falletta Dr E.	ITALY	4	*Wire bending and adhesion investigation with XRD TOMOGRAPHY (CNR-IPCB)*
2024097	Chimenti Dr S.	SPAIN	5	*Analysis of the distribution and morphology of hydrophobic/oleophobic coatings onto textiles by XRD tomography*
2024107	Gentile Prof G.	ITALY	5	*Multi-instrumental characterization of Oniscidean isopods to establish the nature and function of their cuticle and tubercles: XCT measurements*
2024153	Carosi Dr M.	ITALY	5	*Multi-instrumental characterization of primate baculum and baubellum bone tissues to support functional hypotheses: nanometric QCT measurement*

6 Proposals Total Requested Days: 21

AFM

Experiment Proposal

Experiment number GP2024112

Principal investigator	Dr Alessandro Palmioli, University of Milano Bicocca, ITALY
Co-investigator	Professor Cristina Airoldi, University of Milano-Bicocca, ITALY
Co-investigator	Dr Valeria Cassina, University of Milan-Bicocca, ITALY
Co-investigator	Professor Francesco Mantegazza, Universita&039; Milano-Bicocca, ITALY
Co-investigator	Mr Luca Moretti, University of Milano-Bicocca, ITALY
Co-investigator	Dr Linda Molteni, Università degli Studi di Milano-Bicocca, ITALY
Co-investigator (*)	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Experiment title	Training on the use of AFM for morphological analysis of amyloids fibrils
Training MRF	**AFM** **Days requested:** 3
Access Route	Direct Access **Previous GP Number:** -
Science Areas	Biology and Bio-materials, Chemistry, Technique Development **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	This proposal aims to leverage Atomic Force Microscopy (AFM) to study the detailed structures of amyloid protein aggregates, specifically Abeta1-42 and ATX-3, which are associated with neurodegenerative diseases. The project also seeks to verify the effectiveness of various natural extracts and molecules in inhibiting these protein aggregations. The training will equipe two PhD students and two Principal Investigators (PIs) with the necessary skills to operate the AFM, interpret the resulting data, and apply this knowledge to advance their research.
Publications	-

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Training Instrument **AFM** **Days Requested:** 3
Special requirements:

SAMPLE

Material	-	-	-
Formula	-	-	-
Forms			
Volume			
Weight			
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	-	-	-
Pressure Range	-	-	-
Magnetic field range	-	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	-	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	-	-	-
Sensitivity to vapour	-	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	-	-	-

Training on the Use of AFM for Morphological Analysis of Amyloid Fibrils

1. Background and Context

The study of amyloid aggregation is a crucial area of research within the field of neurodegenerative diseases. Amyloid fibrils are misfolded protein structures that aggregate and deposit in tissues, leading to several disorders such as Alzheimer's disease (AD), Parkinson's disease (PD) and spinocerebellar ataxia type-3 (SCA3). Understanding the formation and morphology of these aggregates is essential for developing strategies to prevent or treat these debilitating conditions. This research has wide relevance as it can contribute to the development of preventive and therapeutic approaches for a range of neurodegenerative diseases, impacting public health and the quality of life for affected individuals and their caregivers.

Our group has been engaged for several years in researching compounds that can interfere with the progression of amyloid aggregation. Our focus has been particularly on natural compounds, as dietary intake of these substances can help prevent the primary biochemical mechanisms responsible for the onset of neurodegenerative diseases. This led us to screen various natural extracts obtained from edible sources[1–6]. We have demonstrated that a multidisciplinary approach, combining orthogonal techniques such as MS-based metabolomic screening of complex mixtures, NMR-based molecular recognition studies, *in vitro* biochemical and cell-based assays, and *in vivo* evaluations, is essential for identifying potential amyloid inhibitors and elucidating their mechanisms of action.

Finally, the detailed study of amyloid aggregates requires high-resolution imaging techniques, with AFM being particularly suited for this purpose. AFM allows visualization of the nanometer-scale structures of amyloid fibrils and provides quantitative data on their dimensions and morphology. Training in AFM is necessary to equip the research team with the skills to independently operate the equipment, acquire high-quality data, and accurately interpret the results. This expertise is crucial for advancing our understanding of amyloid aggregation and identifying potential inhibitors.

2. Proposed Training

The proposed training will involve four participants two PhD students and two PIs (academic staff from University of Milano-Bicocca, BtBs) carried out by the instrument scientists of the AFM facility (academic staff from University of Milano-Bicocca, School of Medicine and Surgery) and will cover the following areas over a 3-days period: basic operation and instrument set-up, sample preparation, data acquisition and data processing and interpretation.
The necessity of using the AFM facility arises from our requirement for high-resolution imaging techniques to characterize the morphology of amyloid aggregates at the nanoscale and to evaluate the effects of potential inhibitors. The training will empower participants to operate the AFM independently, thereby enhancing our research capabilities, facilitating future projects, and strengthening the scientific collaboration across different units of the ISIS@MACH ITALIA Research Infrastructure.

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3. Summary of previous instrument time or characterisation

The samples (amyloid proteins and compounds) for the AFM training will be collected and provided by IM@IT unit - Milano Bicocca (BtBs). Our prior research has successfully used AFM for similar studies, demonstrating the importance and feasibility of our proposal.

4. Justification of experimental proposals request

We request three (3) days of time to use the AFM facility (**AFM Nanowizard® 4XP – JPK-Bruker**) available at the IM@IT – Milano Bicocca (School of Medicine and Surgery) and, according with the instrument scientists, we envisage the following training plan:

- **Day 1**: Basic operation of AFM and sample preparation. Participants will learn how to set up the AFM for optimal performance and the appropriate techniques to preparing amyloid aggregate samples for AFM imaging.
- **Day 2**: Imaging and Data Acquisition. Protocols for capturing high-resolution images and acquiring quantitative data on different samples.
- **Day 3**: Data Analysis and Interpretation. Methods for analysing AFM data, including measuring fibril dimensions and aggregate morphology.

Aβ1-42 and ataxin-3 (ATX3), implicated in the aetiology of AD and SCA3 respectively, will be used as different models of amyloidogenic peptide/proteins during the training program. Furthermore, natural compounds and mixtures that have already been shown to inhibit amyloid aggregation during previous studies will be used as positive controls.

References

1 C. Ciaramelli, A. Palmioli, A. De Luigi, L. Colombo, G. Sala, M. Salmona and C. Airoldi, *Food Chem.*, **2021**, 341, 128249.
2 C. Ciaramelli, A. Palmioli, A. De Luigi, L. Colombo, G. Sala, C. Riva, C. P. Zoia, M. Salmona and C. Airoldi, *Food Chem.*, **2018**, 252, 171–180.
3 A. Palmioli, V. Mazzoni, A. De Luigi, C. Bruzzone, G. Sala, L. Colombo, C. Bazzini, C. P. Zoia, M. Inserra, M. Salmona, I. De Noni, C. Ferrarese, L. Diomede and C. Airoldi, *ACS Chem. Neurosci.*, **2022**, 13, 3152–3167.
5 A. Palmioli, S. Bertuzzi, A. De Luigi, L. Colombo, B. La Ferla, M. Salmona, I. De Noni and C. Airoldi, *Bioorganic Chem.*, **2019**, 83, 76–86.
6 C. Ciaramelli, A. Palmioli, I. Angotti, L. Colombo, A. De Luigi, G. Sala, M. Salmona and C. Airoldi, *Front. Chem.*, **2022**, 10, 896253.
7 B. Sciandrone, A. Palmioli, C. Ciaramelli, R. Pensotti, L. Colombo, M. E. Regonesi and C. Airoldi, *ACS Chem. Neurosci.*, **2024**, 15, 278–289.

Experiment Proposal

Experiment number GP2024115

Principal investigator	Dr Claudia Minici, San Raffaele Scientific Institute, ITALY
Co-investigator (*)	Dr Valeria Cassina, University of Milan-Bicocca, ITALY
Co-investigator	Professor Francesco Mantegazza, Universita&039; Milano-Bicocca, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Stiffness characterization of pancreatic ductal adenocarcinoma tissue
MRF Instrument	**AFM** — **Days requested:** 3
Access Route	Direct Access — **Previous GP Number:** no
Science Areas	Biology and Bio-materials — **DOI:** -
Sponsored Grant	Yes — **Sponsor:** Other
Grant Title	Identification of B cell mediated molecular mechanisms driving stromal eaction in pancreatic adenocarcinoma — **Grant Number:** MFAG 26135
Start Date	02/01/2023 — **Finish Date:** 31/03/2028
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

Pancreatic ductal adenocarcinoma (PDAC) is one of the most aggressive and lethal cancers. Cancer cells account for the minority of tumor mass, while the main component of the tumor stroma is represented by the extracellular matrix (ECM): collagens, glycoproteins, proteoglycans, and elastic fibers.

Our preliminary studies identified 57 proteins of the ECM which have a different expression in healthy and PDAC affected matrix, suggesting that tumor development occurs with a modification of the environment that surrounds tumor cells. PDAC biopsies are stiffer than normal pancreatic tissue, and this is considered the reason of PDAC resistance to chemotherapy. The increased stiffness could be due to different factors, as the presence of tumoral cells and the increased interstitial fluid pressure; but the role of the ECM is yet unknown. We aim to measure ECM stiffness of healthy and PDAC derived ECM, to clarify its role in the tumor and possible new therapeutic targets.

Publications -

ISIS neutron and muon source — **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Valeria Cassina, University of Milan-Bicocca, ITALY
MRF Instrument	**AFM** — **Days Requested:** 3
Special requirements:	

SAMPLE

Material	tissues slice	-	-
Formula	-	-	-
Forms	Solid		
Volume	cc		
Weight	mg		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

1. Background and Context

Pancreatic ductal adenocarcinoma (PDAC), one of the most aggressive and lethal cancers, is characterized by a prominent extracellular matrix (ECM) that acts as a physical barrier to drug penetration, accounting for PDAC resistance to chemotherapy. The matrisome is a combination of ECM-core proteins (collagens, glycoproteins, and proteoglycans) and ECM- associated proteins (ECM regulators, mucins, lectins, annexins and secreted factors).
In previous studies conducted in our laboratory, in the context of a 5-year MFAG grant aimed to clarify ECM role in PDAC development, we analysed the matrix composition after pancreatic tissue decellularization by nanoLiquid-chromatography MS/MS and compared the ECM of healthy subjects and PDAC patients before and after undergoing neoadjuvant chemotherapy with folfirinox. We identified 57 proteins differentially expressed in tumor and healthy pancreas. Single-cell RNA sequencing identified the major sources of the disregulated proteins.
Together with the protein composition, the biophysical characterization of ECM appears to be pivotal for the study of cancer development mechanisms and the identification of novel pharmacological targets. We performed preliminary studies of collagen orientation in pancreatic tissue exploiting polarized light microscopy experiments before and after chemotherapy.

2. Proposed experiment

Direct rheological analysis and elastography techniques demonstrate that PDAC biopsies are stiffer than normal pancreatic tissues, but this elevated solid stress could be ascribed to different causes, as the cellular components and the increased interstitial fluid pressure that leads to the collapse of lymphatic and blood vessels; no studies have been conducted yet that specifically focus on the ECM contribution.
Tissue stiffness is crucial for cancer development and metastasis spreading, as cancer cells can migrate according to a gradient of extracellular stiffness, in an event called durotaxis. As different protein composition affects ECM fibers orientation, we are expecting that also PDAC matrix stiffness could differ from the healthy one. By performing AFM measurements, we aim to shed light on this still unknown aspect of PDAC environment. In particular, we propose to use the Nanowizard® 4XP – JPK-Bruker atomic force microscope because it is the stat-of-art instrument for the mechanical characterization at the nanoscale. Indeed, it combines the highest mechanical and thermal stability during long-term experiments with acquisition speed. The measurements consist in the acquisition of at least 4 grid for each sample, with more than 100 force-indentation curves acquired at different positions on the sample surface. The evaluation of tissue stiffness, described quantitatively through the elastic modulus parameter (Young's Modulus), is obtained by force-indentation curve analysis with the Hertz-Sneddon model, considering the shape of the indenter. Each force-indentation curve is fitted by JPK data processing software up to about 2 µm of indentation depth. The elastic modulus of the tissue can be obtained: the higher the elastic modulus value, the stiffer the sample.

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3. Summary of previous experimental proposals or characterisation

This is the first proposal.

4. Justification of experimental time requested

We want to start with a limited number of samples to set some experimental details. Samples to be measured come from 2 healthy donors (**HP**), 2 treatment naïve PDAC tumor patients (**KN**), 2 treatment naïve PDAC peritumoral tissue (**HN**) patients, and 2 patients treated with FOLFIRINOX neoadjuvant therapy (**KNN**). For each sample will be analysed 2 slices of tissue. The total experimental time request is 3 full days. Indeed, for each sample is required up to 15 min of setup time and about 90 min of measurements acquisition. Therefore, we expect to collect measurements on about 5 slice of tissue per day.

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Experiment Proposal

Experiment number GP2024122

Principal investigator	Dr Cristina Scielzo, Università Vita-Salute San Raffaele, ITALY		
Co-investigator	Dr Valeria Cassina, University of Milan-Bicocca, ITALY		
Co-investigator	Professor Francesco Mantegazza, Universita&039; Milano-Bicocca, ITALY		
Co-investigator (*)	Dr Riccardo Campanile, Università di Milano Bicocca, ITALY		
Co-investigator			
Co-investigator			
Co-investigator			
Co-investigator			
Co-investigator			
Experiment title	Nanomechanical characterization of healhy and pathological lymphoid tissue		
MRF Instrument	**AFM**	**Days requested:** 3	
Access Route	Direct Access	**Previous GP Number:** no	
Science Areas	Biology and Bio-materials	**DOI:** -	
Sponsored Grant	Yes	**Sponsor:** Other	
Grant Title	Deciphering and targeting mechanical adaptability for the treatment of chronic lymphocytic leukemia	**Grant Number:** Individual AIRC Grant - IG28750	
Start Date	01/01/2024	**Finish Date:** 31/12/2028	
Similar Submission?	-		
Industrial Links	-		

Non-Technical Abstract

Chronic lymphocytic leukemia (CLL) is an haematological cancer where abnormal B-lymphocytes move between blood, bone marrow, and lymphoid organs, finding safe environments to survive and resist treatments. A key protein called HS1 plays a key role in helping CLL cells to relocate and spread into the lymphoid tissues, thus promoting their expansion. This project aims to investigate how the stiffness of lymphoid tissues, such as the spleen, changes during different stages of CLL, and how the absence of HS1 affects this process. Using advanced technology like atomic force microscopy (AFM), we will measure tissue stiffness in both wild-type and diseased mice, taking into account factors like age, sex, and disease stage. This study could reveal new insights into CLL progression and highlight new therapeutic targets by understanding how the mechanical properties of tissues influence disease development.

Publications -

ISIS neutron and muon source	**E-platform:** No
Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Riccardo Campanile, Università di Milano Bicocca, ITALY		
MRF Instrument	**AFM**	**Days Requested:** 3	
Special requirements:			

SAMPLE

Material	slices of tissues	-	-
Formula	-	-	-
Forms	Solid		
Volume	cc		
Weight	mg		
Container or substrate	-	-	-
Storage Requirements	@-80°C	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

1. Background and Context

Chronic lymphocytic leukemia (CLL) is the most common leukemia in the western world and it is characterised by the expansion of clonal CD5+ B-lymphocytes, which are capable of recirculating between peripheral blood, bone marrow, and secondary lymphoid organs. These lymphocytes originate from specific niches where CLL cells can survive, proliferate, and evade therapies. In this context, the HS1 protein has been identified as a crucial cytoskeletal regulator involved in the homing of CLL cells and in the progression of the disease.
The aim of this study is to investigate the rigidity of lymphoid tissues during various stages of CLL progression and to understand how the absence of the potential mechanosensory protein HS1 influences this process. We hypothesise that factors such as ageing, sex, disease stage, and the knockout (KO) of HS1 in a CLL mouse model could significantly affect the mechanical properties of these tissues over time. To test this, atomic force microscopy (AFM) will be used to analyze spleen sections from wild-type mice (C5/BL), from a CLL mouse model (Eμ-Tcl-1), and from a CLL mouse model with the HS1 gene knocked out (Eμ-Tcl-1-HS1ko). The tissue samples will be collected at three different time points: 3, 6, and 12 months of age, using six mice per group (three males and three females).
This research is supported by the AIRC Individual Grant (IG28750), with the goal of providing a deeper understanding of the mechanobiological dynamics in CLL and identifying potential new therapeutic targets through the study of tissue mechanics.

2. Proposed experiment

According to the literature, leukocytes affected by CLL display reduced stiffness in the actomyosin complex region compared to their healthy counterparts. This reduced stiffness facilitates continuous cytoskeletal remodelling, allowing these cells to recirculate between peripheral blood, bone marrow, and secondary lymphoid organs, as well as perform extravasation processes. The role of secondary lymphoid tissues, particularly the spleen, in CLL remains underexplored. Understanding whether tissue stiffness is influenced by disease stage, sex, and age is crucial for evaluating the potential active involvement of the tissue in disease progression.
To address this, performing force spectroscopy measurements by means of an AFM can help to shed light on this poorly understood role. Specifically, we propose using the Nanowizard® 4XP – JPK-Bruker atomic force microscope, coupled with spherical tips with a 5-micron radius, to obtain an averaged response of the probed tissue. The planned experiments will focus on analysing the stiffness of spleen tissue samples by acquiring grid maps of 11x11 μm^2, containing 11x11 force measurements at each grid point.
The tissue stiffness will be evaluated through the calculation of the Young's Modulus, a parameter derived by fitting the obtained force curves using the Hertz-Sneddon model, processed through the JPK data processing software. This approach will allow us to accurately assess how the rigidity of the lymphoid tissues evolves under the influence of CLL progression, sex, and ageing.

3. Summary of previous experimental proposals or characterisation

This is the first proposal.

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4. Justification of experimental time requested

Samples to be measured come from 6 different mice, 3 males and 3 females being respectively 2 wild-type (WT), 2 affected by CLL (Eμ-Tcl-1) (CLL) and 2 affected by CLL with HS1 gene knocked out (Eμ-Tcl-1-HS1ko) (CLL-HS1KO). For each sample 4-5 square grids will be collected. The total experimental time request is 3 full days. Indeed, for each grid 75 min of measurements acquisition is required. Thus, we expect to collect data on 2 slices of spleen tissue per day.

Experiment Proposal

Experiment number GP2024147

Principal investigator (*) Miss francesca cecilia lauta, humanitas university, ITALY
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator

Experiment title	Exploring surface-induced mechanical forces and macrophage behaviour for microscale interactions with substrate topography		
MRF Instrument	**AFM**	**Days requested:**	6
Access Route	Direct Access	**Previous GP Number:**	No
Science Areas	Biology and Bio-materials	**DOI:**	-
Sponsored Grant	None	**Sponsor:**	-
Grant Title	-	**Grant Number:**	-
Start Date	-	**Finish Date:**	-
Similar Submission?	-		
Industrial Links	-		

Non-Technical Abstract

Successful implant integration is closely tied to the fate of the immune response, and the use of biomaterials that do not trigger a pathological immune response could aid in the integration process. Previous studies have reported changes in macrophage proliferation, motility, and cytokine secretion, but still macrophage behaviour in response to biomaterial physical cues remain obscure.

This project aims to study the influence of micro-scale surface texture on macrophage mechanosensing and subsequent mechanotransduction. These factors are known to influence cytokine secretion and cell recruitment, affecting immune response outcomes. I will culture human monocyte-derived macrophages (hMDMs) from healthy subjects on textured surfaces, analyse the interaction of hMDMs with these surfaces and their potential activation. The goal is to understand if specific biomaterial designs may influence macrophages pro-inflammatory or anti-inflammatory behaviour.

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Miss francesca cecilia lauta, humanitas university, ITALY		
MRF Instrument	**AFM**	**Days Requested:**	6
Special requirements:			

SAMPLE

Material	Polydimethylsiloxane substrate, human monocyte-derived macrophages, cell medium (RPMI, fetal bovine serum, alpha-glutamine, sodium pyruvate, Penicillin-Streptomycin	-	-
Formula	CH3[Si(CH3)2O]nSi(CH3)3	-	-
Forms	Solid		
Volume	0.5 ml		
Weight	10 mg		
Container or substrate	Well plate	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	308 - 310 K	-	-
Pressure Range	1013 - mbar	-	-
Magnetic field range	none - T	-	-
Standard equipment	None	-	-
Special equipment	Cell incubator, 5% CO2, 37°C	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	No	-	-
Special equip. reqs	I will need to use a biological hood for cell seeding and an incubator for cell culturing.	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	No	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

1. Background and Context

Successful implant integration is closely tied to the fate of the immune response, which involves recruitment – amongst others – of macrophages. These are highly plastic cells that respond to environmental cues by switching towards a pro-inflammatory (M1) or pro-healing (M2) phenotype[1]. Previous studies showed that immune cells modulate their cytoskeletal dynamics in response to topographical cues of textured surfaces, which might affect their proliferation, motility, cytokines secretion and cell recruitment, acting as key determinants of the immune response[2,3]. Additionally, a previous study from our lab highlighted that immune cells accumulation in the cavities of macrotextured surfaces may contribute to their activation[4]. These findings suggest that implant surface properties may play a pivotal role in initiating and sustaining immune responses, thereby increasing the risk of chronic inflammation. However, a causal relationship between biomaterial design and macrophage behaviour is still missing.

This project aims to study the influence of micro-scale surface texture on macrophage mechanosensing and subsequent mechanotransduction. These factors are known to influence cytokine secretion and cell recruitment, affecting immune response outcomes.

2. Proposed experiment

This study aims to understand how mechanical cues from topography-driven confinement impact macrophage behaviour and subcellular response. Previous experiments showed that substrate pattern affects human monocyte-derived macrophages (hMDMs) in terms of viability, shape, migration speed and cytokine release. However, mechanical information about hMDMs is missing.

AFM measurements will provide us with insights about how hMDMs deform on wrinkles and will give us information about their elastic modulus, which might be affected by substrate pattern. Mechanical assessment is important because changes in macrophage stiffness are associated with pro-inflammatory and anti-inflammatory phenotypes, which – in turn – relate to chronic inflammation or other severe pathologies, like cancer.

3. Summary of previous experimental proposals or characterisation

Analysis of fluorescent images of human monocyte-derived macrophages cultured on wrinkled patterns for 6 days showed that macrophages align in the main wrinkle direction (Figure 1A) and that wrinkles cause a change in cell viability, with cells being more viable for wrinkles bigger than 2.5 μm (Figure 1B). Analysis of macrophage morphology revealed that cells assume a more elongated, ellipsoid-like shape when cultured on smaller wrinkles ($0 \leq \lambda \leq 2.5\ \mu m$), whereas on bigger wrinkles ($5 \leq \lambda \leq 20\ \mu m$) they are more circular (Figure 1C). Live tracking of macrophages on patterned substrates showed that wrinkles affect migration speed, which is the highest on wrinkles with $\lambda = 2.5\ \mu m$ (Figure 1D).

4. Justification of experimental time requested

We need AFM to get information about macrophages mechanical properties to provide a comprehensive scenario about cells response to substrate pattern cues.

Every experiment needs a maximum of one day to be performed, and at least three replicates for statistics, for a total of 3 days. One additional day is needed for setting up the experimental conditions as well as the machine. The other two days are needed for potential troubleshooting and for acquisition of extra data for statistics.

Bibliography

1. Fujiwara, N., & Kobayashi, K. (2005). Macrophages in inflammation. *Current drug targets. Inflammation and allergy,* 4(3), 281–286. https://doi.org/10.2174/1568010054022024
2. Lv, L., Xie, Y., Li, K., Hu, T., Lu, X., Cao, Y., & Zheng, X. (2018). Unveiling the mechanism of surface hydrophilicity-modulated macrophage polarization. *Advanced Healthcare Materials,* 7(19), 1800675. https://doi.org/10.1002/adhm.201800675
3. McWhorter, F. Y., Wang, T., Nguyen, P., Chung, T., & Liu, W. F. (2013). Modulation of macrophage phenotype by cell shape. *Proceedings of the National Academy of Sciences,* 110(43), 17253-17258. https://www.pnas.org/doi/abs/10.1073/pnas.1308887110
4. Belgiovine, C., Pellegrino, L., Bulgarelli, A., Lauta, F. C., Di Claudio, A., Ciceri, R., ... & Rusconi, R. (2023). Interaction of bacteria, immune cells, and surface topography in periprosthetic joint infections. *International Journal of Molecular Sciences*, 24(10), 9028. https://doi.org/10.3390/ijms24109028

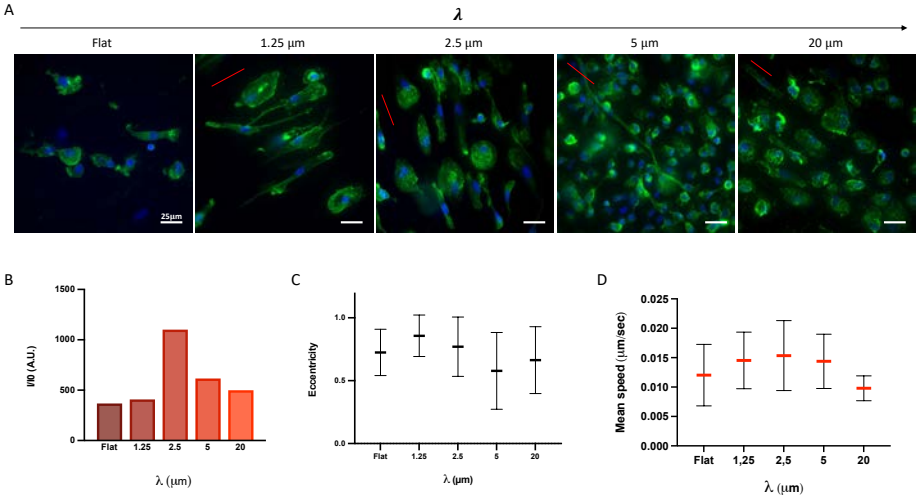

Figure 1. A) *Fluorescent images of human monocyte-derived macrophages (hMDMs) cultured for 6 days on flat PDMS substrates and on wrinkled PDMS. Red lines indicate the main pattern direction. Scale bar = 25 μm. Phalloidin labelled F-actin (green), DAPI nuclear staining (blue).* ***B)*** *hMDMs viability on patterned substrates determined by quantification of phalloidin intensity. The highest the intensity the more cells on the substrate.* ***C)*** *Analysis of hMDMs shape on fluorescent images, reported in terms of eccentricity.* ***D)*** *Measure of hMDMs mean speed. Cells were imaged every 15 minutes for 24 hours. Tracking was performed with MTrackJ in ImageJ.*

Experiment Proposal

Experiment number GP2024151

Principal investigator	Dr Luca Pellegrino, HUMANITAS UNIVERSITY, ITALY
Co-investigator (*)	Dr Valeria Cassina, University of Milan-Bicocca, ITALY
Co-investigator	Dr Riccardo Campanile, Università di Milano Bicocca, ITALY
Co-investigator	Professor Francesco Mantegazza, Universita&039; Milano-Bicocca, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Investigating microbial colonization and removal on bioinspired wrinkled surfaces
MRF Instrument	**AFM** **Days requested:** 5
Access Route	Direct Access **Previous GP Number:** -
Science Areas	Biology and Bio-materials, Materials, Medicine, Physics, Technique Development **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

Microbes have remarkable capabilities to attach to surfaces of natural and artificial systems, eventually leading to the formation of biofilms and associated chronic and persistent infections. It is extremely appealing to understand how bacteria interact with threedimensional surface topographies and how to design smart patterns as a strategy to create antifouling and biocidal materials. Here, we propose a dynamic strategy, merging verstile and large-scale surface modification teqhniques based on mechanical wrinkling of soft bilayers, microfluidics and microbiology. Specifically, we will evaluate the effect of surface topography over bacterial proliferation, motitly and viability, incorporating nano- to micro-scaled wrinkled geometries in microfluidic channels, mimicking biological tissues surfaces and implantable medical devices, testing a series of different clinically relevant bacterial strains (such as Pseudomonas aeruginosa and Staphylococcus aureus).

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Valeria Cassina, University of Milan-Bicocca, ITALY
MRF Instrument	**AFM** **Days Requested:** 5
Special requirements:	

SAMPLE

Material	polydimethylsiloxane	-	-
Formula	CH3[Si(CH3)2O]nSi(CH3)3	-	-
Forms	Solid		
Volume	cc		
Weight	10 g		
Container or substrate	standard glass slide	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	298 - 298 K	-	-
Pressure Range	1000 - 1000 mbar	-	-
Magnetic field range	no - no T	-	-
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	No	-	-
Sample Prep Hazards	no	-	-
Special equip. reqs	no	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	no	-	-
Equipment Hazards	-	-	-
Biological hazards	no	-	-
Radioactive Hazards	no	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Removed By User	-	-

1. Background and Context

Bacteria are ubiquitous and can easily colonize most natural and artificial surfaces. Once irreversibly attached on a substrate or interface, bacteria often grow as matrix-encased colonies known as biofilms, which play a critical role in infections associated with medical devices. Such device-related infections are difficult to treat as a biofilm shields the bacteria, protecting them from the immune response and antibacterial treatments. Although there has been significant research to design naturally-inspired surface topographies and create antifouling materials, fabrication techniques are usually demanding in terms of costs, scalability and lifetime. In this research project, will provide the first combined approach of surface patterning and microfluidics in modelling bacterial adhesion and removal on biomedical devices at the interface of soft matter and microbiology. Specifically, we will provide a scalable, accessible and cost-effective surface patterning technology compared to conventional photolithographic techniques, able to produce fine-tuned topographies to be incorporated in biomedical devices, such as titanium prosthesis and siliconic breast implants. Ultimately, the effectiveness of textured implants in limiting microbial colonization will impact the conventional prophylaxis methodologies deployed in case of microbial infection, such the large use of antibiotics, contributing to the development of associated microbial resistance.
Our research programme is supported by the HORIZON-MSCA-2022-PF-01, MOBILE 101110029 grant and has already gained collaboration with relevant medical centres such as IRCCS Humanitas Research Hospital and other Italian and European universities such as ENS Lyon, ETH Zurich and Università Milano Bicocca.

2. Proposed experiment

We developed a scalable surface modification method based on mechanical wrinkling of polydimethylsiloxane (PDMS) accessing a plethora of different nano- to micro-scale patterns. Sinusoidal wrinkles spontaneously arise in buckled polymeric bilayers when an applied mechanical strain e overcomes a critical threshold. Upon relaxation, a pattern develops characteristic wavelength (λ) and amplitude (A) defined by the mismatch in mechanical properties of the bilayer constituents. We have tested the interaction of different wrinkled geometries with a series of bacterial strains such as *P. aeruginosa*, *E. coli* and *S. aureus*, in microfluidic devices, finding that multi-directional patterns are able to delay proliferation by hindering bacteria motility. The focus of this research is to elucidate the effect of wrinkled topographies characteristic length scales (λ and A) and pattern geometries over a series of clinically relevant bacterial strains;
Atomic force microscopy (ISIS@MACH ITALIA instrument) will be a crucial technique needed to characterize pattern geometries and features, with a resolution not achievable with other techniques. Moreover, the confinement and of fixated bacterial cells probed by means of AFM mechanical force maps will be fundamental to characterize the preferential attachment of bacteria with respect to the surface specific curvature and topographical cues.

3. Summary of previous experimental proposals or characterisation

This is the first proposal.

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4. Justification of experimental time requested

We have requested 5 days for the experiment. We will analyze 5 different wrinkling geometries:

- Simple sinusoidal or 1D, with four different wavelengths (1 day)
- Double sinusoidal 2D checkerboard, with four different wavelengths (1 day)
- Herringbone sinusoidal and Hierarchical sinusoidal (1 day)
- Acquisition of force maps for one selected wavelength (5 μm), (1 day)
- Acquisition of force maps for one selected wavelength (5 μm), (1 day)

For a total of 20 samples. Five days of work will be essential to complete the topographical characterization of the different patterns (air, tapping mode) and the force map acquisition.

AFM BIO

Experiment Proposal

Experiment number GP2024070

Principal investigator	Dr Valentina Nigro, ENEA, ITALY
Co-investigator (*)	Dr BARBARA RUZICKA, ISC CNR, ITALY
Co-investigator	Dr Roberta Angelini, National Research Council
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Study of morphological changes of soft responsive microgels at solid interfaces through Atomic Force Microscopy
Training MRF	**AFM BIO** — **Days requested:** 2
Access Route	Direct Access — **Previous GP Number:** No
Science Areas	Physics — **DOI:** -
Sponsored Grant	None — **Sponsor:** -
Grant Title	- — **Grant Number:** -
Start Date	- — **Finish Date:** -
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

Microgel-formed films are gaining increasing interest for applications in soft nanotechnology and as model systems for studying nanoscale structuring of soft colloids at solid interfaces. Understanding the structure-property relationship of microgel films on solid interfaces is crucial for novel strategies in sensing and biosensing, tissue engineering or as temperature-responsive cell surfaces.

AFM measurements in liquid are essential for investigating morphological properties of adsorbed microgels in their native environment. Therefore, we need specific training on the AFM-BIO instrument to test feasibility on microgels of different sizes and composition spin-coated on various substrates (glass, silica, silicon, etc.). Complementary details will be obtained through comparison with GISAXS measurements on the SAXS GISAXS instrument, requested through a separate Training Proposal. Additionally, this proposal will strengthen the cooperation between ENEA and ISIS@MACH ITALIA.

Publications -

ISIS neutron and muon source — **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr BARBARA RUZICKA, ISC CNR, ITALY	
Training Instrument	**AFM BIO**	**Days Requested:** 2
Special requirements:		

SAMPLE

Material	-	-	-
Formula	-	-	-
Forms			
Volume			
Weight			
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	-	-	-
Pressure Range	-	-	-
Magnetic field range	-	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	-	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	-	-	-
Sensitivity to vapour	-	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	-	-	-

Study of morphological changes of soft responsive microgels at solid interfaces through Atomic Force Microscopy

1. Background and Context

Microgel-formed films are gaining interest due to their potential technological applications and as model systems for studying self-assembly and phase behaviours at interfaces [1]. Recent research has primarily focused on microgels at liquid-liquid or air-liquid interfaces [2], overlooking complex behaviours emerging from their adsorption to solid surfaces. The possibility to arrange microgels in 2D arrays inspires their use as smart coatings in soft nanotechnology utilizing their stimuli-responsive behaviour.

Thermo-responsive microgels based on poly(N-isopropylacrilamide) (PNIPAM) are especially promising for their application in sensing and biosensing, as functional tissue in regenerative medicine or even for drug release, thanks to their Volume Phase Transition Temperature (VPTT) of around 32°C. In the last year, our group has widely investigated their bulk properties by combining Dynamic Light Scattering (DLS), rheology, calorimetry, Small-Angle Neutron Scattering (SANS), Small Angle X-ray Scattering (SAXS), Raman spectroscopy, X-ray Photon Correlation Spectroscopy (XPCS), transmission electron microscopy and electrophoretic measurements [3]. Recently, we proposed PNIPAM microgel films as smart coatings for solid-state radiation detectors for radiobiological experiments on cell cultures (BIOTRACK project, Regione Lazio [4]). Exploiting their thermo-responsiveness, PNIPAM microgel films can be used for fabricating cell surfaces able to modulate cell attachment/detachment with temperature [5]. We have therefore investigated the optical and morphological properties of PNIPAM microgels spin-coated on solid substrates through UV-Vis-NIR spectroscopy, wettability measurements and Atomic Force Microscopy (AFM) [6]. In particular, AFM measurements in the dry state revealed the formation of densely packed layers with particle arrangements depending on microgel size, particle softness, packing density and growth conditions (Fig.1).

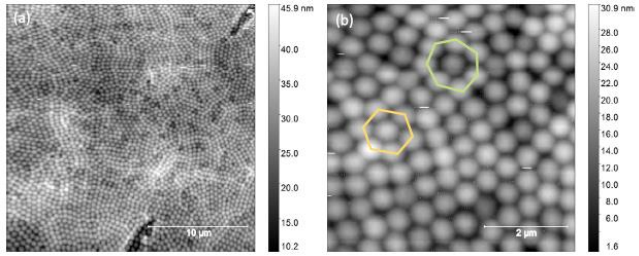

Fig. 1. AFM images on (a) 24×24 µm² and (b) 6×6 µm² area of a film of PNIPAM microgels on glass.

However, in the typically dry conditions of AFM measurements, microgel architecture is affected by water expulsion. Morphological properties of microgels in their native environment must be therefore explored through in-liquid AFM. These measurements are expected to provide reliable information on the impact of a solid substrate on the shape and flexibility of adsorbed microgels in their native state. Comparison with Grazing-Incidence Small-Angle X-ray Scattering (GISAXS) measurements could provide a comprehensive understanding of microgel behaviour at the solid-liquid interface.

2. Proposed Training

Training on the AFM BIO instrument is required to evaluate feasibility of AFM measurements on microgels in a liquid environment at temperatures below and above 32°C. Systematic characterizations have been previously performed on samples in the dry state, while we have never performed in-liquid AFM on adsorbed microgels. Additionally, training is needed to test the feasibility of AFM measurements on cell cultures deposited on microgel-based surfaces. This will help investigate the effects of the mechanical and surface properties of microgel films on cellular behaviour, attachment, and detachment. To gain insight into the in-plane microgel arrangement, it could be useful to combine AFM with GISAXS measurements using the SAXS GISAXS instrument, requested through a separate Training Proposal. This approach will also enable direct comparison with results from DLS, SANS and SAXS previously performed on aqueous suspension of PNIPAM microgels. Training is requested for a maximum of two researchers and will be carried out by MRF staff.

3. Summary of previous training proposals

No previous training proposals have been submitted.

4. Justification of experimental proposals request

AFM BIO is requested for topographical analysis of microgels in liquid in the temperature range T=20-50°C. Training is required for the contact and tapping mode, as well for force spectroscopy to investigate nano-mechanical properties of soft microgels. Considering the following breakdown, 2 full days of training are requested:

- Sample handling + basic operations to set-up the instrument in different operational modes = 1day
- Optimization of measurement conditions + data analysis = 1 day

[1] Shaulli, X. et al. ACS Nano 17 (3) (2023) 2067-2078.
[2] Vialetto J. et al., ACS Nano 15 (8) (2021) 13105-13117.
[3] Nigro, V. et al., Polymers 13 (2021) 1353.
[4] https://www.biotrack.enea.it/it/
[5] Sanzari, I. et al., Sci. Rep. 10 (2020) 6126.
[6] Nigro, V. et al., Colloids Surf. A: Physicochem. Eng. Asp. 674 (2023) 131918.

AFM Raman with Optical Profiler

Experiment Proposal

Experiment number GP2024045

Principal investigator (*) Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator

Experiment title	Investigating the Corrosive Impact of Chrome-Tanned Semi aniline Calf Leather using RAMAN Spectroscopy and Profilometry	
MRF Instrument	**AFM Raman with Optical Profiler**	**Days requested:** 3
Access Route	Direct Access	**Previous GP Number:** NO
Science Areas	Materials	**DOI:** -
Sponsored Grant	None	**Sponsor:** -
Grant Title	-	**Grant Number:** -
Start Date	-	**Finish Date:** -
Similar Submission?	-	
Industrial Links	LBS LUXURY BRANDS SERVICES SRL	
Non-Technical Abstract	The aim of this study is to systematically investigate the composition of semi aniline chrome-tanned calf leather and to identify correlations between adsorbed substances on the leather surface and its aggressiveness towards metal accessories. By understanding these relationships, we can develop better methods for predicting and mitigating corrosion, thereby improving the quality and durability of leather products in the fashion and luxury industries. Understanding the chemical composition of semi aniline chrome-tanned calf leather will lead to significant improvements in product quality ad in particular this research will provide a scientific basis for better quality control practices and contribute to the development of more robust and durable fashion items.	
Publications	-	

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY
MRF Instrument **AFM Raman with Optical Profiler** **Days Requested:** 3
Special requirements:

SAMPLE

Material	Chrome-Tanned Semi aniline Calf Leather	-	-
Formula	Chrome-Tanned Semi aniline Calf Leather	-	-
Forms	Solid		
Volume	4 cc		
Weight	10 g		
Container or substrate	No special need	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	RT - K	-	-
Pressure Range	Atmospheric pressure - mbar	-	-
Magnetic field range	No - T	-	-
Standard equipment	-	-	-
Special equipment	No special equipment needed	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	No	-	-
Special equip. reqs	Profilometer	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	No	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Investigating the Corrosive Impact of Chrome-Tanned Semi aniline Calf Leather

1. Background and context

Semi aniline chrome-tanned calf leather is a highly valued material in the fashion and luxury industries, commonly used for products such as handbags, belts, and footwear. Ensuring the quality and longevity of these products is crucial, particularly in preventing corrosive processes that can damage metallic accessories like buckles, zippers, and decorative elements. The leather can release tanning substances that may react with metals, leading to corrosion and tarnishing, which compromises the aesthetic and functional integrity of the final products.

To assess the corrosive potential of leathers, quality control laboratories typically perform simulated corrosion tests using a reference sample. The extent of oxidation on the sample, after exposure to the leather, is evaluated and the leather is classified on a scale of aggressiveness from 1 to 5 (1 = highly aggressive, 5 = non-aggressive). However, beyond this empirical test, there has been no systematic study to investigate the underlying causes of oxidation and the specific substances responsible for the corrosive effects on metal accessories.

Although some literature provides a foundational understanding of surface composition and residuals chemicals absorption on leather [1-3], a detailed investigation, especially regarding the correlation with aggressiveness and corrosion properties toward metal accessories, is notably absent. This research aims to fill that critical knowledge gap: this study is in fact expected to reveal specific chemical components in the leather that are responsible for initiating or accelerating corrosion in metal accessories. By identifying these substances, the fashion and luxury industries can take proactive measures to treat or modify leather to reduce its corrosive impact, thereby enhancing the durability and quality of leather products.

2. Proposed experiment

This proposal is part of a broader study to investigate the underlying causes of oxidation and the specific substances responsible for the corrosive effects on metal accessories by semi aniline chrome-tanned calf leather. The study involves several instrumental techniques: a) RAMAN Spectroscopy and profilometry; b) FT-IR Spectroscopy; c) Tomography; d) Ion Scattering Spectroscopy; e) SEM-EDX.

Raman confocal microscopy is a pivotal tool for this experiment due to its unique capabilities.

In particular, the XploRA PLUS Raman spectrometer is a powerful tool for analysing the surface composition of chrome-tanned semi aniline calf leather because the analysis will provide detailed information on the chemical composition, distribution of compounds, and structural characteristics of the leather. Here are the key types of data that can be obtained: 1) identification of specific compounds present on the leather surface, including residual tanning agents, fats, oils, and other processing chemicals; 2) detection of chromium and its various oxidation states, which are crucial for understanding the tanning process and potential corrosion issues; 3) spatial distribution: mapping the distribution of identified substances across the leather surface to understand their uniformity or concentration in specific areas and depth profiling to see how these substances are distributed within the surface layers of the leather; 4) correlating the presence and concentration of specific substances with the observed corrosive effects on metallic accessories and identifying potential sources of corrosive agents and their pathways of migration to the leather surface.

Moreover, thanks to the non-destructive nature of the technique, it reserves the samples for possible further investigations with other instruments.

By employing the XploRA PLUS Raman spectrometer, we can therefore obtain comprehensive data on the chemical and structural properties of semi aniline chrome-tanned calf leather. This data is essential for understanding and mitigating the corrosive effects that these leathers can have on metallic accessories, ultimately leading to improved quality and durability of leather products in the fashion and luxury industries.

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In addition to the detailed chemical analysis provided by the XploRA PLUS Raman spectrometer, a thorough understanding of the surface topography and roughness of chrome-tanned semi aniline calf leather is crucial. This information can provide insights into the physical characteristics of the leather surface that may contribute to its interaction with metallic accessories and subsequent corrosion. The Z20 Profilometer by ZETA INSTRUMENTS is an essential ancillary tool for this purpose. In fact, the Z20 Profilometer can provide precise measurements of the leather surface's topography, including its roughness, texture, and any surface irregularities. These measurements are critical for correlating the physical surface properties with the chemical composition data obtained from the Raman spectrometer: 1) Surface Roughness: we will obtain data on the roughness and texture of the leather surface, which can influence the adsorption of substances and their interaction with metallic accessories; 2) Topographical Mapping: High-resolution maps of the leather surface, showing the distribution of peaks, valleys, and other features will help understand their corelation with observed corrosion properties.

3. Summary of previous experimental proposals or characterisation

Historically, the understanding of the aggressiveness of leather toward metal accessories has been constrained by a reliance on empirical methods and trial and error. This approach lacks the detailed insight and predictive capability that sophisticated analytical tools like Raman confocal microscopy can provide. In particular, access to such advanced analysis and the requisite expertise is often limited. Although some literature provides a foundational understanding of surface composition of tanned leather as reported in section 1, a detailed exploration of these elements' interplay, especially regarding the correlation with aggressiveness and corrosion properties toward metal accessories, is notably absent.

4. Justification of experimental time requested

As detailed in section 2, Raman confocal microscopy is a pivotal tool for this experiment due to its unique capabilities. **We request 3 days of experimental time on the AFM Raman with Optical Profilometer to analyse 15 samples** coming from 5 leather batches: we collected **5 types of batches** of semi aniline chrome-tanned calf leather with high level of aggressiveness (level 1 to level 2, with reference to the empirical scale where (1 = highly aggressive, 5 = non-aggressive) as determined by the currently used corrosion test as detailed in section 1. **For each batch we will then collect three samples** for investigating the spatial homogeneity of the composition.

This number ensures diverse representation from different batches of treatment conditions. The first day will be dedicated to collecting low-resolution Raman 2D maps from all the samples. The second day will focus on collecting Raman spectra and high-resolution maps to identify and map the distribution of adsorbed substances, such as fatty acids, residual chromium compounds, and other contaminants. We will then compare the Raman spectroscopic data with the aggressiveness classifications to identify patterns and correlations and determine which substances are most strongly associated with higher levels of corrosion. This step is crucial for effective time management and prioritizing detailed analyses. The final day is reserved for the profilometer analysis, based also on the compilation of initial findings. This structured approach allows for thorough examination while maintaining a strict timeline.

References

[1] Yolanda S. Hedberg, Carola Lidén, Inger Odnevall Wallinder, Correlation between bulk- and surface chemistry of Cr-tanned leather and the release of Cr(III) and Cr(VI), Journal of Hazardous Materials, Volume 280, 2014, Pages 654-661.
[2] Hana Vaskova, Raman microscopic detection of chromium compounds, MATEC Web of Conferences 7 05012 (2016).
[3] Karel Kolomaznik, Michaela Barinova, and Hana Vaskova, Chromium VI Issue in Leather Waste – A Technology for the Processing of Used Leather Goods and Potential of Raman Spectroscopy in Chromium Traces Detection, INTERNATIONAL JOURNAL OF MATHEMATICS AND COMPUTERS IN SIMULATION, Issue 5, Volume 6, 2012, pages 447-455.

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Experiment Proposal

Experiment number GP2024088

Principal investigator (*)	Dr Margaux Bouzin, Università degli Studi di Milano-Bicocca, ITALY	
Co-investigator	Mr Riccardo Bolzoni, University of Milan-Bicocca, ITALY	
Co-investigator	Mrs Letizia Marchesi, Università degli studi di Milano Bicocca, ITALY	
Co-investigator	Professor Maddalena Collini, Università degli Studi di Milano Bicocca, ITALY	
Co-investigator		
Co-investigator		
Co-investigator		
Co-investigator		
Co-investigator		
Experiment title	AFM/Raman & TERS Training for the Biophysics Research Group of UNIMIB	
Training MRF	**AFM Raman with Optical Profiler**	**Days requested:** 2
Access Route	Direct Access	**Previous GP Number:** No
Science Areas	Biology and Bio-materials, Chemistry, Materials, Physics	**DOI:** -
Sponsored Grant	None	**Sponsor:** -
Grant Title	-	**Grant Number:** -
Start Date	-	**Finish Date:** -
Similar Submission?	-	
Industrial Links	-	

Non-Technical Abstract

Multimodal Raman/AFM platforms combine Atomic Force Microscopy and confocal co-located Raman spectroscopy to map the sample surface topology and mechanical properties along with the chemical bonds and structural composition. In Tip Enhanced Raman Scattering, the resolution of such chemical imaging is pushed at the nanoscale. Applications range from the characterization of novel plasmonic nanostructures to the investigation of the elastic properties of hydrogels and polymeric films. Such applications, envisioned within the proponents' main research lines, motivate the proposal for training of the Biophysics group of University of Milano-Bicocca: the training aims at expanding the proponents' expertise in optical (fluorescence, non-linear and super-resolution photo-thermal) microscopy towards nanoscale imaging based on chemical contrast, and is expected to pave the way for the subsequent exploitation of AFM/Raman/TERS techniques for biophysical applications in the near future.

Publications -

ISIS neutron and muon source	**E-platform:** No
Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Margaux Bouzin, Università degli Studi di Milano-Bicocca, ITALY	
Training Instrument	**AFM Raman with Optical Profiler**	**Days Requested:** 2
Special requirements:		

SAMPLE

Material	-	-	-
Formula	-	-	-
Forms			
Volume			
Weight			
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	-	-	-
Pressure Range	-	-	-
Magnetic field range	-	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	-	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	-	-	-
Sensitivity to vapour	-	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	-	-	-

AFM/Raman & TERS Training for the Biophysics Research Group of UNIMIB

1. Background and Context

Raman spectroscopy probes molecular vibrations via inelastically scattered near-ultraviolet to near-infrared monochromatic light. The recorded spectroscopic signatures provide rich information on the sample chemical bonds, composition, structure and molecular conformation[1]. When Raman imaging is combined with Atomic Force Microscopy (AFM) in an integrated multimodal platform, the measurement of the elastic modulus can be superimposed to the chemical map to further evidence the interplay between surface composition, morphology and domains interaction: the chemical information delivered by confocal Raman microscopy on the ~micrometer scale is complemented with physical (topological, mechanical) information at the nanoscale[1]. Such nanometer-sized spatial resolution, pushed by the super-resolution TERS (Tip Enhanced Raman Scattering) configuration, allows the comprehensive characterization of organic and inorganic samples on a spatial scale that is still only partially accessible by fluorescence-based imaging modalities (Fig.1), albeit with low throughput and with limit at the uppermost sample surface as required by the near-field enhancement conveyed by the scanning probe tip[2,3]. Promising applications of AFM-Raman and TERS can be envisioned within active research lines in the proponents' lab[4,5]: these include the investigation of nanostructured plasmonic materials and the quantification of mechanical/elastic properties of proteinaceous hydrogels and polymeric films, thereby motivating the present request for training in the AFM-Raman and TERS fields. While specifically aimed at broadening the proponents' expertise in optical microscopic and spectroscopic techniques towards nanoscale imaging based on chemical contrast, the training is expected to also pave the way for subsequent biophysical applications of state-of-the-art chemical-based imaging, even in the framework of fostered scientific collaborations at the involved academic institutions.

2. Proposed Training

The present training proposal is conceived for members of the Biophysics and Biophotonics research group at the Physics Department of University of Milano-Bicocca. The principal investigator (Margaux Bouzin) has been recently appointed as tenure-track researcher (RTT), with research activity centered on the development and application of (super-resolution) optical microscopy techniques for the quantitative investigation of complex biological systems from the tissue scale down to the sub-cellular level. Training on Raman-AFM and TERS imaging is aimed at extending the group's expertise in the field of confocal fluorescence microscopy, non-linear (two-photon excitation and second harmonic generation) imaging and photo-thermal far-infrared imaging[6-8] towards high-resolution approaches based on chemical contrast, and will allow exploring the strengths and potential applicability of AFM/Raman spectroscopies within the PI's current research lines. The suitability of the techniques for the investigation of newly synthetized photoluminescent and photo-thermal nanoparticles will be explored, as part of a long-standing collaboration of University of Milano-Bicocca and University of Pavia (Prof. P. Pallavicini) in the development and characterization of anisotropic nanostructures for biophysical applications. In parallel, Raman and TERS requirements in terms of sample preparation and substrates will be specifically explored, and current pitfalls and challenges of TERS on soft matter specimens will be evaluated. In addition to the proposal PI, two Ph.D. students from the laboratory will join the training as part of their second-year doctoral activity. Ideally and upon agreement with the local Instrument Scientists, the training will be attended in person at the University of Rome Tor Vergata by the PI, while Ph.D. students may join the training remotely if preferable. Both in-person and remote training formats have proven successful within a previous Call for Direct Access of Medium Range Facilities at the proponents' institution. MRF staff and instrument scientists at Tor Vergata, who have been contacted in advance for the present proposal, will provide guidelines for optimal samples identification and preparation and will carry out the training.

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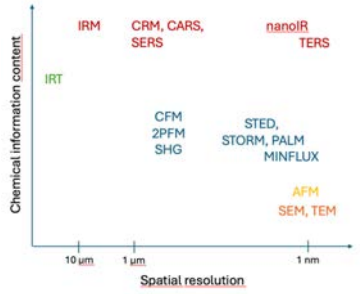

Fig.1. Comparison of different imaging modalities with spatial resolution and chemical information content as figures of merit. TERS bridges the high chemical information of vibrational Raman spectroscopies (confocal Raman, CARS and SERS imaging) and IR microscopy (IRM) to the high resolution of scanning probe techniques (e.g., AFM) and of super-resolution fluorescence-based imaging modalities (STED, STORM, PALM, MINFLUX). Super-resolution imaging, Infrared thermography (IRT) and conventional optical imaging modalities (Confocal and 2-Photon Fluorescence Microscopy, Second Harmonic Generation Imaging) are at the core of the research activity of the Biophysics Group at UNIMIB.

3. Summary of previous training proposals

No previous training proposal has been submitted by the same proponents through the ISIS@MACH ITALIA infrastructure for the *AFM Raman with Optical Profiler* experimental setup.

4. Justification of instrument time request

The instrument identified for the training proposal is the confocal microRaman spectrometer (XploRA Plus, Horiba) of University of Rome Tor Vergata, which combines vibrational Raman spectroscopy with the desired AFM and TERS capabilities. A two-day instrument time is estimated to be sufficient for the scope of the present proposal to cover instrument configuration, exemplary data acquisitions and the fundamentals of data processing.

[1] Belianinov A. et al., *ACS Nano,* 12, 11798-11818 (2018)
[2] Schmid T. et al., *Angew. Chem. Int. Ed.*, 52, 5940-5954 (2013)
[3] Kumar N. et al., *Nat. Prot.*, 11609-1193 (2019)
[4] Marini M. et al., *Lab Chip*, 22 (24), 4917-4932 (2022)
[5] Zeynali A. et al., *Adv. Opt. Mater.*, 8 (13), 2000584 (2020)
[6] Marini M. et al., *Adv. Func. Mater.*, 2213926 (2023)
[7] Bouzin M. et al., *Biophys. J.*, 109, 2246-2258 (2015)
[8] Bouzin M. et al., *Nat. Commun.*, 10:5523 (2019)

Experiment Proposal

Experiment number GP2024089

Principal investigator	Dr Mario Marini, Università degli Studi di Milano-Bicocca, ITALY
Co-investigator (*)	Dr Margaux Bouzin, Università degli Studi di Milano-Bicocca, ITALY
Co-investigator	Professor Maddalena Collini, Università degli Studi di Milano Bicocca, ITALY
Co-investigator	Dr Anna Prioriello, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Optical profilometry experiments on two-photon polymerized microlenses
MRF Instrument	**AFM Raman with Optical Profiler** **Days requested:** 1
Access Route	Direct Access **Previous GP Number:** No
Science Areas	Biology and Bio-materials, Materials, Physics **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	Light scattering and optical aberrations represent major obstacles in the application of non-linear fluorescence microscopy to in-vivo deep tissue imaging. The use of low-numerical-aperture objectives, in combination with high-dioptric-power microlenses implanted in-vivo close to the observation volume, can be beneficial for the reduction of aberrations. In the framework of an EU-funded FET Open project, we are developing novel plano-convex micro-lenses fabricated by two-photon laser polymerization. Following the characterization of the optical performance of spherical micro-lenses and the demonstration of their applicability to two-photon-excitation fluorescence microscopy, we focus here on newly developed parabolic lenses and plan to assess their surface quality, roughness and curvature by optical profilometry, with the rationale of completing the characterization of the micro-lenses developed by the project consortium as well as guiding the optimization of the fabrication protocol.
Publications	-

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Margaux Bouzin, Università degli Studi di Milano-Bicocca, ITALY		
MRF Instrument	**AFM Raman with Optical Profiler**	**Days Requested:** 1	
Special requirements:			
	SAMPLE		
Material	Micro-lenses fabricated by two-photon polymerization on glass coverslips.	-	-
Formula	-	-	-
Forms	Solid		
Volume	cc		
Weight	mg		
Container or substrate	glass slide	-	-
Storage Requirements	-	-	-
	SAMPLE ENVIROMENT		
Temperature Range	293 - K	-	-
Pressure Range	1000 - mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	-	-	-
	SAFETY		
Prep lab needed	No	-	-
Sample Prep Hazards	No	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	No	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Removed By User	-	-

Optical profilometry experiments on two-photon-polymerized microlenses

1. Background and Context

The assessment of the immunological response of a host organism to the implant of a novel biomaterial still relies on the histopathological inspection of ex-vivo stained tissue sections. Current protocols, standardized within the ISO 10993 norm, exploit semi-quantitative scoring systems, are associated to a significant economic burden and appear ethically questionable, thereby pointing out the need for efficient longitudinal (i.e., multiple-time-point), intravital, non-invasive and label-free studies of the animal immune reaction to the implant. Fluorescence microscopy based on non-linear excitation is especially suited for such a task, but the spatial resolution and signal-to-noise ratio of collected fluorescence images rapidly worsen at increasing penetration depth in deep tissues due to both light scattering and optical (mainly spherical) aberrations. In this context, and in the framework of an EU-funded research project (FET-OPEN project IN2SIGHT, G.A. 964481 to PI Prof. G. Chirico at UNIMIB), we are developing a miniaturized sub-cutaneous imaging window, to be implanted in-vivo, composed of micro-scaffolds and arrays of micro-lenses [1]. Scaffolds guide the tissue regeneration at the interface with the implanted material, while the microlenses enable non-linear imaging of the tissue by focusing a collimated or quasi-collimated excitation laser beam right at the observation site [1]. The lenses allow avoiding the use of long-working-distance and high-numerical-aperture microscope objectives, with a reduction of the impact of spherical aberrations on the propagating beams.
The successful exploitation of the microlenses imposes key requirements on their geometry and optical performance – high curvature, high dioptric power, relatively large (hundreds-of-μm) size, smooth surface, low autofluorescence and biocompatibility –, and demands for an extensive assessment of the optical properties, wavefront, modulation transfer function and surface quality of the micro-optics. Since optical profilometry appears to be the ideal technique to investigate the surface roughness and curvature of the microlenses, we apply here for a one-day access at the optical profiling microscope of the MRF1 ISIS@Mach Italia instrument suite with the rationale of both providing a characterization of the micro-lenses under current development by the project consortium and guiding the optimization of the fabrication protocol.

2. Proposed Experiment

We prototype and fabricate the micro-lenses by two-photon laser polymerization of the biocompatible SZ2080 photo-resist. The fabrication of micro-lenses with 0.4 numerical aperture and large (260-μm) diameter has already been reported with ∼8-min fabrication time per lens [1], and the acquisition of magnified fluorescence images through the microlenses coupled to commercial confocal and two-photon excitation scanning microscopes has been demonstrated on stained human fibroblasts (Fig.1). Such validation experiments have been preliminarily performed on plano-convex spherical lenses. It is our purpose here to extend our investigation to aspheric (plano-parabolic) micro-lenses, which allow further increasing the dioptric power while preserving large lens dimensions and field of view. Optical profilometry experiments will be specifically devoted to mapping the lens surface topology, curvature and roughness as a function of the lens geometry, size and fabrication parameters (e.g., laser z-slicing, radial step and speed in the two-photon laser polymerization protocol).
The present request for access to the MRF1 facility will involve members of the Biophysics and Biophotonics research group at the Physics Department of University of Milano-Bicocca. The principal investigator (Dr. Mario Marini) is currently post-doctoral research fellow within the aforementioned FET Open project. MRF staff and instrument scientists at Tor Vergata, who have been contacted in advance for the present proposal, have agreed on the feasibility of the proposed experiments.

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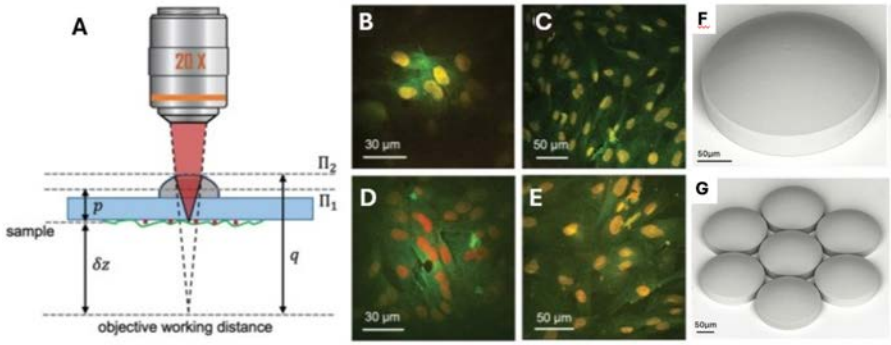

Fig.1. (A): sketch of the configuration adopted to couple a microlens with an optical scanning microscope. The distance between the sample and its virtual image through the microlens is δz. Π_1 and Π_2 are the two principal planes of the microlens. (B)-(E): Fluorescence images of stained human dermal fibroblasts under two-photon excitation ($\lambda_{exc} = 800$ nm). (B): Fluorescence image collected through the microlenses coupled to a 20X dry objective at a distance $\delta z = 135\ \mu m$ (with respect to the objective sample plane), resulting in a total magnification $M_{tot} \simeq 45$. (C): control image obtained through the 20X dry objective at $\delta z \simeq 0$. (D): image of the cells through the microlenses coupled to a 25X water immersion objective obtained at $\delta z \simeq 43\ \mu m$. (E): control image obtained with the 25X water immersion objective at $\delta z \simeq 0$. (F),(G): SEM images of one plano-convex spherical microlens (F) and of an array of 7 lenses symmetrically arranged in a hexagonal configuration. Adapted from ref.1.

3. Summary of previous proposals

The instrument identified for the present proposal is the Z20 (Zeta Instruments) optical profiling microscope of University of Rome Tor Vergata. The optical profilometer is classified on the ISIS@Mach platform as an ancillary instrument of an AFM/Raman setup, and this will also allow for a complementary confocal Raman mapping of the same samples. While no request for access to the same setup has been presented previously by our group, the AFM/Raman instrument is the object of a separate and unrelated two-day training request from our group in the present Call.

4. Justification of experimental proposals request

The sample will consist in 4 glass slides, each of them encompassing 4 arrays of 7 microlenses. Provided the acquisition time is of the order of a few minutes per lens profile, a one-day instrument time is estimated to be sufficient to cover the whole data acquisition and data analysis for the scope of the present proposal.

[1] Marini M. et al., *Adv. Funct. Mater.,* 2213926 (2023)

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Experiment Proposal

Experiment number GP2024095

Principal investigator	Dr Matthew Krzystyniak, STFC, UNITED_KINGDOM
Co-investigator (*)	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Co-investigator	Ms Margherita Simoni, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Roberto Senesi, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Raman determination of molecular composition of industrial-grade polymers for medical applications

MRF Instrument	**AFM Raman with Optical Profiler**	**Days requested:**	2
Access Route	Direct Access	**Previous GP Number:**	-
Science Areas	Materials, Medicine	**DOI:**	-
Sponsored Grant	None	**Sponsor:**	-
Grant Title	-	**Grant Number:**	-
Start Date	-	**Finish Date:**	-
Similar Submission?	-		
Industrial Links	-		

Non-Technical Abstract

At present, the transport and moderation of neutrons in the human body is modelled using specific phantoms, mainly water and PMMA. When choosing the phantom material, one aims to well approximate the human body with respect to the radiation of interest. While the elemental stoichiometry and density are the main parameters in the case of charged hadrons, electrons, and photons, in the case of neutrons the transport is affected by chemical interactions, especially for thermal neutrons. However, when using industrial-grade materials, one loses control over impurities and characterizations of the material itself. To provide the needed input into Monte Carlo simulations, one needs to have a further information on the chemical composition of the materials used as phantoms. We propose to run Raman microscopy and spectroscopy measurements over a series of industrially available polymeric samples to obtain an accurate determination of their molecular composition.

Publications

G. Romanelli et al., J. Phys.: Condens. Matter 33, 285901 (2021).
G. Romanelli et al., EPJ Web of Conferences 284, 17010 (2023)

Instruments	**NILE**	**Days Requested:**	2
Access Route	Direct Access	**Previous RB Number:**	
Science Areas		**DOI:**	
Sponsored Grant	None	**Sponsor:**	
Grant Title	-	**Grant Number:**	
Start Date	-	**Finish Date:**	
Similar Submission?			
Industrial Links			

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Sample record sheet

Principal contact	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY		
MRF Instrument	**AFM Raman with Optical Profiler**	**Days Requested:**	2
Special requirements:			

SAMPLE

Material	polystyrene	polypropylene	polyvinyl chloride
Formula	C8H8	C3H6	C2H3Cl
Forms	Solid	Solid	Solid
Volume	1 cc	1 cc	1 cc
Weight	1 g	1 g	1 g
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	300 - 300 K	- K	- K
Pressure Range	- mbar	- mbar	- mbar
Magnetic field range	- T	- T	- T
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	Yes	Yes
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	No	No
Sensitivity to vapour	No	No	No
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	Disposed by IS

Background and Context

Neutron capture therapy [1] is a cancer-treatment technique based on the irradiation of the human body with epithermal neutrons, their moderation within organic matter, and their eventual absorption by suitable drugs, often rich in boron, taken by the patient and delivered to the cancer region. Following neutron absorption, the heavy ions produced in the nuclear reaction deliver a large amount of energy over small regions of few micrometres, destroying cancer cells without affecting heathy ones. At present, the transport and moderation of neutrons in the human body is modelled using specific phantoms, mainly water and polymethyl methacrylate [2,3]. The purpose of phantom dosimetry is to characterize the adsorbed dose distribution, both primary (in the tumour region) and secondary (in the surrounding healthy tissue). When choosing the phantom material, one aims to well approximate the human body with respect to the radiation of interest. **While the elemental stoichiometry and density are the main parameters in the case of charged hadrons, electrons, and photons, in the case of neutrons the transport is affected by chemical interactions, especially for thermal neutrons.** Industrial 3D printing materials could provide an opportunity to broaden the range of materials (therefore the chemical interactions and functional groups therein) as well as facilitating obtaining geometries better approximating the human body.

Transport simulation of neutrons within phantoms relies on Monte Carlo codes, which feature accurate thermal-neutron libraries for a handful of materials only, while grossly approximating the neutron attenuation coefficient for the rest. However, a method was recently presented, referred to as the Average Functional Group Approximation (AFGA) [5,6], that allows the accurate prediction of transport libraries, such as the mass attenuation factor at thermal neutron energies of hydrogen-rich materials. AFGA is based on a simple rationalisation of an organic material based on its constituent "molecular units". Within this framework, **one can build a procedure whereby the information on the abundances of hydrogen-containing functional groups in different body parts of a patient is given as an input to a transport code, in a "personalized medicine" approach.**

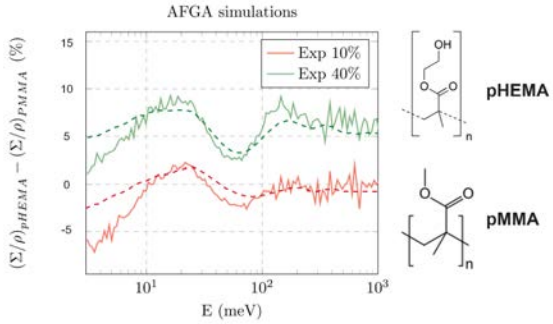

Figure 1 Mass attenuation coefficients of two polymeric materials described using the AFGA model.

Figure 1 shows characterizations, from the VESUVIO beamline at ISIS, on the transport properties of pHEMA as a new promising phantom material. In particular, the differences between the mass attenuation coefficient of pHEMA and of the standard phantom pMMA are shown for two hydration levels of pHEMA (10wt% and 40wt% absorbed water) and compared with the theoretical predictions based on AFGA. One can appreciate how, while having very similar elemental compositions and densities, the attenuation of thermal neutrons can be different up to 10% between an over-simplified phantom material (pMMA) and a more realistic one (e.g., pHEMA).

However, when using industrial-grade materials, one loses control over impurities and characterizations of the material itself. To provide the needed input into Monte Carlo simulations, one needs to have a further information on the chemical composition of the materials used as phantoms. In the spirit of facilitating the process of producing and characterizing new phantoms, the elemental determination should be obtained using benchtop instruments and medium-range facilities.

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Proposed Experiment

We propose to run Raman microscopy and spectroscopy measurements over a series of industrially available polymeric samples to obtain an accurate determination of their molecular composition. Similar samples have already been used for neutron moderation experiments on the VESUVIO beamline at ISIS. Four polymers have been considered as easy-to-access industrial materials, namely polypropylene (PP), polystyrene (PS), PMMA and polyvinyl chloride (PVC). While all materials are expected to be mainly composed of the nominal polymer, differences in the composition of the order of 5% in mass from copolymers can produce inaccuracies in the modelled transport properties.

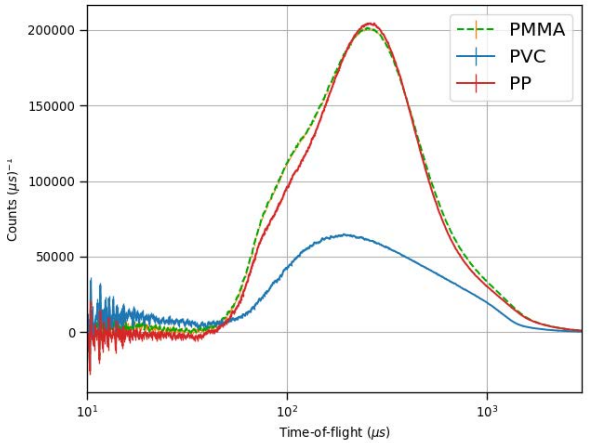

Figure 2 VESUVIO moderation spectra from several polymers considered.

Figure 2 shows recently-run neutron moderation experiments on these polymers at the VESUVIO spectrometer at ISIS, using epithermal and fast neutrons. Moderation experiment were aimed at measuring the many-collision signal of energetic neutrons onto a thick polymeric sample, therefore acting as a moderator. They are, in a way, the next step following experiments to measure total cross sections or mass attenuation factors. Further experiments are planned at the NILE facility, using quasi-monochromatic beams of fast neutrons. At present, preliminary Monte Carlo simulations are being run, which require updated and accurate input files for benchmarking.

Justification of experimental proposals request

We request 2 day of instrument time on the AFM Raman with Optical Profiler, to be divided as follows: 4 hours for each of 4 polymers considered for the neutron experiments.

References

[1] Z.P. Zagorski, *Radiation Physics and Chemistry* 56, 559–565 (1999).
[2] H.B. Liu, D.D. Greenberg, J. Capala and F.J. Wheeler, *Med. Phys.* 23 2051–60 (1996)
[3] C.P. Raaijmakers, E.L. Nottelman, B.J. Mijnheer, *Phys Med Biol.* 2353-61 (2000)
[4] J. Burmeister et al., *Med Phys.* 2560-4 (2000)
[5] G. Romanelli et al., *J. Phys.: Condens. Matter* 33, 285901 (2021).
[6] G. Romanelli et al., EPJ Web of Conferences 284, 17010 (2023)
[7] D. Chiesta et al., Nuc. Inst. Meth. A 902, 11 (2018).

Experiment Proposal

Experiment number GP2024161

Principal investigator	Dr Francesco Saliu, Università Milano Bicocca, ITALY
Co-investigator (*)	Dr Mohammed Monsoor Shaik, University of Milano-Bicocca, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	UV Oxidation and Chemical Catalysis of PET Plastics for the Generation and Characterization of Uniform Nanoplastics
MRF Instrument	**AFM Raman with Optical Profiler**
Days requested:	3
Access Route	Direct Access
Previous GP Number:	-
Science Areas	Environment
DOI:	-
Sponsored Grant	None
Sponsor:	-
Grant Title	-
Grant Number:	-
Start Date	-
Finish Date:	-
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

Plastic pollution is a pressing global issue, with Polyethylene Terephthalate (PET) being one of the most widely use plastic in products like bottles, packaging, and textiles. PET plastics are resistant to natural degradation, leading to their accumulation as micro- and nanoplastics in the environment. These small particles are difficult to study because they vary greatly in size and chemical makeup. The project's key objective is to create uniform PET nanoplastics through a two-step process: first using UV light to induce oxidation, mimicking environmental degradation, and then applying chemical reactions to break down the plastic into consistent, standardized nanoparticles. This uniformity will allow for better research into the health and environmental impacts of nanoplastics. The nanoplastics will be tested for their potential toxic effects on human cells, specifically focusing on their ability to cause cellular damage and trigger inflammatory responses.

Publications -

Internationa MRFs	**Particle Size Analyser**	**Days requested:** 3
ISIS neutron and muon source		**IM@IT E-platform:** No
Instruments		**Days Requested:**
Access Route		**Previous RB Number:**
Science Areas		**DOI:**
Sponsored Grant		**Sponsor:**
Grant Title		**Grant Number:**
Start Date		**Finish Date:**
Similar Submission?		
Industrial Links		

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ISIS Neutron and Muon Source

Sample record sheet

Principal contact	Dr Mohammed Monsoor Shaik, University of Milano-Bicocca, ITALY	
MRF Instrument	**AFM Raman with Optical Profiler**	**Days Requested:** 3
Special requirements:		

SAMPLE

Material	PET nanoplastics - Commercial	PET nanoplastics - PET bottle	PET nanoplastics - PET Film
Formula	-	-	-
Forms	Solid	Solid	Solid
Volume	cc	cc	cc
Weight	10 mg	10 mg	10 mg
Container or substrate	Glass vial	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	- K	- K
Pressure Range	- mbar	- mbar	- mbar
Magnetic field range	- T	- T	- T
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	Yes	Yes
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	No	No
Sensitivity to vapour	No	No	No
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	Disposed by IS

1. Background and Context

Plastic pollution is a pressing global issue with Polyethylene Terephthalate (PET) being one of the most widely used polymers. The durability and resistance of PET to natural degradation result in its accumulation in ecosystems, contributing to the presence of micro- and nanoplastics (MNPs). PET samples from various sources (commercial granules, bottles, and films) have different additives and manufacturing processes, influencing their degradation behavior. While UV-induced photooxidation mimics natural degradation, it is a surface-limited process and cannot uniformly break down PET into smaller nanoparticles. The creation of uniform particles is critical for systematic studies.

This study is crucial because it develops standardized PET nanoparticles across different scenarios to establish a comprehensive library. This will significantly enhance our understanding of the health impacts and environmental behavior of PET nanoplastics. These standardized nanoplastics are essential for consistent toxicological research, offering insights into their cytotoxicity and inflammatory effects.

The researcher involved in the project is supported by the postdoctoral fellowship by the department of biotechnology and biosciences at University of Mialano-Bicocca, Italy.

2. Proposed experiment

The project aims to study UV-induced oxidation of PET plastics followed by chemical catalysis using Trifluoroacetic Acid (TFA). PET samples will be exposed to UV-A lamp for various durations to mimic natural photooxidation [1]. The extent of oxidation will be analyzed using Fourier-transform infrared spectroscopy (FTIR), providing insights into surface-level chemical changes in the polymer structure. Following UV oxidation, the samples will be hydrolyzed using TFA to produce uniform nanoplastics. This method, as described in the literature, dissolves PET and reprecipitates it as nanoparticles ranging from 50 to 200 nm [2]. The chemical catalysis process will help overcome the limitations of surface-based photooxidation and generate standardized PET nanoparticles for future studies (Figure 1).

The AFM-Raman with optical profiler will be used to characterize these nanoplastics, providing critical insights into their morphology and degradation behavior. Data from the AFM-Raman will be processed to create chemical and structural maps, correlating the degree of oxidation with particle size and morphology. This comprehensive analysis is essential for the standardization of nanoplastics and future studies on their environmental and health impacts. The Particle Size Analyzer will be used to determine the size distribution of the nanoplastics produced from the oxidation and hydrolysis processes. The size distribution of these nanoplastics is crucial for understanding their behavior in biological and environmental contexts.

3. Summary of previous experimental proposals or characterisation

FTIR analysis is currently ongoing to evaluate the surface oxidation, but it lack the resolution necessary for detailed chemical and structural analysis. The AFM-Raman optical profiler provides an unparalleled level of detail and accuracy. Additionally, the use of the Particle Size Analyzer will ensure precise size control of the nanoplastics, which is critical for standardization across experiments.

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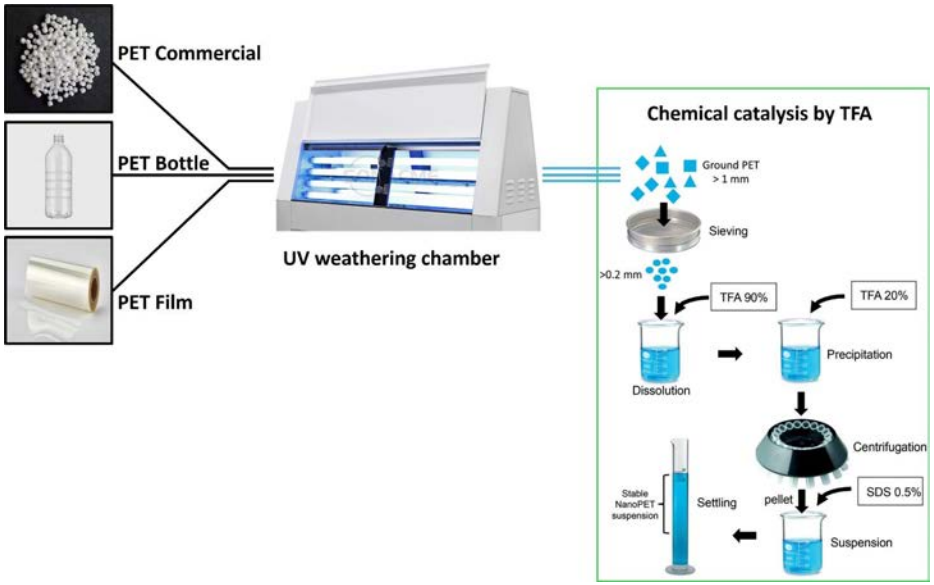

Figure 1. Experimental design to produce different PET nanoplastics by combining UV weathering followed by chemical oxidation.

4. Justification of experimental time requested

The AFM-Raman optical profiler is critical for the successful completion of this project. The unique capabilities of these instruments are essential for characterizing both the UV-oxidized and TFA-catalyzed PET nanoplastics, ensuring a thorough analysis of their properties.

We will examine three types of PET samples (commercial PET granules, bottle standards, and film standards). Each sample will be analyzed for approximately 6 hours, with an additional 4 hours allocated for setup and calibration. A total of 22 hours (~3 days) is requested to ensure high-quality, reproducible data for the chemical and structural analysis of the samples.

In addition, the Particle Size Analyzer will be used to analyze the size distribution of the same three PET samples, requiring 4 hours in total (1 hour per sample and 1 additional hour for preparation). The combination of these two instruments is essential for the comprehensive characterization of the nanoplastics.

[1] Saliu, Francesco, et al. "The release process of microfibers: from surgical face masks into the marine environment." Environmental Advances 4 (2021): 100042.

[2] Rodríguez-Hernández, Ana G., et al. "A novel and simple method for polyethylene terephthalate (PET) nanoparticle production." Environmental Science: Nano 6.7 (2019): 2031-2036.

Experiment Proposal

Experiment number GP2024168

Principal investigator	Professor Claudio Goletti, University of Rome Tor Vergata, ITALY
Co-investigator (*)	Professor Massimo Bonini, CSGI - University of Florence, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Online training on multiple instrumentation for the Students of the GREENANO Erasmus Mundus Joint Master (EMJM) on Materials Science: event #1 organized by Tor Vergata Unit

Training MRF	**AFM Raman with Optical Profiler**	**Days requested:** 1
Access Route	Direct Access	**Previous GP Number:** NO
Science Areas	Materials	**DOI:** No
Sponsored Grant	None	**Sponsor:** -
Grant Title	-	**Grant Number:** -
Start Date	-	**Finish Date:** -
Similar Submission?	-	
Industrial Links	-	

Non-Technical Abstract

This proposal is part of a series of three online events designed to provide specialized training. We propose an online training program specifically for students enrolled in the GREENANO Erasmus Mundus Joint Master (EMJM) in Materials Science. The training focuses on state-of-the-art instrumentation essential for research in materials science, helping students develop skills critical for careers in both academia and industry. Over three events, students will be introduced to advanced tools such as imaging, spectroscopy, and material characterization, provided by three ISIS@MACH ITALIA units and ISIS UK. The program includes demonstrations of real-world case studies and offers students a comprehensive overview of preparing and analyzing samples. The goal is to enhance the students' research capabilities before they begin their practical research stages. The proposed training could serve as a foundation for future users of these facilities and inspire additional training events.

Publications -

Instruments	**INES**	**Days Requested:** 1
Access Route	Direct Access	**Previous RB Number:** No
Science Areas		**DOI:** No
Sponsored Grant	None	**Sponsor:**
Grant Title	-	**Grant Number:**
Start Date	-	**Finish Date:**
Similar Submission?		
Industrial Links		

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Sample record sheet

Principal contact	Professor Massimo Bonini, CSGI - University of Florence, ITALY		
Training Instrument	**AFM Raman with Optical Profiler**	**Days Requested:** 1	
Special requirements:			

SAMPLE

Material	-	-	-
Formula	-	-	-
Forms			
Volume			
Weight			
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	-	-	-
Pressure Range	-	-	-
Magnetic field range	-	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	-	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	-	-	-
Sensitivity to vapour	-	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	-	-	-

Online training on multiple instrumentation for the Students of the GREENANO Erasmus Mundus Joint Master (EMJM) on Materials Science: event #1 organized by University Tor Vergata Unit.

1. Background and Context

The field of materials science is rapidly evolving, with advancements impacting various sectors, including technology, healthcare, and environmental science. Understanding and utilizing state-of-the-art instrumentation is crucial for young researchers to push the boundaries of innovation. The proposed training events are timely as they align with the increasing need for specialized knowledge in high-tech instrumentation, which is essential for cutting-edge research and development. The proposed training is essential for different reasons:

- skill development: introducing students to advanced instrumentation early in their careers will enhance their research capabilities and improve the quality of their experimental work.
- industry relevance: as industry increasingly relies on advanced materials, having a workforce skilled in using high-tech instruments is critical.
- research enhancement: knowledge of these instruments can significantly boost the research output and innovation potential of participating students.

This training proposal facilitates mobility and high-quality education for students across Europe, as the participants will be students enrolled in the **GREENANO Erasmus Mundus Joint Master (EMJM) on Materials Science**.

Figure 1. Partners and pillars of the GREENANO Erasmus Mundus Joint Master (EMJM) on Materials Science (as described at this link).

2. Proposed Training

The proposed training consists of three online events where state-of-the-art instrumentation relevant in the field of materials science will be introduced to the participants through selected case studies. The training participants will be students enrolled in a European master's programme, which involves studying at different universities each semester. The primary goal of "GREENANO" is to foster a new generation of engineers, researchers, and sustainability managers, equipping them to confront the major challenges posed by green and digital transitions.

The University of Rome Tor Vergata is one of the Universities involved and one of the proposers (Prof. Claudio Goletti) is the responsible for the programme. During the first semester (when the proposed training will be held) the students will be hosted by the Université de Lorraine. Helding the event online allows for maximizing the number of students allowed to participate, as well as their training before they are required to perform their own research: in fact, these students are expected to perform a research stage within the Masters. In this framework, the instruments available at ISIS@MACH ITALIA units offer a unique collection of tools for Materials Science, including advanced imaging, spectroscopy, and material characterization tools. The training will be conducted by academic staff and researchers from the following ISIS@MACH ITALIA units:

- University of Rome Tor Vergata Unit (Event #1);
- CSGI - University of Florence Unit (Event #2);
- University of Milano Bicocca Unit (Event #3).

Personnel from the INES beamline at the ISIS UK will also be involved, to present the instrumentation and opportunities available at the facility.

3. Summary of previous training proposals

This is the first proposal for such training events. Therefore, there are no previous training outcomes to summarize. However, we anticipate that this initial training will lay a strong foundation for future, more advanced training sessions.

4. Justification of Training Proposal Request

We ask for three days in total, 1 per each unit involved. The training will be conducted over three separate online events, each lasting approximately 8 hours. During the training, local scientists will demonstrate the use of the instruments online. The time will be allocated as follows:

- Introduction to the portfolio of instruments available at the Tor Vergata unit (2 hours), with special focus on the AFM Raman with Optical Profiler instrument.
- Case study #1: description of a scientific case study carried out at the Tor Vergata unit, with detailed description on how to set up and prepare samples and instruments for the experiment, as well as the analysis of the results (1 hour).
- Case study #2: description of a scientific case study carried out at the Tor Vergata unit, with detailed description on how to set up and prepare samples and instruments for the experiment, as well as the analysis of the results (1 hour).
- Description of the INES beamline at ISIS Facility: each event will present different science cases from t from the portfolio of analysis of materials for application in engineering, cultural heritage, health and earth sciences (2 hours).
- Questions and Answers session (2 hours).

This structure ensures comprehensive coverage of all essential aspects of planning measurements and using the instruments. The timing is designed to maximize learning while being mindful of the online format's limitations.

Confocal Microscope 2

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Experiment Proposal

Experiment number GP2024148

Principal investigator	Dr Margherita Izzi, University of Bari Aldo Moro, ITALY
Co-investigator	Dr Gavino Bassu, Università degli studi di Firenze, ITALY
Co-investigator (*)	Professor Marco Laurati, CSGI, ITALY
Co-investigator	Professor Nicola Cioffi, Università degli Studi di Bari Aldo Moro, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Investigation of the early-stage interaction between bacterial cells and antimicrobial surfaces
MRF Instrument	**Confocal Microscope 2** — **Days requested:** 5
Access Route	Direct Access — **Previous GP Number:** No
Science Areas	Biology and Bio-materials, Chemistry, Materials — **DOI:** -
Sponsored Grant	None — **Sponsor:** -
Grant Title	- — **Grant Number:** -
Start Date	- — **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	The uncontrolled spread of infectious diseases and antibiotic resistance has prompted the development of innovative materials aimed at controlling microbe transmission and combating harmful pathogens. However, it is nowadays essential to estimate correctly the bioactivity mechanisms of antimicrobial agents, with the ultimate goal of developing safer antimicrobials. In this sense, this proposal focuses on investigating the early stages of interaction between antimicrobial ZnO-based polymeric coatings and microorganisms, specifically examining how inorganic bioactive materials influence bacterial motility. The study will involve exposing ZnO-based composite coatings to Bacillus subtilis planktonic suspensions and tracking the evolution of bacterial motion dynamics in the short term, using time-lapse confocal microscopy imaging. For this purpose, the use of Laser Scanning Confocal Microscope Leica TCS SP8 (Confocal Microscope 2) is requested.
Publications	-

ISIS neutron and muon source	**E-platform:** No
Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Professor Marco Laurati, CSGI, ITALY
MRF Instrument **Confocal Microscope 2** **Days Requested:** 5
Special requirements:

SAMPLE

Material	polymeric coatings modified with ZnO nanostructures on silicon sheets	ZnO nanostructures embedded in different biopolymers	-
Formula	-	-	-
Forms	Solid	Solid	
Volume	cc	cc	
Weight	mg	mg	
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	- K	-
Pressure Range	- mbar	- mbar	-
Magnetic field range	- T	- T	-
Standard equipment	None	None	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	Yes	-
Sample Prep Hazards	No, there are no hazards associated with the sample preparation.	No, there are no hazards associated with the sample preparation.	-
Special equip. reqs	-	-	-
Sensitivity to air	No	No	-
Sensitivity to vapour	No	No	-
Experiment Hazards	No, there are no hazards associated with the experiment.	No, there are no hazards associated with experiment.	-
Equipment Hazards	-	-	-
Biological hazards	No, there are no biological hazards associated with the sample.	No, there are no biological hazards associated with the sample.	-
Radioactive Hazards	No, there are no radioactive hazards associated with the sample.	No, there are no radioactive hazards associated with the sample.	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Removed By User	Removed By User	-

Investigation of the early-stage interaction between bacterial cells and antimicrobial surfaces

1. Background and Context

The uncontrolled spread of infectious diseases and antibiotic resistance has prompted the development of innovative materials preventing microbe transmission and combating harmful pathogens. Nanocomposites with metal oxides represent excellent additives for antimicrobial coatings, offering broad-spectrum action against pathogens, across different industries like healthcare, food packaging, and construction [1]. The detailed action mechanisms of these materials are still the subject of investigation. Well accepted bioactivity mechanisms include the production of reactive oxygen species, the release of metal ions, and direct interaction with bacterial membranes through electrostatic forces [2]. Deepening the knowledge of bioactivity mechanisms and surface properties of antimicrobial coatings is crucial for the development of safer and more efficient materials, with a reduced environmental impact.

Aim of this proposal is to investigate the early stages of interaction between antimicrobial agents and microorganisms, focusing on the impact of inorganic bioactive materials on bacterial motility, providing a direct and unconventional way to determine the biostatic dose.

2. Proposed experiment

Motility plays a critical role in the survival, dissemination, and virulence of bacteria [3]. Understanding the potential impact of bioactive agents on bacterial motility can be crucial for controlling the spread of microorganisms. To examine the contact-mediated effect of antimicrobial surfaces on bacterial motility, the Leica TCS SP8 (Confocal Microscope 2) is requested. This instrument allows to follow bacterial motility in 3D at the micron-scale, providing the information required to elucidate the effect of the bioactive species. ZnO-based composite coatings will be put in contact with *Bacillus subtilis* planktonic suspensions and the evolution of bacterial motion dynamics will be followed at short times by time series of confocal microscopy image stacks. Different loadings of bioactive species and different biopolymers will be tested, aiming to evaluate the minimum bacteriostatic dose of selected antimicrobials and minimize their amount in real applications. The single-cell tracking method allows the analysis of thousands of individual bacteria and the resulting average mean squared displacements (MSD). Furthermore, the effect of the developed materials will be evaluated in detail on individual trajectories, extracting characteristic velocities, and splitting trajectories into segments classified as active, normal diffusive, and sub-diffusive.

3. Summary of previous experimental proposals or characterisation

Preliminary tests have demonstrated the effectiveness of this approach, revealing significant differences in how varying amounts of Zn^{2+} released from different bioactive surfaces impact bacterial motility. Electrosynthesized ZnO nanostructures [4] were incorporated into three different polymeric matrices to develop composite bioactive coatings. The antimicrobial materials and bioactive surfaces were spectroscopically and morphologically characterized. Specifically, the amount of the Zn^{2+} ionic release was evaluated by an analytical spectrophotometer [5], demonstrating that Zn^{2+} release depends on the polymeric matrix (Fig. 1). The coatings were put in contact with *B. subtilis* bacterial dispersion, to evaluate the effect of the bioactive species release on the bacteria motility in the early stage of the interaction. The MSDs obtained from 2D images are

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dramatically affected by the exposition to antimicrobial agents resulting in a significant decrease of motility after exposition to all the investigated matrices. Furthermore, the suppression of the bacterial motility is dependent on the entity of the released bioactive Zn^{2+} (Fig. 1).

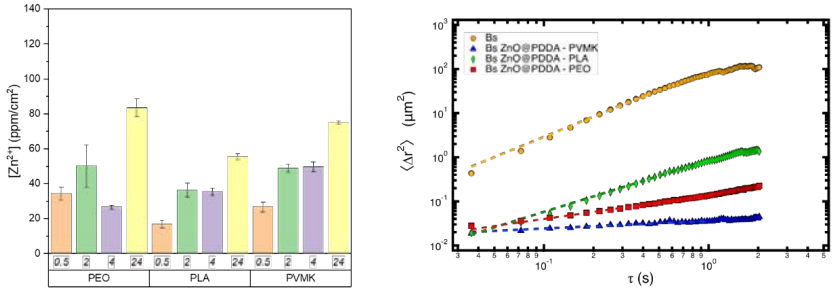

Figure 1. Zn^{2+} release from different ZnO-based composite coatings (PEO, PLA and PVMK) and their relative average mean squared displacements (MSD).

4. Justification of experimental time requested

Upon approval, the proposed experiment will allow to extend the preliminary investigations, exploring the effect of different ZnO concentrations and of other Zn-based nanostructures. Furthermore, the use of novel biopolymer dispersing matrices will be also investigated. The Leica TCS SP8 confocal microscope will be used to collect time series of 3D image stacks with a suitable resolution to statistically analyse the bacterial trajectories.

The experiments will be conducted at room temperature on the following samples:

- three replicates of three different ZnO concentrations embedded in a model polymeric matrix (9 total samples);
- three replicates of a different Zn-based nanostructure embedded in a model polymeric matrix (3 total samples);
- three replicates of ZnO nanostructures embedded in different biopolymers (9 total samples).

This will provide a total of 21 samples for analysis. Considering that an average measurement time of 6 hrs per sample will be needed to setup the experiment and perform a set of image stacks on different portions of a sample, we request 5 days at Confocal Microscope 2: Leica TCS SP8.

References

[1] E.O. Ogunsona et al., Applied Materials Today 18 (2020) 100473.
[2] L. Luo et al., ACS Appl. Nano Mater. 7 (2024) 2529–2545.
[3] J.C. Conrad, Research in Microbiology 163 (2012) 619–629.
[4] M.C. Sportelli et al. Nanomaterials 10 (2020) 473.
[5] M. Izzi et al., ChemElectroChem 10 (2023) e202201132.

Confocal Microscope 3

Experiment Proposal

Experiment number GP2024061

Principal investigator (*)	Dr Enrico Perelli Cippo, Consiglio Nazionale delle Ricerche, ITALY
Co-investigator	Dr Marco Tardocchi, CNR, ITALY
Co-investigator	Dr Marica Rebai, CNR, ITALY
Co-investigator	Dr Stephanie Cancelli, University of Milano-Bicocca, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Study of the bonding of a Diamond detector matrix with the CONFOCAL MICROSCOPE
MRF Instrument	**Confocal Microscope 3**
Days requested:	1
Access Route	Direct Access
Previous GP Number:	No
Science Areas	Engineering, Physics
DOI:	-
Sponsored Grant	None
Sponsor:	-
Grant Title	-
Grant Number:	-
Start Date	-
Finish Date:	-
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

Fast neutron detection is a crucial method for assessing plasma parameters in fusion machines. High-resolution fast neutron spectra measurements provide data on plasma temperature, fuel ion ratio, and other parameters. Solid State Detectors (SSDs) are ideal due to their small size, low cost, fast response times, low energy resolutions, low sensitivity to gamma rays, mechanical resilience, and radiation hardness. Diamond SSDs are noted for their 1% energy resolution, simpler response function, and MHz counting rate. A new diamond SSD detector, a matrix of 4 diamonds (4.5x4.5 mm²), has been developed. The bonding of the detector will be studied using the CONFOCAL MICROSCOPE 3 at ISIS@MACH ITALIA. Additionally, a proposal will be submitted to study the spectroscopic capability of the matrix with SOURIER at ISIS@MACH ITALIA. Final calibration will occur at the NILE facility at ISIS.

Publications

Rebai M et al 2016 Response function of single crystal synthetic diamond detectors to 1-4 MeV neutrons for spectroscopy of D plasmas Rev. Sci. Instrum. 87 11D823

Muraro A et al 2016 First neutron spectroscopy measurements with a pixelated diamond detector at JET Rev. Sci. Instrum. 87 11D833

Cazzaniga C et al 2014 Single crystal diamond detector measurements of deuterium-deuterium and deuteriumtritium neutrons in JET fusion plasmas Rev. Sci. Instrum. 85 043506

Instruments	**NILE**	**Days Requested:**	1
Access Route	Direct Access	**Previous RB Number:**	No
Science Areas		**DOI:**	
Sponsored Grant	None	**Sponsor:**	
Grant Title	-	**Grant Number:**	
Start Date	-	**Finish Date:**	
Similar Submission?			
Industrial Links			

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Sample record sheet

Principal contact	Dr Enrico Perelli Cippo, Consiglio Nazionale delle Ricerche, ITALY		
MRF Instrument	**Confocal Microscope 3**	**Days Requested:**	1
Special requirements:			

SAMPLE

Material	Carbon, aluminium	-	-
Formula	-	-	-
Forms	Solid		
Volume	cc		
Weight	100 g		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	No	-	-
Sample Prep Hazards	None	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	None	-	-
Equipment Hazards	-	-	-
Biological hazards	None	-	-
Radioactive Hazards	None	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	-	-

Study of the bonding of a Diamond detector matrix with the CONFOCAL MICROSCOPE 3

1. Background and Context

Fast neutron detection is one of the prominent methods for assessing the plasma parameters in fusion machines. Measuring the flux of 14 MeV neutrons on a direct line of sight on the plasma can be used to calculate the number of DT fusion reactions, and thus the fusion power; measuring the fast neutron spectra with sufficient energy resolution can be used to obtain information on the plasma ion temperature, on the fuel ion ratio nD/nT and on other quantities; lastly, measuring the neutron flux inside the breeding blanket can be used to calculate the tritium reaction rate and, therefore, the amount of tritium produced.

Solid State Detectors (SSDs) are very good candidates for the application to fusion devices , since they are compact (a few millimetres across) and have comparatively low costs respsct to other neutron sensors, allowing for arrays of detectors to be arranged into tomographic cameras (i.e. along multiple lines of sight). They also feature fast response times, low energy resolutions, low sensitivity to gamma rays, good mechanical resilience and good radiation hardness. Diamond SSDs are widely present in literature, highlighting their strengths (1% energy resolution [1], simple response function and MHz counting rate). Neutron detection in SDD is based on the collection of electron-hole pairs produced by charged particles generated by neutron interaction with 12C carbon nuclei in the detector. The main nuclear reaction channels occurring are: elastic and inelastic scattering 12C(n,n')12C; n-3α reaction (carbon breakup) 12C(n,n')3α (Q_{value}=7.23 MeV) and n-α reaction 12 C(n,α) 9 Be (Q_{value}=5.7 MeV). The latter reaction 12C(n,α)9Be is the selected one for 14 MeV neutron spectroscopy measurements from a Deuterium-Tritium (DT) plasma [2].

A new diamond SSD detector has been developed by ISTP-CNR, a matrix of 4 diamonds (4,5x4,5 mm^2). Aim of this proposal is to study the characteristics of the bonding of the detectors making use of the CONFOCAL MICROSCOPE 3 of ISIS@MACH ITALIA. Moreover, another proposal will be submitted to study the spectroscopic capability of the matrix with the SOURIRE neutron source also available at ISIS@MACH ITALIA. The results from the SOURIRE experiment will be performed at the NILE facility at the ISIS Neutron and Muon Source.

2. Proposed experiment

The present authors propose to use of the CONFOCAL MICROSCOPE 3 to perform an accurate analysis and to obtain high resolved images of the bonding of the detector.

3. Summary of previous experimental proposals or characterisation

The detector has been preliminarily tested with a ^{241}Am source at the ISTP-CNR laboratories to check the detector response and its spectroscopic capability. In particular, ^{241}Am emits α particles with three different energies (5.486, 5.442, 5.388 MeV) with branching ratio (84.5%, 13.06%, 1.62%, respectively).

A dedicated custom electronic chain was used for these measurements; the detector was coupled to a CIVIDEC C6 fast charge preamplifier, which provides a signal with a rise time of 3.5 ns and a shaping time of 25 ns. The preamplifier has a bias current of 25 mA, a gain of 6 mV fC−1 and a bandwidth of 100 MHz. A CAEN high voltage supply model NDT1470 was used to supply a voltage of +400 V to the detector (reported in the following figure).

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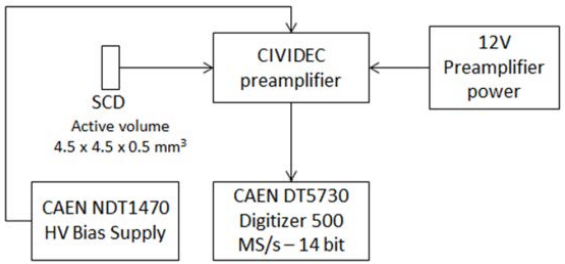

4. Justification of experimental time requested

The sample is a detector made of a matrix of 4 4,5x4,5 mm^2 Diamonds each mounted on a PCB and connected to the electrical bias via "bonds", i.e. micro-soldered micron-size gold wires It is paramount for the performances of the detectors that all the bondings are well realised.

Each detector has 4 electrical bonding and we want to obtain detailed information about the morphological aspects of the electrical contact and of the bonding to check to possible faults.

The images will be taken for all the diamonds located on the PCB, thus we request a full day to perform the experiment.

References

1. D. Rigamonti et al., Neutron spectroscopy measurements of 14 MeV neutrons at unprecedented energy resolution and implications for deuterium–tritium fusion plasma diagnostics, Meas. Sci. Technol. 29 (2018) 045502 (9pp), doi: 10.1088/1361-6501/aaa675
2. A. V. Krasilnikov et al., Study of d-t neutron energy spectra at JETusing natural diamond detectors, Nucl. Instrum. Methods Phys. Res. A476 (2002) 500-505.

Experiment Proposal

Experiment number GP2024170

Principal investigator	Professor Claudio Goletti, University of Rome Tor Vergata, ITALY
Co-investigator (*)	Professor Massimo Bonini, CSGI - University of Florence, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Online training on multiple instrumentation for the Students of the GREENANO Erasmus Mundus Joint Master (EMJM) on Materials Science: event #3 organized by Milano Bicocca Unit

Training MRF	**Confocal Microscope 3**	**Days requested:** 1
Access Route	Direct Access	**Previous GP Number:** NO
Science Areas	Materials	**DOI:** No
Sponsored Grant	None	**Sponsor:** -
Grant Title	-	**Grant Number:** -
Start Date	-	**Finish Date:** -
Similar Submission?	-	
Industrial Links	-	

Non-Technical Abstract

This proposal is part of a series of three online events designed to provide specialized training. We propose an online training program specifically for students enrolled in the GREENANO Erasmus Mundus Joint Master (EMJM) in Materials Science. The training focuses on state-of-the-art instrumentation essential for research in materials science, helping students develop skills critical for careers in both academia and industry. Over three events, students will be introduced to advanced tools such as imaging, spectroscopy, and material characterization, provided by three ISIS@MACH ITALIA units and ISIS UK. The program includes demonstrations of real-world case studies and offers students a comprehensive overview of preparing and analyzing samples. The goal is to enhance the students' research capabilities before they begin their practical research stages. The proposed training could serve as a foundation for future users of these facilities and inspire additional training events.

Publications -

Instruments	**INES**	**Days Requested:** 1
Access Route	Direct Access	**Previous RB Number:** No
Science Areas		**DOI:** No
Sponsored Grant	None	**Sponsor:**
Grant Title	-	**Grant Number:**
Start Date	-	**Finish Date:**
Similar Submission?		
Industrial Links		

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Sample record sheet

Principal contact	Professor Massimo Bonini, CSGI - University of Florence, ITALY	
Training Instrument	**Confocal Microscope 3**	**Days Requested:** 1
Special requirements:		

SAMPLE

Material	-	-	-
Formula	-	-	-
Forms			
Volume			
Weight			
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	-	-	-
Pressure Range	-	-	-
Magnetic field range	-	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	-	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	-	-	-
Sensitivity to vapour	-	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	-	-	-

Online training on multiple instrumentation for the Students of the GREENANO Erasmus Mundus Joint Master (EMJM) on Materials Science: event #3 organized by University of Milano Bicocca Unit.

1. Background and Context

The field of materials science is rapidly evolving, with advancements impacting various sectors, including technology, healthcare, and environmental science. Understanding and utilizing state-of-the-art instrumentation is crucial for young researchers to push the boundaries of innovation. The proposed training events are timely as they align with the increasing need for specialized knowledge in high-tech instrumentation, which is essential for cutting-edge research and development. The proposed training is essential for different reasons:

- skill development: introducing students to advanced instrumentation early in their careers will enhance their research capabilities and improve the quality of their experimental work.
- industry relevance: as industry increasingly relies on advanced materials, having a workforce skilled in using high-tech instruments is critical.
- research enhancement: knowledge of these instruments can significantly boost the research output and innovation potential of participating students.

This training proposal facilitates mobility and high-quality education for students across Europe, as the participants will be students enrolled in the **GREENANO Erasmus Mundus Joint Master (EMJM) on Materials Science**.

Figure 1. Partners and pillars of the GREENANO Erasmus Mundus Joint Master (EMJM) on Materials Science (as described at this link).

2. Proposed Training

The proposed training consists of three online events where state-of-the-art instrumentation relevant in the field of materials science will be introduced to the participants through selected case studies. The training participants will be students enrolled in a European master's programme, which involves studying at different universities each semester. The primary goal of "GREENANO" is to foster a new generation of engineers, researchers, and sustainability managers, equipping them to confront the major challenges posed by green and digital transitions.

The University of Rome Tor Vergata is one of the Universities involved and one of the proposers (Prof. Claudio Goletti) is the responsible for the programme. During the first semester (when the proposed training will be held) the students will be hosted by the Université de Lorraine. Helding the event online allows for maximizing the number of students allowed to participate, as well as their training before they are required to perform their own research: in fact, these students are expected to perform a research stage within the Masters. In this framework, the instruments available at ISIS@MACH ITALIA units offer a unique collection of tools for Materials Science, including advanced imaging, spectroscopy, and material characterization tools. The training will be conducted by academic staff and researchers from the following ISIS@MACH ITALIA units:

- University of Rome Tor Vergata Unit (Event #1);
- CSGI - University of Florence Unit (Event #2);
- University of Milano Bicocca Unit (Event #3).

Personnel from the INES beamline at the ISIS UK will also be involved, to present the instrumentation and opportunities available at the facility.

3. Summary of previous training proposals

This is the first proposal for such training events. Therefore, there are no previous training outcomes to summarize. However, we anticipate that this initial training will lay a strong foundation for future, more advanced training sessions.

4. Justification of Training Proposal Request

We ask for three days in total, 1 per each unit involved. The training will be conducted over three separate online events, each lasting approximately 8 hours. During the training, local scientists will demonstrate the use of the instruments online. The time will be allocated as follows:

- Introduction to the portfolio of instruments available at the Milano Bicocca unit (2 hours), with special focus on the Confocal Microscope 3 instrument.
- Case study #1: description of a scientific case study carried out at the Milano Bicocca unit, with detailed description on how to set up and prepare samples and instruments for the experiment, as well as the analysis of the results (1 hour).
- Case study #2: description of a scientific case study carried out at the Milano Bicocca unit, with detailed description on how to set up and prepare samples and instruments for the experiment, as well as the analysis of the results (1 hour).
- Description of the INES beamline at ISIS Facility: each event will present different science cases from t from the portfolio of analysis of materials for application in engineering, cultural heritage, health and earth sciences (2 hours).
- Questions and Answers session (2 hours).

This structure ensures comprehensive coverage of all essential aspects of planning measurements and using the instruments. The timing is designed to maximize learning while being mindful of the online format's limitations.

DNA Sequencing NGS

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Experiment Proposal

Experiment number GP2024042

Principal investigator	Professor Domenico Lo Vetro, Università di Firenze, ITALY
Co-investigator (*)	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Alessandro Cianchi, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Exploring environmental dynamics in ancient remains before and after the last glacial maximum using aDNA sequencing
MRF Instrument	**DNA Sequencing NGS**
Days requested:	8
Access Route	Direct Access
Previous GP Number:	No
Science Areas	Biology and Bio-materials, Environment
DOI:	No
Sponsored Grant	None
Sponsor:	-
Grant Title	-
Grant Number:	-
Start Date	-
Finish Date:	-
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

Our aim is to study environmental ancient sediments coming from sediments extracted in the Romito cave (Cosenza, Italy) before and after the Last Glacial Maximum by multi-instrumental approach. The characterization of these samples to study the presence of aDNA will be performed by sequencing the DNA using the DNA Sequencing NGS instrument at the Rome Tor Vergata Unit. Data will be cross compared to verify consistency with the one obtained by FT-IR measurements and by retrieving all the proteins in the sediments using the Mass Spectrometer 2 at the University of Milano Bicocca Unit. Hence, we aim here to request access to the DNA Sequencing NGS instrument at the Rome Tor Vergata Unit of IM@IT.

Publications

Berto et al. 2022. Archaeological and Anthropological Sciences. Vol. 14, article N. 127, (2022)

López-García et al. 2014. Palaeogeography, Palaeoclimatology, Palaeoecology, Vol 251, Issues 3–4, Pages 500-526

Scorrano et al. 2022. Communications Biology, Vol 5, Article N 1262 (2022)

Instruments	**INES**	**Days Requested:**	3
Access Route	Direct Access	**Previous RB Number:**	No
Science Areas		**DOI:**	No
Sponsored Grant	None	**Sponsor:**	
Grant Title	-	**Grant Number:**	
Start Date	-	**Finish Date:**	
Similar Submission?			
Industrial Links			

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Sample record sheet

Principal contact	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY		
MRF Instrument	**DNA Sequencing NGS**	**Days Requested:**	8
Special requirements:			

SAMPLE

Material	Sediments/remains	Sediments/remains	Sediments/remains
Formula	organic (collagen) and sand/limestone mixtures	organic (collagen) and sand/limestone mixtures	organic (collagen) and sand/limestone mixtures
Forms	Solid	Solid	Solid
Volume	4-10 cc	4-10 cc	4-10 cc
Weight	2-10 g	2-10 g	2-10 g
Container or substrate	Sterile tube or aluminum foil	Sterile tube or aluminum foil	Sterile tube or aluminum foil
Storage Requirements	Freezer (-20C)	Freezer (-20C)	Freezer (-20C)

SAMPLE ENVIROMENT

Temperature Range	273 - 320 K	273 - 320 K	273 - 320 K
Pressure Range	1000 - 1010 mbar	1000 - 1010 mbar	1000 - 1010 mbar
Magnetic field range	- T	- T	- T
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	Yes	Yes
Sample Prep Hazards	No	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	No	No
Sensitivity to vapour	No	No	No
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	No	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	Returned to user by instrument scientist (when inactive)	Returned to user by instrument scientist (when inactive)

Exploring environmental dynamics in ancient remains before and after the last glacial maximum using aDNA sequencing

Background and Context

Ancient environmental DNA (aDNA) refers to genetic material obtained from environmental samples such as soil, sediment, ice and water, which is thousands or millions of years old [1]. The study of ancient DNA is fascinating because it allows us to reconstruct past ecosystems, understand evolutionary processes and trace the impacts of climate and environmental changes on biodiversity over the millennia. This field is particularly timely, as highlighted by a recent publication in Nature detailing the oldest DNA ever recovered from the environment: 2-million-year-old samples that allowed researchers to reconstruct the ecosystem in Greenland [1].

The broader relevance of ancient DNA research lies in its ability to shed light on the complex interactions between climate, environment and living organisms over geological time scales. By understanding past ecosystems, we gain insights into species resilience and vulnerability, which can guide current biodiversity conservation strategies. This proposal aims to characterize samples coming from sediments extracted in the Romito cave (Cosenza, Italy, see Figure 1) [2, 3] before and after the Last Glacial Maximum (LGM) by multi-instrumental approach. The reason of this is twofold. The LGM, when ice sheets were at their maximum extent, was a period of significant climatic and environmental shifts with profound impacts on global ecosystems and human populations. In an era marked by rapid climate change, insights from the LGM can inform our understanding of how ecosystems and species, including humans, responded to extreme climatic conditions. From the other, the Romito cave is one of the most significant Upper Palaeolithic archaeological sites on the Italian peninsula with a well-dated stratigraphy spanning from ~24,000 to 6,000 years before present (BP) (Figure 1d).

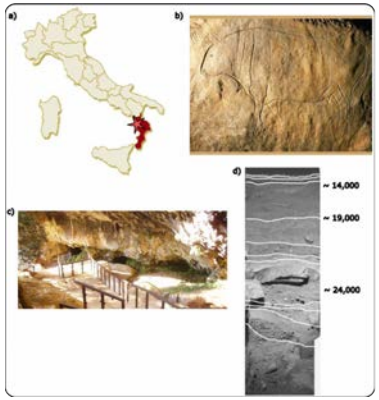

Figure 1. a) Location of Romito cave; b) rock art (Bos primigenius) in the rock-shelter outside the cave; c) cave entrance d) general stratigraphic sequence modified from Blockley et al. (2018).

To this end, we will study two samples from the oldest layer (pre-LGM, ~24000 years BP) of the sediment, two samples from the LGM (~19000 years BP), and two samples post-LGM (~14000 years BP). The characterization of these samples will be assessing first the mineralogy composition of the sediment by means of X-ray diffraction (Multipurpose X-ray Diffractometer instrument, CNR-ICMATE Unit) and Small/Wide Angle X-ray Scattering (SAXS GISAXS instrument, CSGI-Unit) measurements. Complementary neutron diffraction data on the same set of samples will be measured at the INES beamline, ISIS neutron and muon source (UK). The minerals in each sediment sample will be quantified using Rietveld refinement of X-rays and neutron diffraction data and cross-compared to verify consistency. Later, the presence of organic compounds [4] will be inferred by Fourier-transform infrared spectroscopy measurement on the FT-IR Nicolet instrument available at the Rome Tor Vergata Unit. To follow we will perform shotgun sequencing of aDNA, using the DNA Sequencing NGS of Rome Tor Vergata Unit and finally retrieve all the proteins in the remains using Mass Spectrometer 2 at the University of Milano Bicocca Unit which will be useful to support the DNA data [5].

Proposed experiment

In this experiment we aim to perform shotgun sequencing of aDNA using the DNA Sequencing NGS instrument at the Rome Tor Vergata Unit to study the presence of aDNA in 6 samples coming from sediments extracted in the Romito cave. Two samples have been extracted by sediments in the oldest layer (pre-LGM, ~24000 years BP), n. 2 samples from the LGM (~19000 years BP), and two samples post-LGM (~14000 years BP). Results of this experiment will be cross compared to verify consistency with data obtained by the characterization of organic compounds by FT-IR measurement and by retrieving all the proteins in the sediments using the Mass Spectrometer 2 instrument available at the University of Milano Bicocca Unit.

Justification of experimental time requested

Each of the 6 sample will be enclosed in a sterile tube with about 2 -10 g (4-10 cc) of sediments, and it will be maintained at -20 °C temperature to preserve aDNA during the measurements. Before performing the DNA sequencing, for each sample a dedicated complex laboratory workflow must be performed. aDNA must be extracted and for each one library needs to be built and screened by the NGS machine. After screening, the best samples will undergo deep sequencing, which requires additional library preparation and sequencing time. After discussion with the instrument scientist, we estimate that 2 days will be needed for the extraction step, 4 days for library building, and 2 days for sequencing, for a total of 8 days.

References

[1] Kjær et al., Nature **612** (2022), p. 283–291.
[2] Blockley et al. 2018. Quaternary Science Reviews, 184: 5-25.
[3] Craig et al., 2010. Journal of Archaeological Science, 37: 2504-2512.
[4] Scorrano et al., 2015. Annals of Human Biology, 42: 10-19.
[5] Scorrano et al. 2022. Communications Biology, 5: 1262.

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Experiment Proposal

Experiment number GP2024051

Principal investigator	Dr Pier Francesco Fabbri, Museo e Istituto Fiorentino di Preistoria, ITALY
Co-investigator (*)	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Carla Andreani, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Alessandro Cianchi, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Ancient DNA analysis of a Neolithic human tooth from eastern Sicily using NGS: insights and implications

MRF Instrument	**DNA Sequencing NGS**	**Days requested:**	5
Access Route	Direct Access	**Previous GP Number:**	No
Science Areas	Biology and Bio-materials, Cultural Heritage	**DOI:**	-
Sponsored Grant	None	**Sponsor:**	-
Grant Title	-	**Grant Number:**	-
Start Date	-	**Finish Date:**	-
Similar Submission?	-		
Industrial Links	-		

Non-Technical Abstract

This is the last of three proposals for IM@IT tailored to study a tooth belonging to the first Neolithic individual buried at Rocchicella Paliké (CT) in eastern Sicily. The burial is dated back between 5210- 4840 BC (calibrated) (LTL12788A). In two distinct proposals we requested to perform 3D characterisations of the human tooth using XRD TOMOGRAPHY (at the CNR-IPCB Unit), Small/Wide Angle X-ray Scattering (SAXS GISAXS instrument, CSGI-Unit) measurements, and neutron diffraction and neutron tomography at INES and at IMAT beamlines at ISIS Facility (UK). After these characterisations we will focus on the genetic composition, migration patterns, and interaction of this individual with local Mesolithic communities. To this aim in this proposal, we request the use of the NGS instrument, located at the Rome Tor Vergata IM@IT' Unit, for extracting ancient DNA (aDNA) from the tooth and to follow by sequencing the human genome (a destructive analysis).

Publications

Lonoce et al. 2023. Journal of Archaeological Science 155, 105790
Viva et al. 2023. Archaeological and Anthropological Sciences 15:193
Vincenti et al. 2023. American Journal of Biological Anthropology 183: e24911.

ISIS neutron and muon source — **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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ISIS Neutron and Muon Source

Sample record sheet

Principal contact	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY		
MRF Instrument	**DNA Sequencing NGS**	**Days Requested:**	5
Special requirements:			

SAMPLE

Material	intact tooth (enamel, dentine and cementum) about 3x3x1 cm^3	-	-
Formula	Not known	-	-
Forms	Solid		
Volume	8-12 cc		
Weight	200-500 mg		
Container or substrate	-	-	-
Storage Requirements	plastic box	-	-

SAMPLE ENVIROMENT

Temperature Range	273 - 320 K	-	-
Pressure Range	1000 - 1010 mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	No	-	-
Special equip. reqs	No	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	No	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	-	-

Ancient DNA analysis of a Neolithic human tooth from eastern Sicily using NGS: insights and implications

1. Background and Context

The Neolithic period, marking the transition from hunter-gatherer societies to agricultural communities, represents one of the most transformative periods in human history (Barker et al. 2015). This shift, often referred to as the Neolithic Revolution, began around 10,000 years ago in the Near East and gradually spread across Europe and the Mediterranean. During the Neolithic transition, farming communities originated in the Middle East and then started to expand into new territories (Hofmanová et al., 2016). These groups spread through Anatolia and the Balkans (Lazaridis et al., 2014; Mathieson et al., 2018), progressively admixing with local hunter-gatherers (Lipson et al., 2017). There are profound differences in the spatiotemporal patterns of Neolithization across Europe, with significant shifts in genetic ancestry varying by region (Allentoft et al., 2024). This genetic transition was particularly extensive in southern Europe, especially in Italy. In this framework east Sicily, strategically located in the central Mediterranean, offers a unique vantage point for studying the diffusion of Neolithic culture and technology. It holds the potential to shed light on the genetic diversity of early Neolithic farmers, their origins, and their genetic legacy. The peopling of Sicily from the Upper Palaeolithic to the Mesolithic has already shown a peculiar pattern involving migrations and partial replacements of local populations (Mathieson et al., 2018; Catalano et al., 2020; Yu et al., 2022; Scorrano et al., 2022).

This project aims to delve deeper into understanding whether the arrival of Neolithic communities in this area involved complex interactions between incoming agriculturalists and indigenous Mesolithic hunter-gatherers by analysing the ancient DNA (aDNA) of the first Neolithic individual from the site Rocchicella Paliké (CT) in eastern Sicily, dated back to the years 5210-4840 BC (calibrated) (LTL12788A) in East Sicily (Figure 1). These interactions likely facilitated the exchange of ideas, technologies, and genetic material, leading to the establishment of early farming villages.

Figure 1: localization of the site.

To characterise the human tooth, we will firstly perform non-destructive X-ray computed tomography (XCT) by XRD TOMOGRAPHY (at the CNR-IPCB Unit), Small/Wide angle X-ray Scattering (SAXS GISAXS instrument, CSGI-Unit), and neutron tomography and diffraction [at the IMAT and INES beamlines, ISIS (UK)]. The elemental composition of the sample will be quantified using Rietveld refinement of X-rays using XRD TOMOGRAPHY and SAXS GISAXS, and neutron diffraction data using INES and neutron tomography, using IMAT, will be cross compared to verify consistency. XCT scan will be complemented with neutron tomography data, which is more sensitive to detect organic-like material. To follow a destructive shotgun sequencing of aDNA will be performed on the tooth, using the DNA Sequencing NGS of Rome Tor Vergata Unit.

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2. Proposed experiment

In this experiment we aim to perform a destructive shotgun sequencing of aDNA on the human tooth belonging to the first Neolithic individual buried at Rocchicella Paliké (CT) in eastern Sicily, using the DNA Sequencing NGS operating at the IM@IT Unit-University of Rome Tor Vergata. Results of this experiment will be accompanied by those of two distinct non-destructive analysis using IM@IT' instruments which will be requested in separate proposals. The first analysis will request the use of the XRD TOMOGRAPHY (at the CNR-IPCB Unit). 3D rendering and analysis of the reconstructed XCT scan will be compared and complemented by neutron computed tomography reconstructed data that will be acquired at the IMAT@ISIS beamline. The second analysis will request the use of Small/Wide angle X-ray Scattering (using the SAXS GISAXS instrument, CSGI-Unit) and foresee the match of X-ray data with neutron diffraction data to be performed at the INES@ISIS beamline.

4. Justification of experimental time requested

The selected intact tooth sample will be processed using the DNA Sequencing NGS instrument. Before the DNA sequencing, the sample must undergo a dedicated, complex laboratory workflow. This involves extracting aDNA, followed by constructing and screening one library using the NGS machine. After screening, additional libraries will be built and sequenced to achieve sufficient DNA coverage for various population genetic analyses. Based on our experience, we estimate that 2 days will be needed for the extraction step, 1 day for library building, and 2 days for sequencing, for a total of 5 days.

References

Allentoft et al. Nature 625, 301–311 (2024).
Barker, G. & Goucher, C. (Cambridge Univ. Press, 2015).
Catalano et al. Quaternary International 537, 24-32 (2020).
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Lazaridis, I. et al. Nature 513, 409–413 (2014).
Lipson, M. et al. Nature 551, 368–372 (2017).
Mathieson, I. et al. Nature 555, 197 (2018).
Scorrano et al. Communications Biology 5, 1262 (2022)
Yu et al. iScience 25, 104244 (2022)

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Science and Technology Facilities Council
ISIS Neutron and Muon Source

Experiment Proposal

Experiment number GP2024053

Principal investigator	Professor Francesca Bertoldi, Ca&039; Foscari University Venice, ITALY
Co-investigator (*)	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Alessandro Cianchi, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Ancient DNA analysis in a Late Bronze age human child from Doghlauri burial ground (Georgia), affected by tuberculosis
MRF Instrument	**DNA Sequencing NGS** — **Days requested:** 5
Access Route	Direct Access — **Previous GP Number:** NO
Science Areas	Biology and Bio-materials, Cultural Heritage — **DOI:** -
Sponsored Grant	None — **Sponsor:** -
Grant Title	- — **Grant Number:** -
Start Date	- — **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	The proposed experiment proposes a destructive shotgun sequencing of ancient DNA (aDNA) of a tooth and a rib belonging to a non-adult individual from tomb 69 dated 1221-1048 calibrated BC from Aradetis Orgora in the Kareli municipality of the Shida Kartli province of Georgia, utilizing Next-Generation Sequencing (NGS) technology. The remains exhibited pleural bone lesions indicative of tuberculosis. This suggests a potential correlation between the disease and the urban development of Aradetis Orgora during the LBA. The project's primary objective is to extract from these remains the genetic information and reconstruct the child's genetic ancestry, as well as to identify potential genetic evidence of tuberculosis using DNA Sequencing NGS operating at the IM@IT Univ Rome Tor Vergata - Unit.
Publications	Bertoldi et al. 2021. Brepols: 137-152. Milano and Bertoldi (Editors), 2014. (Venezia, 15-17 giugno 2006), Padova, Sargon. De Luca et al. 2010. ournal of Archaeological Sciences, 37: 3048-3058

ISIS neutron and muon source — **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY
MRF Instrument **DNA Sequencing NGS** **Days Requested:** 5
Special requirements:

SAMPLE

Material	intact tooth (2*1*1 cm^3) + rib fragment (4*1*2 cm^3)	-	-
Formula	enamel, dentine and cementum. Hydroxyapatite and collagen	-	-
Forms	Solid		
Volume	5-10 cc		
Weight	3-5 g		
Container or substrate	Sterile tube or aluminum foil	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	273 - 320 K	-	-
Pressure Range	1000 - 1010 mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Ancient DNA analysis in a Late Bronze age human child from Doghlauri burial ground (Georgia), affected by tuberculosis

1. Background and Context

The archaeological complex of Aradetis Orgora (ca 40 ha in total surface) in the Kareli municipality of the Shida Kartli province of Georgia, occupies a strategic position on three mounds in the Kura River valley (Fig. 1). During the Late Bronze/Early Iron Age (LBA/EIA), the settlement reached its maximum expansion. To the north, the Doghlauri cemetery spans 8 hectares, separated from the Main Mound by a small stream (Gagoshidze and Rova 2018). Investigations at this site, initiated by Georgian archaeologists in 1979, have revealed nearly 70 Bronze Age burials, including a notable burial with a chariot and two horses.

Figure 1 localization and map of the site; rib portions showing bone lesions.

Archaeological investigations from 2012 to 2015, prompted by the imminent construction of a new highway, uncovered an additional 450 burials (Gagoshidze and Rova 2020). These belong to the Early Bronze Age (EBA, Kura-Araxes culture) and the LBA/EIA (Lchashen-Tsitelgori culture). From these findings, only a portion part of the human remains brought to light were available for detailed study, comprising remains from 75 burials: 23 from the EBA and 52 from the LBA. Despite the disparity in the number of graves, with EBA graves often being collective, the EBA sample represents 52 individuals, and the LBA sample includes 56 individuals, consisting of males, females, and juveniles (Bertoldi et al. 2016; Rasia et al. 2021).

Anthropological data, cross-referenced with archaeological documentation, revealed changes in burial customs. Collective burials, accommodating multiple primary depositions, were prevalent during the EBA, while single burials became the norm in the LBA. Notably, a child found in tomb 69, dated to 1221-1048 calibrated BC and corroborated by pottery grave goods dated to the LBA, despite poor preservation, showed evidence of pleural bone lesions on the ventral part of ribs, likely indicative of tuberculosis (Fig. 1). These findings suggest a possible link between the disease and the urban context of Aradetis Orgora, which was experiencing significant development during the LBA (Rasia et al. 2021, 2023).

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The primary objective of this project is to analyse the child found in tomb 69 to retrieve its genetic information and to reconstructs his genetic ancestry background. Moreover, we aim to retrieve genetic evidence of tuberculosis to gain insights into the social and environmental factors that influenced these changes. *Mycobacterium tuberculosis* is known to be challenging to diagnose molecularly, even in modern symptomatic patients (Wood et al. 2015, 2019; Shenai et al. 2013; Mesman et al. 2020). Moreover, it shares approximately 99% genetic sequence identity with other common soil *Mycobacteria* (Borowka et al. 2019). However, it has been identified in ancient individuals when bone lesions provide supporting evidence (Scorrano et al., 2022). Given the lesions observed in the bone remains, we believe it is possible to study this pathogen further. This research could contribute to a deeper understanding of the cultural and health dynamics within ancient urban settlements in the South Caucasus region.

2. Proposed experiment

In this experiment we aim to perform the destructive shotgun sequencing of ancient DNA (aDNA). It will be performed on the tooth and on the ribs of the non-adult individual buried in tomb 69 of Doghlauri cemetery, using the DNA Sequencing NGS operating at the IM@IT Unit-University of Rome Tor Vergata.

4. Justification of experimental time requested

The selected intact tooth sample and rib sample will be processed using the DNA Sequencing NGS instrument. Before the DNA sequencing, the sample must undergo a dedicated, complex laboratory workflow. This involves extracting aDNA, followed by constructing and screening one library using the NGS machine. After screening, additional libraries will be built and sequenced to achieve sufficient DNA coverage for various population genetic analyses. Based on our experience, we estimate that 2 days will be needed for the extraction step, 1 day for library building, and 2 days for sequencing, for a total of 5 days.

References

Bertoldi et al. 2016. ISBN 978-9941-13-555-2.
Borowka et al. 2019. GigaScience 8, giz065.
Rasia et al. 2023. 13th ICAANE meeting, Copenhagen.
Gagoshidze and Rova. 2018. Orientalia Lovaniensia Analecta 268, 521-546.
Gagoshidze and Rova. 2020. https://doi.org/10.2307/j.ctv10tq3zv
Mesman et al. 2020. Sci. Rep. 10, 22231.
Rasia et al. 2021. Sci. Rep. 5, 8668 (2015).
Scorrano et al 2022. *Sci Rep* 12, 6468.
Shenai et al. 2013. J. Clin. Microb. 51, 4161–4166.
Wood et al. 2019. Genome Biol. 20, 257–269.

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Experiment Proposal

Experiment number GP2024078

Principal investigator	Professor Mauro Rubini, Sovraintendenza, ITALY
Co-investigator (*)	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	A multidisciplinary aDNA study of the unique ancient Homo cepranensis petrous bone
MRF Instrument	**DNA Sequencing NGS** **Days requested:** 7
Access Route	Direct Access **Previous GP Number:** No
Science Areas	Biology and Bio-materials, Cultural Heritage **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

Our aim is to study a petrous bone of unique ancient remain Homo cepranensis (Ceprano, località Campogrande, Italy) by multi-instrumental approach through a combined series of non-destructive and destructive analyses. For non-destructive analyses we request in distinct proposals the RETINA instrument to perform XRD Tomography, for a 3D reconstruction combined with XRF maps, and the IMAT beamline at ISIS facility for complementary neutron tomography and neutron time of flight Prompt Gamma Activation Analysis (T-PGAA). In these analyses the scope is to obtain a 3D reconstruction of the Homo cepranensis remain as well as the uranium series. For complementary destructive characterizations, in distinct proposals, we request the proteomic analyses and the ancient DNA (aDNA) using the IM@IT' Mass Spectrometer 2 and the DNA Sequencing NGS instruments. The latter analysis is the objective of the present proposal.

Publications

Di Vincenzo. Sci Rep 7, 13974 (2017).
Manzi. J. Hum. Evol. 59, 580-585 (2010).
Rubini. J. Hum. Evol. 77, 204-216 (2014)

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY
MRF Instrument	**DNA Sequencing NGS** **Days Requested:** 7
Special requirements:	

SAMPLE

Material	petrous bone (about 3x2x1 cm3)	-	-
Formula	hydroxyapatite and collagen.	-	-
Forms	Solid		
Volume	6-9 cc		
Weight	350-600 mg		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	273 - 320 K	-	-
Pressure Range	7000 - 1010 mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

A multidisciplinary aDNA study of the unique ancient *Homo cepranensis* petrous bone

1. Background and Context

In this proposal we tender to study a remain of a unique ancient sample, *Homo cepranensis*, by multi-instrumental approach. The artifact was discovered on March 13, 1994, by archaeologist Italo Biddittu during surface reconnaissance along the route of a highway under construction near Ceprano (locality of Campogrande in the province of Frosinone) in the lower Sacco Valley (Figure 1). The bulldozers that facilitated the discovery of the artifact at the same time likely caused its fragmentation. The fossil artifact is limited to the neurocranium (calvarium). The fragments were contained within a series of stratified fluvio-lacustrine deposits. About 50 large fragments were unearthed in a small area near the original discovery, and more than 200 small pieces were collected by sieving the sediments [1]. Unfortunately, most of the facial bones, as well as much of the cranial base and almost the entire left parietal, were not found. Currently, the fossil is located at the Superintendency of Archaeology, Fine Arts, and Landscape for the Provinces of Frosinone and Latina.

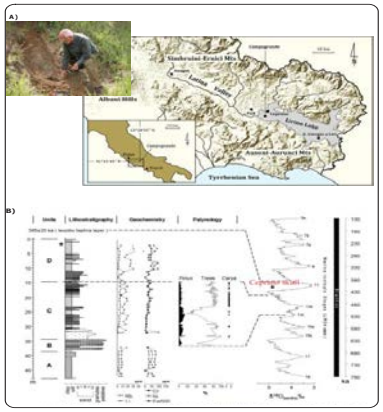
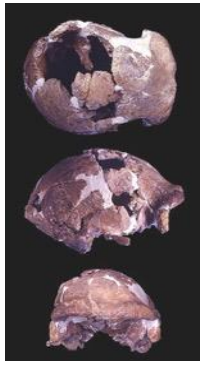


Figure 1.A) geographical localization of the site with Italo Biddittu during the discovery; B) stratigraphy, geochemistry [2], palynological data and $^{40}Ar/^{39}Ar$ dating [3].

The current form of the artifact is the result of a reconstruction initiated in 1994 by Prof. A. Ascenzi continued by Prof. R. J. Clarke and reviewed by paleoanthropologist M. A. de Lumley and Prof. F. Mallegni [4] (Figure 2). Previous characterisations were carried out directly with the original fragments and with extensive use of dental plaster. The calvarium was already analysed using X-ray microtomography (μCT) at the Multidisciplinary Laboratory of the Abdus Salam International Centre for Theoretical Physics in Trieste. Medical CT scans and recent μCT scans of the calvarium revealed the extent of the plaster and discouraged its mechanical removal. Attempts to digitally remove the plaster from the calvarium also failed, using both globally applied threshold filters and manual operations on each tomographic section; only with high-resolution 3D imaging it was possible to digitally remove the dental plaster insertions and separate the fragments [5]. The calvarium is quite well-preserved, although incomplete, there are no absolute dates and relative dating, based on the regional geo-stratigraphic and paleontological framework, place it between 0.9 and 0.8 Ma [6] .Recent magneto-stratigraphic analyses of the lacustrine and fluvial sediments recovered from cores taken at the site of the artifact, however, have provided a different relative dating; according to these studies, the stratigraphic level containing the artifact itself is between 0.5 Ma and 0.35 Ma [3] (Figure 1). The artifact also shows a series of characteristics, such as a cranial capacity of 1180-1200 cm³, typical of the oldest forms of humanity from the Middle Pleistocene. Cladistic studies initially attributed it to the species *H. erectus* and later to *H. heidelbergensis*, though its exact classification remains unclear. In this proposal our aim is to solve this question by analysing one petrous bone of the sample using multimolecular destructive analyses: proteomics and ancient DNA analysis (aDNA). The aDNA analysis on this remain is quite challenging because, to date, the oldest ancient genome published from the same latitude is from Sima de los Huesos (Spain), dated back 430,000 years ago [7]. Moreover, the aDNA analysis will be complemented by the proteomic approach (in a distinct request), which in the last five years has demonstrated the possibility of retrieving molecular information from very old specimens from warm environments, such as *Gigantopithecus* (1.9 million years old [8]) and *Homo antecessor* (between 772,000-949,000 years ago [9]). Of great interest is to date for the first time the uranium series and through the fragments to realise a virtual reconstruction of part of the remain.

Figure 2: *H. cepranensis* skull reconstruction.



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2. Proposed experiment

In this specific proposal we aim to perform shotgun sequencing of aDNA using the NGS instrument at the IM@IT' Unit -University of Rome Tor Vergata to study the unique ancient *Homo cepranensis* petrous bone. Results of this experiment will be cross compared to verify consistency with data obtained by a separate proposal requesting the use of Mass Spectrometer 2 available at IM@IT' Unit - University of Milano Bicocca to perform the proteomic analysis. Moreover, we plan to request access, in separate proposals, to the RETINA MRF instrument to perform X-ray tomography with hard X-ray beams, allowing a 3D reconstruction of an extended object, and the concurrent collection of X-ray fluorescence data for 3D chemical composition map. In a distinct proposal using IMAT and T-PGAA we will obtain the 3D reconstruction a complete digital twin of the sample before its partial destruction in later characterizations and detect any isotopes from the uranium decay chain. This analysis will complementary elemental characterization with respect to X-ray fluorescence.

3. Justification of experimental time requested

Before performing the DNA sequencing, for the sample a dedicated complex laboratory workflow must be performed. aDNA must be extracted and for each one library needs to be built and screened by the NGS machine. After screening, the best samples will undergo deep sequencing, which requires additional library preparation and sequencing time. After discussion with the instrument scientist, we estimate that 2 days will be needed for the extraction step, 3 days for library building, and 2 days for sequencing, a total of 7 days.

References

[1] Ascenzi, Segre. 2000. In The Origin of Humankind: 25-33; [2] Lisiecki, Rayamo. 2005. Paleoceanography Paleoceanography, 20. [3] Nomade et al. 2011. Quetarnary Geochronology, 6: 453-457. [4] Mallegni et al. 2003. Coptes Rendus Palevol, 2: 153-159. [5] Di Vincenzo et al. 2017. Scientific Reports 7: 13974. [6] Manzi et al. 2001. Proc Natl Acad Sci USA (PNAS) 98: 10011-10016. [7] Meyer et al. 2016. Nature 531: 504–507. [8] Welker et al. 2019. Nature 576: 262-265. [9] Welker et al. 2020. Nature 580: 235–238.

Experiment Proposal

Experiment number GP2024079

Principal investigator	Professor Ashot Piliposyan, Armenian State Pedagogical University, ARMENIA
Co-investigator (*)	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Alessandro Cianchi, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Reconstruction of the family relationships, kinship and lineage from Bronze Age collective burials from lake Sevan basin (Armenia)

MRF Instrument	**DNA Sequencing NGS**	**Days requested:**	5
Access Route	Direct Access	**Previous GP Number:**	No
Science Areas	Biology and Bio-materials, Cultural Heritage	**DOI:**	-
Sponsored Grant	None	**Sponsor:**	-
Grant Title	-	**Grant Number:**	-
Start Date	-	**Finish Date:**	-
Similar Submission?	-		
Industrial Links	-		

Non-Technical Abstract

Our aim is to study five well-preserved petrous bone samples from five different Armenian sites: Kanageh, Noratus, Nerkin Getashen, Lchashen, and Zorats Karer, dating back to 2000-1400 BCE. These sites are located on the western and southern shores of Lake Sevan, in the provinces of Gegharkunik and Syunik, Armenia, at elevations of around 1,800-2,000 meters above sea level. With this proposal, we will assess the amount of endogenous ancient DNA through a destructive analysis using the DNA Sequencing NGS instrument at the IM@IT Unit - University of Rome Tor Vergata. This result will be the starting point for extending the analysis to all 51 individuals from these sites. The final goal is to reconstruct the genetic makeup of the Armenian populations during the Bronze Age-Iron Age and to verify possible genetic kinship or family relations among the people excavated from the collective burials.

Publications

Margaryan et al. 2017. Current Biology, vol. 27, pp. 2023-2028
Piliposyan et al. 2020. Archaeopress Archaeology, pp. 251–277
Bertoldi et al. 2021. ICE I, International Congress “The East”, (Edited by Marc Lebeau).

ISIS neutron and muon source — **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY		
MRF Instrument	**DNA Sequencing NGS**	**Days Requested:**	5
Special requirements:			

SAMPLE

Material	Petrous bones (about 3x2x1 cm3)	-	-
Formula	hydroxyapatite and collagen	-	-
Forms	Liquid		
Volume	6-9 cc		
Weight	350-600 mg		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	273 - 320 K	-	-
Pressure Range	1000 - 1010 mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Reconstruction of the family relationships, kinship and lineage from Bronze Age collective burials from lake Sevan basin (Armenia)

1. Background and Context

Kanageh, Noratus, Nerkin Getashen, Lchashen and Zorats Karer are cemeteries mostly containing collective burials, dating back to 2000-1400 BCE, in the western and southern shores of the lake Sevan (province of Gegeharkunik and of Syunik, Armenia) at around 1,800-2,000 m ASL (Figure 1).

Figure 1. geographical localization of the sites.

The mentioned archaeological sites contain burials with stone cysts surrounded by stone cromlechs. Collective burials were carried out in simultaneous burial act, which may indicate that men, women, and children were sacrificed or were brought to the burial chamber at the same time as the central most important deceased in the tomb. The general structure of the tombs, the rich materials and some artlfacts (symbols of power such as maces, daggers, and standards) testify that in some tombs, the burial of a representative of the elite was carried out [1-4].

Such tombs are not numerous throughout Transcaucasia, moreover, the bioarchaeological materials (human and animal bones) of the Sevan basin are well preserved, which allows for comprehensive bioarcheological research. Such research is highly relevant and very important, because in such these collective burials, simultaneous materials imported from Asia Minor, the Iranian plateau and Mesopotamia have been unearthed, which indicate of cultural, economic and spiritual connections of those regions. A comprehensive study of the paleoanthropological materials may also indicate suggest some family relations and kinship among the individuals buried in these tombs, or speaks about some connections in between among ethnic communities of the above mentioned regions [5].

The morphology of the skulls found in the Bronze Age collective burials of Armenia and the presence of the non-metric traits could speak about the morphological similarity of the people buried in each complex. It was supposed that the individuals in the collective burials could be related by kinship [6]. Craniological similarities and the existence of alternative anthropological features are not enough to confirm this thesis. Ancient DNA (aDNA) research of the individuals found in each specific burial complex can clearly prove the genetic kinship or family relations of the people excavated from the collective burials.

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2. Proposed experiment

In this experiment we aim to perform shotgun sequencing of aDNA using the DNA Sequencing NGS instrument at the IM@IT' Unit -University of Rome Tor Vergata to study the presence and the percentage of endogenous aDNA in 5 samples one for each stie from a total number of 51: Kanageh (n=16), Noratus (n=6), Nerkin Getashen (n=17), Lchashen (n=10) and Zorats Karer (n=2) cemeteries of Armenian. We will select the five best preserved petrous bone samples. Results of this experiment will be useful to quantify the amount of endogenous aDNA and will be a starting point to extend the analysis of all the samples and to answer the proposed questions.

3. Justification of experimental time requested

Before performing the DNA sequencing, for each sample a dedicated complex laboratory workflow must be performed. aDNA must be extracted and for each one library needs to be built and screened by the NGS machine. After screening, the best samples will undergo deep sequencing, which requires additional library preparation and sequencing time. After discussion with the instrument scientist, we estimate that 2 days will be needed for the extraction step, 2 days for library building, and 1 days for sequencing, for a total of 5 days.

References

1. Piliposyan et al. 1997. International Scientific Session, Tbilisi, 73-74.
2. Piliposyan et al. 2003. Studi Micenei ed Egeo-Anatolici, Roma, 318-325.
3. Piliposyan et al. 2016. International Workshop, Gorgia, Tbilisi, 101-111.
4. Piliposyan et al. 2021. Bulletin of the Institute of Oriental Studies, vol. I, 23-67.
5. Margaryan et al. 2017. Current Biology, 27: 2023-2028.
6. Bertoldi et al. 2021. International Congress "The East" Turnhout, Belgium, 137-152.

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Experiment Proposal

Experiment number GP2024080

Principal investigator	Professor Francesca Bertoldi, Ca&039; Foscari University Venice, ITALY
Co-investigator (*)	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Alessandro Cianchi, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Inhumated graves from the eastern Venetic necropolis in Padua: a case study
MRF Instrument	**DNA Sequencing NGS** **Days requested:** 5
Access Route	Direct Access **Previous GP Number:** No
Science Areas	Biology and Bio-materials, Cultural Heritage **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	Our aim is to genetically analyse an individual: Tb. 133 from Padova, dated back to around the 6th century BCE, using the DNA Sequencing NGS instrument at the IM@IT Unit - University of Rome Tor Vergata. Tb. 133 is located in Tumulus A, a very impressive structure, near a man buried beside a horse. Almost all the burials related to it (tombs 45, 49, 50, 51, 57, 64, 96, 97, 117, 144) were cremations, while only tombs 117, 133, and 68 were inhumations. All the finds from the grave goods were systematically studied based on graphic and anagraphic documentation to define chronology, typology, comparisons with similar contexts in the city and Veneto, and peculiarities of the funerary ritual. This detailed study highlighted the chronological definition of the use of the structure itself by an extended family unit from the 6th century BCE. We will attempt to confirm this archaeological information through a genetic approach.
Publications	Bertoldi et al. 2021. Brepols: 137-152. Milano and Bertoldi (Editors), 2014. (Venezia, 15-17 giugno 2006), Padova, Sargon. De Luca et al. 2010. ournal of Archaeological Sciences, 37: 3048-3058.

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY		
MRF Instrument	**DNA Sequencing NGS**	**Days Requested:** 5	
Special requirements:			
	SAMPLE		
Material	intact tooth (2*1*1 cm^3) or petrous bone (4*1*2 cm^3)	-	-
Formula	enamel, dentine and cementum. Hydroxyapatite and collagen	-	-
Forms	Solid		
Volume	5-10 cc		
Weight	300-600 mg		
Container or substrate	plastic bag	-	-
Storage Requirements	-	-	-
	SAMPLE ENVIROMENT		
Temperature Range	273 - 320 K	-	-
Pressure Range	1000 - 1010 mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-
	SAFETY		
Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Inhumated graves from the eastern Venetic necropolis in Padua: a case study

1. Background and context

Padova stands as one of the main archaeological sites of the Veneto region, governing both internal and international relationships while maintaining strong Venetic characteristics. Our research group has been conducting investigations on two of the main necropolises of this city, one to the south and one to the east [1, 2]. Cremation burials are the most frequent funerary customs, though inhumations are also present. The pattern of organization in groups and mounds reveals synchronic and diachronic social relations. A distinctive feature of this funerary ritual is the reopening of burials to reunite the remains of two or more individuals (probably relatives among them) (Fig. 1).

Figure 1: map of the site.

In 1990, a large sector measuring 4100 m² was investigated in the eastern part of the city, known since the early 20th century for several occasional findings [3]. The excavation was carried out with emergency procedures due to urgent construction-related reasons. Following geomagnetic surveys confirming the hypothesis of a significant number of burials, a new methodology was developed, which included excavating some burials in the field and extracting a third of the tombs (about 120 out of 314) along with their stratigraphic context in wooden chests for laboratory investigation. This investigation is still ongoing and with the support of the Municipality of Padova, several laboratory excavation campaigns were conducted, but from 2017 to the present, investigations have resumed as part of the research funding of Ca' Foscari University of Venice. A total of 97 burials in 57 chests were excavated, concurrently managing restoration, anthropological and anthracological analyses and study and edition of some phases and groupings [1, 4-9].

The eastern necropolis is a huge sepulchral area where groups of graves and funerary mounds host the deceased. Between the end of the 9th and the 7th century BC the mounds were not too large, with a diameter of 5.8 meters. In the 6th century the situation is quite different with mounds larger than 20 meters. Tumulus A was a very impressive structure, partially preserved in the area, dating to the 6th century B.C. (Fig. 2).

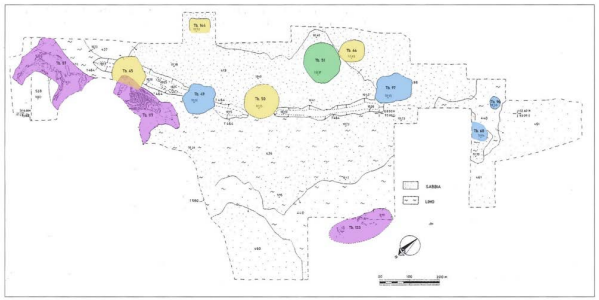

Figure 2. structure of the Tumulus A with the burials.

Padova, via Tiepolo-via San Massimo, Tumulus A: velvet: two horse graves to the south, one with a man beside the horse and one inhumation (Tb. 133) to the north.

At the bottom of the mound lay two horses graves, and inside one of them a man was buried beside the horse. Another inhumation grave was nearby (Tb. 133). Almost all the burials related to it (tombs 45, 49, 50, 51, 57, 64, 96, 97, 117, 144) were cremations, while only 117, 133 and 68 were inhumation. All the finds from the grave goods were systematically studied based on the graphic and anagraphic documentation to define chronology, typology, comparisons with similar contexts in the city and Veneto, and peculiarities of the funerary ritual. In this way we highlighted a detailed chronological definition of the use of the structure itself by an extended family unit from the 6th century BC.

2. Proposed experiment

In this experiment we aim to perform the destructive shotgun sequencing of ancient DNA (aDNA). It will be performed on the tooth or on the petrous bone of the of the grave 133 of the eastern Venetic necropolis in Padua, using the DNA Sequencing NGS operating at the IM@IT Unit-University of Rome Tor Vergata.

3. Justification of experimental time requested

The selected intact human sample will be processed using the DNA Sequencing NGS instrument. Before the DNA sequencing, the sample must undergo a dedicated, complex laboratory workflow. This involves extracting aDNA, followed by constructing and screening one library using the NGS machine. After screening, additional libraries will be built and sequenced to achieve sufficient DNA coverage for various population genetic analyses. Based on our experience, we estimate that 2 days will be needed for the extraction step, 2 days for library building, and 1 days for sequencing, for a total of 5 days.

References

[1] Gamba et al. 2014. Venezia, Regione del Veneto - Dipartimento Cultura. [2] Gamba, Voltolini. 2018. Arimnestos. Ricerche di Protostoria Mediterranea, 1: 209-225. [3] Ruta Serafini. 1990. Padova, Libreria Editrice Zielo. [4] Bortolami. 2023, Identità e società nel Veneto preromano, Mantova, SAP. [5] Gambacurta. 2009. Atti della giornata di studio, Padova, Il Poligrafo, 19-29. [6] Gambacurta 2011. Atti della giornata di studio, Padova, Il Poligrafo, 125-169. [7] Gambacurta et al. 2023. Atti della Giornata di Studi in onore di Anna Maria Chieco Bianchi (Padova 2022), «R.I.S.A.», 1, Padova, SAV, 63-73. [8] Millo. 2021. Studi di archeologia per Angela Ruta Serafini, Mantova, SAP, 105-116. [9] Moscardo. 2018-2019. Tesi di laurea, Università Ca' Foscari di Venezia, rel. prof.ssa G. Gambacurta.

Experiment Proposal

Experiment number GP2024085

Principal investigator	Professor Gabriele Gentile, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Carla Andreani, University of Rome Tor Vergata, ITALY
Co-investigator (*)	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	In-depth analysis of ancient DNA from sub-fossil bones of pink iguana from Galápagos islands using DNA sequencing
MRF Instrument	**DNA Sequencing NGS**
Days requested:	5
Access Route	Direct Access
Previous GP Number:	No
Science Areas	Biology and Bio-materials, Physics
DOI:	-
Sponsored Grant	None
Sponsor:	-
Grant Title	-
Grant Number:	-
Start Date	-
Finish Date:	-
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

Our aim is to study the origin of the pink iguana which is unknown at present from a multi-instrumental characterization of sub-fossil bones of terrestrial iguanas (Conolophus), rarely preserved and found in lava tubes of a few Galapagos islands like Santa Cruz, Isabela, Rabida, and Santiago. Current genetic and paleogeographic data would indicate that the species originated from an ancestor, now extinct, that lived on an island other than the only one (Isabela) where the pink iguana exists at present. As such, we would like to measure the presence of ancient DNA on these samples by sequencing the DNA with the DNA Sequencing NGS instrument available at the Rome Tor Vergata Unit. Data will be cross compared to verify consistency and information completed by measuring the presence of organic compounds by FT-IR data. Hence, we aim here to request access to the DNA Sequencing NGS instrument available at the Rome Tor Vergata Unit of IM@IT.

Publications

G. Gentile et al., Zootaxa (2009), 2201: 1-10.
G. Gentile et al., Problematic Wildlife, a cross-disciplinary approach. Angelici F. (Ed). Springer, pp.315-336 (2016).
G. Gentile et al., Proceedings of the National Academy of Sciences of the United States of America, 106 (2009), 507–511.

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E-platform: No

Instruments	
Days Requested:	
Access Route	
Previous RB Number:	
Science Areas	
DOI:	
Sponsored Grant	
Sponsor:	
Grant Title	
Grant Number:	
Start Date	
Finish Date:	
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY		
MRF Instrument	**DNA Sequencing NGS**	**Days Requested:**	5
Special requirements:			

SAMPLE

Material	Sub-fossil Conolophus bones	-	-
Formula	-	-	-
Forms	Solid		
Volume	0.07-0.37 cc		
Weight	100-500 mg		
Container or substrate	Sterile tube or aluminum foil	-	-
Storage Requirements	Freezer (-20C)	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	-	-

In-depth analysis of ancient DNA from sub-fossil bones of pink iguana from Galápagos islands using DNA sequencing

1. Background and Context

Visited by a young Charles Darwin, the Galápagos islands were crucial locations for the development of the evolutionary thinking. Iguanas are among the most representative reptiles of the archipelago, where four endemic species exist (Fig1), belonging to two genera: *Amblyrhynchus cristatus* (marine iguana), *Conolophus subcristatus*, *C. pallidus* and *C. marthae* (land iguanas). The last - the pink iguana - was recently discovered and described as a new species by the proponent in 2009 [1].

Fig. 1 The four named species of Galápagos iguanas. All of them are endemic to the archipelago.

The Critically Endangered [2] pink iguana rapidly became a flagship species, recognized worldwide as an iconic species capable of attracting general attention toward Biodiversity conservation [3]. With its coloration and unique ecological, physiological, and behavioral traits [4-5], the pink iguana is a source of evolutionary questions, especially related to its origin. Current genetic and paleogeographic data would indicate that the species originated from an ancestor, now extinct, that lived on an island other than the only one (Isabela) where the pink iguana exists at present. In fact, at the time the ancestor lived, Isabela had not emerged yet [6-7]. Indeed, the island where the pink iguana ancestor lived remains unknown at present. The only evidence of past presence of a lineage related to the pink iguana could be provided from sub-fossil bones of terrestrial iguanas (*Conolophus*), rarely preserved and found in lava tubes of a few islands (Santa Cruz, Isabela, Rabida, Santiago). Unfortunately, a correct species assignment cannot be done solely on the base of the morphology of a few bones. This calls for an in-depth analysis of ancient DNA (aDNA) extracted from sub-fossil bones and by means of Fourier-transform infrared spectroscopy (FT-IR) measurement for assessing the presence of organic compounds to complete the characterization. In fact, DNA retains much information about the evolutionary and genealogic relationships between species, allowing also the reconstruction of past demographic dynamics. The correct dating of those bones would provide a precise time frame for pinpointing the evolutionary events of diversification and clarify the dynamics of colonization of the archipelago. In the frame of an international collaboration with the Natural History Museum of Florida and the Galápagos National Park Directorate, we obtained several specimens of sub-fossil *Conolophus* bones, collected in lava tubes. We intend to process 10 of them, dating precisely their age. Secondly, we will perform shotgun sequencing of aDNA and FT-IR spectroscopy measurements using the DNA Sequencing NGS and the FT-IR Nexus instruments available at the Rome Tor Vergata and CSGI - University of Florence Units, respectively. The proposal will also take advantage from the genomic reference database produced by the Consortium for Iguana Genomes (CIG), funded by several sources, coordinated by the PI and participated by the University of Leeds (UK), the Genome Center and the University Kebangsaan (Malaysia). PhD students, co-advised by CIG partners produced reference genomes for several species of iguana, including Galápagos' ones. The proposed project will provide data for at least 2 PhD students.

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2. Proposed experiment

In this experiment we aim to perform shotgun sequencing of aDNA using the DNA Sequencing NGS instrument at the Rome Tor Vergata Unit to study the presence of aDNA in 10 samples coming from sub-fossil *Conolophus* bones, collected in lava tubes of a few Galápagos islands (Santa Cruz, Isabela, Rabida, Santiago). Genome sequencing results of this experiment will take advantage of the use of the DNA library from the genomic reference database produced by the Consortium for Iguana Genomes. Furthermore, findings of this measurements will be cross compared to verify consistency and information completed with data obtained by the characterization of organic compounds by FT-IR measurement (FT-IR Nexus instrument) available at the CSGI - University of Florence Unit.

3. Justification of experimental time requested

Each of the 10 sample will be enclosed in a sterile tube with about 100-500 mg of sub-fossil bone properly treated, and it will be maintained at -20 °C temperature to preserve aDNA during the measurements. Before performing the DNA sequencing, for each sample a dedicated complex laboratory workflow must be performed. aDNA must be extracted and for each one library needs to be built and screened by the NGS machine. After screening, the best samples will undergo deep sequencing, which requires additional library preparation and sequencing time. After discussion with the instrument scientist, we estimate that 2 days will be needed for the extraction step, 2 days for library building, and 1 days for sequencing, for a total of 5 days.

References

[1] G. Gentile et al., Zootaxa (2009), 2201: 1-10.
[2] G. Gentile, The IUCN Red List of Threatened Species 2012: e.T174472A1414375.
[3] G. Gentile et al., Problematic Wildlife, a cross-disciplinary approach. Angelici F. (Ed). Springer, pp.315-336 (2016).
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[5] G.A. Lewbard et al., Acta Zoologica, 00 (2023), 1–10.
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[7] G. Gentile et al., Proceedings of the National Academy of Sciences of the United States of America, 106 (2009), 507–511.

Experiment Proposal

Experiment number GP2024091

Principal investigator	Mr Gontran Sonet, Royal Belgian Institute of Natural Sciences, BELGIUM
Co-investigator (*)	Professor Claudio Ottoni, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Exploring the presence of aurochs from the prehistoric period to medieval times using ancient DNA
MRF Instrument	**DNA Sequencing NGS** — **Days requested:** 5
Access Route	Direct Access — **Previous GP Number:** No
Science Areas	Biology and Bio-materials, Cultural Heritage — **DOI:** -
Sponsored Grant	None — **Sponsor:** -
Grant Title	- — **Grant Number:** -
Start Date	- — **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	European domestic cattle descend from the Near Eastern aurochs that were domesticated and brought westward by the first farmers during the Neolithic period. Then, hybridization events occurred between domestic cattle (imported from Near East) and local European wild aurochs, as shown using DNA data. While most populations of aurochs already declined more than 2000 years ago, the coexistence of domestic cattle and aurochs in Europe, and their admixture are difficult to document osteometrically. Indeed, large-sized domestic cattle are difficult to identify (in particular in remains from the Roman times). This project aims at collecting ancient DNA data to 1) identify bovids dating from the prehistoric period to medieval times, and 2) characterize their genetic relationships with other ancient bovids and modern breeds. This will document past bovid diversity and contribute to the understanding of the agricultural practices of past human populations.
Publications	Goffette et al, 2022, International Journal of Osteoarchaelogy, 32, 38 – 48 Ferrari et al, 2023, Biodiversity Data Journal, 11, e102317 Loog et al, 2020, Molecular Ecology, 29, 1596 – 1610

ISIS neutron and muon source — **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Professor Claudio Ottoni, University of Rome Tor Vergata, ITALY		
MRF Instrument	**DNA Sequencing NGS**	**Days Requested:** 5	
Special requirements:			
	SAMPLE		
Material	Bone fragments (max 4x2x1cm)	-	-
Formula	collagen, hydroxyapatite	-	-
Forms	Solid		
Volume	5-10 cc		
Weight	300-500 mg		
Container or substrate	bag	-	-
Storage Requirements	-	-	-
	SAMPLE ENVIROMENT		
Temperature Range	273 - 320 K	-	-
Pressure Range	1000 - 1010 mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	-	-	-
	SAFETY		
Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	-	-

Exploring the presence of aurochs and cattle from prehistoric period to medieval times using ancient DNA

1. Background and Context

European domestic cattle (*Bos taurus*) descend from the Near Eastern aurochs (*Bos primigenius*) that were domesticated and brought westward by the first farmers during the Neolithic period (Bradley et al. 1996, Troy et al. 2001). Then, hybridization events occurred between domestic cattle (imported from Near East) and local European wild aurochs, as shown using DNA data (Verdugo et al. 2019). The coexistence of domestic cattle and aurochs in Europe, and their admixture are difficult to document osteometrically because large-sized domestic cattle may be misidentified as aurochs. As such, bovid remains from Roman times include exceptionally large specimens which are difficult to identify. Aurochs got extinct at the turn of the 17th century in Eastern Europe, but most populations already declined more than 2000 years ago (Epstein & Mason 1984). Hence, remains of aurochs are found much more rarely in the historical than in the prehistorical record. Characterizing bovids from the prehistoric period to medieval times helps to follow the decline of large wild mammal populations before their extinction and improve our knowledge on the history of domestic cattle. DNA analysis is a powerful approach to study historical bovids because the mitochondria of most European aurochs differ from domestic cattle (haplogroup “P” versus “T”, Troy et al. 2001, Beja-Pereira et al. 2006). In addition, signatures of hybridization can be detected in the nuclear genome (Verdugo et al. 2019).

This project aims at collecting ancient DNA data to 1) identify bovids dating from the prehistoric period to medieval times, and 2) characterize their genetic relationships with other ancient bovids and modern breeds. This will document past bovid diversity and contribute to the understanding of the agricultural practices of past human populations.

2. Proposed experiment

Here we propose to analyse the ancient DNA of 20 bovid bone samples (fragments of long bones) from the prehistoric period to medieval times. Their archaeological context and osteometric identifications suggest that they are aurochs, but they could be very large domestic cattle as well, which is informative about past agricultural practices.

We propose to perform two shotgun sequencing runs of ancient DNA libraries (5 days), using the NextSeq550 Illumina instrument of Rome Tor Vergata-UNIT. Massively parallel sequencing of short DNA fragments is an ideal approach to recover ancient DNA from archaeological remains. Technical advances in DNA extraction and library construction methods, and treatment of big data significantly improved ancient DNA recovery and paleogenomics research.

3. Summary of previous experimental proposals or characterisation

No previous experiment was conducted.

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4. Justification of experimental time requested

A total of 5 days on the NextSeq550 Illumina instrument of Rome Tor Vergata-UNIT would be necessary to perform a genomics analysis of the 20 historical bovid specimens selected.
This time is needed to make one first run to 1) evaluate the DNA quality and level of contamination, and 2) perform a "shallow" genome sequencing (genome skimming) of all 20 bone samples. The data produced will enable the assembly of mitogenomes (or parts of it) and some nuclear DNA loci. This will enable sex determination and distinction between aurochs, domestic cattle and possible hybrids. Then, a second run is planned to sequence a selection of samples showing promising results (hydrids, or divergent individuals) at larger coverage, and provide data from a larger part of the genome. The data produced here will enable genomic analysis and genome wide comparisons with data already available for other ancient specimens and modern cattle.

References

Bradley et al. 1996. PNAS. 93(10):5131–5.
Beja-Pereira et al. 2006. PNAS. 103: 8113–8118.
Lindqvist & Rajora 2019. Springer, 427 p.
Troy et al. 2001. Nature 410: 1088–1091.
Verdugo et al. 2019. Science, 365: 173–176.
Epstein & Mason 1984. in Evolution of Domesticated Animals, ed. Mason, pp 6-27

Experiment Proposal

Experiment number GP2024105

Principal investigator	Professor Stefano Capomaccio, Università degli Studi di Perugia, ITALY
Co-investigator (*)	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Genomic Analysis of an Iron Age Horse: Insights into Ancient Equine Genetics and Modern Conservation Implications
MRF Instrument	**DNA Sequencing NGS**
Access Route	Direct Access
Science Areas	Biology and Bio-materials, Cultural Heritage
Sponsored Grant	None
Grant Title	-
Start Date	-
Similar Submission?	-
Industrial Links	-

Days requested: 5
Previous GP Number: No
DOI: -
Sponsor: -
Grant Number: -
Finish Date: -

Non-Technical Abstract

This project will investigate an Iron Age horse sample from Montecchio del Vallone di San Lorenzo, Italy, using ancient DNA (aDNA) sequencing to uncover the genetic history of equine populations. The research seeks to gain new knowledge on horse genetics during the Iron Age (1200–600 BCE), a period marked by significant cultural and technological shifts. Italy's position as a Mediterranean crossroads suggests a unique genetic heritage influenced by various civilizations. By comparing this new ancient genome with modern and ancient public equine data, the study aims to reveal changes in genetic diversity, infer lost traits, and offer insights into historical breeding practices. The findings will also contribute to modern conservation efforts, ensuring the preservation of genetic diversity in contemporary horse populations.

Publications

Capomaccio, S. et al. Exploring the Italian equine gene pool via high-throughput genotyping. Front. Genet. 14, (2023).
Cappelli, K. et al. Genome-wide epigenetic modifications in sports horses during training as an adaptation phenomenon. Sci Rep 13, 18786 (2023).
Capomaccio, S. et al. Splicing site disruption in the KIT gene as strong candidate for white dominant phenotype in an Italian Trotter. ANIMAL GENETICS 48, 727–728 (2017).

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E-platform: No

Instruments	
Access Route	
Science Areas	
Sponsored Grant	
Grant Title	
Start Date	
Similar Submission?	
Industrial Links	

Days Requested:
Previous RB Number:
DOI:
Sponsor:
Grant Number:
Finish Date:

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Sample record sheet

Principal contact Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY
MRF Instrument **DNA Sequencing NGS** **Days Requested:** 5
Special requirements:

SAMPLE

Material	bone fragment	-	-
Formula	collagen and hydroxyapatite	-	-
Forms	Solid		
Volume	4-10 cc		
Weight	500-1000 mg		
Container or substrate	sterile bag	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	273 - 320 K	-	-
Pressure Range	1000 - 1010 mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	No	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	no	-	-
Special equip. reqs	DNA extraction and libraries preparation	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	no	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	No	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	-	-

Genomic Analysis of an Iron Age Horse: Insights into Ancient Equine Genetics and Modern Conservation Implications

1. Background and Context

With the advent of modern sequencing technologies on ancient DNA (aDNA) and the availability of fast algorithms and high-performance computational resources, the scientific community has started unravelling many questions about the past. Migrations, origins of deleterious alleles and groundbreaking cultural events have been placed in space and time for the human populations [1,2].

Also animal history have been updated using the same approaches as in humans, solving the last doubts on domestication processes [3], particularly in horses [4–6].

While the main history branch appears to be mainly traced and accepted, other interesting histories can contribute to fully understand the complete history of the horse population expansion and the ultimately the breed formation: literature is scarce about horses from Iron Age burial sites from Italy and none has performed robust aDNA analysis.

In this scenario, the sequencing of ancient horse samples from this time span could represent an opportunity to unravel the genetic and historical complexities of equine populations in a region that has served as a nexus of cultural and migratory interactions. Italy's unique position in the Mediterranean, as a historical crossroads for diverse civilizations such as the Etruscans, Greeks, Romans, and various migratory tribes, has likely resulted in a rich genetic melting pot that had influences on horse populations. Understanding the genetic makeup of these ancient horses offers significant insights into historical breeding practices, migration patterns, and cultural exchanges that have shaped the gene pool of modern Italian horses. The Iron Age, indeed, 1200 to 600 BCE, was a time of significant social and technological transformation, marked by the rise and fall of powerful civilizations and the proliferation of trade networks. Horses played a crucial role during this era, not only as means of transportation and warfare but also as symbols of status and power [7]. Sequencing the aDNA of horses from this period can provide critical data to reconstruct the genetic diversity and structure of ancient equine populations, shedding light on how historical events and human activities influenced their evolution. Also, Italian data can be now compared to a vast collection of European (France in particular) sequences that are publicly available [8].

In this proposal, we aim to sequence an Iron Age horse sample from Montecchio del Vallone di San Lorenzo, an Italians Iron Age site dated back 6th century BCE. This will provide a renewed understating of the genetic map of ancient equine populations, offering insights into historical interactions, migration patterns, and breeding practices. This research, benefiting of publicly available data, can fill the gaps in our understanding of the genetic heritage of Italian horses.

The data will then be compared with the modern equine genome to assess changes in genetic diversity over time, offering a robust window into past breeding practices and human intervention, and inferring genetic traits that have been lost or diminished in frequency over time. Furthermore, the genetic information from ancient sample can have practical implications for modern conservation efforts [9,10].

2. Proposed experiment

In this experiment we aim to perform the destructive shotgun sequencing of ancient DNA (aDNA). It will be performed on a bone fragment of one horse buried in Montecchio del Vallone di San Lorenzo, an Italians Iron Age site dated back 6th century BCE, using the DNA Sequencing NGS operating at the IM@IT Unit-University of Rome Tor Vergata.

3. Justification of experimental time requested

The selected intact bone sample will be processed using the DNA Sequencing NGS instrument. Before the DNA sequencing, the sample must undergo a dedicated, complex laboratory workflow. This involves extracting aDNA, followed by constructing and screening one library using the NGS machine. After screening, additional libraries will be built and sequenced to achieve sufficient DNA coverage for various population genetic analyses. Based on our experience, we estimate that 2 days will be needed for the extraction step, 1 day for library building, and 2 days for sequencing, for a total of 5 days.

References

1. Allentoft, M. E. *et al.* Population genomics of post-glacial western Eurasia. *Nature* 625, 301–311 (2024).
2. Barrie, W. *et al.* Elevated genetic risk for multiple sclerosis emerged in steppe pastoralist populations. *Nature* 625, 321–328 (2024).
3. Alberto, F. J. *et al.* Convergent genomic signatures of domestication in sheep and goats. *Nat Commun* 9, 813 (2018).
4. Librado, P. *et al.* The Evolutionary Origin and Genetic Makeup of Domestic Horses. *Genetics* 204, 423–434 (2016).
5. Fages, A. *et al.* Tracking Five Millennia of Horse Management with Extensive Ancient Genome Time Series. *Cell* 177, 1419-1435.e31 (2019).
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9. Librado, P. *et al.* Ancient genomic changes associated with domestication of the horse. *Science* 356, 442–445 (2017).
10. MacHugh, D. E., Larson, G. & Orlando, L. Taming the Past: Ancient DNA and the Study of Animal Domestication. *Annual Review of Animal Biosciences* 5, 329–351 (2017).

Experiment Proposal

Experiment number GP2024113

Principal investigator	Dr Flavio Enei, Polo Museale Civico di Santa Marrinella, ITALY
Co-investigator	Dr Giulia Orefice, University of Rome Tor Vergata, ITALY
Co-investigator (*)	Professor MARIA CRISTINA MARTINEZ-LABARGA, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Genetic characterization of the Medieval community of Santa Severa
MRF Instrument	**DNA Sequencing NGS** — **Days requested:** 5
Access Route	Direct Access — **Previous GP Number:** -
Science Areas	Biology and Bio-materials — **DOI:** -
Sponsored Grant	None — **Sponsor:** -
Grant Title	- — **Grant Number:** -
Start Date	- — **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	This study aims at the genetic characterisation of the medieval population of Santa Severa by combining human population genetics and metagenomic analyses to detect possible pathogens such as a plague, which was very common in the Middle Ages. The site, built in the Middle Ages, overlaps with the ancient Etruscan settlement of Pyrgi. Archaeological excavations brought to light two cemetery areas, dating from the 7th to 15th century CE. A skeleton collection of 455 individuals was found, of which about 100 were selected based on archaeological and anthropological data. The analysis of ancient DNA aims to understand the genetic composition, reconstruct potential family ties to understand the social structure and relationships with other coeval populations. Preliminary sequencing assessed the preservation of the DNA and future steps include further sequencing for more in-depth genomic analysis.
Publications	Enei, F. (2013). Santa Severa - Tra leggenda e realtà storica (Ceccarelli Editrice S.r.l.: Viterbo) Gnes, M., et al. (2018). Bioarchaeological approach to the study of the medieval population of Santa Severa (Rome, 7th–15th centuries). J. Archaeol. Sci. Rep. 18, 11–25 Gismondi, A., et al. (2020). A multidisciplinary approach for investigating dietary and medicinal habits of the Medieval population of Santa Severa. PLoS ONE 15: e0227433

ISIS neutron and muon source — **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Professor MARIA CRISTINA MARTINEZ-LABARGA, University of Rome Tor Vergata, ITALY
MRF Instrument	**DNA Sequencing NGS** — **Days Requested:** 5
Special requirements:	

SAMPLE

Material	Illumina genomic libraries of ancient DNA	-	-
Formula	DNA, adapter, water, index	-	-
Forms	Liquid		
Volume	1 ml		
Weight	1,2 mg		
Container or substrate	DNA LoBind Tubes	-	-
Storage Requirements	refrigerated at 4°C	-	-

SAMPLE ENVIROMENT

Temperature Range	273 - 277 K	-	-
Pressure Range	1013,25 - mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	Yes	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	no	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

1.Background and Context

Molecular anthropology is a scientific discipline that studies human evolution, genetic diversity and the relationships between populations through the analysis of ancient DNA (aDNA) extracted from ancient human remains. This field offers valuable insights into human history by uncovering the dynamics and interactions between populations. The proposed research focuses on the analysis of skeletal remains from the medieval cemetery of the Santa Severa Castle (Rome, Italy) [1]. This study will contribute to the implementation of aDNA studies on the medieval populations of the region and help to piece together the broader puzzle of Italian demographic history. In particular, the research will shed light on the local history of Santa Severa, an area settled for millennia and help clarify the dynamics of population change over time. It is supported by a PhD student, a master's thesis student, and collaborations with industrial partners specializing in genetic sequencing and bioarchaeological analysis. These partnerships provide the technical expertise necessary for advanced molecular studies.


Museum of the Santa Severa Castle

2.Proposed experiment

The main objective of the experiment is to investigate the genetic composition and kinship relationships of individuals in the medieval cemetery in order to understand their social structure and connections with coeval populations. Another objective is to conduct metagenomic analyses to identify pathogens, such as plague, common in the Middle Ages. These results will be decisive for reconstructing population dynamics and understanding the impact of infectious diseases on this community. The use of ISIS@MACH ITALIA tools is essential due to their high-throughput sequencing performance, which is necessary to analyse well-preserved aDNA samples. Other techniques do not have the resolution and sensitivity that the ISIS@MACH platform offers. Preliminary work, including molecular techniques and bioinformatic simulations on a subset of individuals, has yielded promising results. Sequencing at the University of Tor Vergata confirmed that the samples are well-preserved and suitable for further analysis.

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Example of sample processing at the Laboratory of the Centre of Molecular Anthropology for the study of ancient DNA at Villa Mondragone (Monte Porzio Catone, Rome) - University of Rome Tor Vergata

3.Summary of previous experimental proposals or characterisation

Two previous studies have already been conducted on the Santa Severa population. The first[2] analysed demographic and health status based on morphological evidence, defining population structure, life expectancy, and common diseases. The second[3] study focused on dietary patterns and medical habits using a multidisciplinary approach, combining stable isotope analysis from bone proteins with dental calculus investigations, employing DNA analysis, light microscopy, and gas chromatography-mass spectrometry (GC-MS). These studies established a solid foundation for understanding the population's lifestyle, health, and suitability for further genetic and metagenomic analyses. This project aims at the genetic characterisation of individuals to complete the picture of the medieval population of Santa Severa as has already been done in the literature for other Italian medieval populations [4,5].

4.Justification of experimental time requested

We requested the MRFs tool for its accuracy and efficiency in generating high-quality sequencing data from ancient DNA (aDNA) samples. Due to the sensitive nature of aDNA, this instrument is particularly well adapted to process degraded genetic material, providing the resolution needed to accurately capture both genetic composition and metagenomic data. We estimate the study of 50 samples. Working on batches of 10 samples, it will take one week to prepare and pool each batch, and a total of five days for sequencing. This timeline ensures accurate data collection and analysis, allowing for any technical adjustments during the process.

References: [1] Enei, F. (2013). Santa Severa - Tra leggenda e realtà storica (Ceccarelli Editrice S.r.l.: Viterbo); [2] Gnes, M., Baldoni, M., Marchetti, L., Basoli, F., Leonardi, D., Canini, A., Licoccia, S., Enei, F., Rickards, O., Martínez-Labarga, C. (2018). Bioarchaeological approach to the study of the medieval population of Santa Severa (Rome, 7th–15th centuries). J. Archaeol. Sci. Rep. 18, 11–25; [3] Gismondi, A., Baldoni, M., Gnes, M., Scorrano, G., D'Agostino, A., Di Marco, G., Calabria, G., Petrucci, M., Müldner, G., Von Tersch, M., Nardi, A., Enei, F., Canini, A., Rickards, O., Alexander, M., Martínez-Labarga, C. (2020). A multidisciplinary approach for investigating dietary and medicinal habits of the Medieval population of Santa Severa (7th-15th centuries, Rome, Italy). PLoS ONE 15: e0227433; [4] O'Sullivan, N., Posth, C., Coia, V., Schuenemann, V.J., Price, T.D., Wahl, J., Pinhasi, R., Zink, A., Krause, J., Maixner, F. (2018). Ancient genome-wide analyses infer kinship structure in an Early Medieval Alemannic graveyard. Sci. Adv. 4: eaao1262; [5] Coia, V., Paladin, A., Zingale, S., Wurst, C., Croze, M., Maixner, F., Zink, A. (2023). Ancestry and kinship in a Late Antiquity-Early Middle Ages cemetery in the Eastern Italian Alps. iScience 26, 108215.

Experiment Proposal

Experiment number GP2024146

Principal investigator	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY
Co-investigator (*)	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Charles Cockell, University of Edinburgh, UNITED_KINGDOM
Co-investigator	Dr Jens Holtvoeth, Teesside University, UNITED_KINGDOM
Co-investigator	Miss Julia Puputti, Boulby Underground Laboratory STFC, UNITED_KINGDOM
Co-investigator	Professor Carla Andreani, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Biogeochemical Multi-Instrumental Study of Rocks and Microbial Biomass in Zechstein Salt Deposits of Boulby using DNA Sequencing NGS
MRF Instrument	**DNA Sequencing NGS** **Days requested:** 5
Access Route	Direct Access **Previous GP Number:** No
Science Areas	Biology and Bio-materials, Chemistry, Environment, Materials **DOI:** -
Sponsored Grant	Yes **Sponsor:** CNR
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	Our aim is to investigate the potential for preserving biological material in ancient salt deposits, with a focus on the Zechstein salt deposits in Boulby Mine (UK), offering insights into the environmental conditions of the Zechstein Sea ~250 million years ago. The study employs a multi-instrumental approach, combining non-destructive and destructive analyses to correlate biomolecule presence with mineral phases and elemental compositions. By analyzing a range of biomarkers the research aims to create a detailed biogeochemical fingerprint of fossil microbial biomass. This research contributes to both astrobiology and our understanding of ancient terrestrial environments. We propose a multi-instrumental approach involving by combining non-destructive and destructive analyses exploiting the analytical instrumental suite at IM@IT and ISIS facilities. In this specific proposal we propose the DNA Sequencing NGS instrument to perform shotgun sequencing of aDNA in salt samples.
Publications	Cockell, C. et al. (2020) Astrobiology 20, 864-877 Scorrano et al. Communications Biology 5, 1262 (2022) W Barrie, Y Yang, E K Irving-Pease, K E Attfield, G Scorrano et al. Nature 625, 321 (2024)

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
MRF Instrument **DNA Sequencing NGS** **Days Requested:** 5
Special requirements:

SAMPLE

Material	slabs of salt	-	-
Formula	salt (NaCl) sediments and organic phases	-	-
Forms	Friable powder		
Volume	<50 cc		
Weight	50-100 g		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	273 - 320 K	-	-
Pressure Range	1000 - 1010 mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	No	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	Yes	-	-
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Biogeochemical Multi-Instrumental Study of Rocks and Microbial Biomass in Zechstein Salt Deposits of Boulby using the DNA Sequencing NGS

1. Background and Context

Can the remains of biological material be preserved in salts many hundreds of millions of years old and what signatures can be preserved? To answer these questions, we must be able to probe the chemical and physical conditions at small scales in ancient salt deposits to understand the geological context of biomolecular preservation. We aim to produce a biogeochemical and molecular fingerprint of fossil microbial biomass and inorganic rock samples in the Zechstein salt deposits of Boulby Mine, North Yorkshire, UK through analysing biomarkers, specifically, alkyl lipids (alkanes, fatty acids and alcohols, steroids), glycerol dialkyl glycerol tetraethers (GDGTs), ancient DNA (aDNA) and proteins. This research is timely as the outcomes will help to interpret Raman spectroscopy data produced from the same material. Raman spectroscopy will be one of the analytical tools aboard the next generation Mars rovers. Martian evaporites are prime targets in the search for extra-terrestrial life since the last places where microbial life could have existed on Mars would have been the evaporating oceans. In addition to astrobiology, outcomes are expected to contribute insights into late Permian hydrology and paleoecology. Biomarker distributions in the salt at Boulby mine and particularly in backfilled desiccation cracks can provide information on the environmental conditions in and around the Zechstein Sea ~250 million years ago. The site represents a shallow near shore setting of the Zechstein Basin, with exposure of the evaporite surfaces during sea-level low stand. The presence of biomarkers originating from both microbes and plants in the Boulby salt has already been demonstrated [1]. Upscaling of the extraction procedure and an improved extraction protocol is expected to produce sufficient material for compound-specific carbon and hydrogen isotope analyses ($d^{13}C$, $d^{2}H$) of leaf wax-derived compounds, which will provide information on continental plant types and aridity. In this context the knowledge of the exact spatial distribution and inorganic chemical composition of organic matter in the salt on the atomic scale would help much more targeted analyses. For example, leaf waxes may be associated to different material and specific sites compared to microbial membrane lipids. Thus, we aim to use analytical instruments of IM@IT and neutron beamlines of ISIS Facilities. Due to the age of the samples, a good understanding of the exact spatial distribution of the organic matter and its association the inorganic phases of the salt would greatly support targeted analyses. This would allow us to select specific sub-samples with higher organic yields for biomarker investigations and compound-specific isotope analyses, in particular. In addition to targeting the analysis, the physical and chemical context of the biomolecules (or even a lack of them) provides essential information to explain how physical and chemical conditions influence the fate of biomolecules and their potential or long-term preservation over geological time scales. For example, is the presence of any putative biomarkers associated with specific elements or mineral phases? We can answer this by correlating the presence of biomolecules with mineral phases and elemental composition determining using small/wide angle X-ray Scattering by means of SAXS GISAXS; hard X-ray Fluorescence (2D/3D XRF), using RETINA and Multipurpose X-ray diffraction; and neutron diffraction combined with tomography, using INES and IMAT neutron beamlines. Does the oxidation state of elements, for example iron, influence the chemical environment and thus the presence and preservability/stability of biosignatures in the salt over time? We will answer this by correlating the presence of any biosignatures to the elemental oxidation state in the same location at the relevant scales using mineralogical information. Therefore, we propose a multi-instrumental approach involving a combination of non-destructive and destructive analyses exploiting the analytical instrumental suite at IM@IT and ISIS facilities. All analyses will be using the same sample materials, first, for non-destructive and then destructive methods, to maximise the complementary character of the resulting data sets. On one hand, for the non-destructive analyses of the samples, we use the analytical suite of instruments of IM@IT and ISISI Facilities indicated two paragraphs above. The minerals in each salt sample will be quantified using combined Rietveld refinement of X-rays and neutron diffraction data, using laser ablation. On the other hand, due to the very low yields of the organic phase and its fine dispersal in the salt, the samples to be analysed will consist of small slabs of salt of 100-150g, representing two distinct types of material: relatively pure evaporite material and desiccation crack backfill material. The pure evaporite material will be mainly sodium chloride, containing lenses of clay-rich material and isolated potassium chloride crystals. It is expected to include an organic phase originating predominantly from extremophile biomass and some eolian terrigenous input. The slightly darker coloured material from the desiccation cracks, on the other hand, is expected to include higher proportions of terrigenous organic particles and fragments of biofilm from the salt surface that were resuspended and washed into the cracks during rising water level. For the (destructive) analysis of the biomarkers, originating from microorganisms, terrestrial vegetation or processing-related contamination, like alkanes, alcohols, and ketones we propose to use Gas Chromatography – Ion Mobility Spectrometer (GC-IMS), steranes and GDGTs will be analysed by normal-phase UHPLC at Bristol University; for the aDNA analysis, using DNA sequencing NGS, and for the analysis of both fatty acids and ancient proteins using the Mass Spectrometer 2. Genetic, lipid, and proteomic and volatilomic data will then be cross compared to verify their consistency with the possible extremophile species identified. This research is embedded in a wider collaborative attempt to understand extremophile ecology through a comparison with lipid, proteins and DNA data of modern microbes living on the salt surfaces and in brines in Boulby mine. We aim to see if microbial communities adapt to changes in brine salinity and/or ion composition (chloride vs. sulphate, sodium vs. potassium) either by individual species changing their cell membrane properties or by shifts in species distribution. This is a collaboration with Teesside University, the UKRI-STFC Underground Lab. Boulby, the UK Centre for Astrobiology at Edinburgh University, NASA Jet Propulsion Lab, Bristol University, the University of Bern, the IM@IT and ISIS Facilities. It is supported by the Seedcorn Funding scheme of Teesside University to produce pilot data for a larger proposal to fund PhD projects at Teesside and Edinburgh Universities.

2. Proposed experiment using the DNA Sequencing NGS

In this experiment we aim to perform shotgun sequencing of aDNA of salt power samples using the DNA Sequencing NGS instrument at the IM@IT Unit - Rome Tor Vergata to study the presence of environmental aDNA in the slabs of salt. Results of this experiment will be cross compared and integrated with data obtained using the GC-IMS and by the Mass Spectrometer 2 instruments operating at the IM@IT Unit - University of Milano Bicocca.

3. Justification of experimental time requested

Each of the four samples will be enclosed in a sterile tube with about 5 -20 g (8-20 cc) of rock sample and maintained at -20 °C temperature to preserve aDNA during the measurements. Before DNA sequencing, for each sample a dedicated laboratory workflow will be performed: once aDNA is extracted for each sample a library needs to be built and screened by the NGS machine. To follow, the best sample needs to undergo deep sequencing. We estimate that 1 days will be needed for the extraction step, 2 days for library building, and 2 days for sequencing, for a total of **5 days**.

ESCALB QXi

Experiment Proposal

Experiment number GP2024046

Principal investigator (*) Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator

Experiment title	Investigating the Corrosive Impact of Chrome-Tanned Semi aniline Calf Leather using X-ray Photoelectron Spectroscopy (XPS) and Ion Scattering Spectroscopy (ISS)		
MRF Instrument	**ESCALB QXi**	**Days requested:**	4
Access Route	Direct Access	**Previous GP Number:**	NO
Science Areas	Materials	**DOI:**	-
Sponsored Grant	None	**Sponsor:**	-
Grant Title	-	**Grant Number:**	-
Start Date	-	**Finish Date:**	-
Similar Submission?	-		
Industrial Links	LBS LUXURY BRANDS SERVICES SRL		

Non-Technical Abstract

The aim of this study is to systematically investigate the composition of semi aniline chrome-tanned calf leather and to identify correlations between adsorbed elements on the leather surface and its aggressiveness towards metal accessories. By understanding these relationships, we can develop better methods for predicting and mitigating corrosion, thereby improving the quality and durability of leather products in the fashion and luxury industries. Understanding the chemical composition of semi aniline chrome-tanned calf leather will lead to significant improvements in product quality ad in particular this research will provide a scientific basis for better quality control practices and contribute to the development of more robust and durable fashion items.

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY
MRF Instrument **ESCALB QXi** **Days Requested:** 4
Special requirements:

SAMPLE

Material	Chrome-Tanned Semi aniline Calf Leather	-	-
Formula	Chrome-Tanned Semi aniline Calf Leather	-	-
Forms	Solid		
Volume	4 cc		
Weight	10 g		
Container or substrate	No special need	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	RT - K	-	-
Pressure Range	Atmospheric pressure - mbar	-	-
Magnetic field range	No - T	-	-
Standard equipment	-	-	-
Special equipment	No special equipment needed	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	No	-	-
Special equip. reqs	No	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	No	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Investigating the Corrosive Impact of Chrome-Tanned Semi aniline Calf Leather

1. Background and context

Semi aniline chrome-tanned calf leather is a highly valued material in the fashion and luxury industries, commonly used for products such as handbags, belts, and footwear. Ensuring the quality and longevity of these products is crucial, particularly in preventing corrosive processes that can damage metallic accessories like buckles, zippers, and decorative elements. The leather can release tanning substances that may react with metals, leading to corrosion and tarnishing, which compromises the aesthetic and functional integrity of the final products.

To assess the corrosive potential of leathers, quality control laboratories typically perform simulated corrosion tests using a reference sample. The extent of oxidation on the sample, after exposure to the leather, is evaluated and the leather is classified on a scale of aggressiveness from 1 to 5 (1 = highly aggressive, 5 = non-aggressive). However, beyond this empirical test, there has been no systematic study to investigate the underlying causes of oxidation and the specific elements responsible for the corrosive effects on metal accessories.

This proposal aims to investigate the surface composition of the leather using XPS (X-ray Photoelectron Spectroscopy) and ISS (Ion Scattering Spectroscopy) with the ESCALAB QXi, providing detailed elemental and surface information that could be correlated with the leather's corrosive properties. The fashion and luxury industries could than take proactive measures to treat or modify leather to reduce its corrosive impact, thereby enhancing the durability and quality of leather products.

2. Proposed experiment

This proposal is part of a broader study to investigate the underlying causes of oxidation and the specific substances responsible for the corrosive effects on metal accessories by Semi aniline chrome-tanned calf leather. The study involves several instrumental techniques: a) X-ray Photoelectron Spectroscopy (XPS) and Ion Scattering Spectroscopy (ISS); b) XRF Tomography; c) RAMAN Spectroscopy and profilometry; d) FT-IR Spectroscopy.

XPS and ISS with the ESCALAB QXi would give a great contribution to this study since it is an advanced tool for surface analysis, particularly useful for analysing the outermost atomic layers of materials, making it an excellent technique for studying surface composition and interactions that may contribute to corrosion. These techniques would allow insight on the elements present on the surface, their oxidation states and chemical environment providing crucial information for corrosion properties.

In particular the analysis will provide valuable insights on the following aspects:

1. Surface Composition: XPS and ISS are highly sensitive to the surface composition of materials, providing detailed information about the elements present on the outermost atomic layers of the leather. This surface-specific data is critical for identifying potential corrosive agents that could interact with metallic accessories.

2. Elemental Quantification: XPS and ISS allows for the precise quantification of elemental concentrations on the leather surface. By understanding the abundance of specific elements, we can assess their potential impact on corrosion processes.

3. Chemical State Information: XPS, can provide information on the chemical states of elements present on the surface. Understanding the oxidation states and chemical environments of these elements can help elucidate their role in corrosion mechanisms.

4. Depth Profiling: ESCALAB QXi is equipped with a dual mode ion gun (monoatomic and gas cluster ion beam), this will allow depth profiling investigation.

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By conducting these analyses, we will obtain essential data for understanding and mitigating the corrosive effects that these leathers can have on metallic accessories, ultimately leading to improved quality and durability of leather products in the fashion and luxury industries.

3. Summary of previous experimental proposals or characterisation

Historically, the understanding of the aggressiveness of leather toward metal accessories has been constrained by a reliance on empirical methods and trial and error. This approach lacks the detailed insight and predictive capability that sophisticated analytical tools like XPS and ISS investigation can provide. In particular, access to such advanced analysis and the requisite expertise is often limited. Although some literature provides a foundational understanding of the surface composition of chrome-tanned leather [1-3], a detailed exploration of these elements' interplay, has not been conducted extensively so far. This research aims to fill that critical knowledge gap in order to provide a correlation between the surface-specific elements found with the investigation and how these elements might contribute to corrosion, enabling to provide then recommendations for leather treatment and processing methods that could mitigate the corrosive impact on metal accessories.

4. Justification of experimental time requested

As detailed in section 2, X-ray Photoelectron Spectroscopy (XPS) and Ion Scattering Spectroscopy (ISS) are pivotal tools for this experiment due to their unique capabilities.

To achieve a comprehensive surface analysis of the leather samples, we request machine time for the ESCALAB QXi to perform XPS and ISS. This will enable us to obtain detailed elemental and surface information, which can be correlated with the leather's corrosive properties. We request 4 days of experimental time to analyse 15 samples coming from 5 leather batches: we collected 5 types of batches of semi aniline chrome-tanned calf leather with high level of aggressiveness (level 1 to level 2, with reference to the empirical scale where (1 = highly aggressive, 5 = non-aggressive) as determined by the currently used corrosion test as detailed in section 1. For each batch we will then collect three samples for investigating the spatial homogeneity of the composition within the same batch.

XPS and ISS analyses will be also coupled to depth profiling by using the dual mode ion gun to examine changes in elemental composition with depth. Finally, the data analysis will focus on the correlation of XPS and ISS data with empirical corrosion test results.

This schedule ensures efficient use of the ESCALAB QXi, providing detailed insights into the surface composition and chemistry of the leather samples. By understanding the correlation between these factors and the leather's corrosive properties, we aim to enhance the quality control processes and improve the longevity and performance of leather products in the fashion and luxury industries.

[1] Nayan Ranjan Singha, Pijush Kanti Chattopadhyay, Arnab Dutta, Manas Mahapatra, Mousumi Deb, Review on additives-based structure-property alterations in dyeing of collagenic matrices, Journal of Molecular Liquids, Volume 293, 2019, 111470.
[2] Unceta, N., Séby, F., Malherbe, J. et al. Chromium speciation in solid matrices and regulation: a review. Anal Bioanal Chem 397, 1097–1111 (2010).
[3] Stanca, M.; Gaidau, C.; Alexe, C.-A.; Stanculescu, I.; Vasilca, S.; Matei, A.; Simion, D.; Constantinescu, R.-R. Multifunctional Leather Surface Design by Using Carbon Nanotube-Based Composites. Materials 2021, 14, 3003.

Experiment Proposal

Experiment number GP2024054

Principal investigator (*) Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator

Experiment title	Training on X-ray Photoelectron Spectroscopy (XPS) with ESCALAB QXi X-Ray Photoelectron Spectrometer	
Training MRF	**ESCALB QXi**	**Days requested:** 1
Access Route	Direct Access	**Previous GP Number:** NO
Science Areas	Materials	**DOI:** -
Sponsored Grant	None	**Sponsor:** -
Grant Title	-	**Grant Number:** -
Start Date	-	**Finish Date:** -
Similar Submission?	-	
Industrial Links	RGF SRL	

Non-Technical Abstract

Consorzio Physis SRL S.B. is a benefit company collecting societies dedicated to the production of high-quality fashion metallic components and surface treatments for fashion and luxury. To enhance the quality and longevity of the products, we aim to deepen our understanding of surface composition and oxidation states. We are particularly interested in the ESCALB QXi instrument, which utilizes X-ray Photoelectron Spectroscopy (XPS) for detailed surface analysis, providing valuable insights into the elemental and chemical state of materials, crucial for identifying surface contaminants and oxidation states. This technique is especially beneficial for studying corrosion and surface alterations in metal accessories, enabling targeted interventions to improve product performance. To fully leverage this technology, we seek training on the ESCALB QXi instrument, focusing on XPS principles and practical applications.

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY
Training Instrument **ESCALB QXi** **Days Requested:** 1
Special requirements:

SAMPLE

Material	-	-	-
Formula	-	-	-
Forms			
Volume			
Weight			
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	-	-	-
Pressure Range	-	-	-
Magnetic field range	-	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	-	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	-	-	-
Sensitivity to vapour	-	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	-	-	-

Training proposal on X-ray Photoelectron Spectroscopy (XPS) at ESCALAB QXi X-ray (CNR-ICMATE)

1. Background and context

Consorzio Physis SRL S.B. is actively engaged in the production of fashion accessories, since it collects the facilities producing metallic fashion accessories and surface treatments as well as chemicals providers for the surface treatments. As part of our ongoing efforts to enhance our technical capabilities and address critical challenges related to quality and longevity of the items produced, we are seeking to deepen our understanding of surface composition analysis and oxidation states. We are particularly interested in the ESCALB QXi instrument, which utilizes X-ray Photoelectron Spectroscopy (XPS) for detailed surface analysis, providing valuable insights into the elemental and chemical state of materials, crucial for identifying surface contaminants and oxidation states [1]. This technique is especially beneficial for studying corrosion and surface alterations in metal accessories, enabling targeted interventions to improve product performance. To fully leverage this technology, we seek training on the ESCALB QXi instrument, focusing on XPS principles and practical applications since Consorzio Physis SRL S.B. is a benefit company collecting societies dedicated to the production of high-quality fashion metallic components and surface treatments for fashion and luxury. To enhance the quality and longevity of the products, we aim to deepen our understanding of surface composition and oxidation states.

2. Proposed training

X-ray Photoelectron Spectroscopy (XPS) is a powerful analytical technique that offers numerous advantages for surface analysis. It provides quantitative information about the elemental composition, chemical state, and electronic state of the materials being analyzed. By measuring the binding energies of core electrons ejected from the surface atoms when irradiated with X-rays, XPS can identify and quantify elements within the top 1-10 nanometers of a material's surface. This makes it particularly useful for detecting surface contaminants, thin film compositions, and various oxidation states. In the context of corrosion studies, XPS is invaluable for understanding the surface chemistry changes, identifying corrosion products, and investigating non-uniformities and alterations on metallic surfaces. This detailed insight enables more targeted and effective interventions to improve material performance and longevity of metal accessories produced for the fashion and luxury market, such as metal accessories for footwear and leathergoods and jewellery. In more detail, the analysis that would be performed after gaining knowledge and experience with ESCALB QXi instrument will provide valuable insights on the following aspects:

1. Surface Composition

2. Elemental Quantification

3. Chemical State Information/ oxidation states

4. Depth Profiling (thanks to the dual mode ion gun)

Therefore, we request a training on the instrument to gain knowledge in the technique and on the analytical capabilities of the instrument.

The training would be carried out at CNR-ICMATE, with whom we are already in contact and agreed on the proposed training.

3. Summary of previous training proposals

No previous training proposal has been presented. However, this training will empower us to perform more accurate and detailed analyses, ultimately leading to improved quality and durability of the fashion accessories since X-ray Photoelectron Spectroscopy (XPS) stands out as a robust analytical method with a wide array of benefits for surface analysis. It provides invaluable data concerning surface impurities,

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assessing thin film structures, and discerning different oxidation levels. In the realm of corrosion research conducted by Consorzio Physis, XPS plays a key role in comprehending alterations in surface chemistry, pinpointing corrosion byproducts, and exploring irregularities and changes on metal surfaces. Such in-depth knowledge allows for targeted and efficient actions to boost the performance and longevity of metallic accessories designed for the fashion and luxury segment, including footwear, leather goods, and jewellery.

4. Justification of experimental proposals request

We request a training on the instrument to gain knowledge in the technique and on the analytical capabilities of the instrument; specifically, our objectives for the training course would include:

1. Learning the principles of XPS (X-ray Photoelectron Spectroscopy).
2. Instrumental session to apply the technique on some samples.

Therefore we request 2 days of training, as discussed and agreed with the instrument scientists, in order to provide strong know-how on the principles of the technique during the first day of training, with valuable examples on data analysis, while the second day will be dedicated to an experimental session on selected samples in order to explore the analytical capabilities of the instrument in terms of surface composition analysis, elemental quantification, oxidation state analysis and depth profiling.

[1] Review on surface-characterization applications of X-ray photoelectron spectroscopy (XPS): Recent developments and challenges; D. Nanda Gopala Krishna, John Philip; Applied Surface Science Advances; Volume 12, December 2022, 100332

Experiment Proposal

Experiment number GP2024067

Principal investigator (*) Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator

Experiment title	Advanced Analysis of Surface and Composition Variations in Treated Brass Samples with ESCALAB QXi (XPS)		
MRF Instrument	**ESCALB QXi**	**Days requested:**	3
Access Route	Direct Access	**Previous GP Number:**	NO
Science Areas	Materials	**DOI:**	-
Sponsored Grant	None	**Sponsor:**	-
Grant Title	-	**Grant Number:**	-
Start Date	-	**Finish Date:**	-
Similar Submission?	-		
Industrial Links	RGF SRL		

Non-Technical Abstract

Brass-treated accessories are vital for leather goods and footwear, offering both functionality (like zippers and closures) and aesthetic appeal (such as decorative elements on bags and shoes). Ensuring these components remain durable and free from corrosion is essential. Therefore, research must focus on understanding oxidation to improve manufacturing and surface treatments, guaranteeing the long-term durability and appearance of brass accessories. Understanding the chemical composition on the surface of the brass accessories after corrosion will lead to significant improvements in the development of surface protective treatments and will contribute to the development of more robust and durable fashion and luxury items.

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY		
MRF Instrument	**ESCALB QXi**	**Days Requested:**	3
Special requirements:			

SAMPLE

Material	Brass	-	-
Formula	Cu, Zn	-	-
Forms	Solid		
Volume	4 cc		
Weight	10 g		
Container or substrate	No special need	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	RT - K	-	-
Pressure Range	Atmospheric pressure - mbar	-	-
Magnetic field range	No - T	-	-
Standard equipment	-	-	-
Special equipment	No special equipment needed	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	No	-	-
Special equip. reqs	No	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	No	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Advanced Analysis of Surface and Composition Variations in Treated Brass Samples with ESCALAB QXi (XPS)

1. Background and context

Brass-treated accessories are essential in the leather goods and footwear industries due to their dual functional and aesthetic purposes. They serve practical roles as zippers, closures, and handle elements, while also enhancing the visual appeal of the finished products through decorative pieces on bags, charms, and plaques on shoes. It is crucial that these components remain durable and maintain their appearance without corrosion over time. Thus, research and development must focus on understanding surface composition and modification arising from oxidation, in order to optimize manufacturing processes and surface treatments, therefore ensuring long-term durability and functionality of brass accessories themselves and on the finished products, like belts, bags and shoes.

This proposal therefore aims to investigate the surface composition of samples that show corrosive alterations on the surface using XPS (X-ray Photoelectron Spectroscopy) with the ESCALAB QXi, providing detailed elemental and surface information that could be correlated with the mechanisms of corrosion that have occurred on the surface. The fashion and luxury industries could than take proactive measures to treat or modify the product process and the surface finishing applied on the articles, thereby enhancing the durability and quality of the final finished products.

2. Proposed experiment

This proposal is part of a broader study to investigate the modification of the surface composition of treated brass articles after the built-up of oxidation phenomena that gave alteration of the aspect of the surface. The study involves several instrumental techniques: a) X-ray Photoelectron Spectroscopy (XPS); b) Scanning electron microscopy (SEM); c) RAMAN Spectroscopy.

XPS investigation with the ESCALAB QXi would give a great contribution to this study since it is an advanced tool for surface analysis, particularly useful for analysing the outermost atomic layers of materials, making it an excellent technique for studying surface composition arising from corrosion. This technique would allow insight on the elements present on the surface, their oxidation states and chemical environment providing crucial information for corrosion mechanism understanding.

In particular the analysis will provide valuable insights on the following aspects:

1. Surface Composition: XPS is highly sensitive to the surface composition of materials, providing detailed information about the elements present on the outermost atomic layer. This surface-specific data is critical for identifying products arising from corrosive phenomena.

2. Elemental Quantification: XPS allows for the precise quantification of elemental concentrations on the metallic surface. By understanding the abundance of specific elements, we can assess the effects of the corrosion processes.

3. Chemical State Information: XPS, can provide information on the chemical states of elements present on the surface. Understanding the oxidation states and chemical environments of these elements can help elucidate the corrosion mechanisms occurred.

By conducting these analyses, we will obtain essential data for understanding the corrosive effects that led to alterations on the surface of the brass articles, useful for research and development teams to improve production processes and surface finishing ultimately leading to improved quality and durability of the accessories.

3. Summary of previous experimental proposals or characterisation

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Historically, the durability of brass accessories for the fashion and luxury markets has been constrained by a reliance on empirical methods and trial and error. This approach lacks the detailed insight and predictive capability that sophisticated analytical tools like XPS investigation for the investigation of corrosive phenomena can provide. In particular, access to such advanced analysis and the requisite expertise is often limited. Although some literature provides a foundational understanding of the surface composition occurring after corrosion for brass articles used for different fields and applications, [1-2], a detailed exploration of the altered surface of the treated brass articles used in the fashion and luxury market for leathergoods and footwear after the occurring of corrosion, has not been conducted extensively so far. This research aims to fill that critical knowledge gap, to provide a correlation between the surface-specific elements found with the investigation, enabling to provide then recommendations for production methods and surface treatments that could mitigate the corrosive effects on the surface of metal accessories for fashion and luxury markets.

4. Justification of experimental time requested

As detailed in section 2, X-ray Photoelectron Spectroscopy (XPS) is a pivotal tool for this experiment due to its unique capabilities.

To achieve a comprehensive surface analysis of the brass samples, we request machine time for the ESCALAB QXi to perform XPS. This will enable us to obtain detailed elemental and surface information, which can be correlated with the corrosion mechanism. We request 3 days of experimental time to analyse 15 samples coming from 3 accessories batches: we collected 3 types of batches of brass accessories; for each batch we will then collect five samples for investigating the effects of the corrosion within the same batch.

This schedule ensures efficient use of the ESCALAB QXi, providing detailed insights into the surface composition and chemistry of the corrosion. By understanding these phenomena, we aim to improve the production processes and finishing treatments, in order to enhance the longevity and performance of brass accessories in the fashion and luxury industries.

[1] Federica Cocco / Marzia Fantauzzi / Bernhard Elsener / Antonella Rossi
How Surface Analysis Can Contribute to an Understanding of the Preventive Conservation of Brass Instruments
[2] Atmospheric corrosion of brass in outdoor applications. Patina evolution, metal release and aesthetic appearance at urban exposure conditions. S. Goidanich, J. Brunk, G. Herting, M.A. Arenas b, I. Odnevall Wallinder. Science of the Total Environment, 412-413 (2011) 46–57

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Experiment Proposal

Experiment number GP2024093

Principal investigator (*) Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator

Experiment title	Steel alloys behaviours to corrosion testing for metal accessories quality control with ESCALAB QXi (XPS)		
MRF Instrument	**ESCALB QXi**	**Days requested:**	2
Access Route	Direct Access	**Previous GP Number:**	NO
Science Areas	Materials	**DOI:**	-
Sponsored Grant	None	**Sponsor:**	-
Grant Title	-	**Grant Number:**	-
Start Date	-	**Finish Date:**	-
Similar Submission?	-		
Industrial Links	RGF SRL		

Non-Technical Abstract

In luxury leather goods like handbags, metal components are generally made of brass. However, for certain parts such as thin-section plates, brass may lack the necessary mechanical strength, leading to the use of steel instead. Traditionally, steel alloys are chosen for their low cost and ease of machining. Ensuring these components remain durable and free from corrosion is essential. Therefore, research must focus on understanding oxidation to improve the choice of the base materials in the manufacturing process, guaranteeing the long-term durability and appearance of the final leathergoods where the steel articles are mounted (like bags). Understanding the chemical composition on the surface of the steel components after accelerates corrosion testing will lead to significant improvements in the choice of steel base materials and this will contribute to the development of more robust and durable fashion and luxury items.

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY
MRF Instrument **ESCALB QXi** **Days Requested:** 2
Special requirements:

SAMPLE

Material	Steel	-	-
Formula	Fe	-	-
Forms	Solid		
Volume	4 cc		
Weight	10 g		
Container or substrate	No special need	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	RT - K	-	-
Pressure Range	Atmospheric pressure - mbar	-	-
Magnetic field range	No - T	-	-
Standard equipment	-	-	-
Special equipment	No special equipment needed	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	No	-	-
Special equip. reqs	No	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	No	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Steel alloys behaviours to corrosion testing for metal accessories quality control with ESCALAB QXi (XPS)

1. Background and context

In luxury leather goods like handbags, metal components are generally made of brass. However, for certain parts such as thin-section plates, brass may lack the necessary mechanical strength, leading to the use of steel instead. Traditionally, spring steel alloys are often chosen for their low cost and ease of machining, instead of the more expensive inox steels.
To ensure long-term durability, not only of the steel accessory but also of the entire leather item, it's important to understand the steel component's longevity. Therefore, tests exposing these accessories to corrosive environments have been developed to verify their durability over time. This would help in making informed decisions on steel alloys in terms of resistance, cost, and workability.
The primary difference between different steel alloys that are commonly used (spring steel or inox steel) lies in their mechanical properties and resistance to corrosion. Spring steel is more affordable and offers high tensile strength, making it suitable for components under mechanical stress. However, it has low corrosion resistance. In contrast, inox steel provides excellent corrosion resistance and durability, but at a higher cost and with more challenging workability. To determine the optimal choice in terms of cost and performance, a comparative evaluation of corrosion resistance is necessary to ensure the longevity of accessories mounted on leather goods. Understanding the long-term behavior of more economical spring steels compared to stainless steel is crucial for making an informed decision in product design and base material choice.

2. Proposed experiment

This proposal is part of a broader study to investigate the modification of the surface composition of steel articles after the exposure of the samples to corrosive atmospheres tests. The study involves the following instrumental techniques: a) X-ray Photoelectron Spectroscopy (XPS); b) Scanning electron microscopy (SEM).

XPS investigation with the ESCALAB QXi would give a great contribution to this study since it is an advanced tool for surface analysis, particularly useful for analysing the outermost atomic layers of materials, making it an excellent technique for studying surface composition arising from corrosion. This technique would allow insights on the elements present on the surface, their oxidation states and chemical environment providing crucial information for corrosion mechanism understanding and would be extremely helpful for the choice of the best alloy as base material.

In particular, the proposed analysis will provide valuable insights on the following aspects:

1. Surface Composition: XPS would provide detailed information about the elements present on the outermost atomic layer. This surface-specific data is critical for identifying products arising from corrosive phenomena.

2. Elemental Quantification: XPS allows for the precise quantification of elemental concentrations on the metallic surface. By understanding the abundance of specific elements, we can assess the effects of the corrosion processes.

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By conducting these analyses, we will obtain essential data for understanding the corrosive effects that led to alterations on the surface of the steel articles, useful for research and development teams to improve alloy type choice during design and costing of the products, ultimately leading to improved quality and durability of the final products (typically, luxury leather bags).

3. Summary of previous experimental proposals or characterisation

Historically, the durability of accessories for the fashion and luxury markets has been constrained by a reliance on empirical methods and trial and error. This approach lacks the detailed insight and predictive capability that sophisticated analytical tools like XPS investigation for the investigation of corrosive phenomena can provide. In particular, access to such advanced analysis and the requisite expertise is often limited. Although some literature provides a foundational understanding of the corrosion for steel articles used for different fields and applications, [1-2], a detailed exploration of the altered surface for leathergoods applications has not been conducted extensively so far. This research aims to fill that critical knowledge gap, to provide a correlation between the surface-specific elements found with the investigation, enabling to provide then recommendations for steel alloy choice and possible surface treatments that could mitigate the corrosive effects on the surface.

4. Justification of experimental time requested

As detailed in section 2, X-ray Photoelectron Spectroscopy (XPS) is a pivotal tool for this experiment due to its unique capabilities.

To achieve a comprehensive surface analysis of the two sets of steel samples, we request machine time for the ESCALAB QXi to perform XPS. This will enable us to obtain detailed elemental and surface information, which can be correlated with the corrosion mechanism. We request 2 days of experimental time to analyse 3 samples coming from 2 different alloys batches. We will study the difference between two different steel alloys and the samples realized with such alloys, after been subjected to accelerated corrosion trough exposure of corrosive atmosphere, will then be analysed with XPS in order to highlight the surface modifications and oxidation state of the surface elements and compare the results between the two steel alloys used. For each batch we will collect three samples for investigating the homogeneity of the effects of the exposure to corrosive atmospheres within the same batch.

This schedule ensures efficient use of the ESCALAB QXi, providing detailed insights into the surface composition and chemistry of the corrosion. By understanding these phenomena, we aim to improve the choice of base materials in the production processes and finishing treatments, in order to enhance the longevity and performance of steel components in the fashion and luxury industries.

[1] Shi, Wn., Yang, Sf. & Li, Js. Effect of nonmetallic inclusions on localized corrosion of spring steel. *Int J Miner Metall Mater* **28**, 390–397 (2021). https://doi.org/10.1007/s12613-020-2018-z
[2] Shiqiang Chen, Y. Frank Cheng, Gerrit Voordouw, A comparative study of corrosion of 316L stainless steel in biotic and abiotic sulfide environments, International biodeterioration & Biodegradation, Volume 120, 2017, Pages 91-96

Experiment Proposal

Experiment number GP2024128

Principal investigator	Dr Alice Pavan, Università degli Studi di Milano-Bicocca, ITALY
Co-investigator (*)	Dr Marzio Rancan, CNR, ITALY
Co-investigator	Professor Barbara La Ferla, University of Milano Bicocca, ITALY
Co-investigator	Dr Barbara Vercelli, Consiglio Nazionale delle Ricerche CNR, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Hydrothermal Sustainable Approaches for the Preparation of Carbon Quantum Dots: an XPS Insight into the Effect of Different Carbon Sources
MRF Instrument	**ESCALB QXi** — **Days requested:** 5
Access Route	Direct Access — **Previous GP Number:** no
Science Areas	Chemistry, Materials — **DOI:** -
Sponsored Grant	Yes — **Sponsor:** Other
Grant Title	Multifunctional Compounds for a Multitarget Approach against Neurodegenerative Disorders (MULTIFUN) — **Grant Number:** MUR PRIN Project n. 2022N9E847
Start Date	01/10/2023 — **Finish Date:** 30/09/2025
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	The present proposal is part of a broader research program aiming to study and develop reliable and sustainable synthesis approaches for preparing Carbon Quantum Dots (CDs) with robust and reproducible properties. We submit a request for an experimental campaign finalized at performing XPS analyses on a series of CDs samples prepared through the hydrothermal approach employing gallic acid (GA) and citric acid (CA) or both as carbon sources. The main scope is to investigate the possible synergic effect of the two carbon sources on the chemical composition of the CDs, with particular care given to the nitrogen and oxygen content and the analysis of the C1s, O1s, and N1s peaks. We expect to obtain a precise evaluation of the chemical surroundings of O, C, and N atoms, estimate the graphitization degree of the samples, and support the FT-IR analyses in determining the external functionalities.
Publications	B. Vercelli et al., Small Structures, recently submitted

ISIS neutron and muon source — **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Marzio Rancan, CNR, ITALY
MRF Instrument	**ESCALB QXi** — **Days Requested:** 5
Special requirements:	

SAMPLE

Material	Carbon Quantum Dots obtained at 160°C from gallic acid and urea, solvent EtOH (carbon based graphitic structures)	Carbon Quantum Dots obtained at 160°C from gallic acid/citric acid and urea, solvent EtOH (carbon based graphitic structures)	Carbon Quantum Dots obtained at 160°C from citric acid and urea, solvent EtOH (carbon based graphitic structures)
Formula	-	-	-
Forms	Solid	Solid	
Volume	cc	cc	cc
Weight	5 mg	5 mg	5 mg
Container or substrate	vial	vial	vial
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

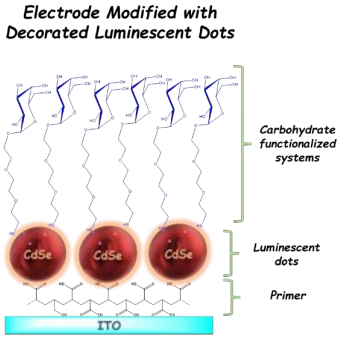
Temperature Range	- K	- K	- K
Pressure Range	- mbar	- mbar	- mbar
Magnetic field range	- T	- T	- T
Standard equipment	None	None	None
Special equipment	no	no	no

SAFETY

Prep lab needed	Yes	Yes	Yes
Sample Prep Hazards	no	no	no
Special equip. reqs	no	no	no
Sensitivity to air	No	No	No
Sensitivity to vapour	No	No	No
Experiment Hazards	no	no	no
Equipment Hazards	-	-	-
Biological hazards	no	no	no
Radioactive Hazards	no	no	no
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	Disposed by IS

1. Background and Context

The group of Prof. La Ferla at the Department of Earth and Environmental Sciences of the University of Milano-Bicocca, in synergic collaboration with CNR in Milan, has developed new self-assembled clacogenide-based lumiscent dots decorated with carbohydrate-bearing ligands (Scheme 1) for medicinal chemistry applications. The blue-sky research activity is documented by a series of pubblications in ISI journals.

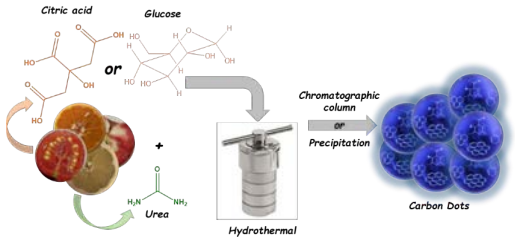


Scheme 1. – Lumiscent dots decorated with carbohydrate-bearing ligands.

Within this research program and considering the growing request for eco-sustainable, non-toxic nanomaterials, the research studies were recently devoted to the employment of carbon quantum dots (CDs) as a "green" and cheap alternative to calcogenide-based dots.

CDs are fluorescent carbon-based nanomaterials, which, since their discovery in 2004, gained grooving interest from the research community, because of their excellent fluorescence properties and surface rich in functionalities, which enables functionalization with a wide range of molecules, including receptors, bio-molecules, molecular semiconductors, etc. Furthermore, they are soluble in water, exhibit an extremely low toxicity, and an excellent biocompatibility useful for real-world biological applications. Their synthesis approaches are simple, sustainable, and could employ cheap and recyclable precursors derived from biomass and agro-industrial waste.

As a first approach, the group focused on the development of a sustainable CDs synthesis strategy, which could be reliable and reproducible. They selected the hydrothermal approach owing to its feasibility for large-scale industrial applications, and they employed precursors that could be obtained from agro-industrial waste, like citric acid (CA), glucose (Glu) as carbon sources and urea as both base and nitrogen sources (Scheme 2).



Scheme 2. – Scheme of hydrothermal preparation of Carbon Quantum Dots

They published two preliminary works dealing with the role played by the nitrogen centers and the thermal post-treatments, respectively, on CDs electrochemical and optical properties[1]. Then they pass to study the influence of the reaction parameters on CDs properties and published a work on the issues encountered in the synthesis/purification of red-emitting CDs[2]. When I joined the group with a research fellowship financed by the MUR PRIN 2022 project n° 2022N9E847 (MULTIFUN), I studied the influence of the process parameters of blue-emitting CDs, focusing on their optical (UV-vis absorption), FT-IR, and morphological (TEM) aspects. The results of my research work are part of a recently submitted more comprehensive paper[3] on the subject. In a further step of my research program, I employed gallic acid (GA) alone and in combination with CA, as carbon source to study a possible synergic effect on the optical and chemical properties of CDs. Preliminary, UV-vis absorption and FT-IR determinations seem to support the hypothesis. In this context, chemical (XPS) analyses are of crucial interest/importance to obtain a comprehensive chemical characterization of the new CDs. In particular, we expect that the CDs obtained from the combination of both carbon sources (GA and CA) will merge the intrinsic properties of the samples prepared employing only one carbon source (GA or CA). We also expect to obtain information about the influence of the temperature process on the CDs prepared employing both GA and CA.

2. Proposed experiment

With the present proposal, we submit the request of a 4/5 days' experimental campaign finalized at performing XPS analyses on a series of CDs samples (optimistically 3/4) prepared through the hydrothermal approach employing GA, CA or both as carbon sources. The main scope is to investigate the influence on the chemical composition of the obtained materials with particular care to the Nitrogen and Oxygen content and the analysis of C_{1s}, O_{1s} and N_{1s} peaks.

The results expected from the proposed experimental campaign are of paramount importance for the development of the research programme briefly described at point 1, because they are expected to provide a precise evaluation of the chemical surroundings of O, C and N atoms, estimate the graphitization degree of the samples and support the FT-IR analyses in the determination of the external surface functionalities, employing a facility (XPS instrument), which is not present in the laboratories of the Department of Earth and Environmental Sciences Department of the University of Milano-Bicocca.

3. Summary of previous experimental proposals or characterisation

Dr. Vercelli submitted the proposal n°GP2024022 for a series of TEM analyses of CDs samples.

4. Justification of experimental time requested

For the development of the present proposal, we selected the ISIS@MACH ITALIA ESCALAB QXi X-ray photoelectron spectrometer facility at the CNR-ICMATE headquarter in Padova, because it meets the above-reported experimental requests. We planned XPS analyses on 3/4 CDs samples for a total of 4/5 days, including eventual time waste related to possible issues in sample preparation, specific instrument settings, and unexpected problems during measurement execution and signal optimization/collection.

5. References

1. Vercelli B. et al. Elec. Acta, **2021**, 138557, https://doi.org/10.1016/j.electacta.2021.138557; Vercelli B. et al. Molecules **2023**, 28(1), 72; https://doi.org/10.3390/molecules28010072.

2. Vercelli B. et al. Nanomaterials **2023**, 13, 1365; https://doi.org/10.3390/nano13101635.

3. Vercelli B. et al. Small Structures, submitted.

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Experiment Proposal

Experiment number GP2024136

Principal investigator (*)	Dr Monica Tonelli, University of Florence, ITALY
Co-investigator	Miss Giulia Mugnaini, University of Florence, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Study of the interactions occurring between halloysite nanotubes and sodium hexametaphosphate in alginate biocomposites towards the preparation of anisotropic structures
MRF Instrument	**ESCALB QXi**
Access Route	Direct Access
Science Areas	Chemistry, Materials
Sponsored Grant	None
Grant Title	-
Start Date	-
Similar Submission?	-
Industrial Links	-
Days requested:	3
Previous GP Number:	GP2024035
DOI:	-
Sponsor:	-
Grant Number:	-
Finish Date:	-

Non-Technical Abstract

This study focuses on the development of biocomposite wires made of calcium cross-linked alginate and halloysite nanotubes (HNTs) through extrusion. The combination of this polysaccharide with inorganic nano fillers results in a material with enhanced physico-chemical properties, attractive across various fields of research. To prevent the aggregation of the nanotubes and obtain homogenous composites, HNTs were dispersed in the presence of sodium hexametaphosphate. According to the preliminary results, some optimised formulations allowed to homogeneously disperse the HNTs, and in turns promoting the alignment of the nanoclays.
XPS analyses will provide paramount information about the potential alteration of HNTs structures, occurring when dispersing these nanotubes in the investigated composites. XPS would also shed lights on the differences among the binding energies of select elements, unravelling the effect of chemical makeup variations.

Publications -

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E-platform: No

Instruments	
Access Route	
Science Areas	
Sponsored Grant	
Grant Title	
Start Date	
Similar Submission?	
Industrial Links	
Days Requested:	
Previous RB Number:	
DOI:	
Sponsor:	
Grant Number:	
Finish Date:	

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Sample record sheet

Principal contact	Dr Monica Tonelli, University of Florence, ITALY
MRF Instrument	**ESCALB QXi**
Special requirements:	
Days Requested:	3

SAMPLE

Material	Alginate	Halloysite nanotubes	sodium hexametaphosphate
Formula	(C6H8O6)n	Al2Si2O5(OH)4 * 2H2O	Na6(PO3)6
Forms	Solid	Solid	Solid
Volume	cc	cc	cc
Weight	3 g	3 g	3 g
Container or substrate	glass vial	glass vial	glass vial
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	RT - K	RT - K	RT - K
Pressure Range	atmospheric pressure - mbar	atmospheric pressure - mbar	atmospheric pressure - mbar
Magnetic field range	none - T	none - T	none - T
Standard equipment	None	None	None
Special equipment	none	none	none

SAFETY

Prep lab needed	No	No	Yes
Sample Prep Hazards	no	no	no
Special equip. reqs	no	no	none
Sensitivity to air	No	No	No
Sensitivity to vapour	No	No	No
Experiment Hazards	no	no	no
Equipment Hazards	-	-	-
Biological hazards	no	no	no
Radioactive Hazards	none	no	no
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	Disposed by IS

1. Background and Context

The development of biocomposite materials with fine-tuned architectures is an attractive challenge across various research fields. The combination of biopolymers and inorganic nanofillers is particularly stimulating, yielding materials with enhanced physico-chemical properties. Nowadays, numerous studies are focused on the use of alginate (Alg), a polysaccharide able to chelate di- and trivalent cations (e.g., Ca^{2+}) resulting in physically cross-linked hydrogel. Alg can be enriched with various additives, either to reinforce it or to include specific functionalities. In this perspective the use of halloysite nanotubes (HNTs) can result in materials with interesting physico-chemical properties. HNTs are naturally occurring biocompatible and economic aluminosilicate clays with a multi-walled tubular structure, able to entrap and gradually release various active molecules. Alg/HNTs biocomposites are promising candidates as innovative multi-functional systems. Due to their elongated structure, HNTs can be oriented through the application of an external shear force, as the one applied by brushing,[1] or during the injection procedure. Nonetheless, the alignment of HNTs during the preparation of micro- and milli-metric wires, is not straightforward, also due to the tendency of these nanotubes to form aggregates. To this purpose, a pre-treatment can be beneficial to select high aspect ratio nanotubes, separate the aggregates, and improve the stability of the dispersions, eventually using specific additives.[1]

This study aims at developing biocomposite wires with oriented nanotubes composed of calcium cross-linked alginate and HNTs, with or without sodium hexametaphosphate as deflocculant. This research involves the work of a PostDoc and a researcher. Overall, the research is supported by a fellowship, and by The National Recovery and Resilience Plan of Italy "3A-Italy Circular and Sustainable Made in Italy" (PNRR, 3A-ITALY - B83C22004890007)

2. Proposed experiment

To study the effect of the interaction between HNT nanotubes and Alg chains on the structure of HNTs, in presence or absence of sodium hexametaphosphate (HMP), the binding energy of some selected elements could be determined with X-ray photoelectron spectroscopy (XPS). This technique could be employed both on HNTs as powders (before and after the pretreatment involving the use of HMP, as shown in Figure 1A), and on the dry composites (Alg/HNT and Alg/HNT-HMP). To this purpose, ESCALAB QXi instrument available in the ISIS@MACH ITALIA could be crucial, allowing to gain information on the present elements and on their chemical makeup.

3. Summary of previous experimental proposals or characterisation

We investigated various wires composed of calcium cross-linked Alg and HNTs, prepared by injecting the Alg/HNTs dispersions in a $CaCl_2$ water solution using an Hyrel 3D printer. To prepare stable clays' dispersions that could promote the orientation of the nanotubes upon injection, we investigated different concentration of HNTs, and we explored multiple methods of mixing, including magnetic stirring, sonication, and ultra-turrax dispersion, as well the incorporation of HMP as dispersant (see Figure 1A). The dispersions were evaluated by means of sedimentation tests, granulometry and ζ-potential measurements, and then used to prepare the wires. Some selected dry Alg/HNTs wires were preliminarily characterized by means of SEM, both looking at the external surfaces and at the inner sections, after cutting these extruded fibres toward the extrusion direction (see Figure 1B). According to the preliminary results, some optimized formulations allowed to homogeneously disperse the HNTs, and in turns promoting the alignment of the nanoclays in specific

conditions, arising to the different chemical makeup encountered by HNTs. We can infer that the attractive interactions that are expected to occur between Na^+ and the negatively charged external surface of the nanotubes, and between PO_4^{3-} and the positively charged internal surface of the nanotubes prevented the formation of HNTs' aggregates (see Fig. 1A top).

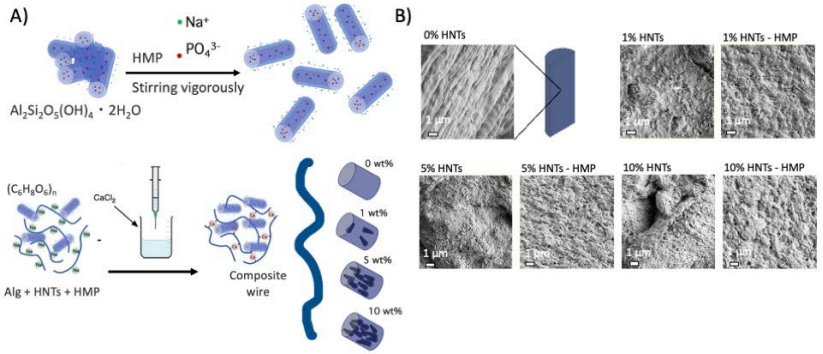

Fig 1. A) Schematic representation of the nanotubes' pretreatment and wires preparation, B) FE-SEM micrographs of the inner sections of alginate wires containing different concentration of HNTs, prepared without or with HMP dispersant.

4. Justification of experimental time requested

The opportunity to use the ESCALAB QXi instrumentation will provide paramount information about the interactions between the components of the composites, potentially affecting HNTs structure and organization. A variation on the binding energy of some selected elements arising to HNTs and HMP (i.e. O, Al, Si, Na, P and C) could be used to evidence differences resulting from Na^+/nanotubes and PO_4^{3-}/nanotubes attractive interactions, occurring between HMP and HNT external and internal surfaces, respectively.[2] It is important to remark here that HNTs display large specific surface area, and the elements involved in these interactions would significantly influence the overall photoemission experiments. At the same time when mixing HNTs with Alg, we could expect a variation on the nanotubes' structure, arising to the interaction between COO^- groups of Alg and the internal surface of HNTs, which would be detectable through XPS analyses.[3]

After discussing the technical details of these experiments with the instrument scientist, we plan to study 6 different samples by XPS surveys coupled with high resolution acquisitions of selected photoemission peaks. At least 3 different points for each sample will be investigated to assess their homogeneity. The samples analysed will be: pure HNTs powder, pure HMP powder, pure Alg powder, HNTs/HMP powder, Alg/HNTs composite, Alg/HNTs-HMP composite. A working time of 3 days is expected.

[1] Zhao et al., *J. Mater. Chem. B*, **2020**, 8, 838-851.
[2] Kubala-Kukuś et al., *Radiation Physics and Chemistry,* **2020**, 175, 108149.
[3] Al-Gaashani et al., Scientific Reports, **2022**,12, 21633.

Experiment Proposal

Experiment number GP2024167

Principal investigator	Dr Monica Tonelli, University of Florence, ITALY
Co-investigator	Miss Giulia Mugnaini, University of Florence, ITALY
Co-investigator (*)	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Study of functionalized Halloysite nanotubes
MRF Instrument	**ESCALB QXi**
Access Route	Direct Access
Science Areas	Chemistry, Materials
Sponsored Grant	None
Grant Title	-
Start Date	-
Similar Submission?	-
Industrial Links	-

Days requested: 1
Previous GP Number: no
DOI: -
Sponsor: -
Grant Number: -
Finish Date: -

Non-Technical Abstract

The aim of this work is the development of halloysite nanotubes (HNTs) functionalized with two different moieties, toward the preparation of composites with tailored physico-chemical properties, attractive across various fields of research:
i) to prevent the aggregation of the nanotubes and obtain homogenous composites, HNTs were functionalized with phosphate, to take advantage of the well-recognized deflocculant properties of phosphate salts.
ii) HNTs where grafted with caffeine to develop hybrid systems endowed with improved antimicrobial properties, as caffeine not only provides antibacterial properties by itself, but it also improves the biological activity of other drugs that could be loaded in the nanotubes.
XPS analyses will provide paramount information about the functionalization of HNTs with phosphate and with caffeine, unravelling the chemical structure of pristine and grafted HNTs.

Publications -

ISIS neutron and muon source

E-platform: No

Instruments	
Access Route	
Science Areas	
Sponsored Grant	
Grant Title	
Start Date	
Similar Submission?	
Industrial Links	

Days Requested:
Previous RB Number:
DOI:
Sponsor:
Grant Number:
Finish Date:

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Sample record sheet

Principal contact
MRF Instrument **ESCALB QXi** **Days Requested:** 1
Special requirements:

SAMPLE

Material	Halloysite	sodium hexametaphosphate	caffeine
Formula	Al2Si2O5(OH)4 * 2H2O	Na6(PO3)6	$C_8H_{10}N_4O_2$
Forms	Solid	Solid	Solid
Volume	cc	cc	cc
Weight	3 g	3 g	3 g
Container or substrate	glass vial	glass vial	glass vial
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	RT - K	RT - K	RT - K
Pressure Range	atmospheric pressure - mbar	atmospheric pressure - mbar	atmospheric pressure - mbar
Magnetic field range	none - T	none - T	none - T
Standard equipment	None	None	None
Special equipment	none	none	none

SAFETY

Prep lab needed	No	No	No
Sample Prep Hazards	no	no	no
Special equip. reqs	no	no	no
Sensitivity to air	No	No	No
Sensitivity to vapour	No	No	No
Experiment Hazards	no	no	no
Equipment Hazards	-	-	-
Biological hazards	no	no	no
Radioactive Hazards	no	no	no
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	Returned to user by instrument scientist (when inactive)	Returned to user by instrument scientist (when inactive)

1. Background and Context

Halloysite nanotubes (HNTs) are naturally occurring biocompatible and economic aluminosilicate clays with a multi-walled tubular structure, with an outer diameter of 50–80 nm, a lumen diameter of 10–15 nm, and length of about 1 µm. HNTs consist of a hollow tubular double-layer of aluminosilicate clay ($Al_2Si_2O_5(OH)_4nH_2O$), chemically resembling kaolin and able to entrap and gradually release various active molecules. HNTs have two key functional groups on external (Si-O-Si) and eternal (Al-OH) surfaces, that lead to different surface charges and pH. As a matter of fact, HNTs have been already proven to be effective as innovative functional systems in various composites. At the same time, several research focused on the selective functionalization of HNTs surface for several reasons, including the prevention of agglomeration phenomena, the improvement of interfacial interactions with polymers and other matrixes, and to influence their binding ability with active molecules, towards applications as nanocarriers.[1,2] As a result, the development of loaded and/or functionalized HNTs is an attractive challenge across various research fields from biomedicine, to environmental science and catalysis. Nowadays, numerous studies are focused on the synthesis of HNTs with specific functional groups, but the characterization of these systems is not always straightforward.[2]

In this study we developed HNTs functionalized with two different functional groups, toward the preparation of nanotubes enriched with specific properties. In the first synthetic pathway (see Fig. 1A) HNTs were functionalized with phosphate groups, to prevent their agglomeration and develop nanoclays easily dispersible in several polymeric matrixes. In the second method (see Fig. 1B) HNTs were grafted with caffeine, toward the development of hybrid systems endowed with improved antimicrobial properties (see also Section 3).

This research involves the work of a PostDoc and a researcher. Overall, the research is supported by a fellowship, and by The National Recovery and Resilience Plan of Italy "3A-Italy Circular and Sustainable Made in Italy" (PNRR, 3A-ITALY - B83C22004890007)

2. Proposed experiment

To study the synthesis of HNT-PO_4 and HNT-caffeine nanotubes, the binding energy of some selected elements could be determined with X-ray photoelectron spectroscopy (XPS). This technique could be employed on HNTs as powders before and after the functionalization, focusing on specific elements that are absent in pristine nanotubes and that would be present on functionalized HNTs (P and N, see also Fig 1). It is worth mentioning here that the functionalization of the nanotubes could also influence the characteristic structure of HNTs, influencing the binding energy and the broadening of the signals ascribed to Al and Si, as already reported when adsorbing nanoparticles on HNTs surface.[2] To this purpose, ESCALAB QXi instrument available in the ISIS@MACH ITALIA could be crucial, allowing to gain information on the present elements and on their chemical makeup.

3. Summary of previous experimental proposals or characterisation

We synthesized HNTs functionalized with two different functional groups. In the first synthetic pathway (see Fig. 1A) HNTs were functionalized with phosphate groups (HNT-PO_4), to take advantage of the well-recognized deflocculant properties of phosphate salts, already demonstrated efficient in preventing HNTs aggregation, acting as dispersants when directly mixed with HNTs powders.[3] In the second method (see Fig. 1B) HNTs were grafted with caffeine (HNT-caffeine), to develop hybrid systems endowed with bio-based long-lasting antimicrobial properties. In fact, caffeine not only provides antibacterial properties by itself, but it also improves the biological activity of other drugs that could be loaded in the lumen of the nanotubes.[4]

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In some preliminary experiments HNTs were characterized by means of zeta potential and fourier transform infrared spectroscopy (FT-IR) experiments, to evaluate the correct grafting of the systems during the various steps of the synthesis, and to confirm the functionalization of the nanotubes. However, FT-IR could not confirm the correct grafting of phosphate groups (Fig. 1A), and thus the employment of XPS would be fundamental. At the same time, also the synthesis of HNT-caffeine was monitored by means of FT-IR, but -C=N and -C=O functionalities could be only spotted in HNTs spectra, thus XPS could be very useful to confirm the correct functionalization of the nanoclays.

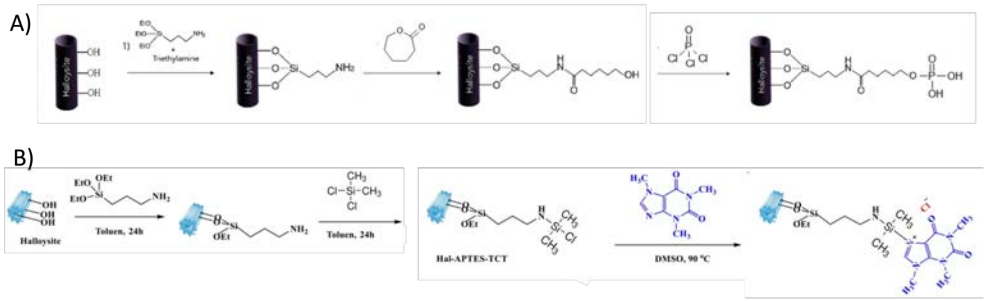

Fig 1. A) Scheme of the functionalization of HNTs with phosphate groups (A) or with grafted caffeine (B).

4. Justification of experimental time requested

The opportunity to use the ESCALAB QXi instrumentation will provide information about the functionalization of HNTs with phosphate and with caffeine. A variation on the abundance of some selected elements arising to HNTs, HMP and caffeine (i.e. O, Al, Si, Na, P, N and C) could be used to evidence differences resulting from the correct grafting of the nanotubes. In fact, XPS could be used to study the chemical structure of HNTs and gain the percent atomic concentrations of HNT-PO_4 and HNT-caffeine samples, focusing on P and N elements to quantify the density of the new groups synthesized on the surface of HNTs, as already reported for analogous systems elsewhere.[5,6]

We plan to study 3 different samples by XPS surveys coupled with high resolution acquisitions of selected photoemission peaks. At least 3 different points for each sample will be investigated to assess their homogeneity. The samples analysed will be: pure HNTs powder, HNT-PO_4 powder, and HNT-caffeine powder. A working time of 1 day is expected.

[1] D. Ganapathy et al., *Journal of Nanomaterials*, **2022**.
[2] R. Al-Gaashani et al., *Scientific Reports,* **2022**, 21633.
[3] E. Durgut et al., *Minerals*, **2022**, 12 (11), 1426.
[4] P. Salas-Ambrosio et al., *ACS Bio & Med Chem*, **2023**, 3 (2), 189–200
[5] M. Hassan Kanani-Jazi et al., *Chemical Engineering Journal,* **2024**, 482, 148746.
[6] B. Szczepanik et al., *Materials*, **2020**,13(24), 5647.

FIB-SEM GAIA 3

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Experiment Proposal

Experiment number GP2024123

Principal investigator	Mr Andrea Pierozzi, Trinity College Dublin, IRELAND
Co-investigator (*)	Professor Francesco Di Benedetto, Università degli Studi di Ferrara, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Ca-Mg-Fe carbonates alteration of basalts at Sverrejfellet Volcano (Svalbard, Norway)
MRF Instrument	**FIB-SEM GAIA 3**
Access Route	Direct Access
Science Areas	Chemistry, Environment, Materials
Sponsored Grant	None
Grant Title	-
Start Date	-
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	The proposal aims to study how certain types of carbonates are formed. We will analyze samples from a volcano in Svalbard, Norway, to understand how basalt breaks down and absorbs CO2. This research is important for developing technologies that can capture CO2 without using a lot of water. The results will help advance CO2 storage technologies.
Publications	-

Days requested: 4
Previous GP Number: no
DOI: -
Sponsor: -
Grant Number: -
Finish Date: -

ISIS neutron and muon source

E-platform: No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Professor Francesco Di Benedetto, Università degli Studi di Ferrara, ITALY
MRF Instrument **FIB-SEM GAIA 3** **Days Requested:** 4
Special requirements:

SAMPLE

Material	Carbonates with basaltic alteration	-	-
Formula	(Ca,Mg,Fe)CO3	-	-
Forms	Solid		
Volume	3 cc		
Weight	5 mg		
Container or substrate	thin section of rock on glass	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	298 - 298 K	-	-
Pressure Range	1 - 1 mbar	-	-
Magnetic field range	0 - 0 T	-	-
Standard equipment	Do Not Know	-	-
Special equipment	No	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	No	-	-
Special equip. reqs	no	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	no	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Removed By User	-	-

Experimental Proposal for MRF Proposal:

1. Background and Context

The proposal aims to investigate the compositional variation of a sequence of Ca-Mg-Fe carbonates to clarify the processes of their formation. The case study involves the analysis of 3 samples from the basalt substrate, weathering layer, and carbonate sequence obtained at the Sverrejfellet volcano (Svalbard, Norway). Deciphering the processes controlling the mechanisms of basalt weathering and CO_2 fixation in such a peculiar test site will have a great relevance in the development of further CCS technologies that do not require large quantities of water. The results of this study will thus contribute to the advancement in the relevant field of CO_2 sequestration technologies.

The Sverrejfellet volcano is the northernmost volcano worldwide, located in the Svalbard archipelago. The degassing of primitive alkali basalts during the subglacial eruptions of Sverrefjellet would have produced notable amounts of carbon dioxide (CO_2) in conjunction with glacial meltwater. Moreover, Sverrejfellet is one of the few natural places where natural carbon capture and storage (CCS) in basaltic materials occurs. The degree to which carbonation happens in the basalts is determined by the release of cations into liquid solutions. Upon dissolving, Ca reacts with carbonate to produce calcite and/or aragonite ($CaCO_3$) leading to precipitation at temperatures below approximately 300 °C [1]. At an interval between 65 °C and 100°C dissolved magnesium reacts with carbonate to form magnesite (MgCO3) and dolomite ($CaMg(CO_3)_2$) [2]. The development of iron-containing carbonates might be restricted based on the oxidation level. Fe^{2+} frequently undergoes oxidation to Fe^{3+} before it can form siderite ($FeCO_3$). As a result, there are no documented Fe^{3+} carbonates found in nature. Instead, when Fe^{2+} undergoes oxidation, it generates Fe oxides and Fe oxyhydroxides. However, under conditions where the pH is sufficiently low to prevent the oxidation of Fe^{2+} to Fe^{3+}, and at moderate temperatures, ankerite ($CaFe(CO_3)_2$) can be produced [3]. An extensive preliminary characterization of the samples, allowed us to assess (Fig.1) the occurrence of 4 distinct regions characterized by different Ca-Mg-Fe composition and textural features. However, due to the small size of the different layers, identifying the individual crystallization stages of the sample and how they change during growth has not yet been possible using laboratory facilities. Basalts are being considered important potential sites for carbon capture and storage (CCS) as an alternative to traditional sedimentary reservoirs due to several advantages: their highly reactive nature to CO2-containing fluids [4] and their significant amounts of divalent metal cations (roughly 25 wt % calcium, magnesium, and iron oxides [4]. Understanding how these carbonates formed at surface conditions could allow.

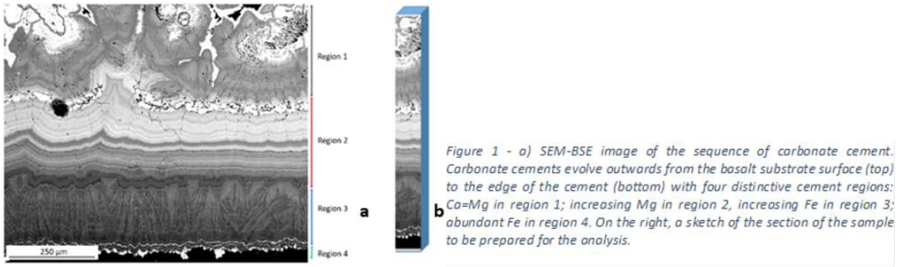

Figure 1 - a) SEM-BSE image of the sequence of carbonate cement. Carbonate cements evolve outwards from the basalt substrate surface (top) to the edge of the cement (bottom) with four distinctive cement regions: Ca=Mg in region 1; increasing Mg in region 2, increasing Fe in region 3; abundant Fe in region 4. On the right, a sketch of the section of the sample to be prepared for the analysis.

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2. Proposed experiment

We plan to re-investigate the intrinsic natural heterogeneity, in terms of crystalline size, chemical composition, and supposedly mineral assemblage of the whole alteration layer, in a core-to-rim approach, by using Scanning Electron Microscopy. We plan to operate on the same thin section well characterized shown in Fig. 1 (see following for further information). Then, we plan to extract using a FIB, some sections (4-6) able to capture the major events in the layered alteration growth of the basalt, so having perfect absorber for further investigation, planned to be executed at the ESRF facility (proposal under evaluation). Sections could be of the order of magnitude of 10 by 10 by 5 µm in size.

3. Summary of previous experimental proposals or characterization

A thin section of the original sample was the object of a thorough microanalytical and electron micromorphological characterization. The results obtained from this detailed characterization will help us to carry out future experiments with the aim of replicating this alteration and this sequence of carbonate layers. As a consequence of all this preliminary study, four layers of carbonate cement differing in terms of Ca, Mg, and Fe content, and of crystal size and texture, were identified. The Ca/Mg ratio in Region 1(see Fig. 1) carbonate is close to 0.50 and decreases to 0.13 in Region 3. In Region 4 instead the Ca content decreases further and there is the presence of distinct phases rich in Fe and Mg respectively. The missing information is the phase composition of the mineral assemblage hosting such variable composition, since using traditional characterization methods does not allow us to achieve information on the mineralogical composition (mineral assemblage and quantification) and the structural and crystal-chemical details of the minerals in the different regions.

4. Justification of experimental time requested

We guess the FIB-SEM GAIA 3 instrumentation is perfectly suitable for the proposed experiment, as it couples the capability of the FIB column, with an excellent SEM instrumentation, coupling FEG technology and several SE and BSE detectors, and finally enabling the use of the X-ray microanalysis. All these tools will be valuable in exploring the natural variability and selecting the proper zone to create the absorbers for SR experiments. We guess 4 days of experimental time is necessary, for the requested task.

5. Bibliography

[1] Ellis, A. J., American Journal of Science, 261(3), pp. 259–267 (1963) [2] Saldi, G. D.; Jordan, G.; Schott, J.; Oelkers, E.H., Geochimica et Cosmochimica Acta. 73(19), pp. 5646–5657 (2009) [3] Gysi, A. P. and Stefánsson, A., Geochimica et Cosmochimica Acta. 81, pp. 129–152 (2012) [4] Gislason, S. and Oelkers, E., Science, 334, pp. 373–374 (2014)

Experiment Proposal

Experiment number GP2024124

Principal investigator (*) Professor Giancarlo Capitani, University of Milano-Bicocca, ITALY
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator

Experiment title	SEM imaging, FIB manipulation and TEM characterization of mineralized granules (“spherites”) found in the honeybees’ midgut		
MRF Instrument	**FIB-SEM GAIA 3**	**Days requested:**	3
Access Route	Direct Access	**Previous GP Number:**	No
Science Areas	Biology and Bio-materials	**DOI:**	-
Sponsored Grant	None	**Sponsor:**	-
Grant Title	-	**Grant Number:**	-
Start Date	-	**Finish Date:**	-
Similar Submission?	-		
Industrial Links	-		

Non-Technical Abstract

The present proposal aims at investigating the mineralized granules, commonly indicated as “spherites” or “spherocrystals”, commonly composed of metal phosphates, found in the midgut of insects. Spherites have been attributed the function of regulating the composition of the internal environment and detoxification when insects are contaminated with toxic metals. Similar mineralized granules form during the development of breast cancer (mammary microcalcifications) in humans. The proposed experiment entails the study by SEM-EDS of spherites found in the midgut of honey bees, their preparation through FIB milling and further study by TEM-EDS.

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Science and Technology Facilities Council
ISIS Neutron and Muon Source

Sample record sheet

Principal contact Professor Giancarlo Capitani, University of Milano-Bicocca, ITALY
MRF Instrument **FIB-SEM GAIA 3** **Days Requested:** 3
Special requirements:

SAMPLE

Material	Honey bees midgut carrying metal phosphate spherites	-	-
Formula	-	-	-
Forms	Solid		
Volume	1 cc		
Weight	1000 mg		
Container or substrate	Aluminium stub	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	ambient - K	-	-
Pressure Range	ambient - mbar	-	-
Magnetic field range	ambient - T	-	-
Standard equipment	None	-	-
Special equipment	none	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	No	-	-
Special equip. reqs	Carbon coater	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	No	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Removed By User	-	-

MRF Proposal: SEM imaging, FIB manipulation and TEM characterization of mineralized granules ("spherites") found in the honeybees' midgut.

1. Background and Context

Many invertebrates are capable of dealing with surplus ions and toxic metals by a process of biomineralization. In several tissues – mostly involved with digestion, storage or excretion – mineralized granules are formed within the Golgi vesicles or the cisternae of the granular endoplasmatic reticulum (Brown, 1982 and reference therein). These mineralized granules, commonly indicated as "spherites" or "spherocrystals", are composed of a variety of metallic atoms like calcium, potassium and zinc, complexed with a phosphorous source (Gomes et al. 2012 and references therein). The aim of the present research is the collection of additional data on the occurrence, origin, formation, composition, structure and possible function of spherites (Fig. 1) detected in honey bees (*Apis mellifera*), and to compare them with literature data on spherites detected in other insects (e.g., Martoja and Ballan-Dufrangais, 1984; Pinheiro de Oliveira et al. 2008; Gomes et al. 2012). Since similar mineralized granules form during the development of breast cancer in humans (mammary microcalcifications have a crucial role in breast cancer detection; Scimeca et al. 2008), the results of this research might reveal of paramount importance for the prevention and treatment of cancer.

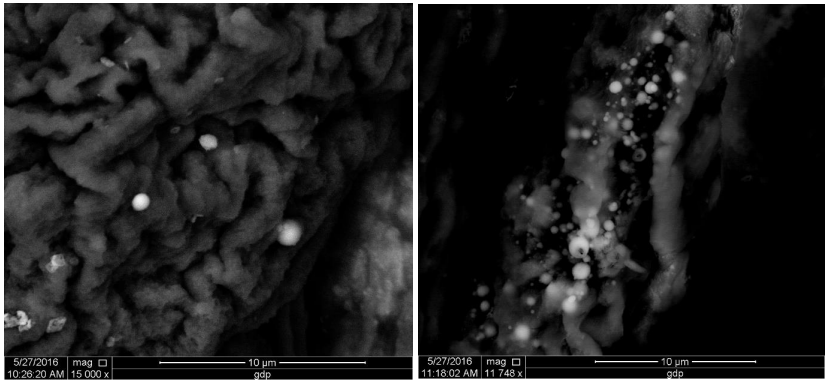

Figure 1. Backscattered SEM image of spherites (bright spheres) in the midgut of honeybees.

2. Proposed experiment

The spherites detected in honey bees and insects in general are heterogeneous in size, but generally comprised within few micrometres (Fig. 1). While such size can be easily detected via scanning electron microscopy (SEM), the observation of the largest spherites with a transmission electron microscope (TEM) – the ultimate technique for the achievement of structure and composition at a sub-microscopic scale – is hampered by the dimension (thickness) along the beam incidence, i.e. they are not electron transparent. On the other hand, smaller spherites are difficult to handle and prepare for TEM, and can provide only partial information. Therefore, the plan is:

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- dissecting honeybees' guts carrying the spherites and spreading them on adhesive carbon pads, in turn sustained by Al stubs;
- imaging the spherites, with a dual beam instrument – a SEM with a focus ion beam column (FIB) such as the Gaia 3 (Tescan);
- gathering morphological and chemical information – the latter though energy dispersive spectroscopy (EDS);
- picking up by a micromanipulator coupled with gas injection system (GIS) the largest spherites;
- sticking them on a TEM-FIB grid and milling down to electron transparency the spherites.

At this stage, the study can continue with TEM, which can be performed in site within the same application with the Talos F200X (ThermoFisher), if the allocated time is sufficient, or in a subsequent application, or elsewhere. The TEM should provide nanoscale structure and composition of spherites.

3. Justification of experimental time requested

Three days are the instrument time requested, exploited as it follows:

- the first day should be employed for the honeybees midgut preparation and imaging and EDS analysis of the spherites with the Gaia 3 SEM-FIB;
- the second day for the picking of spherites with the micromanipulator and their ion milling on the TEM-FIB grid;
- the third day for the observation of the milled spherites with the Talos F200X TEM. For statistical reasons, no less than four milled spherites are required.

4. References

Brown B.E. (1982) The form and function of metal-containing "granules" in invertebrate tissue. Biol. Rev. 57, 621-667.

Gomes F.M., Carvalho D.B., Peron A.C., Saito K., Miranda K., Machado E.A. (2012) Inorganic polyphosphates are stored in spherites within the midgut of *Anticarsia gemmatalis* and play a role in copper detoxification. Journal of Insect Physiology 58, 211–219. https://doi.org/10.1016/j.jinsphys.2011.09.008

Martoja R. and Ballan-Dufrangais C. (1984). The ultrastructure of the digestive and excretory organs. In: Insect Ultrastructure 2 (R.C. King & Akai, eds), Plenum Press, pp. 199-268.

Pinheiro de Oliveira D., Conte H. and Gregório Aparecida E. (2008) Spherites in the midgut epithelial cells of the sugarcane borer parasitized by *Cotesia flavipes*. Biocell, 32(1), 61-67.

Scimeca M., Giannini E., Antonacci C., Pistolese C.A., Spagnoli L.G., Bonanno E. (2014) Microcalcifications in breast cancer: an active phenomenon mediated by epithelial cells with mesenchymal characteristics. BMC Cancer 14, 286. https://doi.org/10.1186/1471-2407-14-286

Experiment Proposal

Experiment number GP2024145

Principal investigator	Professor Davide Lenaz, Università di Trieste, ITALY
Co-investigator (*)	Professor Francesco Di Benedetto, Università degli Studi di Ferrara, ITALY
Co-investigator	Dr Giovanni Orazio Lepore, Università di Firenze, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Thallium and As in sphalerite from Raibl mine (NE Italy): Nano-inclusions or Zn-vicariant?
MRF Instrument	**FIB-SEM GAIA 3**
Days requested:	3
Access Route	Direct Access
Previous GP Number:	No
Science Areas	Environment
DOI:	-
Sponsored Grant	None
Sponsor:	-
Grant Title	-
Grant Number:	-
Start Date	-
Finish Date:	-
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

The Pb-Zn ore deposits of the Raibl mine (Cave del Predil, UD) are characterised by high amount of Tl and As. These elements are toxic and behave differently in the tailings and waters. The aim of this proposal is to determine whether As and Tl are related to some submicro-inclusions within the sphalerite or are dispersed in the structure as vicariant in a double substitution for Zn and/or, in the case of As, S. This could be very important in understanding: 1) the mechanism of enrichment of these two elements during crystallization, 2) how sphalerite weathers and Tl and As become available in the environment. For this task, we plan to perform detailed transmission electron microscopy (TEM) measurements in order to detect the presence of any potential (As,Tl)-bearing nano-inclusions and, eventually, investigate their structural features. This knowledge will have deep consequence in the understanding of the potential environmental anomaly.

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Professor Francesco Di Benedetto, Università degli Studi di Ferrara, ITALY
MRF Instrument	**FIB-SEM GAIA 3** **Days Requested:** 3
Special requirements:	

SAMPLE

Material	Sphalerite thin sections	-	-
Formula	ZnS	-	-
Forms	Solid		
Volume	cc		
Weight	mg		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	Sample Changer	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	No	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	No	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Removed By User	-	-

Thallium and As in sphalerite from Raibl mine (NE Italy): Nano-inclusions or Zn-vicariant?

1. Background and Context

Sphalerite (ZnS), the most abundant Zn-bearing mineral, incorporates a broad range of minor and trace elements. Some of them, such as Ga, Ge, and In, are considered Critical Raw Materials [1] while others are considered as Potentially Toxic Elements such as As, Cd, Hg, Pg and Tl. The Pb-Zn Raibl mine is a dismissed mine located in Cave del Predil (NE Italy) presenting an average ore grade of 5% Zn and 1% Pb. Ongoing analysis on sphalerite crystals shows the presence of considerable amount of Cd, As and other metals such as Fe, Tl, Ge, Sb. Previous research has pointed out that the trace element distribution in sphalerite is influenced by various geological factors, including temperature, pressure, oxygen fugacity, sulfur fugacity, pH, and redox conditions [3-7]. Among the others, [3] discussed the possible mechanism of substitutions in detail recognizing five major element groups: 1) Cu, Sn, and In; 2) Fe, Mn, and Cd; 3) Ag, Sb, As, (Ga), and Pb; 4) Co; and 5) Ge. In some cases, the presence of these elements is actually ascribed to micro- to nano-inclusions, but sometimes they can be dispersed in the structure. [8] proposed an interesting multivariate statistical analysis by using 1336 analyses from 52 different deposits confirming the above major element groups but Tl was not considered. However, in the same study it is pointed out that Tl concentration is usually below 2 ppm in sphalerite apart for some Cretaceous veins in the district of Freiberg (Germany [9]), originated by mixing of highly saline and brines at low temperatures, where it ranges between 1 and 1000 ppm. Arsenic is much more abundant with different types of deposits showing values between 10 and 1000 ppm. The aim of this proposal is to determine whether As and Tl are related to some submicro-inclusions within the sphalerite or are dispersed in the structure as vicariant in a double substitution for Zn and/or, in the case of As, S. This could be very important in understanding: 1) the mechanism of enrichment of these two elements during crystallization, 2) how sphalerite weathers and Tl and As become available in the environment. For this task, we plan to perform detailed transmission electron microscopy (TEM) measurements in order to detect the presence of any potential (As,Tl)-bearing nano-inclusions and, eventually, investigate their structural features. The TEM results will be compared with an X-ray Absorption Spectroscopy study focused on the local structural environment and oxidation state of both As and Tl. TEM investigations will then allow to determine if Tl and As are hosted in the sphalerite crystal structure or in micro- to nanoparticles (NPs) of other Tl-bearing phases. The results achieved will thus shed light on the geochemical trap for two heavily toxic elements, such as As and Tl, which are found in the Reibl ore in concentration absolutely unusual for the mineralogical context. This knowledge will have deep consequence in the understanding of the potential environmental anomaly that is generated by the leaching of such ores, and to promote the most appropriate environmental prevention and remediation actions.

2. Proposed experiment

We ask the access to a complete microanalytical facility as that represented by the combined use, in sequence, of FIB-SEM and HRTEM, to unravel which one, among the proposed crystal chemical solutions, is able to properly describe the unusual Tl-As association in Raibl sphalerite. We propose, in particular, to explore the modes of the Tl-As association through a detailed co-localisation study carried out by X-ray/BSE mapping at the higher detail achievable, using a FEG-SEM instrumentation and compare it with our previous LA-ICP-MS data. Then, once having sorted out the best region to study in detail the association between the two elements, we plan to create a thin lamella, able to be studied by HRTEM. In this way, coupling X-ray analysis over very detailed regions of the lamella in the TEM imaging with electron diffraction and finally with HRTEM imaging, we are confident to be able to identify the occurrence of additional Tl-As nano-inclusions, or to verify structural defects in the sphalerite lattice able to host them. This task can be solved only by an appropriate study, which will be fully achievable only through the ISIS@MACH ITALIA facility.

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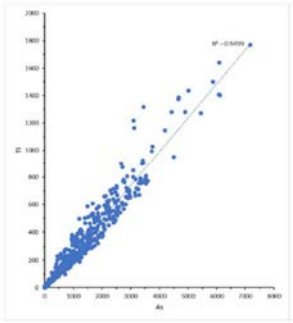
3. Summary of previous experimental proposals or characterisation



In Raibl sphalerite, the concentrations of As and Tl, determined by LA-ICP-MS at ETH-Zurich, are in the range 1-7200 ppm (avg. 1045±1086) and 0.2-1770 (avg. 255±277), respectively, being among the highest worldwide and showing a positive linear correlation with R^2=0.95 (Fig. 1a). Moreover, [2] showed that As and Sb were almost entirely released into the slightly alkaline waters of the mine drainage system, while modelling results infer Tl as being even more mobile, reaching concentrations up to 120 µg/L in waters flowing in the tailings impoundments. SEM investigations did not show evidence for the presence of As/Tl-bearing inclusions at the micro-scale.

4. Justification of experimental time requested

For the proposed experiment we ask access to the FIB-SEM GAIA 3 and Thermo Scientific Talos F200X STEM, both available at ISIS@MACH ITALIA. For completing the proposed task we ask for three days of experimental time.

5. References

1. EU Commission 2020 Study on the EU's list of Critical Raw Materials (final report). European Commission, Brussels, pp 1–158. 2. Barago N. et al. 2023 J. Geochem. Expl., 245, 107129, DOI 10.1016/j.gexplo.2022.107129. 3. Cook, N.J., et al. 2009 Geochim. Cosmochim. Acta 73, 4761–4791, DOI 10.1016/j.gca.2009.05.045. 4. Pfaff, K., et al. 2011. Chem. Geol. 286, 118–134, DOI 10.1016/J.chemgeo.2011.04.018. 5. Keith, M., et al. 2014. Geology 42, 699–702, DOI 10.1130/G35655.1. 6. George, L.L., et al. 2016. Ore Geol. Rev. 77, 97–116, DOI 10.1016/j.oregeorev.2016.02.009. 7. Frenzel, M., et al., 2020. Geology 48, 766–770, DOI 10.1130/G47087.1. 8. Zhang J., et al. 2022 J. Geochem. Expl. 232, 106883, DOI 10.1016/j.gexplo.2021.106883. 9. Bauer, M.E., et al. 2019. Mineral. Deposita 54, 237–262, DOI 10.1007/s00126-017-0773-1.

FT-IR Nexus

Experiment Proposal

Experiment number GP2024047

Principal investigator (*) Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator

Experiment title	Investigating the Corrosive Impact of Chrome-Tanned Semi aniline Calf Leather using FT-IR Spectroscopy		
MRF Instrument	**FT-IR Nexus**	**Days requested:**	1
Access Route	Direct Access	**Previous GP Number:**	NO
Science Areas	Materials	**DOI:**	-
Sponsored Grant	None	**Sponsor:**	-
Grant Title	-	**Grant Number:**	-
Start Date	-	**Finish Date:**	-
Similar Submission?	-		
Industrial Links	LBS LUXURY BRANDS SERVICES SRL		
Non-Technical Abstract	The aim of this study is to systematically investigate the composition of semi aniline chrome-tanned calf leather and to identify correlations between adsorbed substances on the leather surface and its aggressiveness towards metal accessories. By understanding these relationships, we can develop better methods for predicting and mitigating corrosion, thereby improving the quality and durability of leather products in the fashion and luxury industries. Understanding the chemical composition of semi aniline chrome-tanned calf leather will lead to significant improvements in product quality and in particular this research will provide a scientific basis for better quality control practices and contribute to the development of more robust and durable fashion items.		
Publications	-		

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY
MRF Instrument **FT-IR Nexus** **Days Requested:** 1
Special requirements:

SAMPLE

Material	Chrome-Tanned Semi aniline Calf Leather	-	-
Formula	Chrome-Tanned Semi aniline Calf Leather	-	-
Forms	Solid		
Volume	4 cc		
Weight	10 g		
Container or substrate	No special need	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	RT - K	-	-
Pressure Range	Atmospheric pressure - mbar	-	-
Magnetic field range	No - T	-	-
Standard equipment	-	-	-
Special equipment	No special equipment needed	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	No	-	-
Special equip. reqs	No	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	No	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Investigating the Corrosive Impact of Chrome-Tanned Semi aniline Calf Leather

1. Background and context

Semi aniline chrome-tanned calf leather is a highly valued material in the fashion and luxury industries, commonly used for products such as handbags, belts, and footwear. Ensuring the quality and longevity of these products is crucial, particularly in preventing corrosive processes that can damage metallic accessories like buckles, zippers, and decorative elements. The leather can release tanning substances that may react with metals, leading to corrosion and tarnishing, which compromises the aesthetic and functional integrity of the final products.

To assess the corrosive potential of leathers, quality control laboratories typically perform simulated corrosion tests using a reference sample. The extent of oxidation on the sample, after exposure to the leather, is evaluated and the leather is classified on a scale of aggressiveness from 1 to 5 (1 = highly aggressive, 5 = non-aggressive). However, beyond this empirical test, there has been no systematic study to investigate the underlying causes of oxidation and the specific substances responsible for the corrosive effects on metal accessories.

This study is expected to reveal specific chemical components in the leather that are responsible for initiating or accelerating corrosion in metal accessories. By identifying these substances, the fashion and luxury industries can take proactive measures to treat or modify leather to reduce its corrosive impact, thereby enhancing the durability and quality of leather products.

2. Proposed experiment

This proposal is part of a broader study to investigate the underlying causes of oxidation and the specific substances responsible for the corrosive effects on metal accessories by Semi aniline chrome-tanned calf leather. The study involves several instrumental techniques: a) FT-IR Spectroscopy; b) RAMAN Spectroscopy and profilometry; c) Tomography; d) Ion Scattering Spectroscopy.

FT-IR Spectroscopy is a pivotal tool for this experiment due to its unique capabilities.

The Nicolet Nexus 870 FT-IR spectrometer offers high-resolution infrared spectra that can identify and quantify the functional groups and molecular structures present in the leather. This information is critical for understanding the potential corrosive agents on the leather surface and their interactions with metals.

In particular, the instrument will provide the following crucial information:

1. Functional Group Identification: FT-IR spectroscopy is highly effective in detecting specific functional groups such as carbonyls, hydroxyls, and amines. These functional groups are key indicators of the presence of tanning agents, fats, oils, and other substances used in the leather manufacturing process. By identifying these groups, we can gain insights into the chemical composition of the leather and the substances that may contribute to corrosion.

2. Molecular Structure Analysis: FT-IR provides detailed information about the molecular structure of the leather components. This includes understanding the chemical bonds and interactions between different molecules on the leather surface. Such structural insights are crucial for assessing how the leather's chemical makeup might influence its reactivity with metallic accessories, potentially leading to corrosion.

3. Quantitative Analysis: FT-IR spectroscopy allows for the quantification of the relative concentrations of different functional groups present in the leather. This quantitative data helps in understanding the abundance of specific substances and their potential impact on corrosion processes. Knowing the concentration levels of various chemicals can guide us in identifying which components are most likely to cause issues with metal accessories.

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By employing the Nicolet Nexus 870 FT-IR spectrometer in Attenuated Total Reflection (ATR) mode, we can therefore obtain comprehensive data on the chemical and structural properties of semi aniline chrome-tanned calf leather. This data is essential for understanding and mitigating the corrosive effects that these leathers can have on metallic accessories, ultimately leading to improved quality and durability of leather products in the fashion and luxury industries.

3. Summary of previous experimental proposals or characterisation

Historically, the understanding of the aggressiveness of leather toward metal accessories has been constrained by a reliance on empirical methods and trial and error. This approach lacks the detailed insight and predictive capability that sophisticated analytical tools like FT-IR can provide. In particular, access to such advanced analysis and the requisite expertise is often limited. Although some literature provides an understanding of surface composition and surface functional groups [1-2], a detailed exploration of these elements' interplay, especially regarding the correlation with aggressiveness and corrosion properties toward metal accessories, is notably absent. This research aims to fill that critical knowledge gap in order to provide a correlation between surface functional groups responsible for potential interactions with metallic elements, which can lead to corrosion.

4. Justification of experimental time requested

As detailed in section 2, FT-IR Spectroscopy is a pivotal tool for this experiment due to its unique capabilities. We request 1 day of experimental time to analyse 15 samples coming from 5 leather batches: we collected 5 types of batches of semi aniline chrome-tanned calf leather with high level of aggressiveness (level 1 to level 2, with reference to the empirical scale, where 1 = highly aggressive, 5 = non-aggressive as determined by the currently used corrosion test as detailed in section 1. For each batch we will then collect three samples for investigating the spatial homogeneity of the composition.

This number ensures diverse representation from different batches of treatment conditions. The day will be dedicated to a screening on all the five batches in order to highlight the main differences in the spectra and then a fine screening of the samples for each batch provided. Finally, data analysis and comparison will be conducted. This structured approach allows for thorough examination while maintaining a strict timeline.

[1] Gendaszewska, D.; Pipiak, P.; Wieczorek, D.; Sieczyńska, K. Experimental Study on Chrome Tanned Leather Shavings Modification—Properties and Prospective for Future Application. Processes 2024, 12, 228.

[2] E.H.A. Nashy, O. Osman, A.A. Mahmoud, M. Ibrahim, Molecular spectroscopic study for suggested mechanism of chrome tanned leather, Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy, Volume 88, 2012, Pages 171-176.

Experiment Proposal

Experiment number GP2024084

Principal investigator	Professor Gabriele Gentile, University of Rome Tor Vergata, ITALY
Co-investigator (*)	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Carla Andreani, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	In-depth analysis of ancient DNA from sub-fossil bones of pink iguana from Galápagos islands using FT-IR spectroscopy

MRF Instrument	**FT-IR Nexus**	**Days requested:**	3
Access Route	Direct Access	**Previous GP Number:**	No
Science Areas	Biology and Bio-materials, Physics	**DOI:**	-
Sponsored Grant	None	**Sponsor:**	-
Grant Title	-	**Grant Number:**	-
Start Date	-	**Finish Date:**	-
Similar Submission?	-		
Industrial Links	-		

Non-Technical Abstract

Our aim is to study the origin of the pink iguana which is unknown at present from a multi-instrumental characterization of sub-fossil bones of terrestrial iguanas (Conolophus), rarely preserved and found in lava tubes of a few Galapagos islands like Santa Cruz, Isabela, Rabida, and Santiago. Current genetic and paleogeographic data would indicate that the species originated from an ancestor, now extinct, that lived on an island other than the only one (Isabela) where the pink iguana exists at present. As such, we would like to measure the presence of ancient DNA on these samples by sequencing the DNA. Data will be cross compared to verify consistency and information completed by measuring the presence of organic compounds by FT-IR data that will be collected at the FT-IR Nexus instrument available at the CSGI - University of Florence. Hence, we aim here to request access to the FT-IR Nexus instrument available at the CSGI - University of Florence Unit of IM@IT.

Publications

G. Gentile et al., Zootaxa (2009), 2201: 1-10.
G. Gentile et al., Problematic Wildlife, a cross-disciplinary approach. Angelici F. (Ed). Springer, pp.315-336 (2016).
G. Gentile et al., Proceedings of the National Academy of Sciences of the United States of America, 106 (2009), 507–511.

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY		
MRF Instrument	**FT-IR Nexus**	**Days Requested:**	3
Special requirements:			

SAMPLE

Material	Sub-fossil Conolophus bones	-	-
Formula	-	-	-
Forms	Solid		
Volume	0.07-0.37 cc		
Weight	100-500 mg		
Container or substrate	Sterile tube or aluminum foil	-	-
Storage Requirements	Freezer (-20C)	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	-	-

In-depth analysis of ancient DNA from sub-fossil bones of pink iguana from Galápagos islands using FT-IR spectroscopy

1. Background and Context

Visited by a young Charles Darwin, the Galápagos islands were crucial locations for the development of the evolutionary thinking. Iguanas are among the most representative reptiles of the archipelago, where four endemic species exist (Fig1), belonging to two genera: *Amblyrhynchus cristatus* (marine iguana), *Conolophus subcristatus*, *C. pallidus* and *C. marthae* (land iguanas). The last - the pink iguana - was recently discovered and described as a new species by the proponent in 2009 [1].

Fig. 1 The four named species of Galápagos iguanas. All of them are endemic to the archipelago.

The Critically Endangered [2] pink iguana rapidly became a flagship species, recognized worldwide as an iconic species capable of attracting general attention toward Biodiversity conservation [3].
With its coloration and unique ecological, physiological, and behavioral traits [4-5], the pink iguana is a source of evolutionary questions, especially related to its origin. Current genetic and paleogeographic data would indicate that the species originated from an ancestor, now extinct, that lived on an island other than the only one (Isabela) where the pink iguana exists at present. In fact, at the time the ancestor lived, Isabela had not emerged yet [6-7]. Indeed, the island where the pink iguana ancestor lived remains unknown at present. The only evidence of past presence of a lineage related to the pink iguana could be provided from sub-fossil bones of terrestrial iguanas (*Conolophus*), rarely preserved and found in lava tubes of a few islands (Santa Cruz, Isabela, Rabida, Santiago). Unfortunately, a correct species assignment cannot be done solely on the base of the morphology of a few bones. This calls for an in-depth analysis of ancient DNA (aDNA) extracted from sub-fossil bones and by means of Fourier-transform infrared spectroscopy (FT-IR) measurement for assessing the presence of organic compounds to complete the characterization. In fact, DNA retains much information about the evolutionary and genealogic relationships between species, allowing also the reconstruction of past demographic dynamics. The correct dating of those bones would provide a precise time frame for pinpointing the evolutionary events of diversification and clarify the dynamics of colonization of the archipelago.
In the frame of an international collaboration with the Natural History Museum of Florida and the Galápagos National Park Directorate, we obtained several specimens of sub-fossil *Conolophus* bones, collected in lava tubes. We intend to process 10 of them, dating precisely their age. Secondly, we will perform shotgun sequencing of aDNA and FT-IR spectroscopy measurements using the DNA Sequencing NGS and the FT-IR Nexus instruments available at the Rome Tor Vergata and CSGI - University of Florence Units, respectively. The proposal will also take advantage from the genomic reference database produced by the Consortium for Iguana Genomes (CIG), funded by several sources, coordinated by the PI and participated by the University of Leeds (UK), the Genome Center and the University Kebangsaan (Malaysia). PhD students, co-advised by CIG partners produced reference genomes for several species of iguana, including Galápagos' ones. The proposed project will provide data for at least 2 PhD students.

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2. Proposed experiment

In this experiment we aim to perform the characterization on 10 samples coming from sub-fossil *Conolophus* bones, collected in lava tubes of a few Galápagos islands (Santa Cruz, Isabela, Rabida, Santiago) of organic compounds by Fourier-transform infrared spectroscopy measurement using the FT-IR Nexus instrument available at the CSGI - University of Florence Unit. Furthermore, findings of these measurements will complete information and verify consistency of shotgun sequencing of aDNA using the DNA Sequencing NGS instrument at the Rome Tor Vergata Unit used to study the genomic of aDNA.

3. Justification of experimental time requested

Each of the n. 10 sample will be enclosed in a sterile tube with about 100-500 mg of sub-fossil bone properly treated, and it will be maintained at -20 °C temperature to preserve aDNA during the measurements. We envisage, after discussion with the instrument scientist, to measure n. 4 samples per day on the instrument. Hence, we request a total of 3 days of instrument time including set-up and calibration time.

References

[1] G. Gentile et al., Zootaxa (2009), 2201: 1-10.
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Experiment Proposal

Experiment number GP2024101

Principal investigator (*) Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator

Experiment title	Stripping of surface treatments from ABS/metal composites: an FT-IR investigation		
MRF Instrument	**FT-IR Nexus**	**Days requested:**	1
Access Route	Direct Access	**Previous GP Number:**	NO
Science Areas	Materials	**DOI:**	-
Sponsored Grant	None	**Sponsor:**	-
Grant Title	-	**Grant Number:**	-
Start Date	-	**Finish Date:**	-
Similar Submission?	-		
Industrial Links	RGF SRL		

Non-Technical Abstract

In fashion and luxury markets, ABS (Acrylonitrile Butadiene Styrene) components are chosen for their lightweight and cost-effective properties. These components undergo a galvanization process to achieve a metal-like appearance, such as that of brass. This approach provides the aesthetic qualities of metal accessories at a reduced cost and weight, which is particularly important for large decorative elements on shoes or bags. If made from brass, these elements could be too heavy and potentially damage the final product. With increasing emphasis on sustainability, the industry is focused on recycling defective or scrap products to support circular production. The challenge is to effectively separate the metal coatings from ABS and recover both materials, ensuring minimal impact on the environment while maintaining product quality. By conducting this study, we will obtain essential data for understanding the surface modifications occurring on the ABS after removal of the plated layers.

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY
MRF Instrument **FT-IR Nexus** **Days Requested:** 1
Special requirements:

SAMPLE

Material	ABS	-	-
Formula	(C8H8·C4H6·C3H3N)n	-	-
Forms	Solid		
Volume	4 cc		
Weight	10 g		
Container or substrate	No special need	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	RT - K	-	-
Pressure Range	Atmospheric pressure - mbar	-	-
Magnetic field range	No - T	-	-
Standard equipment	-	-	-
Special equipment	No special equipment needed	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	No	-	-
Special equip. reqs	No	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	No	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Stripping of surface treatments from ABS/metal composites: an FT-IR investigation

1. Background and context

In the fashion and luxury markets, Acrylonitrile Butadiene Styrene (ABS) components are sometimes used due to their lightweight properties. These components undergo a specific electroplating process to allow for plastic galvanization. The resulting accessories are aesthetically identical to their counterparts made from base metals, such as brass, but have the advantages of being much more cost-effective and lighter. This is particularly important for large decorative elements on shoes or bags, which would be too heavy if made from brass, potentially damaging the final product.
Given the increasing focus on sustainability, manufacturing industries have been striving to recover and recycle as many defective or scrap products as possible to promote production circularity. For accessories made from such diverse materials as ABS and electroplated metal layers, developing an effective method to separate the metals from the ABS substrate and recover both materials are a challenge [1, 2].
The goal of this analysis is to verify the effectiveness of removing various electroplated layers from ABS and to characterize the resulting "bare" ABS to understand how the stripping process has affected it.

2. Proposed experiment

Until now, the production of accessories for leather goods has primarily focused on items made from base metals, like brass. For the recovery of various metal components, conventional methods of metals stripping and refining have therefore been employed. Recently, there has been an increase in the use of base materials such as ABS in accessory manufacturing. This shift offers significant advantages in terms of cost reduction and lighter weight for accessories. However, it also introduces challenges related to recycling: accessories made with ABS require a specialized process to remove the metal layers without damaging the underlying ABS, ensuring it remains suitable for recycling.

For this study, we will use a combination of two techniques: 1) Fourier Transform Infrared Spectroscopy (FT-IR) and 2) Scanning Electron Microscopy (SEM).

FT-IR will allow to obtain comprehensive data on the chemical properties of ABS [3,4] and compare the findings on its structure before and after the stripping process. In fact, the Nicolet Nexus 870 FT-IR is a powerful instrument for the characterization of polymers such as ABS, offering high-resolution infrared spectra that can identify and quantify the relative ratio of the typical functional groups present in the ABS: acrylonitrile has the nitrile group showing a characteristic peak around 2240-2260 cm^{-1}; butadiene can be identified by the peaks around 965 and 911 cm^{-1} (C-H out-of-plane vibrations) and styrene shows the peaks of the benzyl group around 700-750 cm^{-1} (C-H out-of-plane bending) and 1450-1600 cm^{-1} (aromatic ring vibrations).

In summary, FT-IR is crucial in this experiment for its features and advantages for ABS characterisation: a) Speed: the measurement is quick and time effective; b) Specificity: it specifically identifies the functional groups present in the material; c) non-destructive technique, preserving sample integrity for analysis with multiple techniques.

By conducting these analyses, we will obtain essential data for understanding the surface modifications on the ABS composition after the removal of the electroplated layers to understand if the resulting stripped ABS maintains its features and therefore ensuring recycling of all the components.

3. Summary of previous experimental proposals or characterisation

The use of ABS as base material for fashion accessories introduces new challenges, particularly concerning recycling. Accessories made from ABS must undergo a specialized treatment process to effectively remove the metal layers without damaging the ABS substrate. This is crucial to ensure that the ABS remains suitable for recycling. Currently, there is a lack of comprehensive data and studies that characterize these materials and their recycling processes. As such, this study aims to fill this gap by thoroughly analysing the stripping process and evaluating the suitability of the recovered ABS for recycling. By doing so, it seeks to provide valuable insights into the recycling of ABS-based accessories and contribute to more sustainable manufacturing practices.

4. Justification of experimental time requested

As detailed in section 2, FT-IR is a pivotal tool for this experiment due to its unique capabilities.

We request 1 day of experimental time to analyse 5 samples, analysing 3 points of interest for each sample. We will compare the structure of ABS in the samples before the stripping procedure (on cross-cutted plated ABS samples) and after the stripping steps (on the bare ABS left after the stripping process), in order to analyse the modification to its composition caused by the removal procedure. As agreed with the instrument scientist, the day will be dedicated to a screening on all the samples to highlight the main differences in the spectra, find an internal standard to relatively quantify the ratio between the ABS constituents and then fine screen all the samples provided to check the homogeneity. Finally, data analysis and comparison will be conducted. This structured approach allows for efficient use of the instrument while maintaining a strict timeline, providing detailed insights into the characterisation of the ABS before and after the stripping procedure giving insights in the possibility for the recovered ABS to be reused and recyclable.

[1] J.S. Seo et al. Peeling mechanism of interlocked interface between etched acrylonitrile-butadiene-styrene and electroplated metal layer, Surfaces and Interfaces, 26, 2021, 101337.
[2] R. Tao et al.Contributions of chemical interactions and mechanical interlocking for the adhesion of electroplated copper to ABS in the Cr(VI) etching process, International Journal of Adhesion and Adhesives, 126, 2023, 103450.
[3] J.Wang et al. Surface treatment with Fenton for separation of acrylonitrile-butadiene-styrene and polyvinylchloride waste plastics by flotation, Waste Management, 67, 2017, 20-26.
[4] J. Wang et al. Separation of acrylonitrile-butadiene-styrene and polystyrene waste plastics after surface modification using potassium ferrate by froth flotation, Waste Management, 78, 2018, Pages 829-840

Experiment Proposal

Experiment number GP2024109

Principal investigator	Professor Gabriele Gentile, University of Rome Tor Vergata, ITALY
Co-investigator (*)	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Carla Andreani, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Multi-instrumental characterization of Oniscidean isopods to establish the nature and function of their cuticle and tubercles: FTIR measurements
MRF Instrument	**FT-IR Nexus** — **Days requested:** 3
Access Route	Direct Access — **Previous GP Number:** No
Science Areas	Biology and Bio-materials, Physics — **DOI:** -
Sponsored Grant	None — **Sponsor:** -
Grant Title	- — **Grant Number:** -
Start Date	- — **Finish Date:** -
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

Andronicus is a genus of oniscidean isopods that includes less than a dozen species, most of which are confined to caves in the Italian Pre-Alps. Andronicus offers the unique opportunity to investigate the nature of the cuticle in cave (A. brentanus) and non-cave-dwelling species (A. dentiger), within the same evolutionary lineage, characterizing mineralization in the different species. The proposed investigation will also extend to including tubercles on the surface of the body, the nature of which, currently unknown, could be of a sensorial nature. Preliminary observations would suggest the presence of a possible innervation at the base of these tubercles, which if confirmed, would prove their nature and function. For the characterization on the mineralization of the cuticle and tubercles we envisage to use EDX, FT-IR, Raman, and X-ray diffraction, whereas the morphology characterization will be done by SEM, TEM and nano-XCT. Here, this proposal is focussed on the FTIR analysis.

Publications

Vittori, M. et al., Arthropod Struct Dev. 46 (2016), pp. 96-107.
Gentile, G. and Allegrucci, G., International Journal of Speleology 26 (1997), pp. 47-61.
Neues, F. et al., Cryst. Eng. Comm. 9 (2007), pp. 1245-1251.

ISIS neutron and muon source — **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY		
MRF Instrument	**FT-IR Nexus**	**Days Requested:** 3	
Special requirements:			

SAMPLE

Material	Oniscidean isopod	-	-
Formula	Organic material, Calcite	-	-
Forms	Solid		
Volume	0.03 cc		
Weight	1-2 g		
Container or substrate	-	-	-
Storage Requirements	Freezer (-20C)	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	-	-

Multi-instrumental characterization of Oniscidean isopods to establish the nature and function of their cuticle and tubercles: FTIR measurements

1. Background and Context

Among Crustaceans, oniscidean isopods are uniquely adapted to terrestrial life, exhibiting strongly mineralized cuticles. Onisceideans include several species adapted to the caves. Among the most important evolutionary adaptations found in troglobitic onisceideans (i.e. bound to cave environments, from which they cannot escape due to strict ecological and physiological constraints) are the thinning of the cuticle with a reduce layer of calcite, although calcium carbonate is present in the exocuticle and the endocuticle [1]. Additionally, other adaptations include the lengthening of the appendages, the loss of the eyes, the development of sensory systems alternative to sight such as hygrosensors and chemosensors, usually located in different areas of the body. *Androniscus* (Fig. 1) is a genus of oniscidean isopods that includes less than a dozen species, most of which are confined to caves in the Italian Pre-Alps. Among these, *Androniscus dentiger* is the one that shows the least constraints, being present even in the most superficial layers of the soil in non-cave environments and showing a wide geographical distribution [2]. Indeed, by combining atomic absorption spectroscopy, thermogravimetry and X-ray diffraction, the composition of cuticles in several isopods has been analyzed [3-4]. The use of high-resolution Raman microscopy enabled the determination of the distribution of different mineral phases in the tergal cuticles of some rollers, clingers, and runners [5,6].

Fig. 1 *Androniscus dentiger* (a) and *Androniscus brentanus* (b). Contrary to the second, the first is not troglobite, is pigmented, has thick cuticle, and shows a prominent single-ommatidium eye (arrow).

Androniscus offers the unique opportunity to investigate the nature of the cuticle in cave (*A. brentanus* and more) and non-cave-dwelling species (*A. dentiger*), within the same evolutionary lineage, characterizing mineralization in the different species. The proposed investigation will also extend to including tubercles (tricorns) on the surface of the body, the nature of which, currently unknown, could be of a sensorial nature. Some preliminary observations would suggest the presence of a possible innervation at the base of these tubercles, which if confirmed, would prove their nature and function. For the characterization on the mineralization of the cuticle and tubercles in the two different species (*A. brentanus* and *A. dentiger*) we plan to use Energy Dispersive X-ray Analysis (SEM-EDX), Fourier-transform infrared spectroscopy (FT-IR), Raman spectroscopy and X-ray diffraction, whereas the morphology characterization will be done by means of electron microscopy techniques (SEM, TEM) and X-ray nano tomography. The benefit of using a multi-instrumental approach would allow us not only to cross compared results to verify their consistency, but also to investigate the degree of resorption/failure to develop the eye in these isopods species, allowing us to observe the presence of vestigial or residual structures, such as for example the presence of an optic nerve, in the absence of the ommatidium (eyeball).

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2. Proposed experiment

In this specific proposal we aim to use the FT-IR Nexus instrument available at the CSGI - University of Florence Unit for assessing the degree of mineralization of the cuticle and tubercles on a n. 6 *Androniscus brentanus and n. 6 Androniscus dentiger* isopods samples. Results of this experiment will be cross compared to verify consistency with data obtained by separate proposals where we request XRD, Raman, SEM-EDX, TEM, and nano XCT measurements on the same set of samples.

3. Justification of experimental time requested

Each of the n. 12 samples of the two Oniscidean isopod species (n. 6 *Androniscus brentanus and n.6 Androniscus dentiger)* will be washed for 1–2 s in double distilled water to remove tissue saline at the surface and then for 2–5 s in 100% methanol to remove water. Specimens will be air dried and stored at −20 °C until its use on the instrument. For the FT-IR measurement, cuticle and tubercles will be isolated from the sample and reduce in powder form by grinding. Results will be compared with pure calcite and amorphous calcium carbonate reference samples. We envisage, after discussion with the instrument scientist, to measure n. 4 samples per day on the instrument. Hence, we request a total of 3 days of instrument time including sample preparation, set-up and calibration time.

References

[1] Vittori, M., Tusek-Žnidarič, M. & Štrus, J. (2016) Exoskeletal cuticle of cavernicolous and epigean terrestrial isopods: a review and perspectives. Arthropod Struct Dev. 46(1): 96-107.
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[3] Neues, F., Ziegler, A. & Epple, M. (2007) The composition of the mineralized cuticle in marine and terrestrial isopods: a comparative study Cryst. Eng. Comm. 9: 1245-1251.
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[6] Štrus, J., Žnidaršič, N., Hild, S. & Ziegler, A. (2008) Microscopic anatomy and mineral composition of cuticle in amphibious isopods Ligia italica and Titanethes albus (Crustacea: Isopoda) A. Aretz, B. Hermanns-Sachweh, J. Mayer (Eds.), EMC 2008: Life Sciences, Springer Verlag, Berlin:185-186.
[7] Hornung, E. (2011). Evolutionary adaptation of oniscidean isopods to terrestrial life: Structural-physiological-behavioural aspects. Terrestrial Arthropod Reviews. 4: 95-130.

Experiment Proposal

Experiment number GP2024157

Principal investigator	Dr Monica Carosi, Università Roma Tre, ITALY
Co-investigator	Dr Federica Spani, Università Campus Bio-Medico di Roma, ITALY
Co-investigator (*)	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Multi-instrumental characterization of primate baculum and baubellum bone tissues to support functional hypotheses: FTIR measurement case
MRF Instrument	**FT-IR Nexus** — **Days requested:** 1
Access Route	Direct Access — **Previous GP Number:** No
Science Areas	Biology and Bio-materials, Physics — **DOI:** -
Sponsored Grant	None — **Sponsor:** -
Grant Title	- — **Grant Number:** -
Start Date	- — **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	To better understand the function of primate bacula and baubella bone tissues, we aim to study the micro-architecture of the bone focussed for the first time on characteristics related to either observed shapes and physical-chemical features of this tissue. Key aspects include trabecular density and orientation, bone mineral composition, and the distribution of minerals along the bones. The proposed study will involve and multi-instrumental characterization of different bones which will allow us to fine reconstruct a digital twin of these tissues, and for open exploring image-based finite element analysis to assess the mechanical forces involved during copulation. Here, this proposal is focussed on the FTIR analysis.
Publications	Spani F, Morigi MP, Bettuzzi M, Scalici M, Carosi M, PLoS ONE 15(1): e0228131. Spani, F., Morigi, M., Bettuzzi, M. et al., Sci Rep 11 (2021), 11245. Reznikov, N., Bilton, M., Lari, L., Stevens, M. M. & Kröger,Science 360 (2018), eaao2189.

ISIS neutron and muon source — **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
MRF Instrument	**FT-IR Nexus** — **Days Requested:** 1
Special requirements:	

SAMPLE

Material	Primate bone tissue	-	-
Formula	Ca10(PO4)6(OH)2	-	-
Forms	Solid		
Volume	0.3 cc		
Weight	0.5 g		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	-	-

Multi-instrumental characterization of primate baculum and baubellum bone tissues to support functional hypotheses: FTIR measurement case

1. Background and Context

Inside the external genitals of some placental mammals, including Primates, genital bones are present in one or both sexes: the *baculum* (penile bone; pl. *bacula*) and the *baubellum* (clitoral bone; pl. *baubella*). *Bacula* are common in most primate species, whereas *baubella* are rare. Both bones occur in some species of Hominoidea (the human evolutionary lineage), but not in humans. Although homologous, *baubellum* is only present in species where males have a *baculum,* whereas species with *bacula* may lack *baubella*. Various functions have been proposed for the *baculum* (none for the *baubellum*), however only one is supported by correlational data: baculum supports erection and prevents urethral collapse, aiding sperm transport in species with prolonged copulations and high levels of sexual competition. *In fact, baculum* length positively correlates with copulation duration. Recent studies published the most comprehensive dataset on primate *bacula* and *baubella* occurrence, collecting data from primary literature and samples from fresh cadavers and museum specimens (Natural History Museum La Specola in Italy, the American Museum of Natural History in New York, and the National Museum of Natural History in Washington, DC). Using 3D high-resolution, non-invasive micro-Computed Tomography and a new landmark-free shape analysis (the *alpha*-shape technique), these studies identified three distinct internal and external morphologies in primate *bacula* for the first time.

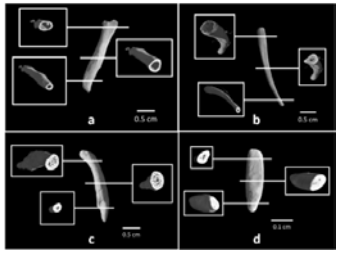

Fig. 1 *3D virtual volumes of 4 different types of primate genital bones, three bacula and one baubellum. For each type, internal structures and cross sections of epiphyses and diaphysis are shown. A: totally hollow structure (Chlorocebus aethiops). B: hollow epiphyses and solid diaphysis with few channels (Otolemur crassicaudatus). C: totally solid structure in both epiphyses and diaphysis with a network of Haversian channels (Papio cynocephalus). D: totally solid structure of baubellum (Sapajus apella) with some Haversian channels*

To better understand the function of primate bacula, the micro-architecture of baculum bone tissue should be investigated focusing for the first time on characteristics related to either observed shapes and mechanical forces exerted on bacula during copulation. Key aspects include trabecular density and orientation, bone mineral composition, and the distribution of minerals along the bones. Potential sex-based differences in these characteristics will aid in interpreting the results. Hence, the proposed study will involve and multi-instrumental approach as follows:

- trabecular density will be assessed by means of quantitative computed tomography (QCT) at different length scale and photon energy which will enable us reconstructing the 3-D bone geometry and volumetric bone mineral density (vBMD);
- trabecular orientation of the bone tissue will be studied in the bulk of the sample by small-angle X-ray scattering (SAXS) to measure crystal shape, their average crystal thickness and their crystal orientation;

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- the structure of bone mineral will be assessed by means of X-ray diffraction (XRD) which is considered as the gold standard for this type of measurement;
- for the compositional characterization of the bone tissue we aim to use nuclear magnetic resonance (NMR) which is uses the responses of isotopes to an external magnetic field to generate compositional information about the sample being scanned, and results will be verified for consistency checks with Raman spectroscopy, Fourier transform infrared spectroscopy (FTIR), X-ray Fluorescence (XRF) spectroscopy, and Energy Dispersive X-ray Analysis (SEM-EDX) data that are used here to determine the chemical and molecular signature of the sample.

2. Proposed experiment

In this specific proposal we aim to use the FT-IR Nexus instrument available at the CSGI - University of Florence Unit to perform Fourier transform infrared spectroscopy on n. 5 samples (n. 3 baculum bone tissue and n. 2 baubella bone tissue) to measure the characteristic frequencies absorbed by the molecular bonds of the bone tissue. The peaks in the resulting FTIR spectrum will be analysed to identify molecular species, qualitatively determine the amount of specific components, and provide information on the stoichiometry of a particular molecular species. In bone, FT-IR spectroscopy (and even Raman spectroscopy) can differentiate the molecular signals of the organic matrix components (collagen, proteoglycans, lipids, etc.) from the signals arising from the constituents of the hydroxyapatite (phosphate, carbonate). Hence, results of this experiment will be cross compared to verify consistency with Raman spectroscopy data measured on the same set of samples with the Raman Confocal Microscope instrument available at the CSGI - University of Florence Unit, and requested by means of a separate proposal.

3. Justification of experimental time requested

Each of the n. 5 samples will be measured in different region of interest of the sample to establish if any disuniformity is present in the material composition. Hence, after discussion with the instrument scientist, we request 1 days of instrument time including sample preparation, set-up and calibration time.

References

[1] Reznikov, N., Bilton, M., Lari, L., Stevens, M. M. & Kröger, R. Fractal-like hierarchical organization of bone begins at the nanoscale. Science 360, eaao2189 (2018).
[2] Glimcher, M. J. Bone: Nature of the Calcium Phosphate Crystals and Cellular, Structural, and Physical Chemical Mechanisms in Their Formation.*Rev. Mineral. Geochem.*64, 223–282 (2006).
[3] Delmas, P. D., Tracy, R. P., Riggs, B. L. & Mann, K. G. Identification of the noncollagenous proteins of bovine bone by two-dimensional gel electrophoresis. *Calcif. Tissue Int.***36**, 308–316 (1984).
[4] Schultz NG, Lough-Stevens M, Abreu E, Orr T, Dean MD. The Baculum was Gained and Lost Multiple Times during Mammalian Evolution. Integr Comp Biol. 2016 Oct;56(4):644-56. doi: 10.1093/icb/icw034. Epub 2016 Jun 1. PMID: 27252214; PMCID: PMC6080509.
[5] Spani, F., Morigi, M., Bettuzzi, M. et al. The ultimate database to (re)set the evolutionary history of primate genital bones. Sci Rep 11, 11245 (2021). https://doi.org/10.1038/s41598-021-90787-2

FT-IR Nicolet

Experiment Proposal

Experiment number GP2024040

Principal investigator	Professor Domenico Lo Vetro, Università di Firenze, ITALY
Co-investigator (*)	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Alessandro Cianchi, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Exploring environmental dynamics in ancient remains before and after the last glacial maximum using FT-IR measurements
MRF Instrument	**FT-IR Nicolet**
Days requested:	2
Access Route	Direct Access
Previous GP Number:	No
Science Areas	Biology and Bio-materials, Environment
DOI:	No
Sponsored Grant	None
Sponsor:	-
Grant Title	-
Grant Number:	-
Start Date	-
Finish Date:	-
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

Our aim is to study environmental ancient sediments coming from sediments extracted in the Romito cave (Cosenza, Italy) before and after the Last Glacial Maximum by multi-instrumental approach. The characterization of these samples to study organic compounds and the possible presence of bones will be perform by Fourier-transform infrared spectroscopy measurements which will be cross compared to verify consistency with data obtained by sequencing the aDNA using the DNA Sequencing NGS instrument at the Rome Tor Vergata Unit and by retrieving all the proteins in the sediments using the Mass Spectrometer 2 at the University of Milano Bicocca Unit. Hence, we aim here to request access to the FT-IR Nicolet instrument available at the Rome Tor Vergata Unit of IM@IT.

Publications

Berto et al. 2022. Archaeological and Anthropological Sciences. Vol. 14, article N. 127, (2022)
López-García et al. 2014. Palaeogeography, Palaeoclimatology, Palaeoecology, Vol 251, Issues 3–4, Pages 500-526
Scorrano et al. 2022. Communications Biology, Vol 5, Article N 1262 (2022)

Instruments	**INES**	**Days Requested:**	3
Access Route	Direct Access	**Previous RB Number:**	No
Science Areas		**DOI:**	No
Sponsored Grant	None	**Sponsor:**	
Grant Title	-	**Grant Number:**	
Start Date	-	**Finish Date:**	
Similar Submission?			
Industrial Links			

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Sample record sheet

Principal contact	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY		
MRF Instrument	**FT-IR Nicolet**	**Days Requested:**	2
Special requirements:			

SAMPLE

Material	Sediments/remains	Sediments/remains	Sediments/remains
Formula	organic (collagen) and sand/limestone mixtures	organic (collagen) and sand/limestone mixtures	organic (collagen) and sand/limestone mixtures
Forms	Solid	Solid	Solid
Volume	4-10 cc	4-10 cc	4-10 cc
Weight	2-10 g	2-10 g	2-10 g
Container or substrate	Sterile tube or aluminum foil	Sterile tube or aluminum foil	Sterile tube or aluminum foil
Storage Requirements	Freezer (-20C)	Freezer (-20C)	Freezer (-20C)

SAMPLE ENVIROMENT

Temperature Range	273 - 320 K	273 - 320 K	273 - 320 K
Pressure Range	1000 - 1010 mbar	1000 - 1010 mbar	1000 - 1010 mbar
Magnetic field range	- T	- T	- T
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	Yes	Yes
Sample Prep Hazards	No	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	No	No
Sensitivity to vapour	No	No	No
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	No	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	Returned to user by instrument scientist (when inactive)	Returned to user by instrument scientist (when inactive)

Exploring environmental dynamics in ancient remains before and after the last glacial maximum using FT-IR measurements

Background and Context

Ancient environmental DNA (aDNA) refers to genetic material obtained from environmental samples such as soil, sediment, ice and water, which is thousands or millions of years old [1]. The study of ancient DNA is fascinating because it allows us to reconstruct past ecosystems, understand evolutionary processes and trace the impacts of climate and environmental changes on biodiversity over the millennia. This field is particularly timely, as highlighted by a recent publication in Nature detailing the oldest DNA ever recovered from the environment: 2-million-year-old samples that allowed researchers to reconstruct the ecosystem in Greenland [1].

The broader relevance of ancient DNA research lies in its ability to shed light on the complex interactions between climate, environment and living organisms over geological time scales. By understanding past ecosystems, we gain insights into species resilience and vulnerability, which can guide current biodiversity conservation strategies. This proposal aims to characterize samples coming from sediments extracted in the Romito cave (Cosenza, Italy, see Figure 1) [2, 3] before and after the Last Glacial Maximum (LGM) by multi-instrumental approach. The reason of this is twofold. The LGM, when ice sheets were at their maximum extent, was a period of significant climatic and environmental shifts with profound impacts on global ecosystems and human populations. In an era marked by rapid climate change, insights from the LGM can inform our understanding of how ecosystems and species, including humans, responded to extreme climatic conditions. From the other, the Romito cave is one of the most significant Upper Palaeolithic archaeological sites on the Italian peninsula with a well-dated stratigraphy spanning from ~24,000 to 6,000 years before present (BP) (Figure 1d).

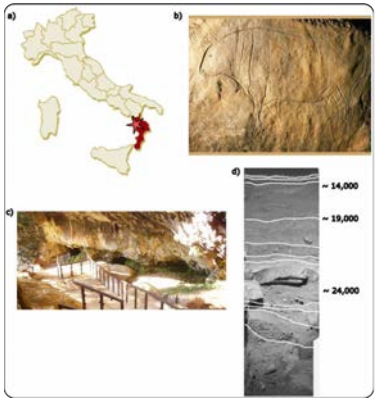

Figure 1. a) Location of Romito cave; b) rock art (Bos primigenius) in the rock-shelter outside the cave; c) cave entrance d) general stratigraphic sequence modified from Blockley et al. (2018).

To this end, we will study two samples from the oldest layer (pre-LGM, ~24000 years BP) of the sediment, two samples from the LGM (~19000 years BP), and two samples post-LGM (~14000 years BP). The characterization of these samples will be assessing first the mineralogy composition of the sediment by means of X-ray diffraction (Multipurpose X-ray Diffractometer instrument, CNR-ICMATE Unit) and Small/Wide Angle X-ray Scattering (SAXS GISAXS instrument, CSGI-Unit) measurements. Complementary neutron diffraction data on the same set of samples will be measured at the INES beamline, ISIS neutron and muon source (UK). The minerals in each sediment sample will be quantified using Rietveld refinement of X-rays and neutron diffraction data and cross-compared to verify consistency. Later, the presence of organic compounds [4] will be inferred by Fourier-transform infrared spectroscopy measurement on the FT-IR Nicolet instrument available at the Rome Tor Vergata Unit. To follow we will perform shotgun sequencing of aDNA, using DNA Sequencing NGS of Rome Tor Vergata Unit and finally retrieve all the proteins in the remains using Mass Spectrometer 2 at the University of Milano Bicocca Unit which will be useful to support the DNA data [5].

Proposed experiment

In this experiment we aim to perform Fourier-transform infrared spectroscopy measurement on the FT-IR Nicolet instrument available at the Rome Tor Vergata Unit to study organic compounds and the possible presence of bones in n. 6 samples coming from sediments extracted in the Romito cave. Two samples have been extracted by sediments in the oldest layer (pre-LGM, ~24000 years BP), n. 2 samples from the LGM (~19000 years BP), and two samples post-LGM (~14000 years BP). Results of this experiment will be cross compared to verify consistency with data obtained by sequencing the aDNA on the same set of samples, using the DNA Sequencing NGS instrument at the Rome Tor Vergata Unit and by retrieving all the proteins in the sediments using the Mass Spectrometer 2 at the University of Milano Bicocca Unit.

Justification of experimental time requested

Each of the n. 6 sample will be enclosed in a sterile tube with about 2 g (4 cc) of sediment, and it will be maintained at -20 °C temperature to preserve aDNA during the measurements. We envisage, after discussion with the instrument scientist, to measure n. 3 samples per day on the instrument. Hence, we request a total of 2 days of instrument time including set-up and calibration time.

References

[1] Kjær et al., Nature **612** (2022), p. 283–291.
[2] Blockley et al. 2018. Quaternary Science Reviews, 184: 5-25.
[3] Craig et al., 2010. Journal of Archaeological Science, 37: 2504-2512.
[4] Scorrano et al., 2015. Annals of Human Biology, 42: 10-19.
[5] Scorrano et al. 2022. Communications Biology, 5: 1262.

Experiment Proposal

Experiment number GP2024063

Principal investigator	Professor Giuseppe Pomarico, La Sapienza University, ITALY
Co-investigator	Professor Roberto Paolesse, University of Rome Tor Vergata, ITALY
Co-investigator (*)	Dr Gabriele Magna, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	IR spectroscopic analysis of electropolymerized Corroles
MRF Instrument	**FT-IR Nicolet**
Days requested:	3
Access Route	Direct Access
Previous GP Number:	No
Science Areas	Chemistry
DOI:	-
Sponsored Grant	None
Sponsor:	-
Grant Title	-
Grant Number:	-
Start Date	-
Finish Date:	-
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

Corroles, a family of contracted porphyrinoids, exhibit broad chemical interactions, undergo straightforward synthetic preparation and functionalization, and enable versatile thin film deposition. Corroles are then promising candidates for use in chemical sensors but the inherently limited conductivity of corrole solid films constrains their application in mass and optical sensors. There is however a great interest in matching the sensitive properties of corrole with the features of facile miniaturization and integration into low-cost electronic circuits. The electropolymerization of Cu corrolates can allow the formation of semiconducting films onto interdigitated electrodes. Remarkably, the electropolymerization protocol allows the selection of the semiconductive nature (p- or n-type) of these films. The different structure of the polymeric film can be investigated by IR spectroscopy, which can give useful insights on the molecular backbone-semiconductor nature of the deposited films.

Publications

A. Milone, A. Monteduro, S. Rizzato, A. Leo, C. Di Natale, S. Sub Kim, G. Maruccio, Adv Sustainable Syst. 2023, 7, 2200083

Anfar, Z., Magna, G., Di Filippo, I., Monti, D., Naitana, M.L., Stefanelli, M., Paolesse, R. J. Phys. Chem. B, 2024, 128 , 1550

Sivalingam, Y., Velappa Jayaraman, S., Magna, G., Kiran, M.S.R.N., Vesce, L., Paolesse, R., Di Natale, C. ACS Appl. Nano Materials, 2024, 7, 9324

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E-platform: No

Instruments	
Days Requested:	
Access Route	
Previous RB Number:	
Science Areas	
DOI:	
Sponsored Grant	
Sponsor:	
Grant Title	
Grant Number:	
Start Date	
Finish Date:	
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Gabriele Magna, University of Rome Tor Vergata, ITALY
MRF Instrument	**FT-IR Nicolet**
Days Requested:	3
Special requirements:	

SAMPLE

Material	polymeryzed corroles	-	-
Formula	organic inorganic polymers	-	-
Forms	Solid		
Volume	10 cc		
Weight	200 mg		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	No	-	-
Sample Prep Hazards	none	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	none	-	-
Equipment Hazards	-	-	-
Biological hazards	none	-	-
Radioactive Hazards	none	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	-	-

IR spectroscopic analysis of electropolymerized Corroles

1. Background and Context

The demand for high-performance gas sensors is continuously growing, especially prompted by prominent technological concepts like wireless electronics, flexible support, and wearable devices. Gas sensors in these fields are expected to boost the capabilities of strategic applications such as those aimed at monitoring physiological parameters and environmental conditions.[1] These emerging use cases require devices that, besides the self-evident sensitivity and selectivity, are small, cost-effective, low-power, and easily integrated into electronic platforms. These demands often conflict with the properties of many state-of-the-art sensors. For example, metal- oxide semiconductors might match the requirements for integrated gas sensors, but their high operating temperatures make them unsuitable for low-power applications.[2] On the other hand, certain molecular materials possess exceptional sensing properties, but their scarce conductivity necessitates a more complicated, voluminous, and power-consuming electronic interface setup to accommodate devices based, for example, on optical or mass-sensing transduction.

To solve this drawback, we explored an alternative approach to producing conductive sensors based on porphyrinoids straightforwardly by exploiting the properties of electropolymerized corroles characterized by electron-active substituents. A typical example is the electrochemical oxidation of 5,10,15,20 - tetrakis - (4-aminophenyl) porphyrins or 5,10,15 – tris - (4-aminophenyl) corroles, which leads to the formation of different types of linkers as the phenazine bridge [3-4]. We investigated the sensing properties of the conductometric sensors based on polymers of [5,10,15- (4-aminophenyl) corrolato] copper.

2. Proposed experiment

We propose to perform a AFT-IR analysis of the polymeric films, in order to determine the influence of the protonation of the polymeric backbone on the possible formation of quinoidal groups that can influence the electrical properties of the film. To do this, we plan to perform measurements using the FT-IR Nicolet instrument at the Tor Vergata unit of IM@IT. Samples of the materials will be prepared by electropolymerization techniques, modulating the experimental conditions to verify their influence on the polymeric structures and the related semiconductor character.

3. Summary of previous experimental proposals or characterisation

Previous characterizations were carried out by SEM analysis of the polymers after the polymerization onto IDE substrates. the polymeric film fills the gap between the electrons to form a conductometric layer, which is uniform over the whole IDE surface. Further magnification confirms that the film mainly covers the glass between the electrodes. The film over the electrodes is characterized by nanoparticles and sporadic bigger nanostructures with more complex morphology. With the increase of scanning, the film starts to coat all the electrode surfaces with the appearance of rod-like structures, submerging the underneath IDEs. The deprotonated polymer morphology showed a film more homogenous over the gold electrode, and the rod-like elements connect the electrodes. Noteworthy, the protonation state of the polymer affects the semiconducting type, as evidenced by the fact that the two sensors show an opposite response direction to the gas: the adsorption of NO increases the resistance of the protonated polymer and decreases the resistance of that deprotonated. Considering the electron donor character of NO, the sensor responses indicate that the films are p-type and n- type polymers, respectively.

4. Justification of experimental time requested

The FT-IR Nicolet is particularly useful for the analysis of polymeric films as those investigated in our group.

Samples of different polymers, done by different CV scans and in the presence of different bases, will be measured. Considering a total of 20 samples, we request 3 instrument days of the FT-IR Nicolet.

References

[1] A. Milone, A. Monteduro, S. Rizzato, A. Leo, C. Di Natale, S. Sub Kim, G. Maruccio, Adv Sustainable Syst. 2023, 7, 2200083.

[2] H. Ji, W. Zeng, Y. Li, Nanoscale, 2019, 11, 22664-22684.

[3] M. G. Walter, C. C. Wamser, J. Phys. Chem. C 2010, 114, 7563-7574.

[4] N. U. Day, M. G. Walter, C. C. Wamser, J. Phys. Chem. C 2015, 119, 17378-17388.

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Fluorescence Microscopy

Experiment Proposal

Experiment number GP2024083

Principal investigator	Dr Anna Prioriello, University of Rome Tor Vergata, ITALY
Co-investigator (*)	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Laura Fazi, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Pietro Morales, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Margaux Bouzin, Università degli Studi di Milano-Bicocca, ITALY
Co-investigator	Professor Roberto Senesi, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Silvia Licoccia, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Experiment title	Fluorescence Microscopy investigation of Polyurethane-Single Wall Carbon Nanotubes composite
MRF Instrument	**Fluorescence Microscopy**
Days requested:	1
Access Route	Direct Access
Previous GP Number:	no
Science Areas	Materials
DOI:	-
Sponsored Grant	None
Sponsor:	-
Grant Title	-
Grant Number:	-
Start Date	-
Finish Date:	-
Similar Submission?	GP2024052
Industrial Links	-

Non-Technical Abstract

Composite materials based on self-grafted carbon nanotubes (CNTs) in polymers have a variety of applications ranging from advanced medical devices to sensor technologies. Their study is often based on dynamical, mechanical, electrochemical, and surface probes. Though, it is difficult to assess the degree of penetration of CNT bundles within the polymer and the type of interaction at the interface. We propose therefore an experimental access to perform Fluorescence Microscopy investigation of composite materials based on self-grafted CNTs in polyurethane (PU). In particular, this experiment will allow us to expand our knowledge of the concentration and penetration gradient of CNTs in the PU polymer matrix.

Publications -

ISIS neutron and muon source	**E-platform:** No
Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
MRF Instrument	**Fluorescence Microscopy** **Days Requested:** 1
Special requirements:	

SAMPLE

Material	Polyurethane (PU)	polyurethane-single wall carbon nanotubes composite (PU/SWCNT)	-
Formula	-	-	-
Forms	Solid	Solid	
Volume	cc	cc	
Weight	mg	mg	
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	- K	-
Pressure Range	- mbar	- mbar	-
Magnetic field range	- T	- T	-
Standard equipment	None	None	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	Yes	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	No	-
Sensitivity to vapour	No	No	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	-

Fluorescence Microscopy investigation of Polyurethane-Single Wall Carbon Nanotubes composite

1. Background and Context

The last decades have seen a growing interest in composite materials because of their wide-ranging applications, ranging from advanced medical devices to sensor technologies. The different applications require specific electro-mechanical properties, which depend on the interaction between the components of the composite material considered.

Our studies [1,2] are focused on the Single Wall Carbon Nanotubes (SWCNT)-polymer substrates composites. By combining the electrical conductivity, the robustness, and the elasticity of SWCNT "as grown" bundles with the viscoelastic properties of polymer films, such as their stretchability and mouldability, a promising category of composite materials useful in the biomedical field have been obtained. For example, by self-grafting SWCNT bundles onto the fairly plastic and mouldable poly-ethylene (PE), we achieved closely spaced microelectrode arrays to monitor, and possibly control, the cortical brain activity of laboratory model animals [1]. On the other hand, a SWCNT/poly-dimethyl-siloxane (PDMS) self-assembled composite, which is much more elastic, allowed the realization of an artificial bladder prototype [2].

The interface properties between polymer and nanotube bundles have been investigated, since it is understood that the properties of composite materials depend on the nanotubes grafting into the polymeric matrices. Preliminary information about the composite material morphology and conformation have been obtained by Scanning Electron Microscopy and confocal micro-Raman spectroscopy; while the mechanical and electrical properties of the composite have been investigated by measurements of stress vs. strain, of resistance vs. strain and of current vs voltage applied to thin film stripes of SWCNT/polymer composites [3]. Our preparatory experiments reveal a deeper penetration of SWCNT into the polymer bulk for thermosetting elastomer substrates with respect to previous investigation on thermoplastic substrates; the former showing diffusion of the SWCNT limited to few micrometers, while the latter allowing much deeper diffusion. Confocal Micro-Raman spectroscopy shows that SWCNT have drifted into the polymer matrices, even if the limited spatial resolution does not allow detection of single ropes or small coils of nanotubes and therefore their spatial distribution.

Hence, additional details on the nanotubes distribution into the polymer bulk are required to better understand the correlation between the composite properties and the polymer chains – nanotube bundles interaction. For this reason, by using Fluorescence Microscopy, we aim to study the distribution of SWCNT that have been rooted into polymeric substrates of one new composite specimen based on the self-grafting of the SWCNT into the polyurethane (PU) matrix.

2. Proposed experiment

In the present proposal, we wish to measure the CNT concentration gradient (along the z coordinate normal to the film surface) into the SWCNT-PU composite. Specimens will be prepared by hot deposition of the CNT suspension onto the polymeric substrate, and the region of greatest physical interest is that of the interface, where the interaction between the nanotubes and the polymer chains of the substrate takes place. The Fluorescence Microscope is available at IM@IT University of Milano Bicocca Unit. Since the commercial clear PU elastomer used as a substrate has not been spectroscopically characterized, we shall also provide a blank specimen to determine the most

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suitable excitation and emission frequency windows to be used. Comparing the pure PU and the PU/SWCNT composite samples will allow distinguishing the signals relating to the polymer and the nanotubes, as done with our samples analysed in the context of the previous training proposal (GP2023073).

3. Summary of previous experimental proposals or characterization

This kind of sample have been analysed by micro-Raman spectroscopy at the UTOV Unit of ISIS@MACH Italia, obtaining preliminary information on the nanotubes penetration in the polymer bulk, showing a penetration depth ranging from 70 to 90 µm. Moreover, these kinds of samples have been analysed from the electro-mechanical point of view showing interesting properties; this composite material can be used as sensor and actuator in biomedical field, as artificial muscle, or as strain sensor to monitor the state of cracks on the walls of buildings following seismic events.

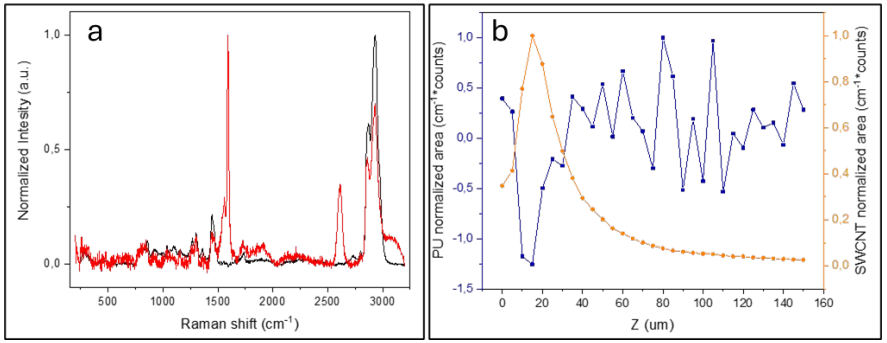

Figure 1: (a) Inelastic Raman spectra of the SWCNT/PU composite material (in red) compared to heated PU substrate (in black); (b) concentration of SWCNT (orange) and PU molecules (blue) inside the composite film vs. distance from the deposition surface.

4. Justification of experimental time requested

We request one day of time to use the Fluorescence Microscope available at the IM@IT – Milano-Bicocca unit. The instrument time will allow the collection of a statistically significant number of images to characterize the nanotubes dispersion into the polymer bulk. In particular, the PU and PU-SWCNT composite samples will be measured by fluorescence and photoluminescence scans by varying and optimizing both the excitation wavelength, ranging from 720 nm to 900 nm, and the collection band.

References

[1] L. Pavone, et al.; Journal of Neural Engineering. 2020 Jul 3;17(3):036032.
[2] EMMA-D-24-00093R3
[3] L. Fazi, et al., Molecules 2023 Feb 13; 28(4):1764.

Experiment Proposal

Experiment number GP2024130

Principal investigator	Dr Barbara Vercelli, Consiglio Nazionale delle Ricerche CNR, ITALY
Co-investigator	Professor Barbara La Ferla, University of Milano Bicocca, ITALY
Co-investigator	Dr Alice Pavan, Università degli Studi di Milano-Bicocca, ITALY
Co-investigator (*)	Professor Maddalena Collini, Università degli Studi di Milano Bicocca, ITALY
Co-investigator	Professor Giuseppe Chirico, Università degli Studi di Milano-Bicocca, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Hydrothermal Sustainable Approaches for the Preparation of Carbon Quantum Dots: a Photo-Physical Insight into the Effect of Different Carbon Sources
MRF Instrument	**Fluorescence Microscopy** — **Days requested:** 5
Access Route	Direct Access — **Previous GP Number:** no
Science Areas	Chemistry, Materials — **DOI:** -
Sponsored Grant	Yes — **Sponsor:** Other
Grant Title	Multifunctional Compounds for a Multitarget Approach against Neurodegenerative Disorders (MULTIFUN) — **Grant Number:** MUR PRIN Project n. 2022N9E847
Start Date	01/10/2023 — **Finish Date:** 30/09/2025
Similar Submission?	GP No: GP2024128; PI: Dr. Alice Pavan and GP No: GP2024129; PI: barbara vercelli
Industrial Links	-
Non-Technical Abstract	The present proposal is part of a broader research program aiming to study and develop reliable and sustainable synthesis approaches for preparing Carbon Quantum Dots (CDs) with robust and reproducible properties. We submit a request for an experimental campaign finalized at performing a photo-physical characterization (decay curves, and life-times determinations) on a series of CDs samples prepared through the hydrothermal approach employing gallic acid (GA) and citric acid (CA) or both as carbon sources. The main scope is to investigate a possible synergic effect of the two carbon sources on the CDs. In particular, we expect that the CDs obtained from the combination of both carbon sources (GA and CA) will merge the intrinsic properties of the samples prepared employing only one carbon source (GA or CA).
Publications	B. Vercelli et al., Small Structures, recently submitted

ISIS neutron and muon source — **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Science and Technology Facilities Council
ISIS Neutron and Muon Source

Sample record sheet

Principal contact Professor Maddalena Collini, Università degli Studi di Milano Bicocca, ITALY
MRF Instrument **Fluorescence Microscopy** **Days Requested:** 5
Special requirements:

SAMPLE

Material	Carbon Quantum Dots obtained at 160°C from gallic acid and urea, solvent EtOH (carbon based graphitic structures)	Carbon Quantum Dots obtained at 160°C from gallic acid/citric acid and urea, solvent EtOH (carbon based graphitic structures)	Carbon Quantum Dots obtained at 160°C from citric acid and urea, solvent EtOH (carbon based graphitic structures)
Formula	-	-	-
Forms	Solid	Solid	
Volume	cc	cc	cc
Weight	5 mg	5 mg	5 mg
Container or substrate	vial	vial	vial
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

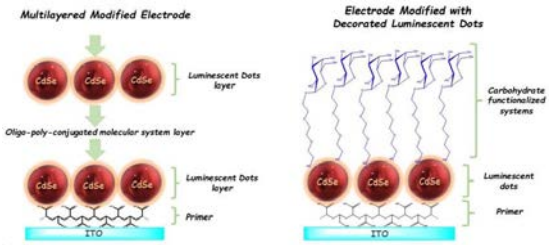
Temperature Range	- K	- K	- K
Pressure Range	- mbar	- mbar	- mbar
Magnetic field range	- T	- T	- T
Standard equipment	None	None	None
Special equipment	no	no	no

SAFETY

Prep lab needed	Yes	Yes	Yes
Sample Prep Hazards	no	no	no
Special equip. reqs	no	no	no
Sensitivity to air	No	No	No
Sensitivity to vapour	No	No	No
Experiment Hazards	no	no	no
Equipment Hazards	-	-	-
Biological hazards	no	no	no
Radioactive Hazards	no	no	no
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	Disposed by IS

1. Background and Context

The group of Electrochemistry and Nanomaterials of the Icmate unit of Milano has a long-time experience in the realization and characterization of self-assembled nano-systems, obtained by the alternation of semiconductor nanocrystals or noble metals clusters and oligo-polyconjugates molecular systems or carbohydrates functionalized systems (Scheme 1), for optoelectronic, photovoltaic and biomedical applications. This is a consolidated blue-sky research activity documented by a series of publications in high-impact ISI journals.

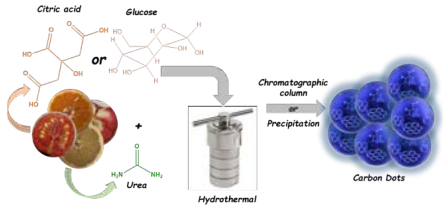


Scheme 1. – Self-assembled Nanosystems

Within this research program and considering the growing request for eco-sustainable, non-toxic nanomaterials, the research studies were recently devoted to the employment of carbon quantum dots (CDs) as a "green" and cheap alternative to noble metal and calcogenide-based dots.

CDs are fluorescent carbon-based nanomaterials, which, since their discovery in 2004, gained grooving interest from the research community, because of their excellent fluorescence properties and surface rich in functionalities, which enables functionalization with a wide range of molecules, including receptors, bio-molecules, molecular semiconductors, etc. Furthermore, they are soluble in water, exhibit an extremely low toxicity, and an excellent biocompatibility useful for real-world biological applications. Their synthesis approaches are simple, sustainable, and could employ cheap and recyclable precursors derived from biomass and agro-industrial waste.

As a first approach, we focused on the development of a sustainable CDs synthesis strategy, which could be reliable and reproducible. We selected the hydrothermal approach owing to its feasibility for large-scale industrial applications, and we employed precursors that could be obtained from agro-industrial waste, like citric acid (CA) or glucose (Glu) as carbon sources and urea as both base and nitrogen sources (Scheme 2).



Scheme 2. – Scheme of hydrothermal preparation of Carbon Quantum Dots

We published two preliminary works dealing with the role played by the nitrogen centers and the thermal post-treatments, respectively, on CDs electrochemical and optical properties[1]. Then we studied the influence of the reaction parameters on CDs properties: we published a work on the issues encountered in the synthesis/purification of red-emitting CDs[2], and we recently submitted a comprehensive work on the influence of the process parameters, particularly temperature, on the properties of CDs[3]. In a further step of our research program, we employed gallic acid (GA) alone and in combination with CA, as carbon source to study a possible synergic effect on the optical and properties of CDs. Preliminary, UV-vis absorption and FT-IR determinations seem to support the hypothesis. In this context, a photo-physical characterization (decay curves, and life-times determinations) is of crucial interest/importance to obtain a comprehensive characterization of the new CDs. In particular, we expect that the CDs obtained from the combination of both carbon sources (GA and CA) will merge the intrinsic properties of the samples prepared employing only one carbon source (GA or CA). Optimistically, we also expect to obtain information about the influence of the temperature process on the photo-physical properties of the CDs prepared employing both GA and CA.

2. Proposed experiment

With the present proposal, we submit the request of a 5 days' experimental campaign finalized at performing a photo-physical characterization (including decay curves, and life-times determinations) on a series of CDs samples (optimistically 3/4) prepared through the hydrothermal approach employing GA, CA or both as carbon sources. The main scope is to investigate the influence of the carbon source on the photo-physical properties of the obtained materials.

The results expected from the proposed experimental campaign are of paramount importance for the development of the research programme briefly described at point 1, because they are expected to support and enforce the preliminary obtained UV-vis and FT-IR results, employing a facility (Fluorescence Microscopy), which is not present in the laboratories of the Icmate unit of Milano.

3. Summary of previous experimental proposals or characterisation

We previously submitted the proposal n°GP2024022 for a series of TEM analyses of CDs samples.

4. Justification of experimental time requested

For the development of the present proposal, we selected the ISIS@MACH ITALIA Fluorescence Microscopy facility at the University of Milano-Bicocca because it meets the experimental requests and is located in the proximity of the Icmate unit of Milano. So, in case of analysis problems or specific sample preparations, it is possible to promptly intervene in the near Icmate laboratories. We planned the photophysical characterization of 3/4 CDs samples for a total of 5 days, including eventual time waste related to possible issues in sample preparation, specific instrument settings, and unexpected problems during measurement execution and signal optimization/collection.

5. References

1. Vercelli B. et al. Elec. Acta, **2021**, 138557, https://doi.org/10.1016/j.electacta.2021.138557; Vercelli B. et al. Molecules **2023**, 28(1), 72; https://doi.org/10.3390/molecules28010072.

2. Vercelli B. et al. Nanomaterials **2023**, 13, 1365; https://doi.org/10.3390/nano13101635.

3. Vercelli B. et al., Small Structures, submitted.

Gas Chromatography-Ion Mobility Spectrometer

Experiment Proposal

Experiment number GP2024142

Principal investigator	Dr Jens Holtvoeth, Teesside University, UNITED_KINGDOM
Co-investigator	Professor Charles Cockell, University of Edinburgh, UNITED_KINGDOM
Co-investigator (*)	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Silvia Licoccia, University of Rome Tor Vergata, ITALY
Co-investigator	Miss Julia Puputti, Boulby Underground Laboratory STFC, UNITED_KINGDOM
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Biogeochemical Multi-Instrumental Study of Rocks and Microbial Biomass in Zechstein Salt Deposits of Boulby using Gas Chromatography – Ion Mobility Spectrometer
MRF Instrument	**Gas Chromatography - Ion Mobility Spectrometer** **Days requested:** 2
Access Route	Direct Access **Previous GP Number:** No
Science Areas	Biology and Bio-materials, Chemistry, Environment, Materials **DOI:** -
Sponsored Grant	Yes **Sponsor:** CNR
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	Our aim is to investigate the potential for preserving biological material in ancient salt deposits, with a focus on the Zechstein salt deposits in Boulby Mine (UK), offering insights into the environmental conditions of the Zechstein Sea ~250 million years ago. The study employs a multi-instrumental approach, combining non-destructive and destructive analyses to correlate biomolecule presence with mineral phases and elemental compositions. By analyzing a range of biomarkers the research aims to create a detailed biogeochemical fingerprint of fossil microbial biomass. This research contributes to both astrobiology and our understanding of ancient terrestrial environments. We propose a multi-instrumental approach involving by combining non-destructive and destructive analyses exploiting the analytical instrumental suite at IM@IT and ISIS facilities. In this specific proposal we propose to use GC-IMS instrument for analysis of the biomarkers, specifically volatile organic compounds.
Publications	Cockell, C. et al. (2020) Astrobiology 20, 864-877 Gasparri, R. et al (2022) J. of Breath Research 16.4, 046008

ISIS neutron and muon source	**E-platform:** No
Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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ISIS Neutron and Muon Source

Sample record sheet

Principal contact	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY		
MRF Instrument	**Gas Chromatography - Ion Mobility Spectrometer**	**Days Requested:** 2	
Special requirements:			
	SAMPLE		
Material	slabs of salt	-	-
Formula	salt (NaCl) sediments and organic phases	-	-
Forms	Friable powder		
Volume	100-150 cc		
Weight	100-150 g		
Container or substrate	-	-	-
Storage Requirements	-	-	-
	SAMPLE ENVIROMENT		
Temperature Range	273 - 320 K	-	-
Pressure Range	1000 - 1010 mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	-	-	-
	SAFETY		
Prep lab needed	No	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	Yes	-	-
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Biogeochemical Multi-Instrumental Study of Rocks and Microbial Biomass in Zechstein Salt Deposits of Boulby using Gas Chromatography – Ion Mobility Spectrometer

1. Background and Context

Can the remains of biological material be preserved in salts many hundreds of millions of years old and what signatures can be preserved? To answer these questions, we must be able to probe the chemical and physical conditions at small scales in ancient salt deposits to understand the geological context of biomolecular preservation. We aim to produce a biogeochemical and molecular fingerprint of fossil microbial biomass and inorganic rock samples in the Zechstein salt deposits of Boulby Mine, North Yorkshire, UK through analysing biomarkers, specifically, alkyl lipids (alkanes, fatty acids and alcohols, steroids), glycerol dialkyl glycerol tetraethers (GDGTs), ancient DNA (aDNA) and proteins. This research is timely as the outcomes will help to interpret Raman spectroscopy data produced from the same material. Raman spectroscopy will be one of the analytical tools aboard the next generation Mars rovers. Martian evaporites are prime targets in the search for extra-terrestrial life since the last places where microbial life could have existed on Mars would have been the evaporating oceans. In addition to astrobiology, outcomes are expected to contribute insights into late Permian hydrology and paleoecology. Biomarker distributions in the salt at Boulby mine and particularly in backfilled desiccation cracks can provide information on the environmental conditions in and around the Zechstein Sea ~250 million years ago. The site represents a shallow nearshore setting of the Zechstein Basin, with exposure of the evaporite surfaces during sea-level low stand. The presence of biomarkers originating from both microbes and plants in the Boulby salt has already been demonstrated [1]. Upscaling of the extraction procedure and an improved extraction protocol is expected to produce sufficient material for compound-specific carbon and hydrogen isotope analyses ($d^{13}C$, $d^{2}H$) of leaf wax-derived compounds, which will provide information on continental plant types and aridity. In this context the knowledge of the exact spatial distribution and inorganic chemical composition of organic matter in the salt on the atomic scale would help much more targeted analyses. For example, leaf waxes may be associated to different material and specific sites compared to microbial membrane lipids. Thus, we aim to use a set of analytical instruments of IM@IT and neutron beamlines of ISIS Facilities. Due to the age of the samples, a good understanding of the exact spatial distribution of the organic matter and its association the inorganic phases of the salt would greatly support targeted analyses. This would allow us to select specific sub-samples with higher organic yields for biomarker investigations and compound-specific isotope analyses, in particular. In addition to targeting the analysis, the physical and chemical context of the biomolecules (or even a lack of them) provides essential information to explain how physical and chemical conditions influence the fate of biomolecules and their potential or long-term preservation over geological time scales. For example, is the presence of any putative biomarkers associated with specific elements or mineral phases? We can answer this by correlating the presence of biomolecules with mineral phases and elemental composition through using small/wide angle X-ray Scattering by means of SAXS GISAXS; hard X-ray Fluorescence (2D/3D XRF), using RETINA and Multipurpose X-ray diffraction; and neutron diffraction combined with tomography, using INES and IMAT neutron beamlines. Does the oxidation state of elements, for example iron, influence the chemical environment and thus the presence and preservability/stability of biosignatures in the salt over time? We will answer this by correlating the presence of any biosignatures to the elemental oxidation state in the same location at the relevant scales using mineralogical information. Therefore, we propose a multi-instrumental approach involving a combination of non-destructive and destructive analyses exploiting the analytical instrumental suite at IM@IT and ISIS facilities. All analyses will be using the same sample materials, first, for non-destructive and then destructive methods, to maximise the complementary character of the resulting data sets. On one hand, for the non-destructive analyses of the samples, we use the analytical suite of instruments of IM@IT and ISIS facilities indicated. The minerals in each salt sample will be quantified using combined Rietveld refinement of X-rays and neutron diffraction data, using laser ablation. On the other hand, due to the very low yields of the organic phase and its fine dispersal in the salt, the samples to be analysed will consist of total lipid extracts from small slabs of salt of 100-150g, representing two distinct types of material: relatively pure evaporite material and desiccation crack backfill material. The pure evaporite material will be mainly sodium chloride, containing lenses of clay-rich material and isolated potassium chloride crystals. It is expected to include an organic phase originating predominantly from extremophile biomass and some eolian terrigenous input. The slightly darker coloured material from the desiccation cracks, on the other hand, is expected to include higher proportions of terrigenous organic particles and fragments of biofilm from the salt surface that were resuspended and washed into the cracks during rising water level. For the (destructive) analysis of the biomarkers, originating from microorganisms, terrestrial vegetation or processing-related contamination, like alkanes, alcohols, and ketones we propose to use Gas Chromatography – Ion Mobility Spectrometer (GC-IMS), steranes and GDGTs will be analysed by normal-phase UHPLC at Bristol University; for the aDNA analysis, using DNA sequencing NGS, and for the analysis of both fatty acids and ancient proteins using the Mass Spectrometer 2. Genetic, lipid, and proteomic and volatilomic data will then be cross compared to verify their consistency with the possible extremophile species identified. This research is embedded in a wider collaborative attempt to understand extremophile ecology through a comparison with lipid, proteins and DNA data of modern microbes living on the salt surfaces and in brines in Boulby mine. We aim to see if microbial communities adapt to changes in brine salinity and/or ion composition (chloride vs. sulphate, sodium vs. potassium) either by individual species changing their cell membrane properties or by shifts in species distribution. This is a collaboration with Teesside University, the UKRI-STFC Underground Lab. Boulby, the UK Centre for Astrobiology at Edinburgh University, NASA Jet Propulsion Lab, Bristol University, the University of Bern, the University of Rome Tor Vergata, the IM@IT and ISIS facilities. It is supported by the Seedcorn Funding scheme of Teesside University to produce pilot data for a larger proposal to fund PhD projects at Teesside and Edinburgh Universities

2. Proposed experiment using GC-IMS

In this proposal we plan to use of Gas Chromatography – Ion Mobility Spectrometer (GC-IMS) to study the origin of eventually detected volatile organic compounds, originating from microorganisms, presence of vegetation or environmental contamination; these data will be compared with wide-angle information from SAXS GISAXS and laser ablation EA and integrated with those obtained by DNA sequencing NGS and with results from Mass Spectrometer 2 and eventually with results on steranes and GDGTs to be analysed by normal-phase UHPLC at Bristol University.

3. Justification of experimental time requested

Specimens will be analysed using headspace sampling technique combined with Gas Chromatography – Ion Mobility Spectrometer (GC-IMS). Rock samples, small quantity (< 10 g) salt powder, representing two distinct types of material, will be sealed in 20ml headspace vials and heated. The headspace will be sampled by an autosampler, and VOCs analysed by the instrumentation. For the analysis of four samples, we request **2 days** for acquisition and data analysis.

MONeutron

Experiment Proposal

Experiment number GP2024094

Principal investigator	Dr Francesco Pintacuda, STMicrocelectronics, ITALY
Co-investigator (*)	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Fabio PRINCIPATO, Università degli Studi di Palermo, ITALY
Co-investigator	Miss Virginia Pietrosanti, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Carla Andreani, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	A power transistor monitor to record high energy neutrons
MRF Instrument	**MONeutron** **Days requested:** 90
Access Route	Direct Access **Previous GP Number:** No
Science Areas	Engineering, ICT, Physics **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	STMicroelectronics

Non-Technical Abstract

We aim in this experiment to expose some Si, SiC MOSFETs (by STMicroelectronics) and diamond-based semiconductors to atmospheric neutrons and measuring the charge deposited on them. Our devices will be placed near the 3He proportional counters of the MONeutron ground-level neutron monitor which is capable to measure the neutron spectrum of atmospheric neutrons. The aim of this experiment is to use the different devices as a neutron detector to measure the neutron spectrum and compare the outcomes with the one register by the MONeutron instrument. Currently, Si and SiC power MOSFETs are irradiated with broad-spectrum neutrons supplied by neutron facilities, so it is not known how the response of the SiC MOSFET depends on the neutron energy measured by a proper neutron monitor design for atmospheric neutrons.

Publications

Pintacuda, F. et al., Microelectronics Reliability, Volume 150, 2023, 115175.
Principato, F.; Cazzaniga, C.; Kastriotou, M.; Frost, C.; Abbene, L.; Pintacuda, F., Radiation 2023, 3, 110-122.
Principato, F. et al. Investigation of the Impact of Neutron Irradiation on SiC Power MOSFETs Lifetime by Reliability Tests. Sensors 2021, 21, 5627.

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ISIS Neutron and Muon Source

Sample record sheet

Principal contact	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY		
MRF Instrument	**MONeutron**	**Days Requested:** 90	
Special requirements:			

SAMPLE

Material	SiC MOSFET	Si detector	-
Formula	SiC	Si	-
Forms	Solid	Solid	
Volume	0.00045 cc	0.00045 cc	
Weight	1.44 mg	1.2 mg	
Container or substrate	selfstanding	selfstanding	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	- K	-
Pressure Range	- mbar	- mbar	-
Magnetic field range	- T	- T	-
Standard equipment	None	None	-
Special equipment	None	-	-

SAFETY

Prep lab needed	Yes	Yes	-
Sample Prep Hazards	No	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	No	-
Sensitivity to vapour	No	No	-
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	No	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	Returned to user by instrument scientist (when inactive)	-

A power transistor monitor to record high energy neutrons

1. Background and Context

Interactions of cosmic rays (mostly comprised of protons and electrons but also of heavy ions with relatively low abundance) with gases in the upper atmosphere result in spallation and ionization events with byproducts of liberated atoms, neutrons, pions, muons, electromagnetic waves, and photons. Among these, neutrons have large mass, are not deflected by the earth's magnetic field, have interaction cross sections larger than muons but less than photons, and finally, are capable of giving rise to further neutron creation. Starting with initial neutron creation in the upper atmosphere, the neutron flux increases with decreasing altitude due to the increased number of cosmic ray-gas interactions, and neutron-gas interactions. The neutron flux peaks at roughly 1 n cm^{-2} s^{-1} approximately at 20 km altitude. The terrestrial neutron flux then starts decreasing with decreasing altitude due to cosmic rays getting deflected and neutrons losing energy during atmospheric gas interactions. The terrestrial neutron flux drops by roughly one order of magnitude at 10-km altitude and an additional two orders of magnitude at sea level [1]. Silicon and silicon carbide (SiC) power MOSFETs are electronic devices used in many applications such as electric vehicles, power grids, and railway traction [2] and in avionic applications [3]. These devices are susceptible to atmospheric neutrons, which induce failure by single-event burnout (SEB) mechanisms [1]. This failure mechanism has impact on power MOSFET reliability and nowadays it has become mandatory in many applications to estimate the failure rate due to neutrons. Usually, the ruggedness of the power MOSFET to atmospheric neutrons is estimated by accelerated neutron tests, by using facilities that provide neutron beams with an atmospheric spectrum and fluxes many orders of magnitude greater than the atmospheric one [3,4]. The STMicroelectronics and Department of Physics and Chemistry (DiFC) of Palermo University (Italy) have collaborated for several years on the study of the effects of neutrons on Si and SiC power MOSFETs and have carried out many neutron irradiation campaigns [5-8]. This research activity is supported by STMicroelectronics, which funded the research activity, manufactures and supplies the devices, by DiFC, which developed the instrumentation and analysis methodologies techniques and by the Chpir-ISIS team, which have collaborated on this research activity.

2. Proposed experiment

The experiment consists of exposing some Si, SiC MOSFETs (by STMicroelectronics) and diamond-based semiconductors to atmospheric neutrons and measuring the charge deposited on them. Our devices will be placed near the ^{3}He proportional counters of the MONeutron ground-level neutron monitor which is capable to measure the atmospheric neutron spectrum. The aim of this experiment is to use the different devices as a neutron detector to measure the spectrum of the collected charge [6] and compare the outcomes with the spectrum register by the MONeutron neutron monitor. Currently, Si and SiC power MOSFETs are irradiated with broad-spectrum neutrons supplied by the facilities, so it is not known how the response of the SiC MOSFET depends on the neutron energy measured by a proper neutron monitor design for atmospheric neutrons. The correlation between the spectrum of the collected charge by the different semiconductors and the spectrum measured by the ^{3}He detectors can give information about the neutron energy-range to which the devices are sensitive. Moreover, we expect that this energy range depends on the material and the technology of the power MOSFET. Fig. 1 shows the setup used in the experiment, where the current pulse induced by neutrons in the semiconductors is measured by high-speed digitizers.

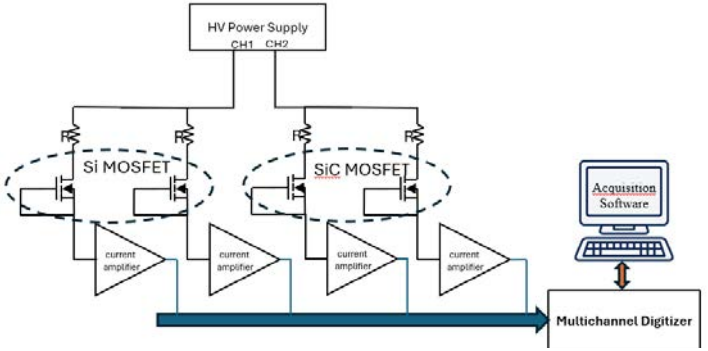

Figure 1: Schematic setup of the proposed experiment.

Two sample of Si and two of SiC MOSFETs will be used to monitor the atmospheric neutrons. We will stack the DUTs one on top of the other to study possible coincidences between neutron interactions in different devices. In addition, we aim to add a diamond-based neutron detector in our system. The trigger of the waveforms acquisition occurs when the impulse is present in at least one of the detectors. The analysis of the acquired waveforms to estimate the spectra of the collected charge will be performed off-line. Similar setups have been used in previous experiments with artificial neutron beams [6].

3. Justification of experimental time requested

Due to the low flux of the atmospheric neutrons at sea level (of about 13 n cm^{-2} h^{-1}) [7] and the unknown efficiency response of the detectors to such neutron energy range, we request to leave the devices outdoors for collecting at least a thousand neutron hits detected in each device to get statistical significance of the data.

4. References

[1] Akturk, A. et al., IEEE Transactions on Nuclear Science **2018**, 65, 1248‑1254. [2] Davidson, C.; Blackmore, E.; Hess, J. Failures of MOSFETs in terrestrial power electronics due to single event burnout. In Proceedings of the INTELEC 2004. 26th Annual International Telecommunications Energy Conference, 2004, pp. 503–507. [3] Galloway, K. F. et al. Estimates for SiC Power MOSFETs in Space Electronics. Aerospace 2018, 5. [4] Martinella. C. et al., IEEE Transactions on Nuclear Science **2021**, 68, 634‑641. [5] Principato F. et al, Sensors **2020**, 20, 3021. [6] Principato F. et al., *Radiation* **2023**, *3*, 110-122. [7] Principato, F. et al., *Sensors* **2021**, *21*, 5627. [8] Pintacuda, F. et al., Microelectronics Reliability, Volume 150, 2023, 115175.

Mass Spectrometer 2

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Science and Technology Facilities Council
ISIS Neutron and Muon Source

Experiment Proposal

Experiment number GP2024041

Principal investigator	Professor Domenico Lo Vetro, Università di Firenze, ITALY
Co-investigator (*)	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Alessandro Cianchi, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Exploring environmental dynamics in ancient remains before and after the last glacial maximum using mass spectrometry measurements
MRF Instrument	**Mass Spectrometer 2** — **Days requested:** 3
Access Route	Direct Access — **Previous GP Number:** No
Science Areas	Biology and Bio-materials, Environment — **DOI:** No
Sponsored Grant	None — **Sponsor:** -
Grant Title	- — **Grant Number:** -
Start Date	- — **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	Our aim is to study environmental ancient sediments coming from sediments extracted in the Romito cave (Cosenza, Italy) before and after the Last Glacial Maximum by multi-instrumental approach. The characterization of these samples to study all the protein content will be performed by mass spectrometry measurements using the Mass Spectrometer 2 at the University of Milano Bicocca Unit. Data will be cross compared to verify consistency with the one obtained by FT-IR measurements and by sequencing the DNA using the DNA Sequencing NGS instrument at the Rome Tor Vergata Unit. Hence, we aim here to request access to the Mass Spectrometer 2 instrument at the University of Milano Bicocca Unit of IM@IT.
Publications	Berto et al. 2022. Archaeological and Anthropological Sciences. Vol. 14, article N. 127, (2022) López-García et al. 2014. Palaeogeography, Palaeoclimatology, Palaeoecology, Vol 251, Issues 3–4, Pages 500-526 Scorrano et al. 2022. Communications Biology, Vol 5, Article N 1262 (2022)

Instruments	**INES** — **Days Requested:** 3
Access Route	Direct Access — **Previous RB Number:** No
Science Areas	— **DOI:** No
Sponsored Grant	None — **Sponsor:**
Grant Title	- — **Grant Number:**
Start Date	- — **Finish Date:**
Similar Submission?	
Industrial Links	

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Science and Technology Facilities Council
ISIS Neutron and Muon Source

Sample record sheet

Principal contact	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY
MRF Instrument	**Mass Spectrometer 2** — **Days Requested:** 3
Special requirements:	

SAMPLE

Material	Sediments/remains	Sediments/remains	Sediments/remains
Formula	organic (collagen) and sand/limestone mixtures	organic (collagen) and sand/limestone mixtures	organic (collagen) and sand/limestone mixtures
Forms	Solid	Solid	Solid
Volume	4-10 cc	4-10 cc	4-10 cc
Weight	2-10 g	2-10 g	2-10 g
Container or substrate	Sterile tube or aluminum foil	Sterile tube or aluminum foil	Sterile tube or aluminum foil
Storage Requirements	Freezer (-20C)	Freezer (-20C)	Freezer (-20C)

SAMPLE ENVIROMENT

Temperature Range	273 - 320 K	273 - 320 K	273 - 320 K
Pressure Range	1000 - 1010 mbar	1000 - 1010 mbar	1000 - 1010 mbar
Magnetic field range	- T	- T	- T
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	Yes	Yes
Sample Prep Hazards	No	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	No	No
Sensitivity to vapour	No	No	No
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	No	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	Returned to user by instrument scientist (when inactive)	Returned to user by instrument scientist (when inactive)

Exploring environmental dynamics in ancient remains before and after the last glacial maximum using mass spectrometry measurements

Background and Context

Ancient environmental DNA (aDNA) refers to genetic material obtained from environmental samples such as soil, sediment, ice and water, which is thousands or millions of years old [1]. The study of ancient DNA is fascinating because it allows us to reconstruct past ecosystems, understand evolutionary processes and trace the impacts of climate and environmental changes on biodiversity over the millennia. This field is particularly timely, as highlighted by a recent publication in Nature detailing the oldest DNA ever recovered from the environment: 2-million-year-old samples that allowed researchers to reconstruct the ecosystem in Greenland [1].

The broader relevance of ancient DNA research lies in its ability to shed light on the complex interactions between climate, environment and living organisms over geological time scales. By understanding past ecosystems, we gain insights into species resilience and vulnerability, which can guide current biodiversity conservation strategies. This proposal aims to characterize samples coming from sediments extracted in the Romito cave (Cosenza, Italy, see Figure 1) [2, 3] before and after the Last Glacial Maximum (LGM) by multi-instrumental approach. The reason of this is twofold. The LGM, when ice sheets were at their maximum extent, was a period of significant climatic and environmental shifts with profound impacts on global ecosystems and human populations. In an era marked by rapid climate change, insights from the LGM can inform our understanding of how ecosystems and species, including humans, responded to extreme climatic conditions. From the other, the Romito cave is one of the most significant Upper Palaeolithic archaeological sites on the Italian peninsula with a well-dated stratigraphy spanning from ~24,000 to 6,000 years before present (BP) (Figure 1d).

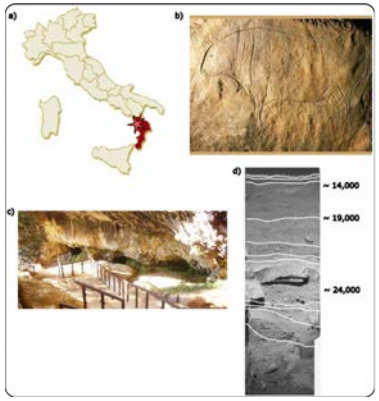

Figure 1. a) Location of Romito cave; b) rock art (Bos primigenius) in the rock-shelter outside the cave; c) cave entrance d) general stratigraphic sequence modified from Blockley et al. (2018).

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To this end, we will study two samples from the oldest layer (pre-LGM, ~24000 years BP) of the sediment, two samples from the LGM (~19000 years BP), and two samples post-LGM (~14000 years BP). The characterization of these samples will be assessing first the mineralogy composition of the sediment by means of X-ray diffraction (Multipurpose X-ray Diffractometer instrument, CNR-ICMATE Unit) and Small/Wide Angle X-ray Scattering (SAXS GISAXS instrument, CSGI-Unit) measurements. Complementary neutron diffraction data on the same set of samples will be measured at the INES beamline, ISIS neutron and muon source (UK). The minerals in each sediment sample will be quantified using Rietveld refinement of X-rays and neutron diffraction data and cross-compared to verify consistency. Later, the presence of organic compounds [4] will be inferred by Fourier-transform infrared spectroscopy measurement on the FT-IR Nicolet instrument available at the Rome Tor Vergata Unit. To follow we will perform shotgun sequencing of aDNA, using the DNA Sequencing NGS of Rome Tor Vergata Unit and finally retrieve all the proteins in the remains using Mass Spectrometer 2 at the University of Milano Bicocca Unit which will be useful to support the DNA data [5].

Proposed experiment

In this experiment we aim to perform mass spectrometry measurements of aDNA using the Mass Spectrometer 2 instrument available at the University of Milano Bicocca Unit to study all the protein content in n. 6 samples coming from sediments extracted in the Romito cave. Two samples have been extracted by sediments in the oldest layer (pre-LGM, ~24000 years BP), n. 2 samples from the LGM (~19000 years BP), and two samples post-LGM (~14000 years BP). Results of this experiment will be cross compared to verify consistency with data obtained by the characterization of organic compounds by FT-IR measurement and by sequencing aDNA in the sediments using the DNA Sequencing NGS instrument available at the Rome Tor Vergata Unit.

Justification of experimental time requested

Each of the n. 6 sample will be enclosed in a sterile tube with about 2 -10 g (4 -10 cc) of sediment, and it will be maintained at -20 °C temperature to preserve aDNA during the measurements. We envisage, after discussion with the instrument scientist, to measure n. 2 samples per day on the instrument. Hence, we request a total of 3 days of instrument time including set-up and calibration time.

References

[1] Kjær et al., Nature **612** (2022), p. 283–291.
[2] Blockley et al. 2018. Quaternary Science Reviews, 184: 5-25.
[3] Craig et al., 2010. Journal of Archaeological Science, 37: 2504-2512.
[4] Scorrano et al., 2015. Annals of Human Biology, 42: 10-19.
[5] Scorrano et al. 2022. Communications Biology, 5: 1262.

Experiment Proposal

Experiment number GP2024077

Principal investigator	Professor Mauro Rubini, Sovraintendenza, ITALY
Co-investigator	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY
Co-investigator (*)	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	A multidisciplinary proteomic study of the unique ancient Homo cepranensis petrous bone using Mass Spectrometer instrument
MRF Instrument	**Mass Spectrometer 2**
Days requested:	2
Access Route	Direct Access
Previous GP Number:	No
Science Areas	Biology and Bio-materials, Cultural Heritage
DOI:	-
Sponsored Grant	None
Sponsor:	-
Grant Title	-
Grant Number:	-
Start Date	-
Finish Date:	-
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

Our aim is to study a petrous bone of unique ancient remain Homo cepranensis (Ceprano, località Campogrande, Italy) by multi-instrumental approach through a combined series of non-destructive and destructive analyses. For non-destructive analyses we request in two distinct proposals the RETINA instrument to perform XRD Tomography, for a 3D reconstruction combined with XRF maps, and the IMAT beamline at ISIS facility for complementary neutron tomography and neutron time of flight Prompt Gamma Activation Analysis (T-PGAA). The scope is to obtain a 3D reconstruction of the Homo cepranensis remain as well as the uranium series. Complementary destructive characterisations, which will be request in distinct proposals, include the ancient DNA (aDNA) and proteomic analyses using the IM@IT' DNA Sequencing NGS and the Mass Spectrometer 2 instruments. The latter analysis is the objective of the present proposal.

Publications

Di Vincenzo. Sci Rep 7, 13974 (2017).
Manzi. J. Hum. Evol. 59, 580-585 (2010).
Rubini. J. Hum. Evol. 77, 204-216 (2014)

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E-platform: No

Instruments	
Days Requested:	
Access Route	
Previous RB Number:	
Science Areas	
DOI:	
Sponsored Grant	
Sponsor:	
Grant Title	
Grant Number:	
Start Date	
Finish Date:	
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
MRF Instrument	**Mass Spectrometer 2** **Days Requested:** 2
Special requirements:	

SAMPLE

Material	petrous bone (about 3x2x1 cm3)	-	-
Formula	hydroxyapatite and collagen.	-	-
Forms	Solid		
Volume	6-9 cc		
Weight	350-600 mg		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	273 - 320 K	-	-
Pressure Range	7000 - 1010 mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

A multidisciplinary proteomic study of the unique ancient *Homo cepranensis* petrous bone using Mass Spectrometer instrument

1. Background and Context

In this proposal we tender to study a remain of a unique ancient sample, *Homo cepranensis*, by multi-instrumental approach. The artifact was discovered on March 13, 1994, by archaeologist Italo Biddittu during surface reconnaissance along the route of a highway under construction near Ceprano (locality of Campogrande in the province of Frosinone) in the lower Sacco Valley (Figure 1). The bulldozers that facilitated the discovery of the artifact at the same time likely caused its fragmentation. The fossil artifact is limited to the neurocranium (calvarium). The fragments were contained within a series of stratified fluvio-lacustrine deposits. About 50 large fragments were unearthed in a small area near the original discovery, and more than 200 small pieces were collected by sieving the sediments [1]. Unfortunately, most of the facial bones, as well as much of the cranial base and almost the entire left parietal, were not found. Currently, the fossil is located at the Superintendency of Archaeology, Fine Arts, and Landscape for the Provinces of Frosinone and Latina. The current form of the artifact is the result of a reconstruction initiated in 1994 by Prof. A. Ascenzi continued by Prof. R. J. Clarke and reviewed by paleoanthropologist M. A. de Lumley and Prof. F. Mallegni [4] (Figure 2). Previous characterisations were carried out directly with the original fragments and with extensive use of dental plaster. The calvarium was already analysed using X-ray microtomography (μCT) at the Multidisciplinary Laboratory of the Abdus Salam International Centre for Theoretical Physics in Trieste. Medical CT scans and recent μCT scans of the calvarium revealed the extent of the plaster and discouraged its mechanical removal. Attempts to digitally remove the plaster from the calvarium also failed, using both globally applied threshold filters and manual operations on each tomographic section; only with high-resolution 3D imaging it was possible to digitally remove the dental plaster insertions and separate the fragments [5]. The calvarium is quite well-preserved, although incomplete, there are no absolute dates and relative dating, based on the regional geo-stratigraphic and paleontological framework, place it between 0.9 and 0.8 Ma [6] .Recent magneto-stratigraphic analyses of the lacustrine and fluvial sediments recovered from cores taken at the site of the artifact, however, have provided a different relative dating; according to these studies, the stratigraphic level containing the artifact itself is between 0.5 Ma and 0.35 Ma [3] (Figure 1). The artifact also shows a series of characteristics, such as a cranial capacity of 1180-1200 cm³, typical of the oldest forms of humanity from the Middle Pleistocene. Cladistic studies initially attributed it to the species *H. erectus* and later to *H. heidelbergensis*, though its exact classification remains unclear. In this proposal our aim is to solve this question by analysing one petrous bone of the sample using multimolecular destructive analyses: proteomics, which is the scope of the present proposal and to follow ancient DNA analysis (aDNA), that we request in a distinct proposal. With the proteomics we retrieve all the proteins in the remains using Mass Spectrometer 2 which will be useful to support the DNA data. The aDNA analysis on this remain is quite challenging because, to date, the oldest ancient genome published from the same latitude is from Sima de los Huesos (Spain), dated back 430,000 years ago [7]. Moreover, the aDNA analysis will be complemented by the proteomic approach (in a distinct request), which in the last five years has demonstrated the possibility of retrieving molecular information from very old specimens from warm environments, such as *Gigantopithecus* (1.9 million years old [8]) and *Homo antecessor* (between 772,000-949,000 years ago [9]). Of great interest is to date for the first time the uranium series and through the fragments to realise a virtual reconstruction of part of the remain.

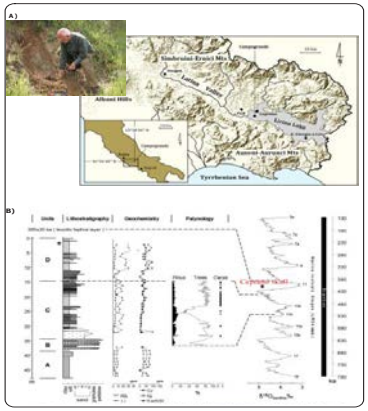
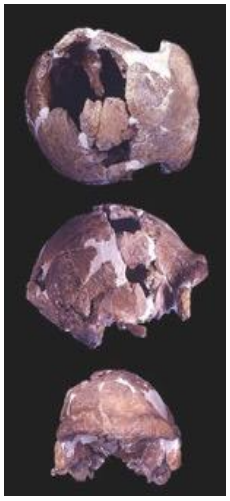


Figure 1.A) geographical localization of the site with Italo Biddittu during the discovery; B) stratigraphy, geochemistry [2], palynological data and $^{40}Ar/^{39}Ar$ dating [3].

Figure 2: *H. cepranensis* skull reconstruction.



2. Proposed experiment

In this specific proposal we aim to sequence the proteins extracted from the ancient remain using the Mass Spectrometer 2 instrument operating at the IM@IT' Unit- University of Milano Bicocca to study the unique ancient *Homo cepranensis* petrous bone from the Ceprano calvarium. Results of this experiment will be cross compared to verify consistency with data obtained by separate proposals where we request NGS instrument operating at the IM@IT' Unit -University of Rome Tor Vergata to perform the ancient DNA analysis (aDNA). Moreover, we request in other proposal a non-destructive analysis: a) RETINA instrument for X-ray tomography with hard X-ray beams, allowing a 3D reconstruction of an extended object, and the concurrent collection of X-ray fluorescence data for 3D chemical composition map; b) IMAT beamline and T-PGAA instrument to measurement before and after the 3D imaging, to obtain a complete digital twin of the sample.

3. Justification of experimental time requested

The protein extraction will be performed by us following the method proposed by Cappellini et al. [10], and the home-made C18 StageTips will be send to the IM@IT' Unit- University of Milano Bicocca for the mass spectrometry analysis. After discussion with the instrument scientist, we plan to measure n. 4 separate extractions plus one blank. Therefore, we request a total of 2 days of instrument time, including set-up and calibration time.

References

[1] Ascenzi, Segre. 2000. In The Origin of Humankind: 25-33; [2] Lisiecki, Rayamo. 2005. Paleoceanography Paleoceanography, 20. [3] Nomade et al. 2011. Quetarnary Geochronology, 6: 453-457. [4] Mallegni et al. 2003. Coptes Rendus Palevol, 2: 153-159. [5] Di Vincenzo et al. 2017. Scientific Reports 7: 13974. [6] Manzi et al. 2001. Proc Natl Acad Sci USA (PNAS) 98: 10011-10016. [7] Meyeret al. 2016. Nature 531: 504–507. [8] Welker et al. 2019. Nature 576: 262-265. [9] Welker et al. 2020. Nature 580: 235–238. [10] Cappellini et al. 2019. Nature 574: 103-107.

Experiment Proposal

Experiment number GP2024125

Principal investigator	Professor Stefania Brocca, Università degli Studi di Milano-Bicocca, ITALY
Co-investigator (*)	Dr Carlo Santambrogio, University of Milano-Bicocca, ITALY
Co-investigator	Professor Rita Grandori, University of Milano-Bicocca, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Assessing interaction of alpha-synuclein with copper

MRF Instrument	**Mass Spectrometer 2**	**Days requested:** 10	
Access Route	Direct Access	**Previous GP Number:** No	
Science Areas	Biology and Bio-materials, Medicine	**DOI:** -	
Sponsored Grant	None	**Sponsor:** -	
Grant Title	-	**Grant Number:** -	
Start Date	-	**Finish Date:** -	
Similar Submission?	-		
Industrial Links	-		

Non-Technical Abstract

Intrinsically disordered proteins (IDPs)) play a key biological role and pose major challenges to structural characterization due to their conformational heterogeneity. This proposal aims to analyze the conformational ensemble of an IDP (a-synuclein) and to assess the impact of post-translational modifications (such as N-terminal acetylation) and the interaction with metal ions (such as copper) on the structural properties of the protein. Native mass spectrometry (native MS) is a powerful tool for the structural and binding analysis of proteins. Therefore, native MS will be employed to analyze the conformational components populated by a-synuclein (with and without acetylation) under several environmental conditions and at different copper concentrations. We expect to identify conformers of different global compactness with distinct binding properties (e.g. stoichiometry, affinity etc.)

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Carlo Santambrogio, University of Milano-Bicocca, ITALY		
MRF Instrument	**Mass Spectrometer 2**	**Days Requested:** 10	
Special requirements:			

SAMPLE

Material	Human alpha-synuclein (unmodified N-terminus)	Human alpha-synuclein (acetylated N-terminus)	-
Formula	C627H1012N166O216S4	C629H1014N166O217S4	-
Forms	Friable powder	Friable powder	
Volume	1.5 ml	1.5 ml	
Weight	1 mg	1 mg	
Container or substrate	plastic tube (1.5 mL)	palstic tube (1.5 mL)	-
Storage Requirements	Temperature -20°C	Temperature -20 °C	-

SAMPLE ENVIROMENT

Temperature Range	- K	- K	-
Pressure Range	- mbar	- mbar	-
Magnetic field range	- T	- T	-
Standard equipment	None	None	-
Special equipment	-	-	-

SAFETY

Prep lab needed	No	No	-
Sample Prep Hazards	None	None	-
Special equip. reqs	None	None	-
Sensitivity to air	No	No	-
Sensitivity to vapour	No	No	-
Experiment Hazards	None	None	-
Equipment Hazards	-	-	-
Biological hazards	None	None	-
Radioactive Hazards	None	None	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	-

1. Background and Context

Intrinsically disordered proteins (IDPs) play key regulatory roles in biological systems and pose major challenges to biophysical characterization due to intrinsic structural dynamics (Bondos et al. 2022). Native mass spectrometry (native MS) is a powerful tool for characterization of conformational ensembles and conformational transitions and also gives information on structural compactness and ligand binding (Mitra 2019).
Our group is involved in the investigation of conformational transitions in natural and synthetic IDPs, with a special focus on the role of electrostatic interactions in structural compactness (Bianchi et al. 2024).
This line of research is currently funded by the grant “Supramolecular assemblies in cell invasion as targets for cancer therapy” (2021-ATEQC-0048) and involves the activity of 1 post-doc and a couple of master students.

2. Proposed experiment

The aims of the proposed experiments are: i) assessing the exact mass and confirming the amino acid sequence of the recombinant, human alpha-synuclein (AS) produced in our laboratory with and without N-terminal acetylation; ii) confirming the N-terminal acetylation state; iii) identifying the conformational components of the ensembles in solution and provide quantitative estimates of the relative amount and solvent-accessible surface area of each component; iv) probing the role of solvent conditions (organic solvents, pH and ionic strength) on the ensemble composition; v) probing the role of N-acetylation on copper binding.
These aims require a native-MS approach because of its unique capability of dissecting and characterizing components of heterogenous systems. In particular, the MS2 unit of ISIS@MACH ITALIA conjugates the possibility to perform such analyses with the ultra-high resolution offered by the Orbitrap technology, which is advantageous for molecular control of recombinant products. Finally, the MS2 team has strong experience in the analysis of IDPs by native MS (Santambrogio et al. 2022).
Previous modelling by molecular-dynamics simulations predict an effect of N-terminal acetylation of human AS, a native modification of the protein expressed in the brain (Rossetti et al. 2016). It is therefore important that in-vitro structural characterization of the recombinant product takes into account these effects.
The data will be analysed by the MS2 unit, providing the quantitative parameters described above by commercial software and in-house developed tools.

3. Summary of previous experimental proposals or characterisation

This is our first experiment with ISIS@MACH ITALIA.
The recombinant proteins will be purified in our laboratory by established procedures and purity will be checked by denaturing gel electrophoresis before sample delivery for MS experiments.

4. Justification of experimental time requested

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MS2 unit provides native-MS measurements, ultra-high resolution, top-down fragmentation possibility, together with software for data analysis and interpretation.
We will have a total of two samples (AS with and without the modification).
For each sample we expect:
One day of instrumental setup
Two days of measurements under variable solvent conditions
Two days of measurements with variable copper concentrations
For a total of 10 working days

5. References

Bianchi G, Mangiagalli M, Ami D, Ahmed J, Lombardi S, Longhi S, Natalello A, Tompa P, Brocca S. (2024) Int J Biol Macromol. 254(Pt 1):127754.

Bondos SE, Dunker AK, Uversky VN. (2022) Cell Commun Signal. 20(1):20.

Mitra G. (2019) Biochim Biophys Acta Proteins Proteom. 1867(11):140260.

Rossetti G, Musiani F, Abad E, Dibenedetto D, Mouhib H, Fernandez CO, Carloni P. (2016) Phys Chem Chem Phys. 18(8):5702-6.

Santambrogio C, Ponzini E, Grandori R. (2022) Biochim Biophys Acta Proteins Proteom. 1870(10):140828.

Experiment Proposal

Experiment number GP2024126

Principal investigator	Professor Emiliano Biasini, University of Trento, ITALY
Co-investigator (*)	Dr Carlo Santambrogio, University of Milano-Bicocca, ITALY
Co-investigator	Professor Rita Grandori, University of Milano-Bicocca, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Characterization of Protein Folding Intermediates of the Cellular Prion Protein by Native Mass Spectrometry
MRF Instrument	**Mass Spectrometer 2**
Days requested:	10
Access Route	Direct Access
Previous GP Number:	No
Science Areas	Biology and Bio-materials, Medicine
DOI:	-
Sponsored Grant	None
Sponsor:	-
Grant Title	-
Grant Number:	-
Start Date	-
Finish Date:	-
Similar Submission?	-
Industrial Links	Sibylla Biotech

Non-Technical Abstract

The misfolding of the cellular prion protein (PrP) into an infectious form (PrPSc) is a crucial event of prion diseases. Our group has developed a novel drug discovery method called Pharmacological Protein Inactivation by Folding Intermediate Targeting (PPI-FIT), which uses Molecular Dynamics (MD) simulations to target and degrade protein folding intermediates. The PPI-FIT approach led to the discovery of SM875, a small molecule that reduces PrP levels, hindering prion propagation. However, the characterization of SM875-PrP interaction is challenging, since the traditional methods of structural biology are often unsuitable for protein folding intermediates. This proposal aims to leverage Native Mass Spectrometry to characterize the interaction between SM875 and the PrP folding intermediate, exploiting the ability of this technique to directly assess the stoichiometry and the conformational selectivity of ligand-binding, even for poorly populated protein conformational states.

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Carlo Santambrogio, University of Milano-Bicocca, ITALY
MRF Instrument	**Mass Spectrometer 2** **Days Requested:** 10
Special requirements:	

SAMPLE

Material	Prion protein	Ligand 1	Ligand 2
Formula	C688H1031N203O212S9	C19H17BrN3O3	C18H14Br2N3O
Forms	Liquid	Friable powder	Friable powder
Volume	2 ml	1.5 ml	1.5 ml
Weight	2 mg	1 mg	1 mg
Container or substrate	Plastic tube	Plastic tube	Plastic tube
Storage Requirements	Storage temperature -80°C	Storage temperature -20°C	Storage temperature -20°C

SAMPLE ENVIROMENT

Temperature Range	- K	- K	- K
Pressure Range	- mbar	- mbar	- mbar
Magnetic field range	- T	- T	- T
Standard equipment	None	None	None
Special equipment	None	None	None

SAFETY

Prep lab needed	No	No	No
Sample Prep Hazards	None	None	None
Special equip. reqs	None	None	None
Sensitivity to air	No	No	No
Sensitivity to vapour	No	No	No
Experiment Hazards	None	None	None
Equipment Hazards	-	-	-
Biological hazards	None	None	None
Radioactive Hazards	None	None	None
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	Disposed by IS

1. Background and Context

Prion diseases involve the misfolding of the cellular prion protein (PrP) into an infectious form (PrPSc) that accumulates in the brain and propagates by converting normal PrP into PrPSc (Prusiner, doi: 10.1073/pnas.95.23.13363). Traditional therapies targeting PrPSc have been unsuccessful, highlighting the need for new approaches. Our group has developed a novel drug discovery method called Pharmacological Protein Inactivation by Folding Intermediate Targeting (PPI-FIT), which uses Molecular Dynamics (MD) simulations to target and degrade protein folding intermediates (Spagnolli doi: 10.1038/s42003-020-01585-x). Native mass spectrometry (native MS) will be key in characterizing PPI-FIT-derived compounds by revealing their binding affinities and conformational effects, essential for drug development.
The PPI-FIT approach led to the discovery of SM875, a small molecule that reduces PrP levels, hindering prion propagation. During preclinical development, understanding a compound's interaction with its target protein is crucial. Traditional methods like X-ray crystallography and NMR are unsuitable for protein folding intermediates due to their tendency to aggregate. Native MS, however, can directly assess ligand-binding even for poorly soluble targets. This research aims to leverage native MS to characterize the interaction between SM875 and the PrP folding intermediate.
This line of research is currently funded by the following grants:
1. Telethon Multi-round Call, Grant n. GGP20043.
2. PRIN, Bando 2022. Grant n. 2022PP8WNZ.
Two PhD students of the University of Trento are currently working on the project.
Links with industry:
The PPI-FIT technology is currently exploited by the startup company Sibylla Biotech (www.sibyllabiotech.it). The journey of PPI-FIT's discovery, as well as the subsequent establishment and growth of Sibylla, have been recently documented in two Nature Outlook articles: (https://www.nature.com/articles/d41586-021-01668-7; https://www.nature.com/articles/d41586-023-01649-y).

2. Proposed experiment

The aims of the proposed experiments are: i) assessing the exact mass and confirming the amino acid sequence of the recombinant, soluble domain of PrP^C produced in our laboratory; ii) identifying the conformational components of the ensembles in solution and provide quantitative estimates of the relative amount and solvent-accessible surface area of each component; iii) probing the interaction with ligands assessing stoichiometry, relative affinity and conformational specificity.
These aims require a native-MS approach because of its unique capability of dissecting and characterizing components of heterogenous systems. In particular, the MS2 unit conjugates the possibility to perform such analyses with the ultra-high resolution offered by the Orbitrap technology. Finally, the MS2 team has strong experience in the analysis of ligand binding by native MS (Santambrogio, doi.org/10.1002/jms.3237).
Previous computational modelling predicts ligand binding by a PrP folding intermediate at atomic resolution (Spagnolli, doi: 10.1038/s42003-020-01585-x) and in-cell biochemical validation has been provided. It is therefore important to provide direct in-vitro evidence and structural characterization of the complex.

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The data will be analysed by the MS2 unit, providing the qualitative and quantitative characterization described above.

3. Summary of previous experimental proposals or characterisation

This is our first experiment with ISIS@MACH ITALIA.
The recombinant protein and the ligands will be produced in our laboratory by established procedures. Purity will be checked by denaturing gel electrophoresis and liquid chromatography. The activity of the ligands on in-vitro protein aggregation and in-vivo protein expression will be assessed.

4. Justification of experimental time requested

MS2 unit provides native-MS measurements, ultra-high resolution, top-down fragmentation possibility, together with software for data analysis and interpretation.
We will have a total of one protein sample (soluble domain of PrP^C) and two small molecules (active ligand and inactive ligands). The protein will be tested alone and with each ligand for a total of four experimental conditions (non-denaturing, partially-denaturing, fully-denaturing and reducing). For each condition, we expect 4 h instrumental setup and conformational characterization, and 8 h measurements at variable ligand concentration (for each ligand), for a total of 10 working days.

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Experiment Proposal

Experiment number GP2024127

Principal investigator	Dr Fabrizio Gelain, Fondazione IRCCS Casa Sollievo della Sofferenza, ITALY
Co-investigator (*)	Dr Carlo Santambrogio, University of Milano-Bicocca, ITALY
Co-investigator	Professor Rita Grandori, University of Milano-Bicocca, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Self-assembling peptidonucleic acids as novel biomaterials for tissue engineering
MRF Instrument	**Mass Spectrometer 2**
Days requested:	10
Access Route	Direct Access
Previous GP Number:	No
Science Areas	Biology and Bio-materials
DOI:	-
Sponsored Grant	None
Sponsor:	-
Grant Title	-
Grant Number:	-
Start Date	-
Finish Date:	-
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

Peptidonucleic acids (PNAs) are synthetic analogues of DNA and RNA, featuring a peptide backbone instead of the sugar-phosphate backbone. This unique structure grants PNAs high stability and strong binding affinity to complementary nucleic acid sequences. PNAs are valuable in biomedical applications due to their resistance to enzymatic degradation and ability to form stable hybrids with DNA or RNA. They are utilized in diagnostics, antisense therapies, and molecular biology research for gene regulation and detection of genetic mutations. In order to create innovative constructs for promising bio-materials, we designed complementary PNA molecules connected by rigid peptidic linkers (PNA-L-PNA), with the aim of preventing intra-hybridization and favor the formation of inter-molecular interactions. The goal of this proposal is the employment of native mass spectrometry to assess the self assembly properties of distinct PNA-L-PNAs and study their stability in gas-phase.

Publications -

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E-platform: No

Instruments	
Days Requested:	
Access Route	
Previous RB Number:	
Science Areas	
DOI:	
Sponsored Grant	
Sponsor:	
Grant Title	
Grant Number:	
Start Date	
Finish Date:	
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Carlo Santambrogio, University of Milano-Bicocca, ITALY
MRF Instrument	**Mass Spectrometer 2**
Days Requested:	10
Special requirements:	

SAMPLE

Material	PNA1	PNA2	PNA3
Formula	C117H151N63O31	C115H150N58O32	C170H214N88O48
Forms	Friable powder	Friable powder	Friable powder
Volume	1.5 ml	1.5 ml	1.5 ml
Weight	1 mg	1 mg	1 mg
Container or substrate	Plastic tube	Plastic tube	Plastic tube
Storage Requirements	Storage temperature -20°C	Storage temperature -20°C	Storage temperature -20°C

SAMPLE ENVIROMENT

Temperature Range	- K	- K	- K
Pressure Range	- mbar	- mbar	- mbar
Magnetic field range	- T	- T	- T
Standard equipment	None	None	None
Special equipment	None	None	None

SAFETY

Prep lab needed	No	No	No
Sample Prep Hazards	None	None	None
Special equip. reqs	None	None	None
Sensitivity to air	No	No	No
Sensitivity to vapour	No	No	No
Experiment Hazards	None	None	None
Equipment Hazards	-	-	-
Biological hazards	None	None	None
Radioactive Hazards	None	None	None
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	Disposed by IS

1. Background and Context

Peptidonucleic acids (PNAs) are synthetic analogues of DNA and RNA, featuring a peptide backbone instead of the sugar-phosphate backbone. This unique structure grants PNAs high stability and strong binding affinity to complementary nucleic acid sequences. PNAs are valuable in biomedical applications due to their resistance to enzymatic degradation and ability to form stable hybrids with DNA or RNA. They are utilized in diagnostics, antisense therapies, and molecular biology research for gene regulation and detection of genetic mutations.
Our group is involved in the investigation of PNAs to develop innovative biomaterials for cell-growth templates and biosensors.
This line of research has received funding from the European Union's Horizon Europe research and innovation program under the EIC grant agreement No. 101046894 “Synergy Project”.
In this project area work 1 junior postdoc (molecular modelling), 2 senior postdocs (molecular modeling and peptide/PNA synthesis & characterization).

2. Proposed experiment

The aims of the proposed experiments are: i) assessing the exact mass and confirming the sequence of several PNAs produced in our laboratory; ii) probing the interaction between PNAs with complementary sequences and determining stoichiometry and specificity; iii) probing the stability of PNA complexes to induced dissociation in the gas phase, as a function of PNA length and sequence.
These aims require a native-MS approach because of its unique capability of dissecting and characterizing non-covalent complexes of macromolecules in heterogeneous systems. In particular, the MS2 unit of ISIS@MACH ITALIA conjugates the possibility to perform such analyses with the ultra-high resolution offered by the Orbitrap technology. Finally, the MS2 team has strong experience in the analysis of macromolecular binding by native-MS.
Previous AA-MDs assessed the propensity of peptide-PNA molecules to spontaneously form aggregates in water solutions at neutral pH. Later on, CG-MDs were also introduced to examine the degree of flexibility of different peptidic linkers in bigger systems. We selected two promising candidates to be used as rigid linkers flanked by PNA molecules, capable of preventing intra-hybridization. These results have to be confirmed in the empirical world.
The data will be analysed by the MS2 unit, providing the qualitative and quantitative characterization described above.

3. Summary of previous experimental proposals or characterisation

This is our first experiment with ISIS@MACH ITALIA.
The PNAs will be produced in our laboratory by established procedures. Purity will be checked by HPLC and LC-MS. Complementary techniques performed in our lab comprise also AFM, SEM and ATR-FTIR.

4. Justification of experimental time requested

MS2 unit provides native-MS measurements, ultra-high resolution, top-down fragmentation possibility, together with software for data analysis and interpretation.

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We will have three PNA sequences. The PNAs will be tested alone and mixed, for a total of 6 conditions. We expect 1 day of general instrumental setup, 6 days of measurements at variable PNA concentration, and 3 days for the experiments of complex dissociation. The entire experimental plan will cover 10 working days.

Experiment Proposal

Experiment number GP2024144

Principal investigator	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY
Co-investigator (*)	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Charles Cockell, University of Edinburgh, UNITED_KINGDOM
Co-investigator	Dr Jens Holtvoeth, Teesside University, UNITED_KINGDOM
Co-investigator	Professor Silvia Licoccia, University of Rome Tor Vergata, ITALY
Co-investigator	Miss Julia Puputti, Boulby Underground Laboratory STFC, UNITED_KINGDOM
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Biogeochemical Multi-Instrumental Study of Rocks and Microbial Biomass in Zechstein Salt Deposits of Boulby using the Mass Spectrometer 2
MRF Instrument	**Mass Spectrometer 2** — **Days requested:** 2
Access Route	Direct Access — **Previous GP Number:** No
Science Areas	Biology and Bio-materials, Chemistry, Environment, Materials — **DOI:** -
Sponsored Grant	Yes — **Sponsor:** CNR
Grant Title	- — **Grant Number:** -
Start Date	- — **Finish Date:** -
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

Our aim is to investigate the potential for preserving biological material in ancient salt deposits, with a focus on the Zechstein salt deposits in Boulby Mine (UK), offering insights into the environmental conditions of the Zechstein Sea ~250 million years ago. The study employs a multi-instrumental approach, combining non-destructive and destructive analyses to correlate biomolecule presence with mineral phases and elemental compositions. By analyzing a range of biomarkers the research aims to create a detailed biogeochemical fingerprint of fossil microbial biomass. This research contributes to both astrobiology and our understanding of ancient terrestrial environments. We propose a multi-instrumental approach involving by combining non-destructive and destructive analyses exploiting the analytical instrumental suite at IM@IT and ISIS facilities. In this specific proposal we propose to use of Mass Spectrometer 2 to detect both fatty acids and ancient proteins.

Publications

Cockell, C. et al. (2020) Astrobiology 20, 864-877
Gasparri, R. et al (2022) J. of Breath Research 16.4, 046008

Instruments	**INES** — **Days Requested:** 3
Access Route	Direct Access — **Previous RB Number:**
Science Areas	— **DOI:**
Sponsored Grant	Yes — **Sponsor:** CNR
Grant Title	- — **Grant Number:**
Start Date	- — **Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
MRF Instrument	**Mass Spectrometer 2** — **Days Requested:** 2
Special requirements:	

SAMPLE

Material	slabs of salt	-	-
Formula	salt (NaCl) sediments and organic phases	-	-
Forms	Friable powder		
Volume	100-150 cc		
Weight	100-150 g		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	273 - 320 K	-	-
Pressure Range	1000 - 1010 mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	No	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	Yes	-	-
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Biogeochemical Multi-Instrumental Study of Rocks and Microbial Biomass in Zechstein Salt Deposits of Boulby using Mass Spectrometer 2

1. Background and Context

Can the remains of biological material be preserved in salts many hundreds of millions of years old and what signatures can be preserved? To answer these questions, we must be able to probe the chemical and physical conditions at small scales in ancient salt deposits to understand the geological context of biomolecular preservation. We aim to produce a biogeochemical and molecular fingerprint of fossil microbial biomass and inorganic rock samples in the Zechstein salt deposits of Boulby Mine, North Yorkshire, UK through analysing biomarkers, specifically, alkyl lipids (alkanes, fatty acids and alcohols, steroids), glycerol dialkyl glycerol tetraethers (GDGTs), ancient DNA (aDNA) and proteins. This research is timely as the outcomes will help to interpret Raman spectroscopy data produced from the same material. Raman spectroscopy will be one of the analytical tools aboard the next generation Mars rovers. Martian evaporites are prime targets in the search for extra-terrestrial life since the last places where microbial life could have existed on Mars would have been the evaporating oceans. In addition to astrobiology, outcomes are expected to contribute insights into late Permian hydrology and paleoecology. Biomarker distributions in the salt at Boulby mine and particularly in backfilled desiccation cracks can provide information on the environmental conditions in and around the Zechstein Sea ~250 million years ago. The site represents a shallow near shore setting of the Zechstein Basin, with exposure of the evaporite surfaces during sea-level low stand. The presence of biomarkers originating from both microbes and plants in the Boulby salt has already been demonstrated [1]. Upscaling of the extraction procedure and an improved extraction protocol is expected to produce sufficient material for compound-specific carbon and hydrogen isotope analyses ($d^{13}C$, $d^{2}H$) of leaf wax-derived compounds, which will provide information on continental plant types and aridity. In this context the knowledge of the exact spatial distribution and inorganic chemical composition of organic matter in the salt on the atomic scale would help much more targeted analyses. For example, leaf waxes may be associated to different material and specific sites compared to microbial membrane lipids. Thus, we aim to use analytical instruments of IM@IT and neutron beamlines of ISIS Facilities. Due to the age of the samples, a good understanding of the exact spatial distribution of the organic matter and its association the inorganic phases of the salt would greatly support targeted analyses. This would allow us to select specific sub-samples with higher organic yields for biomarker investigations and compound-specific isotope analyses, in particular. In addition to targeting the analysis, the physical and chemical context of the biomolecules (or even a lack of them) provides essential information to explain how physical and chemical conditions influence the fate of biomolecules and their potential or long-term preservation over geological time scales. For example, is the presence of any putative biomarkers associated with specific elements or mineral phases? We can answer this by correlating the presence of biomolecules with mineral phases and elemental composition determining using small/wide angle X-ray Scattering by means of SAXS GISAXS; hard X-ray Fluorescence (2D/3D XRF), using RETINA and Multipurpose X-ray diffraction; and neutron diffraction combined with tomography, using INES and IMAT neutron beamlines. Does the oxidation state of elements, for example iron, influence the chemical environment and thus the presence and preservability/stability of biosignatures in the salt over time? We will answer this by correlating the presence of any biosignatures to the elemental oxidation state in the same location at the relevant scales using mineralogical information. Therefore, we propose a multi-instrumental approach involving a combination of non-destructive and destructive analyses exploiting the analytical instrumental suite at IM@IT and ISIS facilities. All analyses will be using the same sample materials, first, for non-destructive and then destructive methods, to maximise the complementary character of the resulting data sets. On one hand, for the non-destructive analyses of the samples, we use the analytical suite of instruments of IM@IT and ISISI Facilities indicated two paragraphs above. The minerals in each salt sample will be quantified using combined Rietveld refinement of X-rays and neutron diffraction data, using laser ablation. On the other hand, due to the very low yields of the organic phase and its fine dispersal in the salt, the samples to be analysed will consist of small slabs of salt of 100-150g, representing two distinct types of material: relatively pure evaporite material and desiccation crack backfill material. The pure evaporite material will be mainly sodium chloride, containing lenses of clay-rich material and isolated potassium chloride crystals. It is expected to include an organic phase originating predominantly from extremophile biomass and some eolian terrigenous input. The slightly darker coloured material from the desiccation cracks, on the other hand, is expected to include higher proportions of terrigenous organic particles and fragments of biofilm from the salt surface that were resuspended and washed into the cracks during rising water level. For the (destructive) analysis of the biomarkers, originating from microorganisms, terrestrial vegetation or processing-related contamination, like alkanes, alcohols, and ketones we propose to use Gas Chromatography – Ion Mobility Spectrometer (GC-IMS), steranes and GDGTs will be analysed by normal-phase UHPLC at Bristol University; for the aDNA analysis, using DNA sequencing NGS and for the analysis of the ancient proteins using the Mass Spectrometer 2. Genetic, lipid, and proteomic and volatilomic data will then be cross compared to verify their consistency with the possible extremophile species identified. This research is embedded in a wider collaborative attempt to understand extremophile ecology through a comparison with lipid, proteins and DNA data of modern microbes living on the salt surfaces and in brines in Boulby mine. We aim to see if microbial communities adapt to changes in brine salinity and/or ion composition (chloride vs. sulphate, sodium vs. potassium) either by individual species changing their cell membrane properties or by shifts in species distribution. This is a collaboration with Teesside University, the UKRI-STFC Underground Lab. Boulby, the UK Centre for Astrobiology at Edinburgh University, NASA Jet Propulsion Lab, Bristol University, the University of Bern, University of Rome Tor Vergata, the IM@IT and ISIS Facilities. It is supported by the Seedcorn Funding scheme of Teesside University to produce pilot data for a larger proposal to fund PhD projects at Teesside and Edinburgh Universities

2. Proposed experiment using the Mass Spectrometer 2

In this experiment we aim to perform mass spectrometry measurements to study both fatty acids and ancient proteins in the slabs of salt samples coming from Boulby Mine, North Yorkshire, UK. Results of this experiment will be cross compared to verify consistency with data obtained by GC-IMS and by DNA Sequencing NGS instruments operating.

3. Justification of experimental time requested

The propose to use the Mass Spectrometer 2 to detect both fatty acids and ancient proteins in a total of four salt samples. After discussion with instrument scientist, we request **2 days** for acquisition and data analysis.

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Multipurpose X-Ray Diffractometer

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Experiment Proposal

Experiment number GP2024038

Principal investigator	Professor Domenico Lo Vetro, Università di Firenze, ITALY
Co-investigator (*)	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Alessandro Cianchi, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Exploring environmental dynamics in ancient remains before and after the last glacial maximum using X-ray Diffraction
MRF Instrument	**Multipurpose X-ray Diffractometer** **Days requested:** 3
Access Route	Direct Access **Previous GP Number:** No
Science Areas	Biology and Bio-materials, Environment **DOI:** No
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	Our aim is to study environmental ancient sediments coming from sediments extracted in the Romito cave (Cosenza, Italy) before and after the Last Glacial Maximum by multi-instrumental approach. The characterization of these samples will assess first the mineralogy composition of the sediment by means of X-ray diffraction and Small/Wide Angle X-ray scattering measurements. Complementary neutron diffraction data on the same set of samples will be measured at the INES beamline, ISIS neutron and muon source (UK). The minerals in each sediment sample will be quantified using Rietveld refinement of X-rays and neutron diffraction data and cross- compared to verify consistency. Hence, we aim here to request access to the X-ray diffraction instrument (Multipurpose X-ray Diffractometer) operating at the CNR-ICMATE Unit of IM@IT.
Publications	Berto et al. 2022. Archaeological and Anthropological Sciences. Vol. 14, article N. 127, (2022) López-García et al. 2014. Palaeogeography, Palaeoclimatology, Palaeoecology, Vol 251, Issues 3–4, Pages 500-526 Scorrano et al. 2022. Communications Biology, Vol 5, Article N 1262 (2022)

Instruments	**INES**	**Days Requested:** 3
Access Route	Direct Access	**Previous RB Number:** No
Science Areas		**DOI:** No
Sponsored Grant	None	**Sponsor:**
Grant Title	-	**Grant Number:**
Start Date	-	**Finish Date:**
Similar Submission?		
Industrial Links		

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Sample record sheet

Principal contact Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY
MRF Instrument **Multipurpose X-ray Diffractometer** **Days Requested:** 3
Special requirements:

SAMPLE

Material	Sediments/remains	Sediments/remains	Sediments/remains
Formula	organic (collagen) and sand/limestone mixtures	organic (collagen) and sand/limestone mixtures	organic (collagen) and sand/limestone mixtures
Forms	Solid	Solid	Solid
Volume	4-10 cc	4-10 cc	4-10 cc
Weight	2-10 g	2-10 g	2-10 g
Container or substrate	Sterile tube or aluminum foil	Sterile tube or aluminum foil	Sterile tube or aluminum foil
Storage Requirements	Freezer (-20C)	Freezer (-20C)	Freezer (-20C)

SAMPLE ENVIROMENT

Temperature Range	273 - 320 K	273 - 320 K	273 - 320 K
Pressure Range	1000 - 1010 mbar	1000 - 1010 mbar	1000 - 1010 mbar
Magnetic field range	- T	- T	- T
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	Yes	Yes
Sample Prep Hazards	No	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	No	No
Sensitivity to vapour	No	No	No
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	No	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	Returned to user by instrument scientist (when inactive)	Returned to user by instrument scientist (when inactive)

Exploring environmental dynamics in ancient remains before and after the last glacial maximum using X-ray Diffraction

Background and Context

Ancient environmental DNA (aDNA) refers to genetic material obtained from environmental samples such as soil, sediment, ice and water, which is thousands or millions of years old [1]. The study of ancient DNA is fascinating because it allows us to reconstruct past ecosystems, understand evolutionary processes and trace the impacts of climate and environmental changes on biodiversity over the millennia. This field is particularly timely, as highlighted by a recent publication in Nature detailing the oldest DNA ever recovered from the environment: 2-million-year-old samples that allowed researchers to reconstruct the ecosystem in Greenland [1].

The broader relevance of ancient DNA research lies in its ability to shed light on the complex interactions between climate, environment and living organisms over geological time scales. By understanding past ecosystems, we gain insights into species resilience and vulnerability, which can guide current biodiversity conservation strategies. This proposal aims to characterize samples coming from sediments extracted in the Romito cave (Cosenza, Italy, see Figure 1) [2, 3] before and after the Last Glacial Maximum (LGM) by multi-instrumental approach. The reason of this is twofold. The LGM, when ice sheets were at their maximum extent, was a period of significant climatic and environmental shifts with profound impacts on global ecosystems and human populations. In an era marked by rapid climate change, insights from the LGM can inform our understanding of how ecosystems and species, including humans, responded to extreme climatic conditions. From the other, the Romito cave is one of the most significant Upper Palaeolithic archaeological sites on the Italian peninsula with a well-dated stratigraphy spanning from ~24,000 to 6,000 years before present (BP) (Figure 1d).

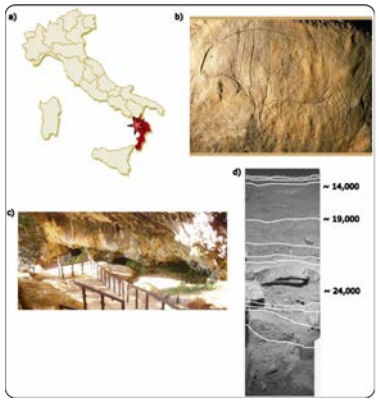

Figure 1. a) Location of Romito cave; b) rock art (Bos primigenius) in the rock-shelter outside the cave; c) cave entrance d) general stratigraphic sequence modified from Blockley et al. (2018).

To this end, we will study two samples from the oldest layer (pre-LGM, ~24000 years BP) of the sediment, two samples from the LGM (~19000 years BP), and two samples post-LGM (~14000 years BP). The characterization of these samples will be assessing first the mineralogy composition of the sediment by means of X-ray diffraction (Multipurpose X-ray Diffractometer instrument, CNR-ICMATE Unit) and Small/Wide Angle X-ray Scattering (SAXS GISAXS instrument, CSGI-Unit) measurements. Complementary neutron diffraction data on the same set of samples will be measured at the INES beamline, ISIS neutron and muon source (UK). The minerals in each sediment sample will be quantified using Rietveld refinement of X-rays and neutron diffraction data and cross-compared to verify consistency. Later, the presence of organic compounds [4] will be inferred by Fourier-transform infrared spectroscopy measurement on the FT-IR Nicolet instrument available at the Rome Tor Vergata Unit. To follow we will perform shotgun sequencing of aDNA, using DNA Sequencing NGS of Rome Tor Vergata Unit and finally retrieve all the proteins in the remains using Mass Spectrometer 2 at the University of Milano Bicocca Unit which will be useful to support the DNA data [5].

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Proposed experiment

In this experiment we aim to perform X-ray diffraction measurements on the Multipurpose X-ray Diffractometer instrument (CNR-ICMATE Unit) to study the mineralogy composition of n. 6 samples coming from sediments extracted in the Romito cave. Two samples have been extracted by sediments in the oldest layer (pre-LGM, ~24000 years BP), n. 2 samples from the LGM (~19000 years BP), and two samples post-LGM (~14000 years BP). Results of this experiment will be compared with Small/Wide Angle X-ray Scattering measurements performed on the same set of samples with the SAXS GISAXS instrument of the CSGI-Unit and with complementary neutron diffraction data still on the same samples collected at the INES beamline, ISIS neutron and muon source (UK). The minerals in each sediment sample will be quantified using Rietveld refinement of X-rays and neutron diffraction data and cross-compared to verify consistency.

Justification of experimental time requested

Each of the n. 6 sample will be enclosed in a sterile tube with about 2 g (4 cc) of sediment, and it will be maintained at -20 °C temperature to preserve aDNA during the measurements. We envisage, after discussion with the instrument scientist, to measure n. 2 samples per day on the instrument. Hence, we request a total of 3 days of instrument time including set-up and calibration time.

References

[1] Kjær et al., Nature **612** (2022), p. 283–291.
[2] Blockley et al. 2018. Quaternary Science Reviews, 184: 5-25.
[3] Craig et al., 2010. Journal of Archaeological Science, 37: 2504-2512.
[4] Scorrano et al., 2015. Annals of Human Biology, 42: 10-19.
[5] Scorrano et al. 2022. Communications Biology, 5: 1262.

Experiment Proposal

Experiment number GP2024092

Principal investigator	Professor Marco Taddei, University of Pisa, ITALY
Co-investigator (*)	Dr Jacopo Perego, University of Milano-Bicocca, ITALY
Co-investigator	Professor Valentina Crocellà, University of Torino, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	In situ powder X-ray diffraction of flexible metal-organic frameworks during gas adsorption
MRF Instrument	**Multipurpose X-ray Diffractometer** **Days requested:** 5
Access Route	Direct Access **Previous GP Number:** No
Science Areas	Chemistry **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

We propose to perform an in situ powder X-ray diffraction experiment on metal-organic frameworks (MOFs) that display step-shaped CO2 adsorption isotherms, with the aim of gaining insights into the structural response induced by gas adsorption. The MOFs object of the experiment were recently discovered by us and belong to two classes: the first one is the perfluorinated analogue of Al-based MIL-53, the second is a series of isoreticular Cu-based bisimidazoles containing monovalent inorganic anions. This is motivated by the interest in understanding how the nature of the organic influences the flexible behaviour of these MOFs, so as to gain the ability to tune their adsorption properties and gas separation performance.

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Jacopo Perego, University of Milano-Bicocca, ITALY
MRF Instrument	**Multipurpose X-ray Diffractometer** **Days Requested:** 5
Special requirements:	

SAMPLE

Material	Al, tetrafluoroterephthalic acid	CuClO4, 2-trifluoromethyl-1,4-Bis(1H-imidazol-1-yl)benzene	CuClO4, 2-methyl-1,4-Bis(1H-imidazol-1-yl)benzene
Formula	AlF4C8O5H	CuC26H20N8F6Cl2O4	CuC26H24N8Cl2O4
Forms	Friable powder	Friable powder	Friable powder
Volume	1 cc	1 cc	1 cc
Weight	200 mg	200 mg	200 mg
Container or substrate	None	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	195 - 298 K	195 - 298 K	195 - 298 K
Pressure Range	0 - 2000 mbar	0 - 2000 mbar	0 - 2000 mbar
Magnetic field range	- T	- T	- T
Standard equipment	Gas Handdling, Furnace	Gas Handdling, Furnace	Gas Handdling, Furnace
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	Yes	Yes
Sample Prep Hazards	No	No	No
Special equip. reqs	No	No	No
Sensitivity to air	No	No	No
Sensitivity to vapour	No	No	No
Experiment Hazards	No	No	No
Equipment Hazards	-	-	-
Biological hazards	No	NO	No
Radioactive Hazards	No	NO	No
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	Disposed by IS

1. Background and Context

"Phase-change" adsorbents are attracting considerable interest for gas separation processes, because they display step-shaped adsorption isotherms with steep gas uptake when a threshold partial pressure of adsorbate is reached, with benefits in terms of achievable working capacity, selectivity and energy efficient regeneration. In the case of metal-organic frameworks (MOFs), this behaviour is determined by an adsorption-induced structural rearrangement, which can be either a breathing effect, a cooperative adsorption mechanism, rotation of organic rings within the framework, or combinations thereof.[1]

Within the frame of the national PRIN2020 project "doMino", we are pursuing the development of novel "phase-change" MOFs based on different combinations of metals and organic linkers to be employed as fillers for mixed-matrix membranes for CO_2 capture. We are interested in understanding the structural factors underpinning the peculiar adsorption behaviour displayed by these materials, with the ultimate aim of gaining the ability to finely tune the atomic structure and, as a result, the gas separation performance.

One approach is to develop (per-)fluorinated analogues of known MOFs, which recently led to the discovery of the Al^{III}-based MOF F4_MIL-53(Al).[2] This MOF displays purely temperature-induced breathing behaviour and step-shaped N_2, Ar and CO_2 isotherms with small hysteresis in desorption (Figure 1). A comparison with the parent, non-fluorinated MIL-53(Al), suggests that the breathing behaviour is strongly affected by the nature of the linker.

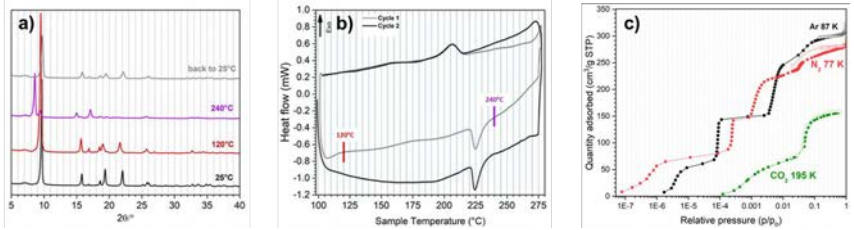

Figure 1. Variable temperature PXRD patterns collected with Cu Kα radiation (a), differential scanning calorimetry curves (b), and adsorption isotherms of Ar (87 K, black), N_2 (77 K, red) and CO_2 (195 K, green) (c) collected on F4_MIL-53(Al).

Another approach is based on the combination of Cu^{II}, neutral bisimidazolate ligands and inorganic anions, such as BF_4^-, ClO_4^- and NO_3^-. Five isoreticular MOFs with a pillared-layered structure were obtained, which display "phase-change" CO_2 adsorption behaviour dependent on the nature of the functional groups present on the organic linker. (Figure 2).

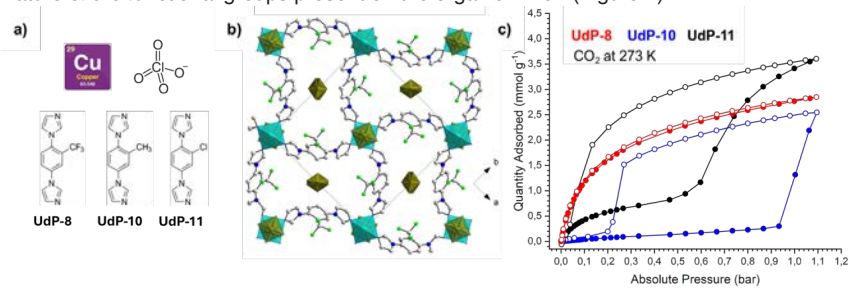

Figure 2. Building blocks for the synthesis of UdP-X series of MOFs (a), crystal structure (b), and CO_2 adsorption isotherms collected at 273 K on UdP-8 (red), UdP-10 (blue) and UdP-11(black).

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2. Proposed experiment

We propose to perform an *in situ* powder X-ray diffraction (PXRD) study using the multipurpose X-ray diffractometer available in ISIS@MACH ITALIA to investigate the structural response of the above mentioned MOFs when they are activated and exposed to CO_2. We will first follow the activation process, conducted under vacuum at elevated temperature, and then dose CO_2 at controlled pressure in the range between 10^{-3} and 1 bar at three temperatures comprised between 195 and 298 K. Should the materials retain their long-range order, the data collected will be analysed by performing indexing and *ab initio* whole powder pattern fitting to identify the symmetry and unit cell parameters of the crystalline phases observed during the study. The data collected during the study will be combined with evidence from spectroscopic techniques (infrared, solid state nuclear magnetic resonance), adsorption microcalorimetry and density functional theory calculations to provide a clear picture of the adsorption mechanisms existing in the two different classes of materials under investigation.

3. Summary of previous experimental proposals or characterisation

The samples have preliminarily been characterised by powder/single crystal XRD, gas sorption analysis, scanning electron microscopy, thermogravimetric analysis, differential scanning calorimetry.

4. Justification of experimental time requested

We request five days of machine time: one day for each sample described in section 1, *i.e.*, F4_MIL-53(Al), UdP-8, UdP-10 and UdP-11, plus one additional day for the collection of high-resolution patterns in specific conditions to perform indexing and profile fitting.

References

[1] Scheeman et al. *Chem. Soc. Rev.,* **2014**, *43,* 6062-6096.
[2] Morelli Venturi et al. *Mol. Syst. Des. Eng.*, **2023**, *8*, 586-590.

Experiment Proposal

Experiment number GP2024110

Principal investigator	Professor Gabriele Gentile, University of Rome Tor Vergata, ITALY
Co-investigator (*)	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Carla Andreani, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Multi-instrumental characterization of Oniscidean isopods to establish the nature and function of their cuticle and tubercles: XRD measurements
MRF Instrument	**Multipurpose X-ray Diffractometer** **Days requested:** 3
Access Route	Direct Access **Previous GP Number:** No
Science Areas	Biology and Bio-materials, Physics **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

Andronicus is a genus of oniscidean isopods that includes less than a dozen species, most of which are confined to caves in the Italian Pre-Alps. Andronicus offers the unique opportunity to investigate the nature of the cuticle in cave (A. brentanus) and non-cave-dwelling species (A. dentiger), within the same evolutionary lineage, characterizing mineralization in the different species. The proposed investigation will also extend to including tubercles on the surface of the body, the nature of which, currently unknown, could be of a sensorial nature. Preliminary observations would suggest the presence of a possible innervation at the base of these tubercles, which if confirmed, would prove their nature and function. For the characterization on the mineralization of the cuticle and tubercles we envisage to use EDX, FT-IR, Raman, and X-ray diffraction, whereas the morphology characterization will be done by SEM, TEM and nano-XCT. Here, this proposal is focussed on the XRD analysis.

Publications

Vittori, M. et al., Arthropod Struct Dev. 46 (2016), pp. 96-107.
Gentile, G. and Allegrucci, G., International Journal of Speleology 26 (1997), pp. 47-61.
Neues, F. et al., Cryst. Eng. Comm. 9 (2007), pp. 1245-1251.

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY		
MRF Instrument	**Multipurpose X-ray Diffractometer**	**Days Requested:** 3	
Special requirements:			

SAMPLE

Material	Oniscidean isopod	-	-
Formula	Organic material, Calcite	-	-
Forms	Solid		
Volume	0.03 cc		
Weight	1-2 g		
Container or substrate	-	-	-
Storage Requirements	Freezer (-20C)	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	-	-

Multi-instrumental characterization of Oniscidean isopods to establish the nature and function of their cuticle and tubercles: XRD measurements

1. Background and Context

Among Crustaceans, oniscidean isopods are uniquely adapted to terrestrial life, exhibiting strongly mineralized cuticles. Oniscideans include several species adapted to the caves. Among the most important evolutionary adaptations found in troglobitic oniscideans (i.e. bound to cave environments, from which they cannot escape due to strict ecological and physiological constraints) are the thinning of the cuticle with a reduce layer of calcite, although calcium carbonate is present in the exocuticle and the endocuticle [1]. Additionally, other adaptations include the lengthening of the appendages, the loss of the eyes, the development of sensory systems alternative to sight such as hygrosensors and chemosensors, usually located in different areas of the body. *Androniscus* (Fig. 1) is a genus of oniscidean isopods that includes less than a dozen species, most of which are confined to caves in the Italian Pre-Alps. Among these, *Androniscus dentiger* is the one that shows the least constraints, being present even in the most superficial layers of the soil in non-cave environments and showing a wide geographical distribution [2]. Indeed, by combining atomic absorption spectroscopy, thermogravimetry and X-ray diffraction, the composition of cuticles in several isopods has been analyzed [3-4]. The use of high-resolution Raman microscopy enabled the determination of the distribution of different mineral phases in the tergal cuticles of some rollers, clingers, and runners [5,6].

Fig. 1 *Androniscus dentiger* (a) and *Androniscus brentanus* (b). Contrary to the second, the first is not troglobite, is pigmented, has thick cuticle, and shows a prominent single-ommatidium eye (arrow).

Androniscus offers the unique opportunity to investigate the nature of the cuticle in cave (*A. brentanus* and more) and non-cave-dwelling species (*A. dentiger*), within the same evolutionary lineage, characterizing mineralization in the different species. The proposed investigation will also extend to including tubercles (tricorns) on the surface of the body, the nature of which, currently unknown, could be of a sensorial nature. Some preliminary observations would suggest the presence of a possible innervation at the base of these tubercles, which if confirmed, would prove their nature and function. For the characterization on the mineralization of the cuticle and tubercles in the two different species (*A. brentanus and A. dentiger*) we plan to use Energy Dispersive X-ray Analysis (SEM-EDX), Fourier-transform infrared spectroscopy (FT-IR), Raman spectroscopy and X-ray diffraction, whereas the morphology characterization will be done by means of electron microscopy techniques (SEM, TEM) and X-ray nano tomography. The benefit of using a multi-instrumental approach would allow us not only to cross compared results to verify their consistency, but also to investigate the degree of resorption/failure to develop the eye in these isopods species, allowing us to observe the presence of vestigial or residual structures, such as for example the presence of an optic nerve, in the absence of the ommatidium (eyeball).

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2. Proposed experiment

In this specific proposal we aim to use the Multipurpose X-ray Diffractometer instrument available at the CNR-ICMATE Unit for assessing the degree of mineralization of the cuticle and tubercles on a n. 6 *Androniscus brentanus and n. 6 Androniscus dentiger* isopods samples. Results of this experiment will be cross compared to verify consistency with data obtained by separate proposals where we request FT-IR, Raman spectroscopy, SEM-EDX, TEM, and nano XCT measurements on the same set of samples.

3. Justification of experimental time requested

Each of the n. 12 samples of the two Oniscidean isopod species (n. 6 *Androniscus brentanus and n.6 Androniscus dentiger)* will be washed for 1–2 s in double distilled water to remove tissue saline at the surface and then for 2–5 s in 100% methanol to remove water. Specimens will be air dried and stored at −20 °C until its use on the instrument. For the XRD measurement, cuticle and tubercles will be isolated from the sample and reduce in powder form by grinding. We envisage, after discussion with the instrument scientist, to measure n. 4 samples per day on the instrument. Hence, we request a total of 3 days of instrument time including sample preparation, set-up and calibration time.

References

[1] Vittori, M., Tusek-Žnidarič, M. & Štrus, J. (2016) Exoskeletal cuticle of cavernicolous and epigean terrestrial isopods: a review and perspectives. Arthropod Struct Dev. 46(1): 96-107.
[2] Gentile, G. & Allegrucci, G. (1997) Geographic variation and genetic relationships in populations of the *Androniscus dentiger* complex from Central Italy (Isopoda, Oniscidea, Trichoniscidae). International Journal of Speleology, 26: 47-61.
[3] Neues, F., Ziegler, A. & Epple, M. (2007) The composition of the mineralized cuticle in marine and terrestrial isopods: a comparative study Cryst. Eng. Comm. 9: 1245-1251.
[4] Hild, S., Neues, F., Žnidaršič, N., Štrus, J., Epple, M., Marti,O. & Ziegler, Z. (2009) Ultrastructure and mineral distribution in the tergal cuticle of the terrestrial isopod *Titanethes albus* . Adaptations to a karst cave biotope. Journal of Structural Biology 168 (3): 426 – 436.
[5] Hild, S., Marti, O. & Ziegler, Z. (2008) Spatial distribution of calcite and amorphous calcium carbonate in the cuticle of the terrestrial crustaceans *Porcellio scaber* and *Armadillidium vulgare*J. Struct. Biol. 163: 100-108.
[6] Štrus, J., Žnidaršič, N., Hild, S. & Ziegler, A. (2008) Microscopic anatomy and mineral composition of cuticle in amphibious isopods Ligia italica and Titanethes albus (Crustacea: Isopoda) A. Aretz, B. Hermanns-Sachweh, J. Mayer (Eds.), EMC 2008: Life Sciences, Springer Verlag, Berlin:185-186.
[7] Hornung, E. (2011). Evolutionary adaptation of oniscidean isopods to terrestrial life: Structural-physiological-behavioural aspects. Terrestrial Arthropod Reviews. 4: 95-130.

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Experiment Proposal

Experiment number GP2024141

Principal investigator	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Jens Holtvoeth, Teesside University, UNITED_KINGDOM
Co-investigator	Professor Charles Cockell, University of Edinburgh, UNITED_KINGDOM
Co-investigator	Professor Silvia Licoccia, University of Rome Tor Vergata, ITALY
Co-investigator	Miss Julia Puputti, Boulby Underground Laboratory STFC, UNITED_KINGDOM
Co-investigator (*)	Professor Alessandro Cianchi, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Biogeochemical Multi-Instrumental Study of Rocks and Microbial Biomass in Zechstein Salt Deposits of Boulby using the Multipurpose X-ray Diffractometer
MRF Instrument	**Multipurpose X-ray Diffractometer** **Days requested:** 2
Access Route	Direct Access **Previous GP Number:** No
Science Areas	Biology and Bio-materials, Chemistry, Environment, Materials **DOI:** -
Sponsored Grant	Yes **Sponsor:** CNR
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	Our aim is to investigate the potential for preserving biological material in ancient salt deposits, with a focus on the Zechstein salt deposits in Boulby Mine (UK), offering insights into the environmental conditions of the Zechstein Sea ~250 million years ago. The study employs a multi-instrumental approach, combining non-destructive and destructive analyses to correlate biomolecule presence with mineral phases and elemental compositions. By analyzing a range of biomarkers the research aims to create a detailed biogeochemical fingerprint of fossil microbial biomass. This research contributes to both astrobiology and our understanding of ancient terrestrial environments. We propose a multi-instrumental approach involving by combining non-destructive and destructive analyses exploiting the analytical instrumental suite at IM@IT and ISIS facilities. In this specific proposal we propose to use of Multipurpose X-ray Diffractometer to study the mineralogy composition of the samples.
Publications	Cockell, C. et al. (2020) Astrobiology 20, 864-877 Gasparri, R. et al (2022) J. of Breath Research 16.4, 046008

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Professor Alessandro Cianchi, University of Rome Tor Vergata, ITALY
MRF Instrument **Multipurpose X-ray Diffractometer** **Days Requested:** 2
Special requirements:

SAMPLE

Material	slabs of salt	-	-
Formula	salt (NaCl) sediments and organic phases (solid and/or power)	-	-
Forms	Solid		
Volume	100-150 cc		
Weight	100-150 g		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	273 - 320 K	-	-
Pressure Range	1000 - 1010 mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	No	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	Yes	-	-
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Biogeochemical Multi-Instrumental Study of Rocks and Microbial Biomass in Zechstein Salt Deposits of Boulby using Multipurpose X-ray diffractometer

1. Background and Context

Can the remains of biological material be preserved in salts many hundreds of millions of years old and what signatures can be preserved? To answer these questions, we must be able to probe the chemical and physical conditions at small scales in ancient salt deposits to understand the geological context of biomolecular preservation. We aim to produce a biogeochemical and molecular fingerprint of fossil microbial biomass and inorganic rock samples in the Zechstein salt deposits of Boulby Mine, North Yorkshire, UK through analysing biomarkers, specifically, alkyl lipids (alkanes, fatty acids and alcohols, steroids), glycerol dialkyl glycerol tetraethers (GDGTs), ancient DNA (aDNA) and proteins. This research is timely as the outcomes will help to interpret Raman spectroscopy data produced from the same material. Raman spectroscopy will be one of the analytical tools aboard the next generation Mars rovers. Martian evaporites are prime targets in the search for extra-terrestrial life since the last places where microbial life could have existed on Mars would have been the evaporating oceans. In addition to astrobiology, outcomes are expected to contribute insights into late Permian hydrology and paleoecology. Biomarker distributions in the salt at Boulby mine and particularly in backfilled desiccation cracks can provide information on the environmental conditions in and around the Zechstein Sea ~250 million years ago. The site represents a shallow near shore setting of the Zechstein Basin, with exposure of the evaporite surfaces during sea-level low stand. The presence of biomarkers originating from both microbes and plants in the Boulby salt has already been demonstrated [1]. Upscaling of the extraction procedure and an improved extraction protocol is expected to produce sufficient material for compound-specific carbon and hydrogen isotope analyses ($d^{13}C$, $d^{2}H$) of leaf wax-derived compounds, providing information on continental plant types and aridity.

In this context the knowledge of the exact spatial distribution and inorganic chemical composition of organic matter in the salt on the atomic scale would help much more targeted analyses. For example, leaf waxes may be associated to different material and specific sites compared to microbial membrane lipids. Due to the age of the samples, a good understanding of the exact spatial distribution of the organic matter and its association the inorganic phases of the salt would greatly support targeted analyses. This would allow us to select specific sub-samples with higher organic yields for biomarker investigations and compound-specific isotope analyses, in particular. In addition to targeting the analysis, the physical and chemical context of the biomolecules (or even a lack of them) provides essential information to explain how physical and chemical conditions influence the fate of biomolecules and their potential or long-term preservation over geological time scales. For example, is the presence of any putative biomarkers associated with specific elements or mineral phases? We can answer this by correlating the presence of biomolecules with mineral phases and elemental composition determining using small/wide angle X-ray Scattering by means of SAXS GISAXS; hard X-ray Fluorescence (2D/3D XRF), using RETINA and Multipurpose X-ray diffractometer; and neutron diffraction combined with tomography, using respectively INES and IMAT neutron beamlines. Does the oxidation state of elements, for example iron, influence the chemical environment and thus the presence and preservability/stability of biosignatures in the salt over time? We will answer this by correlating the presence of any biosignatures to the elemental oxidation state in the same location at the relevant scales using mineralogical information. Therefore, we propose a multi-instrumental approach involving a combination of non-destructive and destructive analyses exploiting the analytical instrumental suite at IM@IT and ISIS facilities. All analyses will be using the same sample materials, first, for non-destructive and then destructive methods, to maximise the complementary character of the resulting data sets. On one hand, for the non-destructive analyses of the samples, we use the analytical suite of instruments of IM@IT and ISIS Facilities indicated. The minerals in each salt sample will be quantified using combined Rietveld refinement of X-rays and neutron diffraction data, using laser ablation. On the other hand, due to the very low yields of the organic phase and its fine dispersal in the salt, the samples to be analysed will consist of small slabs of salt of 100-150g, representing two distinct types of material: relatively pure evaporite material and desiccation crack backfill material. The pure evaporite material will be mainly sodium chloride, containing lenses of clay-rich material and isolated potassium chloride crystals. It is expected to include an organic phase originating predominantly from extremophile biomass and some eolian terrigenous input. The slightly darker coloured material from the desiccation cracks, on the other hand, is expected to include higher proportions of terrigenous organic particles and fragments of biofilm from the salt surface that were resuspended and washed into the cracks during rising water level. For the (destructive) analysis of the biomarkers, originating from microorganisms, terrestrial vegetation or processing-related contamination, like alkanes, alcohols, and ketones we propose to use Gas Chromatography – Ion Mobility Spectrometer (GC-IMS), steranes and GDGTs will be analysed by normal-phase UHPLC at Bristol University; for the aDNA analysis, using DNA sequencing NGS, and for the analysis of both fatty acids and ancient proteins using the Mass Spectrometer 2. Genetic, lipid, and proteomic and volatilomic data will then be cross compared to verify their consistency with the possible extremophile species identified. This research is embedded in a wider collaborative attempt to understand extremophile ecology through a comparison with lipid, proteins and DNA data of modern microbes living on the salt surfaces and in brines in Boulby mine. We aim to see if microbial communities adapt to changes in brine salinity and/or ion composition (chloride vs. sulphate, sodium vs. potassium) either by individual species changing their cell membrane properties or by shifts in species distribution. This is a collaboration with Teesside University, the UKRI-STFC Underground Lab. Boulby, the UK Centre for Astrobiology at Edinburgh University, NASA Jet Propulsion Lab, Bristol University, the University of Bern, the University of Rome Tor Vergata, the IM@IT and ISIS facilities. It is supported by the Seedcorn Funding scheme of Teesside University to produce pilot data for a larger proposal to fund PhD projects at Teesside and Edinburgh Universitie.

2. Proposed experiment using Multipurpose X-ray diffractometer

In this experiment we aim to perform X-ray diffraction measurements on the Multipurpose X-ray Diffractometer instrument to study the mineralogy composition of the samples. The minerals in each sediment sample will be quantified using Rietveld refinement of X-rays and neutron diffraction data (from INES) and cross-compared to verify consistency with SAXS GISAX outcomes and XRF data taken with RETINA. Furthermore, a complete digital twin of the sample will be obtained by XCT at RETINA and neutron tomography at IMAT before its partial destruction in later characterizations. After that we will process the sample for analysis (destructive) of biomarkers using GC-IMS.

3. Justification of experimental time requested

Salt specimens are small slabs of salt of 100-150g, representing two distinct types of material. We propose to collect the diffraction pattern a duplicate sample of each type of material: relatively pure evaporite material and desiccation crack backfill material. After discussion with the instrument scientist, we request **2 days** of instrument time including set-up and calibration time.

Experiment Proposal

Experiment number GP2024156

Principal investigator	Dr Monica Carosi, Università Roma Tre, ITALY
Co-investigator	Dr Federica Spani, Università Campus Bio-Medico di Roma, ITALY
Co-investigator (*)	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Multi-instrumental characterization of primate baculum and baubellum bone tissues to support functional hypotheses: XRD measurement case
MRF Instrument	**Multipurpose X-ray Diffractometer** **Days requested:** 3
Access Route	Direct Access **Previous GP Number:** No
Science Areas	Biology and Bio-materials, Physics **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	To better understand the function of primate bacula and baubella bone tissues, we aim to study the micro-architecture of the bone focussed for the first time on characteristics related to either observed shapes and physical-chemical features of this tissue. Key aspects include trabecular density and orientation, bone mineral composition, and the distribution of minerals along the bones. The proposed study will involve and multi-instrumental characterization of different bones which will allow us to fine reconstruct a digital twin of these tissues, and for open exploring image-based finite element analysis to assess the mechanical forces involved during copulation. Here, this proposal is focussed on the XRD analysis.
Publications	Spani F, Morigi MP, Bettuzzi M, Scalici M, Carosi M, PLoS ONE 15(1): e0228131. Spani, F., Morigi, M., Bettuzzi, M. et al., Sci Rep 11 (2021), 11245. Reznikov, N., Bilton, M., Lari, L., Stevens, M. M. & Kröger,Science 360 (2018), eaao2189.

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
MRF Instrument **Multipurpose X-ray Diffractometer** **Days Requested:** 3
Special requirements:

SAMPLE

Material	Primate bone tissue	-	-
Formula	Ca10(PO4)6(OH)2	-	-
Forms	Solid		
Volume	0.3 cc		
Weight	0.5 g		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	-	-

Multi-instrumental characterization of primate baculum and baubellum bone tissues to support functional hypotheses: XRD measurement case

1. Background and Context

Inside the external genitals of some placental mammals, including Primates, genital bones are present in one or both sexes: the *baculum* (penile bone; pl. *bacula*) and the *baubellum* (clitoral bone; pl. *baubella*). *Bacula* are common in most primate species, whereas *baubella* are rare. Both bones occur in some species of Hominoidea (the human evolutionary lineage), but not in humans. Although homologous, *baubellum* is only present in species where males have a *baculum,* whereas species with *bacula* may lack *baubella*. Various functions have been proposed for the *baculum* (none for the *baubellum*), however only one is supported by correlational data: baculum supports erection and prevents urethral collapse, aiding sperm transport in species with prolonged copulations and high levels of sexual competition. *In fact, baculum* length positively correlates with copulation duration. Recent studies published the most comprehensive dataset on primate *bacula* and *baubella* occurrence, collecting data from primary literature and samples from fresh cadavers and museum specimens (Natural History Museum La Specola in Italy, the American Museum of Natural History in New York, and the National Museum of Natural History in Washington, DC). Using 3D high-resolution, non-invasive micro-Computed Tomography and a new landmark-free shape analysis (the *alpha*-shape technique), these studies identified three distinct internal and external morphologies in primate *bacula* for the first time.

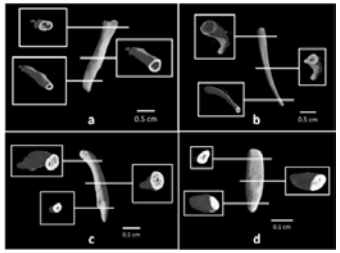

Fig. 1 *3D virtual volumes of 4 different types of primate genital bones, three bacula and one baubellum. For each type, internal structures and cross sections of epiphyses and diaphysis are shown. A: totally hollow structure (Chlorocebus aethiops). B: hollow epiphyses and solid diaphysis with few channels (Otolemur crassicaudatus). C: totally solid structure in both epiphyses and diaphysis with a network of Haversian channels (Papio cynocephalus). D: totally solid structure of baubellum (Sapajus apella) with some Haversian channels*

To better understand the function of primate bacula, the micro-architecture of baculum bone tissue should be investigated focusing for the first time on characteristics related to either observed shapes and mechanical forces exerted on bacula during copulation. Key aspects include trabecular density and orientation, bone mineral composition, and the distribution of minerals along the bones. Potential sex-based differences in these characteristics will aid in interpreting the results. Hence, the proposed study will involve and multi-instrumental approach as follows:

- trabecular density will be assessed by means of quantitative computed tomography (QCT) at different length scale and photon energy which will enable us reconstructing the 3-D bone geometry and volumetric bone mineral density (vBMD);
- trabecular orientation of the bone tissue will be studied in the bulk of the sample by small-angle X-ray scattering (SAXS) to measure crystal shape, their average crystal thickness and their crystal orientation;

- the structure of bone mineral will be assessed by means of X-ray diffraction (XRD) which is considered as the gold standard for this type of measurement;
- for the compositional characterization of the bone tissue we aim to use nuclear magnetic resonance (NMR) which is uses the responses of isotopes to an external magnetic field to generate compositional information about the sample being scanned, and results will be verified for consistency checks with Raman spectroscopy, Fourier transform infrared spectroscopy (FTIR), X-ray Fluorescence (XRF) spectroscopy, and Energy Dispersive X-ray Analysis (SEM-EDX) data that are used here to determine the chemical and molecular signature of the sample.

2. Proposed experiment

In this specific proposal we aim to use the Multipurpose X-ray Diffractometer instrument available at the CNR-ICMATE Unit to perform X-ray diffraction measurement that will allow us characterizing the structure of bone mineral on a n. 3 baculum bone tissue and n. 2 baubella bone tissue. Results of this experiment will be cross compared to verify consistency with SAXS, FT-IR, Raman, and EDX data measured on the same set of samples with different instrument available at the ISIS@MACH ITALIA research infrastructure and requested in separate proposals.

3. Justification of experimental time requested

Each of the n. 5 samples of bone tissue will be fixed on the instrument sample stage, and the crystal phase present in the measured diffractogram will be quantified using Rietveld refinement. Hence, after discussion with the instrument scientist, we request 3 days of instrument time including sample preparation, set-up and calibration time.

References

[1] Reznikov, N., Bilton, M., Lari, L., Stevens, M. M. & Kröger, R. Fractal-like hierarchical organization of bone begins at the nanoscale. Science 360, eaao2189 (2018).
[2] Glimcher, M. J. Bone: Nature of the Calcium Phosphate Crystals and Cellular, Structural, and Physical Chemical Mechanisms in Their Formation.*Rev. Mineral. Geochem.*64, 223–282 (2006).
[3] Delmas, P. D., Tracy, R. P., Riggs, B. L. & Mann, K. G. Identification of the noncollagenous proteins of bovine bone by two-dimensional gel electrophoresis. *Calcif. Tissue Int.***36**, 308–316 (1984).
[4] Schultz NG, Lough-Stevens M, Abreu E, Orr T, Dean MD. The Baculum was Gained and Lost Multiple Times during Mammalian Evolution. Integr Comp Biol. 2016 Oct;56(4):644-56. doi: 10.1093/icb/icw034. Epub 2016 Jun 1. PMID: 27252214; PMCID: PMC6080509.
[5] Spani, F., Morigi, M., Bettuzzi, M. et al. The ultimate database to (re)set the evolutionary history of primate genital bones. Sci Rep 11, 11245 (2021). https://doi.org/10.1038/s41598-021-90787-2

NMR 600 MHz

Experiment Proposal

Experiment number GP2024131

Principal investigator (*) Dr Ilaria Negri, Università Cattolica del Sacro Cuore, ITALY
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator

Experiment title	Study of the chemical and structural characteristics of biodegradable microplastics in contaminated honey bees through NMR spectroscopy		
MRF Instrument	**NMR 600 MHz**	**Days requested:**	3
Access Route	Direct Access	**Previous GP Number:**	-
Science Areas	Biology and Bio-materials, Chemistry, Environment, Materials	**DOI:**	-
Sponsored Grant	None	**Sponsor:**	-
Grant Title	-	**Grant Number:**	-
Start Date	-	**Finish Date:**	-
Similar Submission?	The proposal GP2024121 aims to characterize the size and morphology of the biodegradable microplastics delivered to the bees through SEM/EDX. In particular, the gut content of bees exposed to the biodegradable microplastics by ingestion and the body of bees exposed to the biodegradable microplastics by contact will be analysed with SEM/EDX. This technique can indeed provide the following information: i) size down to the nanoscale of the plastic fragments; ii) details on the morphology and topography of the sample's surface; iii) presence/absence of inorganic contaminants in the plastic fragments. On the contrary, NMR spectroscopy will allow us to provide the chemical and structural characteristics of the MNPs and possible modifications after the exposure tests.		
Industrial Links	-		
Non-Technical Abstract	Honeybees are key pollinators and provide humans with various products. This insect is also a well-known bioindicator of environmental pollution, including micro- and nano-plastics (MNPs), i.e. small plastic particles originating from the degradation of plastics. MNPs may harm the health of bees by ingestion and skin contact. Previous works demonstrated that honeybees can collect a significant amount of airborne plastic fragments, which become trapped by the hairy body of the insects. The present proposal aims to investigate the chemical and structural characteristics of biodegradable MNPs in bees subject to oral and contact exposure. Nuclear Magnetic Resonance spectroscopy will be used as an analytical tool for accurate chemical identification and quantification of the MNPs contaminating the bees highlighting possible modifications after the exposure tests.		

Publications

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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ISIS Neutron and Muon Source

Sample record sheet

Principal contact Dr Ilaria Negri, Università Cattolica del Sacro Cuore, ITALY
MRF Instrument **NMR 600 MHz** **Days Requested:** 3
Special requirements:

SAMPLE

Material	PBAT (Polybutylene Adipate Terephthalate) - based biodegradable plastic	Insect (honeybees) bodies	Insect (honeybees) guts
Formula	C20H30O10	-	-
Forms	Friable powder	Solid	Solid
Volume	cc	cc	cc
Weight	100 mg	10 mg	1 mg
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	- K	- K
Pressure Range	- mbar	- mbar	- mbar
Magnetic field range	- T	- T	- T
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	Yes	Yes
Sample Prep Hazards	No	No	No
Special equip. reqs	No	No	No
Sensitivity to air	No	No	No
Sensitivity to vapour	No	No	No
Experiment Hazards	No	No	No
Equipment Hazards	-	-	-
Biological hazards	No	No	No
Radioactive Hazards	No	No	No
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	Disposed by IS

MRF Proposal: study of the chemical and structural characteristics of biodegradable microplastics in contaminated honey bees through NMR spectroscopy

1. Background and Context

Micro- and nano-plastics (MNPs), i.e. small plastic particles originating from the degradation of plastics, are ubiquitous contaminants in aquatic and terrestrial environments, able to pose significant risks to human and ecosystem health. MNPs are particularly mobile and may easily reach small organisms, especially invertebrates, which may ingest or transport them on their skin, contributing to their dispersion through ecological interactions. Honeybees are of great importance as key pollinators in terrestrial ecosystems, for the production of honey and beeswax, and for biomedicine. Honeybees are highly sensitive to pollutants, and their health can be affected by various environmental contaminants, including plastic pollution. Indeed, MNPs can disrupt the bees' digestive systems and affect the gut microbiota, leading to health issues and weakening the insects' immunity system. Furthermore, previous works demonstrated that, during the foraging activity, honeybees can collect airborne particulate matter, including plastic fragments, which may be trapped by the hairy body of the insects (Negri et al., 2015; Edo et al. 2021). Microplastics can therefore enter the food web through predation, leading to bioaccumulation and further ecological impacts affecting not only bees, but also other species within the ecosystem. Understanding these interactions is therefore important for assessing the broader environmental impact of microplastics, especially biodegradable ones, whose effects on human health and the environment are less known. Indeed, while ecotoxicological studies of MNPs have almost exclusively focused on conventional plastics, little information is available on the environmental toxicity of biodegradable plastic fragments. Moreover, knowledge gaps exist with regard to the fate of biodegradable MNPs in the environment, including possible chemical and structural modifications after exposure to biological systems.

The present proposal aims to investigate the fate of biodegradable MNPs in honeybees after oral and contact exposure. Nuclear Magnetic Resonance (NMR) spectroscopy will be used as an analytical tool for an accurate chemical identification and quantification of the microplastics contaminating the bees by applying advanced metabolomics protocols and structural studies.

This proposal falls within the frame of the project Minagris (MIcro- and NAno-plastics in AGRIcultural Soils) funded by the European Union's Horizon 2020 Programme for Research & Innovation under Grant Agreement number: 101000407. The aim of the project Minagris is to address the environmental impacts of MNPs (both conventional and biodegradable) used in agricultural systems, particularly focusing on their effects on soil health, biodiversity, and ecosystem services, including pollination.

2. Proposed experiment

Honeybees will be exposed orally and by contact (OECD 1998a, b) to MNPs made of PBAT (Polybutylene Adipate Terephthalate), a common biodegradable plastic made from renewable resources, such as corn starch and sugarcane (Fig.1).

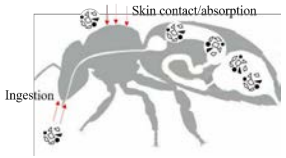

Fig.1 – exposure routes of microplastics to bees

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Bees will be exposed for 48 h to an artificial diet contaminated with the biodegradable MNPs (OECD 1998a). Then, the gut of bees will be dissected and the gut content analysed to detect the presence of the plastic fragments. The aim of the experiment is to verify if the MNPs are ingested by bees and excreted with the faeces; if modifications in the chemical nature of MNPs occur during oral exposure. Exposure by contact will be carried out with anesthetized bees (OECD 1998b). After 48h, the body of the bees will be analysed. The aim of the experiment is to verify if the MNPs are actively removed and/or manipulated by the bees through the typical cleaning behaviour and modifications in the chemical nature of MNPs occur after manipulation.

These data will expand our knowledge about the biotic transportation routes and possible chemical/structural modifications of biodegradable MNPs, contributing to their aerial dispersal and increased risk of MNPs to enter into the food web through predation, where they may bioaccumulate and bio-magnify in ecosystem top predators.

The ISIS@MACH ITALIA Bruker Avance III 600 MHz NMR spectrometer will allow to characterise chemically and structurally the MNPs prior and after the experiments.

NMR spectroscopy offers several advantages over other methods used for analyzing microplastics, such as Raman and FTIR spectroscopy, as it has lower detection limits and may provide more reproducible results, from both the qualitative and quantitative point of view, overcoming possible interference from other organic substances (e.g., the body of the bee, the gut content, etc.).

4. Justification of experimental time requested

I'm requesting the use of the ISIS@MACH ITALIA Bruker Avance III 600 MHz NMR spectrometer because I believe it's the unique instrument that allows me to reach my proposal's aim.

I'm requesting 3 working days to be used as follow:

Day 1: preparation of the MNPs (about 10 g x 5 replicates) and NMR analysis for the characterization of the chemical nature before the exposure test. NMR analysis of freshly prepared MNPs (about 10 g x 5 replicates) to characterize their chemical nature before the exposure test.

Day 2: preparation of the insects' guts (3 guts from plastic-treated bees and 3 guts from control bees). NMR analysis of the gut content for the identification and characterization of the MNPs after the oral exposure test.

Day 3: preparation of the insects' bodies (3 bodies from plastic-treated bees and 3 bodies from control bees). NMR analysis of the body surface for the identification and characterization of the MNPs after the contact exposure test.

References

- Edo C, Fernandez-Alba AR, Vejsnæs F, van der Steen JJM, Fernandez-Pinas F, Rosal R (2021). Honeybees as active samplers for microplastics. Sci. Total Environ. 767, 144481.
- Negri I, Mavris C, Di Prisco G, Caprio E, Pellecchia M (2015). Honey Bees (*Apis mellifera*, L.) as Active Samplers of Airborne Particulate Matter. PLoS ONE 10(7): e0132491.
- OECD (1998a), *Test No. 213: Honeybees, Acute Oral Toxicity Test*, OECD Guidelines for the Testing of Chemicals, Section 2, OECD Publishing, Paris.
- OECD (1998b), *Test No. 214: Honeybees, Acute Contact Toxicity Test*, OECD Guidelines for the Testing of Chemicals, Section 2, OECD Publishing, Paris.

Experiment Proposal

Experiment number GP2024159

Principal investigator	Dr Stefania Zappia, Consiglio Nazionale delle Ricerche, ITALY
Co-investigator (*)	Dr Anna Maria Ferretti, CNR SCITEC, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Metal NP supported Over Microporous Polymelamine Framework for the CO2 absorption and reduction
MRF Instrument	**NMR 600 MHz**
Days requested:	1
Access Route	Direct Access
Previous GP Number:	-
Science Areas	Chemistry, Energy, Materials
DOI:	-
Sponsored Grant	None
Sponsor:	-
Grant Title	-
Grant Number:	-
Start Date	-
Finish Date:	-
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

Porous organic polymers (POPs) have long been considered as prime candidates for CO2) capture, separation, and conversion. Their permanent porosity, structural tunability, stability and relatively low cost are key factors in such considerations. Whereas heteroatom-rich microporous networks have been actively exploited to boost the CO2 affinity of POPs, recently, the focus has shifted to engineering the pore environment, resulting in a new generation of highly microporous POPs rich in heteroatoms and featuring abundant catalytic sites for the capture and conversion of CO2 into value-added products. We have just tested the capability of the POP, obtained by the coupling between melamine and benzene-1,3,5-tricarboxaldehyde, to absorb CO2 and generate Re@POP derivative, that show catalytic activity for (CO2RR). We synthesized a new family of melamine-based POP, coordinated with noble metal nanoparticles to test them as catalysts for the CO2 reduction that must be characterized.

Publications -

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E-platform: No

Instruments	
Access Route	
Science Areas	
Sponsored Grant	
Grant Title	
Start Date	
Similar Submission?	
Industrial Links	
Days Requested:	
Previous RB Number:	
DOI:	
Sponsor:	
Grant Number:	
Finish Date:	

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Sample record sheet

Principal contact	Dr Anna Maria Ferretti, CNR SCITEC, ITALY
MRF Instrument	**NMR 600 MHz**
Days Requested:	1
Special requirements:	

SAMPLE

Material	Microporous Polymelamine Framework (POP)	-	-
Formula	melamine and aldehyde	-	-
Forms	Friable powder		
Volume	cc		
Weight	5-10 mg		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	No	-	-
Sample Prep Hazards	no	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	no	-	-
Equipment Hazards	-	-	-
Biological hazards	nono	-	-
Radioactive Hazards	no	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	-	-

1. Background and Context

Porous organic polymers (POPs) have long been considered as prime candidates for CO_2) capture, separation, and conversion. Their permanent porosity, structural tunability, stability and relatively low cost are key factors in such considerations. Whereas heteroatom-rich microporous networks have been actively exploited to boost the CO_2 affinity of POPs, recently, the focus has shifted to engineering the pore environment, resulting in a new generation of highly microporous POPs rich in heteroatoms and featuring abundant catalytic sites for the capture and conversion of CO_2 into value-added products. We have just tested the capability of the POP, obtained by the coupling between melamine and benzene-1,3,5-tricarboxaldehyde, to absorb CO_2 and generate Re@POP derivative, that show a catalytic activity for (CO2RR). We synthesized a new family of melamine based POP changing the aldehydic components in order to see if changing the size and the density of the pore the CO_2 absorption capability increase. Moreover, we plan to coordinate the POP with metal nanoparticles (MNPs) using NPs that have a good catalytic activity for the CO2 reduction like Pt, Ag but also Ni to test them as catalysts for the CO_2 reduction simultaneously.

In order to get a good characterization of the POP network chemical structure must be characterize with solid state NMR.

2. Proposed experiment

The ^{13}C cross-polarization magic angle spinning (CP-MAS) NMR spectrum of the obtained polymer with the assignments of the resonances is necessary to confirm formation of the polymer network and confirm its structure

3. Summary of previous experimental proposals or characterization

This is the first proposal submitted at ISIS@MACH, anyway previous analysis are reported in Zappia S. et al. Polymers 2022, 14, 5472

4. Justification of experimental time requested

We would like to test just one sample so we estimated that one day is enough.

RETINA

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Experiment Proposal

Experiment number GP2024044

Principal investigator	Professor Alessandro Cianchi, University of Rome Tor Vergata, ITALY
Co-investigator (*)	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Mario Galletti, Universitá di Roma Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Study of TArget Resistance to damaGE and contamination (TARGET) by means XCT and XRF measurements

MRF Instrument	**RETINA**	**Days requested:**	3
Access Route	Direct Access	**Previous GP Number:**	-
Science Areas	Materials, Physics, Technique Development	**DOI:**	-
Sponsored Grant	None	**Sponsor:**	-
Grant Title	-	**Grant Number:**	-
Start Date	-	**Finish Date:**	-
Similar Submission?	-		
Industrial Links	-		

Non-Technical Abstract

Our aim is to study the damages produced by high-intensity laser, photon and particle beams on targets currently used as diagnostics at particle accelerators. The characterization of both a particle beams damaged sample and a pristine one used as a reference will be done at the surface level by means of scanning electron microscopy and optical profilometry which it will allow verify the consistency of the results. We also plan to assess eventual implantation of different ion species that develops after radiation bombardment of the target diagnostic by means of energy dispersive spectroscopy (EDS) microanalysis and X-ray Fluorescence spectroscopy, whereas bulk damage of the sample will be assessed by X-ray computed tomography (XCT) which will allow us reconstructing in 3D the entire sample with micrometric spatial resolution in one shot. Hence, we aim here to request access to the XCT and X-ray Fluorescence spectroscopy instrument RETINA operating at the POLIMI Unit of IM@IT.

Publications

Gschwendtner, Edda, and Patric Muggli. "Plasma wakefield accelerators." Nature Reviews Physics 1.4 (2019): 246-248.

Rule, D. W. "Transition radiation diagnostics for intense charged particle beams."NIM in Phys. Research B: Beam Interactions with Materials and Atoms 24 (1987): 901-904.

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E-platform: No

Instruments	
Access Route	
Science Areas	
Sponsored Grant	
Grant Title	
Start Date	
Similar Submission?	
Industrial Links	

Days Requested:	
Previous RB Number:	
DOI:	
Sponsor:	
Grant Number:	
Finish Date:	

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Sample record sheet

Principal contact	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY		
MRF Instrument	**RETINA**	**Days Requested:**	3
Special requirements:			

SAMPLE

Material	Silicon crystal Aluminium film	-	-
Formula	-	-	-
Forms	Solid		
Volume	0,03 cc		
Weight	1000 mg		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	No	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	-	-

Study of TArget Resistance to damaGE and contamination (TARGET) by means XCT and XRF measurements

Background and Context

Material survival in a hostile environment is a fundamental requirement for beam instrumentation in a particle accelerator. Intercepting diagnostics is the most critical part, as particle beams continuously hit it. A new generation of particle accelerators is developing, based on the interaction of strong laser (>10^{18} W/cm^2) with plasma or particle beam, to boost the particle energy or the photon energy via inverse Compton scattering [1].

In a photon-electron collider, such as one used in a gamma-ray source, electron and high-intensity photon beams can hit the diagnostic. View screens, e.g., small targets that emit light via optical transition radiation [2], when struck by particles or reflect photons, are widely used to image the photon and electron beam. This proposal investigates the damages produced by high-intensity laser and photon beams on such diagnostics and identifies the critical aspect of the current design.

These screens are usually realized with a bulk substrate of crystalline Silicon, about 300 μm thick, with an aluminium deposition of several hundreds of micrometers to enhance the reflectivity.

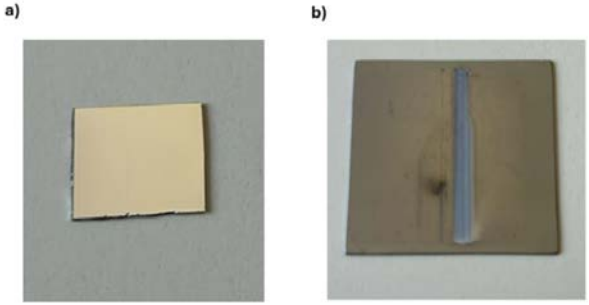

Figure 1: Diagnostic target of 10 mm x 10 mm area, and of 300 µm thickness used at particle accelerator to image the beam profile. a) Pristine target never been exposed to any particle or laser beam radiation damage. b) Target sample with evident damage done after particle radiation.

High-intensity electron beams can deposit relevant amounts of energy, leading to possible surface damage. High-intensity laser beams can accidentally hit the screen during the alignment process, also giving surface or bulk damage. Both electron and laser beams can also hit the vacuum pipe or other device inside the vacuum, producing evaporation of ions that can have enough energy to be implanted in the screen material.

To better understand the damage on such diagnostic targets, we propose a multi-instrumental approach study. From one end, we aim to assess the damage occurring at the surface of the target by means of scanning electron microscopy and optical profilometry. Eventual craters, cracks and defects produced by particle beams on the sample will be measured by SEM and optical microscopy to verify the outcomes. This can be done by means of the SEM&C-AFM with Optical Profiler instrument available at the Roma Tor Vergata Unit of ISIS@MACH ITALIA. We also plan to assess

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eventual implantation of different ion species that develops after radiation bombardment of the target diagnostic by means of energy dispersive spectroscopy (EDS) microanalysis that is an ancillary equipment available on the same instrument. For the characterization of the bulk damage of the sample we plan to use X-ray computed tomography (XCT) which will allow us reconstructing in 3D the entire sample with micrometric spatial resolution in one shot. Defects detected on the sample by XCT data can be compared with results obtained by SEM and optical microscopy data. To do that, we aim to perform XCT scans of the two samples at the RETINA instrument available at the POLIMI-National Medium Range Facilities Unit. The choice of this instrument potentially opens the possibility to do X-ray Fluorescence (XRF) spectroscopy measurements, which we can use here as a second investigation tool for studying the ion implantation on the surface of the sample already assessed by SEM-EDS data. The expected results, in terms of morphological study, surface contamination, and structure damage, can offer important insight into the choice of our diagnostic target material, the production system, and the long-term survival in a harsh environment.

Proposed experiment

In this experiment we aim to perform XCT scan on a damaged and pristine diagnostic target for accelerator application by using the RETINA instrument available at the POLIMI-National Medium Range Facilities Unit. Eventual defects detected by the XCT data will be compared by results obtained by SEM and optical microscopy data. Furthermore, we envisage here mapping the elemental composition of damage and pristine samples by XRF measurements, which results will be benchmarked by surface similar maps extract by SEM-EDS data.

Justification of experimental time requested

The radiation damaged and reference target diagnostic samples have dimensions of about 10mm x 10mm and thickness of about 300 μm. We aim to acquire XCT scan for each sample using a field of view of 10 mm x 10 mm, pixel size of 25 μm, and about 600 projections to fulfil the Niquist-Shannon sampling theorem. With an exposure time per projection of 10 s, each tomography will last about 2 hours.

Furthermore, we envisage here mapping the elemental composition of both a damage and pristine sample by XRF measurements performed on n. 15 localised points where the proponents have already collected SEM-EDS data which will allowed a straight result comparison.

Hence, after discussion with the instrument scientist, we request 3 days of instrument time including set-up and calibration time.

References

[1] Gschwendtner, Edda, and Patric Muggli. "Plasma wakefield accelerators." *Nature Reviews Physics* 1.4 (2019): 246-248.

[2] Rule, D. W. "Transition radiation diagnostics for intense charged particle beams." *Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms* 24 (1987): 901-904.

Experiment Proposal

Experiment number GP2024059

Principal investigator (*) Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator

Experiment title	Investigating the Corrosive Impact of Chrome-Tanned Semi aniline Calf Leather using XRF		
MRF Instrument	**RETINA**	**Days requested:**	2
Access Route	Direct Access	**Previous GP Number:**	NO
Science Areas	Materials	**DOI:**	-
Sponsored Grant	None	**Sponsor:**	-
Grant Title	-	**Grant Number:**	-
Start Date	-	**Finish Date:**	-
Similar Submission?	-		
Industrial Links	LBS LUXURY BRANDS SERVICES SRL		

Non-Technical Abstract

The aim of this study is to systematically investigate the composition of semi aniline chrome-tanned calf leather and to identify correlations between adsorbed elements on the leather surface and its aggressiveness towards metal accessories. By understanding these relationships, we can develop better methods for predicting and mitigating corrosion, thereby improving the quality and durability of leather products in the fashion and luxury industries. Understanding the chemical composition of semi aniline chrome-tanned calf leather will lead to significant improvements in product quality ad in particular this research will provide a scientific basis for better quality control practices and contribute to the development of more robust and durable fashion items.

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY
MRF Instrument **RETINA** **Days Requested:** 2
Special requirements:

SAMPLE

Material	Chrome-Tanned Semi aniline Calf Leather	-	-
Formula	Chrome-Tanned Semi aniline Calf Leather	-	-
Forms	Solid		
Volume	4 cc		
Weight	10 g		
Container or substrate	No special need	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	RT - K	-	-
Pressure Range	Atmospheric pressure - mbar	-	-
Magnetic field range	No - T	-	-
Standard equipment	-	-	-
Special equipment	No special equipment needed	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	No	-	-
Special equip. reqs	No	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	No	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Investigating the Corrosive Impact of Chrome-Tanned Semi aniline Calf Leather with XRF

1. Background and context

Semi-aniline chrome-tanned calf leather is a highly valued material in the fashion and luxury industries, commonly used for products such as handbags, belts, and footwear. Ensuring the quality and longevity of these products is crucial, particularly in preventing corrosive processes that can damage metallic accessories like buckles, zippers, and decorative elements. The leather can release tanning substances that may react with metals, leading to corrosion and tarnishing, which compromises the aesthetic and functional integrity of the final products.

To assess the corrosive potential of leathers, quality control laboratories typically perform simulated corrosion tests using a reference sample. The extent of oxidation on the sample, after exposure to the leather, is evaluated qualitatively through visual tests. The leather is classified on a scale of aggressiveness from 1 to 5 (1 = highly aggressive, 5 = non-aggressive). However, beyond this empirical test, there has been no systematic study to investigate the underlying causes of oxidation and the specific elements responsible for the corrosive effects on metal accessories.

This proposal aims to investigate the composition of the leather using X-ray fluorescence (XRF) mapping with RETINA, providing detailed elemental and spatial distribution data that could be correlated with the leather's corrosive properties. The fashion and luxury industries could then take proactive measures to treat or modify leather to reduce its corrosive impact, thereby enhancing the durability and quality of leather products.

2. Proposed experiment

This proposal is part of a broader study to investigate the underlying causes of oxidation and the specific substances responsible for the corrosive effects on metal accessories by Semi aniline chrome-tanned calf leather. The study involves several instrumental techniques: a) XRF mapping; b) RAMAN Spectroscopy and profilometry; c) FT-IR Spectroscopy; d) Ion Scattering Spectroscopy.

X-ray Fluorescence analysis at RETINA will provide valuable insights on the elemental composition of the leather products and their in-plane distribution along the material. Specifically, we can extract information on:

1. Elemental Composition: XRF analysis can identify the presence of elements such as chromium that is one of the main components used so far in the tanning industry. By understanding the elemental composition, we can pinpoint specific substances that may be responsible for initiating corrosion in metal accessories.

2. Spatial Distribution: 2D in-plane scans of the leather product allow for the visualization of the spatial distribution of elements within the material. This information is crucial for understanding how these elements are distributed across the leather's surface, which can influence their reactivity with metals.

3. Concentration Gradients: Through the 2D XRF scans, the concentration gradients of different elements can be analysed quantitatively. Such gradients may provide insights into the diffusion processes and how certain areas of the leather may be more prone to causing corrosion.

4. Identification of Corrosive Agents: By correlating the elemental data with known corrosive agents, we can identify specific elements or compounds in the leather that are likely to contribute to corrosion. This identification is key to understanding the mechanisms behind the leather's aggressiveness towards metal accessories.

By conducting this analysis, we will obtain essential data for understanding and mitigating the corrosive effects that these leathers can have on metallic accessories, ultimately leading to improved quality and durability of leather products in the fashion and luxury industries.

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3. Summary of previous experimental proposals or characterisation

Historically, the understanding of the aggressiveness of leather toward metal accessories has been constrained by a reliance on empirical methods and trial and error. This approach lacks the detailed insight and predictive capability that sophisticated analytical tools like XRF Tomography investigation can provide. In particular, access to such advanced analysis and the requisite expertise is often limited. Although some literature provides a foundational understanding of elemental composition and distribution of the elements on semi aniline chrome-tanned calf leather, [1] a detailed exploration of these elements' interplay, especially regarding the correlation with aggressiveness and corrosion properties toward metal accessories, has not been conducted extensively so far. This research aims to fill that critical knowledge gap in order to provide a correlation between elemental composition and spatial distribution of chemical elements on the surface of semi-aniline chrome-tanned calf leather and the observed corrosive properties when in contact with metal accessories, giving the possibility of linking specific elements to increased corrosion risk.

4. Justification of experimental time requested

As detailed in section 2, X-ray Fluorescence analysis is a pivotal tool for this experiment due to its unique capabilities.

To achieve a comprehensive analysis of the leather samples, we request machine time for RETINA to perform XRF analysis. This will enable us to obtain detailed elemental and spatial distribution data, which can be correlated with the leather's corrosive properties. We request 2 days of experimental time to analyse 15 samples coming from 5 leather batches: we collected 5 types of batches of semi aniline chrome-tanned calf leather with high level of aggressiveness (level 1 to level 2, with reference to the empirical scale where (1 = highly aggressive, 5 = non-aggressive) as determined by the currently used corrosion test as detailed in section 1. For each batch we will then collect three samples for investigating the spatial homogeneity of the composition within the same batch.

This number ensures diverse representation from different batches of treatment conditions. The first day will be dedicated to the 2D scanning of the first batch of samples and the data collected will be analysed to identify key elements and their distribution. Then a detailed data analysis, focusing on elemental quantification and spatial mapping, will be conducted on the other batches. A focus will be made on elements known to influence corrosion such as chromium. The mapping of the spatial distribution of these elements within the leather samples will help to understand their localization and concentration gradients. On the second day, a correlation of XRF data with empirical corrosion test results will be conducted and reproducibility measures will be conducted if necessary. Finally, a formulation of recommendations based on the integrated analysis will be outlined in order to improve the processing methods that could mitigate the corrosive impact of the leather on metal accessories.

[1] Bruno P. Pouliot, Dr. Jennifer Mass, Lara Kaplan; USING XRF FOR THE IDENTIFICATION OF CHROME TANNING IN LEATHER; Poste, American Institute for Conservation 43rd Annual Meeting • Miami, Florida • May 2015

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Experiment Proposal

Experiment number GP2024064

Principal investigator	Dr Elena Colombo, Politecnico di Milano, ITALY
Co-investigator	Mr Francesco Casamichiela, Politecnico di Milano, ITALY
Co-investigator	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Co-investigator (*)	Ms Aixeen Manuel Fontanilla, RETINA Politecnico di Milano, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Ion Migration Studies in Polymer Electrolyte Membranes Fuel Cells
MRF Instrument	**RETINA** **Days requested:** 4
Access Route	Direct Access **Previous GP Number:** No
Science Areas	Energy **DOI:** -
Sponsored Grant	Yes **Sponsor:** Other
Grant Title	Progetto Permanent - Bando MITE PNRR Missione 2 Investimento 3.5 A **Grant Number:** RSH2A_0O0012
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

Ce is usually adopted as a chemical stabilizer in membranes for polymer electrolyte membrane fuel cells (PEMFCs) to inhibit the free radicals attack through the redox reactions of Ce(III)/Ce(IV) ions. However, Ce can redistribute over the active area during operation, resulting in a heterogenous pattern. XRF can help in understanding the Ce mobility and obtain physically relevant coefficients (e.g., diffusivity), thus investigating the dynamics of Ce migration in a cation-exchanged ionomer membrane. We propose to establish the Ce concentration gradient by applying a controlled potential gradient in a hydrogen pump, measure a linear concentration profile via XRF, and fit the physical diffusion parameters of the membrane, using the RETINA instrument at Politecnico di Milano.

Publications

https://doi.org/10.1016/j.jpowsour.2022.232246
https://iopscience.iop.org/article/10.1149/1945-7111/abf4eb/meta
https://doi.org/10.1016/j.jpowsour.2023.233376

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Ms Aixeen Manuel Fontanilla, RETINA Politecnico di Milano, ITALY		
MRF Instrument	**RETINA**	**Days Requested:** 4	
Special requirements:			

SAMPLE

Material	Nafion membranes: sulfonated tetrafluoroethylene polymer membranes soaked in a Ce solution.	-	-
Formula	C7HF13O5S C2F4	-	-
Forms	Solid		
Volume	cc		
Weight	mg		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	No	-	-
Sample Prep Hazards	No	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	No	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Removed By User	-	-

Ion Migration Studies in Polymer Electrolyte Membranes Fuel Cells

1. Background and Context

Internal combustion engines (ICEs) burning fossil fuels contribute to approximately 20% of global CO2 emissions [1]. To mitigate greenhouse gases and combat climate change, fuel cell electric vehicles (FCEVs) have emerged as a promising renewable technology to replace ICE vehicles. FCEVs have a much lower environmental impact, emitting only water vapor as a by-product and producing no harmful pollutants. Major automotive companies like Toyota, Honda, and Hyundai are now commercially leasing and selling FCEVs, which offer performance comparable or superior to traditional ICEs in terms of speed, acceleration, re-fueling time, and driving range. However, challenges such as performance at high current densities, durability, and cost still need to be addressed. [2] PEM fuel cells, commonly using Nafion® polymer electrolyte membranes, conduct protons while separating gaseous reactants at the anode and cathode, and rely on platinum-based electrocatalysts for lower temperature electrochemical reactions. These cells operate at low temperatures, provide high power output, and are scalable, making them suitable for vehicle applications. Free radicals generated during fuel cell operation degrade the catalyst and fuel cell stability. To inhibit free radical attacks, Cerium is typically used as a chemical stabilizer in membranes through Ce(III)/Ce(IV) redox reactions, but its migration during operation can lead to uneven distribution. This uneven migration results in areas with insufficient cerium, increasing susceptibility to oxidative degradation and reducing membrane lifespan and performance [3]. Research at the MRT Fuel Cell & Battery Laboratory focuses on developing innovative PEMFC components, optimizing operation, and extending lifespan to scale up for automotive applications. Current efforts include numerical modelling to understand and mitigate cerium transport phenomena, which accelerate fuel cell aging.

In this proposal, the primary goal is to investigate the cation-exchanged ionomer membranes through an in-plane X-ray fluorescence analysis scanning using the RETINA Instrument, operating at the Politecnico di Milano. With the migration profiles derived from the XRF analysis, we can obtain valuable insights in the Ce mobility and diffusivity.

2. Proposed experiment

Cerium migration in PEM fuel cells is influenced by electric potential and ion concentration gradients, with mobility and diffusivity varying with temperature, humidity, and membrane properties. Migration models, essential for predicting and mitigating cerium movement, enhance fuel cell performance and durability through optimized material selection and design. XRF analysis provides detailed data on cerium migration profiles, allowing quantitative characterization of ion concentration within membranes. RETINA's X-ray fluorescence analysis enables precise elemental mapping, tracking cerium ion distribution and migration over time, thanks to XRF's non-destructive nature. This capability supports repeated measurements on the same PEM fuel cell, facilitating continuous monitoring of element movement and long-term studies on migration patterns and degradation mechanisms, crucial for enhancing the durability and reliability of PEM fuel cells.

In this proposal, we will investigate cerium ion distribution in the membranes using in-plane XRF analysis at the RETINA MRF of IM@IT. The study consists in two parts: 1) membrane preparation with controlled Ce(III) and Ce(IV) concentrations in sulfuric acid solutions and application of potential gradients in a hydrogen pump under specified conditions to track Ce in-plane distribution; 2) controlled doping of PEM fuel cells to assess the impact on the performance by electrochemical and performance characterization. In-plane XRF scanning will be done post-adhoc experiments to quantify Ce area density profiles across membrane length [4]. Ce concentration gradients in samples of practical size extend along few cm. A spatial resolution of about 1 mm is sufficient to visualize the main features of the Ce concentration profile as, according to the existing literature, we do not expect gradients occurring at smaller scales. Our preliminary 1-D model, based on the Nernst-Planck equation, will be refined using XRF data correlated with temperature, humidity, and potential gradients. Moreover, in this first study we are only interested in 1D gradients, so to reduce the number of modelling parameters needed to fit the experimental profile. The resolution and scanning requirements are all satisfied by RETINA, as it can provide linear concentration gradients along planar samples with a resolution down to 1 mm. In addition, Ce should be trackable by the instrument as it is within the declared range of detectable elements. RETINA could also be used to measure the initial uniform concentration of Ce absorbed by the membranes. Besides cerium, platinum presence from electrodes will also be monitored to optimize membrane assembly. Overall, results of the study will be beneficial in advancing our understanding of cerium migration in PEM fuel cells.

3. Summary of previous experimental proposals or characterization

Previous characterization on fuel cell membranes has been conducted in external testing facilities using an SEM instrument to visualize morphological structures and determine the thickness of the PEM layers. This allowed us to effectively characterize the membrane thickness, which is essential to determine the density of the sample and retrieve the elemental concentration via the fundamental parameter method.

4. Justification of experimental time requested

Applying this combined approach to a range of samples with carefully calibrated experimental conditions (in terms of Ce absorption and diffusion time) could provide critical insights in Ce absorption and migration in cation exchange membranes. We plan to analyze a total of 16 samples, covering 4 different potential gradients, 2 soaking times, 2 different temperatures and relative humidities. Several migration profiles derived from samples under varying experimental conditions are needed to effectively calibrate the Ce migration model and identify the optimal fitting parameters. Moreover, some of the model parameters can depend on the ambient conditions, thus requiring changing the temperature and relative humidity. Our samples will be 3 cm long, which amounts to 30 points with a 1 mm step. Considering 3 minutes per point, each scan will take 1.5 h. Thus, we request 3 days of beamtime, plus 8 more hours for setup. We will bring the equipment needed to impose a Ce concentration gradient on the membranes.

References

[1] International Energy Agency (2018). "World Energy Outlook."
[2] Z.P. Cano., et al. (2018). Nature Energy 3, 279-289
[3] Yeh-Hung Lai et al., J. Electrochem. Soc. 165 F3217, 2018.
[4] Andrew M. Baker et al., J. Electrochem. Soc. 164 F1272, 2017.

Experiment Proposal

Experiment number GP2024076

Principal investigator	Professor Mauro Rubini, Sovraintendenza, ITALY
Co-investigator	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY
Co-investigator (*)	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	A multidisciplinary proteomic study of the unique ancient Homo cepranensis petrous bone using RETINA
MRF Instrument	**RETINA**
Days requested:	1
Access Route	Direct Access
Previous GP Number:	-
Science Areas	Biology and Bio-materials, Cultural Heritage
DOI:	No
Sponsored Grant	None
Sponsor:	-
Grant Title	-
Grant Number:	-
Start Date	-
Finish Date:	-
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

Our aim is to study a petrous bone of unique ancient remain Homo cepranensis (Ceprano, località Campogrande, Italy) by multi-instrumental approach through a combined series of non-destructive and destructive analyses. For non-destructive analyses in this proposal, we request the RETINA instrument to perform XRD Tomography, for a 3D reconstruction combined with XRF maps, and, in a distinct proposal, the IMAT beamline at ISIS facility for complementary neutron tomography and neutron time of flight Prompt Gamma Activation Analysis (T-PGAA). The scope is to obtain a 3D reconstruction of the Homo cepranensis remain as well as the uranium series. Additionally complementary destructive characterisations, which will be request in two distinct proposals, include the ancient DNA (aDNA) and proteomic analyses using the IM@IT' DNA Sequencing NGS and the Mass Spectrometer 2 instruments.

Publications

Di Vincenzo. Sci Rep 7, 13974 (2017)
Manzi. J. Hum. Evol. 59, 580-585 (2010).
Rubini. J. Hum. Evol. 77, 204-216 (2014)

Instruments	**IMAT**	**Days Requested:**	3
Access Route	Direct Access	**Previous RB Number:**	No
Science Areas	**DOI:** No		
Sponsored Grant	None	**Sponsor:**	
Grant Title	-	**Grant Number:**	
Start Date	-	**Finish Date:**	
Similar Submission?			
Industrial Links			

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Sample record sheet

Principal contact	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY		
MRF Instrument	**RETINA**	**Days Requested:**	1
Special requirements:			

SAMPLE

Material	petrous bone (about 3x2x1 cm3)	-	-
Formula	hydroxyapatite and collagen.	-	-
Forms	Solid		
Volume	6-9 cc		
Weight	350-600 mg		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	273 - 320 K	-	-
Pressure Range	7000 - 1010 mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	It is requested the use of T-PGAA before and after IMAT measurements	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	No	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

A multidisciplinary proteomic study of the unique ancient *Homo cepranensis* petrous bone using RETINA

1. Background and Context

In this proposal we tender to study a remain of a unique ancient sample, *Homo cepranensis*, by multi-instrumental approach. The artifact was discovered on March 13, 1994, by archaeologist Italo Biddittu during surface reconnaissance along the route of a highway under construction near Ceprano (locality of Campogrande in the province of Frosinone) in the lower Sacco Valley (Figure 1). The bulldozers that facilitated the discovery of the artifact at the same time likely caused its fragmentation. The fossil artifact is limited to the neurocranium (calvarium). The fragments were contained within a series of stratified fluvio-lacustrine deposits. About 50 large fragments were unearthed in a small area near the original discovery, and more than 200 small pieces were collected by sieving the sediments [1]. Unfortunately, most of the facial bones, as well as much of the cranial base and almost the entire left parietal, were not found. Currently, the fossil is located at the Superintendency of Archaeology, Fine Arts, and Landscape for the Provinces of Frosinone and Latina.

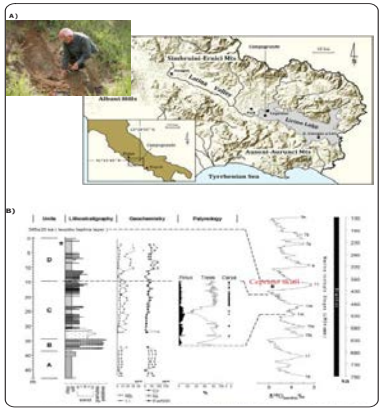
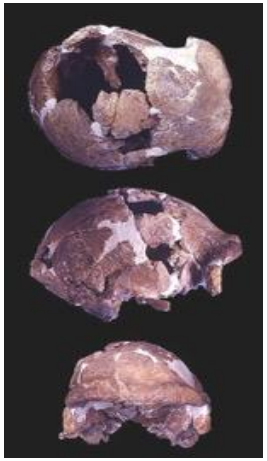


Figure 1.A) geographical localization of the site with Italo Biddittu during the discovery; B) stratigraphy, geochemistry [2], palynological data and $^{40}Ar/^{39}Ar$ dating [3].

Figure 2: *H. cepranensis* skull reconstruction.



The current form of the artifact is the result of a reconstruction initiated in 1994 by Prof. A. Ascenzi continued by Prof. R. J. Clarke and reviewed by paleoanthropologist M. A. de Lumley and Prof. F. Mallegni [4] (Figure 2). Previous characterisations were carried out directly with the original fragments and with extensive use of dental plaster. The calvarium was already analyzed using X-ray microtomography (µCT) at the Multidisciplinary Laboratory of the Abdus Salam International Centre for Theoretical Physics in Trieste. Medical CT scans and recent µCT scans of the calvarium revealed the extent of the plaster and discouraged its mechanical removal. Attempts to digitally remove the plaster from the calvarium also failed, using both globally applied threshold filters and manual operations on each tomographic section; only with high-resolution 3D imaging it was possible to digitally remove the dental plaster insertions and separate the fragments [5]. The calvarium is quite well-preserved, although incomplete, there are no absolute dates and relative datings, based on the regional geo-stratigraphic and paleontological framework, place it between 0.9 and 0.8 Ma [6]. Recent magneto-stratigraphic analyses of the lacustrine and fluvial sediments recovered from cores taken at the site of the artifact, however, have provided a different relative dating; according to these studies, the stratigraphic level containing the artifact itself is between 0.5 Ma and 0.35 Ma [3] (Figure 1). The artifact also shows a series of characteristics, such as a cranial capacity of 1180-1200 cm³, typical of the oldest forms of humanity from the Middle Pleistocene. Cladistic studies initially attributed it to the species *H. erectus* and later to *H. heidelbergensis*, though its exact classification remains unclear. In this proposal our aim is to solve this question by analyzing one petrous bone of the sample using multimolecular destructive analyses: proteomics, which is the scope of the present proposal and to follow ancient DNA analysis (aDNA), that we request in a distinct proposal. With the proteomics we retrieve all the proteins in the remains using Mass Spectrometer 2 which will be useful to support the DNA data. The aDNA analysis is quite challenging because, to date, the oldest ancient genome published from the same latitude is from Sima de los Huesos (Spain), dated back 430,000 years ago [7]. It is also interesting the possibility to date the uranium series and through the fragments realise a virtual reconstruction of the remain.

2. Proposed experiment

In this specific proposal we aim to use the RETINA instrument for a non-destructive X-ray tomography with hard X-ray beams, allowing a 3D reconstruction of an extended object, and the concurrent collection of X-ray fluorescence data for 3D chemical composition map. Results of this experiment will be cross compared to verify consistency with data obtained by separate proposals where we request IMAT beamline and Germanium instrument at ISIS for neutron tomography experiment and to perform a T-PGAA measurement before and after the 3D reconstruction at the IMAT beamline at ISIS, to obtain a complete digital twin of the sample before its partial destruction in later characterizations. After that we will process the sample by destruction analysis: the sequencing of the proteins using the Mass Spectrometer 2 instrument operating at the IM@IT' Unit-University of Milano Bicocca and the aDNA sequencing by NGS instrument operating at the IM@IT' Unit -University of Rome Tor Vergata.

3. Justification of experimental time requested

To achieve a comprehensive characterisation of the remains, we request XRF analysis with RETINA with the use of X-ray pencil beam collimated on the sample with millimetric spatial resolution to obtain the map of its elements. The results will be correlated with neutron 3D tomography and T-PGAA measurements. Hence, after discussion with the instrument scientist, we request 1 days of instrument time including set-up and calibration time.

References

[1] Ascenzi, Segre. 2000. In The Origin of Humankind: 25-33; [2] Lisiecki, Rayamo. 2005. Paleoceanography Paleoceanography, 20. [3] Nomade et al. 2011. Quetarnary Geochronology, 6: 453-457. [4] Mallegni et al. 2003. Coptes Rendus Palevol, 2: 153-159. [5] Di Vincenzo et al. 2017. Scientific Reports 7: 13974. [6] Manzi et al. 2001. Proc Natl Acad Sci USA (PNAS) 98: 10011-10016. [7] Meyeret al. 2016. Nature 531: 504–507.

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Experiment Proposal

Experiment number GP2024133

Principal investigator	Mr Dario Rastelli, RAYLAB, ITALY
Co-investigator	Mr Francesco Casamichiela, Politecnico di Milano, ITALY
Co-investigator (*)	Ms Aixeen Manuel Fontanilla, RETINA Politecnico di Milano, ITALY
Co-investigator	Ms Chiara Caprioli, RAYLAB, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Characterization of Silicon Avalanche Photon Detectors (APD) for X-ray Fluorescence analysis
MRF Instrument	**RETINA** **Days requested:** 3
Access Route	Direct Access **Previous GP Number:** -
Science Areas	Technique Development **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	Raylab s.r.l.

Non-Technical Abstract

Avalanche photodiodes (APDs) provide a robust, compact solution for detecting X-rays, due to their internal signal amplification (caused by electron cascades), which improves the signal-to-noise ratio (SNR), and good energy resolution (especially in cooled environments). At Raylab Solutions, an innovative Italian company and spin-off of Politecnico di Milano, we have recently expanded our applications to include the development of silicon-based detection units for X-ray fluorescence analysis. Our system includes a Peltier-cooled reach-through Si APD with active area of 25 mm^2 and crystal thickness of 130 um, and an in-house electronic system to manage the readout of the signals and to control the thermoelectric cooling of the APD. In this proposal, our goal is to characterize the resolution, SNR and limits of detection of our system to perform quantitative X-ray fluorescence analysis, by deploying it at the RETINA Instrument, operating at Politecnico di Milano.

Publications

http://dx.doi.org/10.1016/j.nima.2020.164078
http://dx.doi.org/10.1038/s41598-022-21113-7
http://dx.doi.org/10.1016/j.nima.2016.01.076

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Ms Aixeen Manuel Fontanilla, RETINA Politecnico di Milano, ITALY
MRF Instrument	**RETINA** **Days Requested:** 3
Special requirements:	

SAMPLE

Material	Cu, Ti, Fe targets	-	-
Formula	-	-	-
Forms	Solid		
Volume	cc		
Weight	mg		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Characterization of Silicon Avalanche Photon Detectors (APD) for X-ray Fluorescence analysis

1. Background and Context

Avalanche photodiodes (APDs) are silicon-based solid-state devices that transform photons into electrical currents. They provide a robust, compact solution for detecting light and X-rays, featuring high gains and quick response times while being insensitive to magnetic fields. These distinct characteristics make APDs extensively used in diverse areas including physics, medicine, and aerospace. [1]

For material analysis, APDs are an excellent choice for direct X-ray detection due to their internal signal amplification, caused by electron cascades, which improves the signal-to-noise ratio drastically. APDs can also be operated even at room temperature or in slightly cooled environments. [2] They also offer good energy resolution, allowing effective discrimination of different fluorescent X-ray energies, and their fast response times are beneficial in high-throughput applications. Additionally, modern APDs are designed to have low dark current and electronic noise. [3] Most importantly, in XRF, at low X-ray energies, the characteristic K shell emission lines of light elements may partially overlap with the L lines of transition metals. [4] This overlap makes the quantification difficult and sometimes, inaccurate. Therefore, the energy resolution of detector systems is crucial and must be maximized without compromising detector efficiency.

At Raylab Solutions, an innovative Italian company and spin-off of Politecnico di Milano, we focus on developing and designing high-performance radiation detectors for radiation protection analysis and monitoring. Recently, we have expanded our applications to include the development of silicon-based detection units for X-ray fluorescence analysis. Our system includes a Peltier-cooled reach-through Si APD with active area of 25 mm^2 and crystal thickness of 130 um, and an in-house electronic system to manage the readout of the signals and to control the thermoelectric cooling of the APD. In our device, we aim to achieve a good energy resolution in the low-energy X-rays to enable quantification of low-Z elements, which is a limitation for high-Z detectors such as CdZnTe and Ge.

In this proposal, our goal is to characterize the capability of our detectors and its associated electronic system to perform quantitative X-ray fluorescence analysis by deploying it at the RETINA Instrument, operating at the Politecnico di Milano. The performance of APDs as a soft X-ray detector will be studied in terms of its energy resolution, minimum detectable energy (MDE), signal-to-noise ratio, and its limits of detection.

2. Proposed experiment

Exploiting the high-power X-ray source of the RETINA facility, we can install our APDs and investigate their fundamental properties as an X-ray detector. This is made possible due to the versatility of the facility, allowing the easy placement of detector setups, and the high precision positioning system that enables reproducibility in the measurements. The energy resolution of the APDs will be determined upon the irradiation of mono-elemental reference samples available in the facility, by measuring the FWHM of the peaks due to soft X-rays in the range between 2 and 12 keV. The FWHM determines the signal discrimination capability of the APD which is crucial for X-ray fluorescence applications. Another feature of interest of this APD is the low peak background plateau of the measured X-ray energy spectrum, which enhances the signal-to-noise ratio. In reach-through APDs such as this one, the internal structure should reduce this plateau to extremely low counts compared to other APD designs, e.g., bevelled-edge or reversed-APDs [5]. We intend to verify this by taking the peak-to-background ratio from the measured spectrum.

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In the investigation of the APD properties for quantitative XRF analysis, the APD will be mounted in the detector base, and will measure the X-rays emitted by the thin reference XRF standard available at the RETINA facility. The standard includes a wide selection of non-overlapping XRF lines (both K and L lines) over a large energy range. Elemental quantification of the standard will be inferred from the spectra using the algorithms already employed at RETINA. From these quantification procedures, we can derive the MDE and the limits of detection of the APDs. Furthermore, we will compare elemental quantifications of standards and/or samples obtained from the APDs and the output using the in-house CdZnTe detector to evaluate their coherence. Results of the study will be helpful in evaluating the APD's potential as a soft X-ray detector for X-ray fluorescence application.

3. Summary of previous experimental proposals or characterisation

The detector leakage current, also known as dark current (ID), and detector capacitance are crucial performance parameters of any silicon detector. The dark current sets the noise limit associated with the photodiode, which is dependent on the applied bias voltage. To measure the current-voltage (I-V) characteristics, we used a Keithley 6484 Picoammeter/Voltage Source. Additionally, we assessed the capacitance-voltage characteristics using an LCR Bridge HM8118. This assessment allowed us to determine the full depletion voltage and detector capacitance accurately.

4. Justification of experimental time requested

Our beamtime will be divided in two main parts, one related to the measurements of pure elemental standards, and a second focusing on the quantification of multi-element standard and samples. For the quantifications, we will use the software already available at RETINA. We plan to test multiple APDs (4-8 units) of the same manufacturer, to evaluate the device-to-device variation of the response, which could be an issue for this kind of detector. A setup will be devised in which 4 APDs can be mounted and tested together, to save time. The APDs will be equidistant from the point of X-ray emission for a thorough comparison of their responses to the same radiation field. We ask for three days of beamtime: one day for setup and test on mono-elemental standards, a second day for the measurements on multi-elemental samples and a third day for the quantification of the measured samples.

References

[1] Marc Diepold, et al., (2015). *Rev. Sci. Instrum.* 86 (5): 053102.
[2] A. Ochi, Y. Nishi, T. Tanimori (1996). Nucl. Instr. and Meth. A, 378, p. 267
[3] Baron A.Q, et al., (2006). J Synchrotron Radiat. 2006 Mar;13(Pt 2):131-42.
[4] Alessandra Gianoncelli et al., (2016). Nucl. Instr. and Meth. A, 816, 113-118.
[5] Y. Yatsu, et al., (2006). Nucl. Instr. and Meth. A, 564 (1), 134-143.

Experiment Proposal

Experiment number GP2024139

Principal investigator	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Jens Holtvoeth, Teesside University, UNITED_KINGDOM
Co-investigator	Professor Charles Cockell, University of Edinburgh, UNITED_KINGDOM
Co-investigator	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY
Co-investigator (*)	Professor Alessandro Cianchi, University of Rome Tor Vergata, ITALY
Co-investigator	Miss Julia Puputti, Boulby Underground Laboratory STFC, UNITED_KINGDOM
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Biogeochemical Multi-Instrumental Study of Rocks and Microbial Biomass in Zechstein Salt Deposits of Boulby using RETINA
MRF Instrument	**RETINA** **Days requested:** 4
Access Route	Direct Access **Previous GP Number:** No
Science Areas	Biology and Bio-materials, Chemistry, Environment, Materials **DOI:** -
Sponsored Grant	Yes **Sponsor:** CNR
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	Our aim is to investigate the potential for preserving biological material in ancient salt deposits, with a focus on the Zechstein salt deposits in Boulby Mine (UK), offering insights into the environmental conditions of the Zechstein Sea ~250 million years ago. The study employs a multi-instrumental approach, combining non-destructive and destructive analyses to correlate biomolecule presence with mineral phases and elemental compositions. By analyzing a range of biomarkers the research aims to create a detailed biogeochemical fingerprint of fossil microbial biomass. This research contributes to both astrobiology and our understanding of ancient terrestrial environments. We propose a multi-instrumental approach involving by combining non-destructive and destructive analyses exploiting the analytical instrumental suite at IM@IT and ISIS facilities. In this specific proposal we propose to use RETINA instrument for a 3D reconstruction of the extended samples and collection of XRF data.
Publications	Cockell, C. et al. (2020) Astrobiology 20, 864-877 Gasparri, R. et al (2022) J. of Breath Research 16.4, 046008

Instruments	**IMAT**	**Days Requested:** 2
Access Route	Direct Access	**Previous RB Number:**
Science Areas		**DOI:**
Sponsored Grant	Yes	**Sponsor:** CNR
Grant Title	-	**Grant Number:**
Start Date	-	**Finish Date:**
Similar Submission?		
Industrial Links		

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Sample record sheet

Principal contact	Professor Alessandro Cianchi, University of Rome Tor Vergata, ITALY		
MRF Instrument	**RETINA**	**Days Requested:** 4	
Special requirements:			
	SAMPLE		
Material	slabs of salt	-	-
Formula	salt (NaCl) sediments and organic phases	-	-
Forms	Solid		
Volume	100-150 cc		
Weight	100-150 g		
Container or substrate	-	-	-
Storage Requirements	-	-	-
	SAMPLE ENVIROMENT		
Temperature Range	273 - 320 K	-	-
Pressure Range	1000 - 1010 mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	-	-	-
	SAFETY		
Prep lab needed	No	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	Yes	-	-
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Biogeochemical Multi-Instrumental Study of Rocks and Microbial Biomass in Zechstein Salt Deposits of Boulby using RETINA

1. Background and Context

Can the remains of biological material be preserved in salts many hundreds of millions of years old and what signatures can be preserved? To answer these questions, we must be able to probe the chemical and physical conditions at small scales in ancient salt deposits in order to understand the geological context of biomolecular preservation. We aim to produce a biogeochemical and molecular fingerprint of fossil microbial biomass and inorganic rock samples in the Zechstein salt deposits of Boulby Mine, North Yorkshire, UK through analysing biomarkers, specifically, alkyl lipids (alkanes, fatty acids and alcohols, steroids), glycerol dialkyl glycerol tetraethers (GDGTs), ancient DNA (aDNA) and proteins. This research is timely as the outcomes will help to interpret Raman spectroscopy data produced from the same material. Raman spectroscopy will be one of the analytical tools aboard the next generation Mars rovers. Martian evaporites are prime targets in the search for extra-terrestrial life since the last places where microbial life could have existed on Mars would have been the evaporating oceans. In addition to astrobiology, outcomes are expected to contribute insights into late Permian hydrology and paleoecology. Biomarker distributions in the salt at Boulby mine and particularly in backfilled desiccation cracks can provide information on the environmental conditions in and around the Zechstein Sea ~250 million years ago. The site represents a shallow near shore setting of the Zechstein Basin, with exposure of the evaporite surfaces during sea-level low stand. The presence of biomarkers originating from both microbes and plants in the Boulby salt has already been demonstrated [1]. Upscaling of the extraction procedure and an improved extraction protocol is expected to produce sufficient material for compound-specific carbon and hydrogen isotope analyses ($d^{13}C$, $d^{2}H$) of leaf wax-derived compounds, providing information on continental plant types and aridity.

In this context the knowledge of the exact spatial distribution and inorganic chemical composition of organic matter in the salt on the atomic scale would help much more targeted analyses. For example, leaf waxes may be associated to different material and specific sites compared to microbial membrane lipids. Due to the age of the samples, a good understanding of the exact spatial distribution of the organic matter and its association the inorganic phases of the salt would greatly support targeted analyses. This would allow us to select specific sub-samples with higher organic yields for biomarker investigations and compound-specific isotope analyses, in particular. In addition to targeting the analysis, the physical and chemical context of the biomolecules (or even a lack of them) provides essential information to explain how physical and chemical conditions influence the fate of biomolecules and their potential or long-term preservation over geological time scales. For example, is the presence of any putative biomarkers associated with specific elements or mineral phases? We can answer this by correlating the presence of biomolecules with mineral phases and elemental composition determining using small/wide angle X-ray Scattering by means of SAXS GISAXS; hard X-ray Fluorescence (2D/3D XRF), using RETINA and Multipurpose X-ray diffractometer; and neutron diffraction combined with tomography, using respectively INES and IMAT neutron beamlines. Does the oxidation state of elements, for example iron, influence the chemical environment and thus the presence and preservability/stability of biosignatures in the salt over time? We will answer this by correlating the presence of any biosignatures to the elemental oxidation state in the same location at the relevant scales using mineralogical information. Therefore, we propose a multi-instrumental approach involving a combination of non-destructive and destructive analyses exploiting the analytical instrumental suite at IM@IT and ISIS facilities. All analyses will be using the same sample materials, first, for non-destructive and then destructive methods, to maximise the complementary character of the resulting data sets. On one hand, for the non-destructive analyses of the samples, we use the analytical suite of instruments of IM@IT and ISIS Facilities indicated. The minerals in each salt sample will be quantified using combined Rietveld refinement of X-rays and neutron diffraction data, using laser ablation. On the other hand, due to the very low yields of the organic phase and its fine dispersal in the salt, the samples to be analysed will consist of small slabs of salt of 100-150g, representing two distinct types of material: relatively pure evaporite material and desiccation crack backfill material. The pure evaporite material will be mainly sodium chloride, containing lenses of clay-rich material and isolated potassium chloride crystals. It is expected to include an organic phase originating predominantly from extremophile biomass and some eolian terrigenous input. The slightly darker coloured material from the desiccation cracks, on the other hand, is expected to include higher proportions of terrigenous organic particles and fragments of biofilm from the salt surface that were resuspended and washed into the cracks during rising water level. For the (destructive) analysis of the biomarkers, originating from microorganisms, terrestrial vegetation or processing-related contamination, like alkanes, alcohols, and ketones we propose to use Gas Chromatography – Ion Mobility Spectrometer (GC-IMS), steranes and GDGTs will be analysed by normal-phase UHPLC at Bristol University; for the aDNA analysis, using DNA sequencing NGS, and for the analysis of both fatty acids and ancient proteins using the Mass Spectrometer 2. Genetic, lipid, and proteomic and volatilomic data will then be cross compared to verify their consistency with the possible extremophile species identified. This research is embedded in a wider collaborative attempt to understand extremophile ecology through a comparison with lipid, proteins and DNA data of modern microbes living on the salt surfaces and in brines in Boulby mine. We aim to see if microbial communities adapt to changes in brine salinity and/or ion composition (chloride vs. sulphate, sodium vs. potassium) either by individual species changing their cell membrane properties or by shifts in species distribution. This is a collaboration with Teesside University, the UKRI-STFC Underground Lab. Boulby, the UK Centre for Astrobiology at Edinburgh University, NASA Jet Propulsion Lab, Bristol University, the University of Bern, the University of Rome Tor Vergata, the IM@IT and ISIS facilities. It is supported by the Seedcorn Funding scheme of Teesside University to produce pilot data for a larger proposal to fund PhD projects at Teesside and Edinburgh Universities.

2. Proposed experiment using RETINA

In this proposal we propose the use of RETINA instrument for a non-destructive X-ray tomography analysis of the samples with hard X-ray beams, allowing a 3D reconstruction of the extended samples, and the concurrent collection of X-ray fluorescence data for 3D chemical composition map. The elemental characterisation of samples will be done by Rietveld refinement of X-ray XRD data acquired with RETINA, small/wide angle X-ray Scattering data SAXS GISAXS instrument, hard X-ray Fluorescence (2D/3D XRF), Multipurpose X-ray diffractometer (distinct proposals), and by means of neutron diffraction and tomography performed at INES@ISIS, IMAT@ISIS beamlines and by T-PGAA measurements before and after the 3D reconstruction on IMAT. These will allow a complete digital twin of the sample before its partial destruction in later characterizations. After that we will process the sample for the analysis of biomarker data obtained from destructive analyses of the sample using GC-IMS, for DNA analysis, using DNA sequencing NGS, and for both fatty acids and ancient proteins, using the Mass Spectrometer 2.

3. Justification of experimental time requested

Salt specimens are small slabs of salt of 100-150g, representing two distinct types of material. We aim to measure the sample using a field of view of 20 mm x 40 mm, pixel size of 10 μm, and about 3150 projections to fulfil the Niquist-Shannon sampling theorem. With an exposure time per projection of 5 s, the tomography will last about 4.5 hours. Hence, after discussion with the instrument scientist, we request **4 days** of instrument time including set-up and calibration time.

Experiment Proposal

Experiment number GP2024154

Principal investigator	Dr Monica Carosi, Università Roma Tre, ITALY
Co-investigator	Dr Federica Spani, Università Campus Bio-Medico di Roma, ITALY
Co-investigator (*)	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Multi-instrumental characterization of primate baculum and baubellum bone tissues to support functional hypotheses: micrometric QCT-XRF measurement
MRF Instrument	**RETINA** **Days requested:** 5
Access Route	Direct Access **Previous GP Number:** No
Science Areas	Biology and Bio-materials, Physics **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	To better understand the function of primate bacula and baubella bone tissues, we aim to study the micro-architecture of the bone focussed for the first time on characteristics related to either observed shapes and physical-chemical features of this tissue. Key aspects include trabecular density and orientation, bone mineral composition, and the distribution of minerals along the bones. The proposed study will involve and multi-instrumental characterization of different bones which will allow us to fine reconstruct a digital twin of these tissues, and for open exploring image-based finite element analysis to assess the mechanical forces involved during copulation. Here, this proposal is focussed on the micrometric QCT and XRF analysis.
Publications	Spani F, Morigi MP, Bettuzzi M, Scalici M, Carosi M, PLoS ONE 15(1): e0228131. Spani, F., Morigi, M., Bettuzzi, M. et al., Sci Rep 11 (2021), 11245. Reznikov, N., Bilton, M., Lari, L., Stevens, M. M. & Kröger,Science 360 (2018), eaao2189.

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
MRF Instrument	**RETINA** **Days Requested:** 5
Special requirements:	

SAMPLE

Material	Primate bone tissue	-	-
Formula	Ca10(PO4)6(OH)2	-	-
Forms	Solid		
Volume	0.3 cc		
Weight	0.5 g		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	-	-

Multi-instrumental characterization of primate baculum and baubellum bone tissues to support functional hypotheses: micrometric QCT-XRF measurement

1. Background and Context

Inside the external genitals of some placental mammals, including Primates, genital bones are present in one or both sexes: the *baculum* (penile bone; pl. *bacula*) and the *baubellum* (clitoral bone; pl. *baubella*). *Bacula* are common in most primate species, whereas *baubella* are rare. Both bones occur in some species of Hominoidea (the human evolutionary lineage), but not in humans. Although homologous, *baubellum* is only present in species where males have a *baculum,* whereas species with *bacula* may lack *baubella*. Various functions have been proposed for the *baculum* (none for the *baubellum*), however only one is supported by correlational data: baculum supports erection and prevents urethral collapse, aiding sperm transport in species with prolonged copulations and high levels of sexual competition. *In fact, baculum* length positively correlates with copulation duration. Recent studies published the most comprehensive dataset on primate *bacula* and *baubella* occurrence, collecting data from primary literature and samples from fresh cadavers and museum specimens (Natural History Museum La Specola in Italy, the American Museum of Natural History in New York, and the National Museum of Natural History in Washington, DC). Using 3D high-resolution, non-invasive micro-Computed Tomography and a new landmark-free shape analysis (the *alpha*-shape technique), these studies identified three distinct internal and external morphologies in primate *bacula* for the first time.

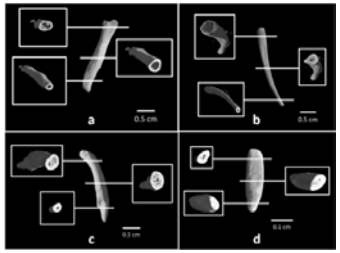

Fig. 1 *3D virtual volumes of 4 different types of primate genital bones, three bacula and one baubellum. For each type, internal structures and cross sections of epiphyses and diaphysis are shown. A: totally hollow structure (Chlorocebus aethiops). B: hollow epiphyses and solid diaphysis with few channels (Otolemur crassicaudatus). C: totally solid structure in both epiphyses and diaphysis with a network of Haversian channels (Papio cynocephalus). D: totally solid structure of baubellum (Sapajus apella) with some Haversian channels*

To better understand the function of primate bacula, the micro-architecture of baculum bone tissue should be investigated focusing for the first time on characteristics related to either observed shapes and mechanical forces exerted on bacula during copulation. Key aspects include trabecular density and orientation, bone mineral composition, and the distribution of minerals along the bones. Potential sex-based differences in these characteristics will aid in interpreting the results. Hence, the proposed study will involve and multi-instrumental approach as follows:

- trabecular density will be assessed by means of quantitative computed tomography (QCT) at different length scale and photon energy which will enable us reconstructing the 3-D bone geometry and volumetric bone mineral density (vBMD);
- trabecular orientation of the bone tissue will be studied in the bulk of the sample by small-angle X-ray scattering (SAXS) to measure crystal shape, their average crystal thickness and their crystal orientation;

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- the structure of bone mineral will be assessed by means of X-ray diffraction (XRD) which is considered as the gold standard for this type of measurement;
- for the compositional characterization of the bone tissue we aim to use nuclear magnetic resonance (NMR) which is uses the responses of isotopes to an external magnetic field to generate compositional information about the sample being scanned, and results will be verified for consistency checks with Raman spectroscopy, Fourier transform infrared spectroscopy (FTIR), X-ray Fluorescence (XRF) spectroscopy, and Energy Dispersive X-ray Analysis (SEM-EDX) data that are used here to determine the chemical and molecular signature of the sample.

2. Proposed experiment

In this specific proposal we aim to use the RETINA instrument available at the POLIMI Unit to perform quantitative computed tomography (QCT) measurement that will allow us reconstructing the 3-D bone geometry, volumetric bone mineral density (vBMD), and chemical composition at micrometric length scale on a n. 3 baculum bone tissue and n. 2 baubella bone tissue. Standard sample solutions based on calcium hydroxyapatite (CHA) on distilled water at different concentration will be measured as well for calibration purpose (required for vBMD). Results of this experiment will be cross compared to verify consistency with a finer spatial resolution (nanometric) QCT data measured on the same set of samples with the XRD TOMOGRAPHY instrument available at the CNR IPCB Unit, and requested by means of a separate proposal.

3. Justification of experimental time requested

Each of the n. 5 samples will be fixed to prevent any sample movement on the tomography rotation stage with a field of view adjust to the sample size, and a number of projections required to fulfil the Niquist-Shannon sampling theorem. Eventual XCT scans with a reduced field of view for increasing spatial resolution will be evaluated if necessary. Further, X-ray fluorescence will be performed on the overall sample and on relevant region of interest to map disuniformities in the chemical composition of the tissue. Hence, after discussion with the instrument scientist, we request 5 days of instrument time including sample preparation, set-up and calibration time.

References

[1] Reznikov, N., Bilton, M., Lari, L., Stevens, M. M. & Kröger, R. Fractal-like hierarchical organization of bone begins at the nanoscale. Science 360, eaao2189 (2018).
[2] Glimcher, M. J. Bone: Nature of the Calcium Phosphate Crystals and Cellular, Structural, and Physical Chemical Mechanisms in Their Formation.*Rev. Mineral. Geochem.*64, 223–282 (2006).
[3] Delmas, P. D., Tracy, R. P., Riggs, B. L. & Mann, K. G. Identification of the noncollagenous proteins of bovine bone by two-dimensional gel electrophoresis. *Calcif. Tissue Int.***36**, 308–316 (1984).
[4] Schultz NG, Lough-Stevens M, Abreu E, Orr T, Dean MD. The Baculum was Gained and Lost Multiple Times during Mammalian Evolution. Integr Comp Biol. 2016 Oct;56(4):644-56. doi: 10.1093/icb/icw034. Epub 2016 Jun 1. PMID: 27252214; PMCID: PMC6080509.
[5] Spani, F., Morigi, M., Bettuzzi, M. et al. The ultimate database to (re)set the evolutionary history of primate genital bones. Sci Rep 11, 11245 (2021). https://doi.org/10.1038/s41598-021-90787-2

Raman Confocal Microscope

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Experiment Proposal

Experiment number GP2024086

Principal investigator (*) Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator

Experiment title	Advanced Analysis of Surface and Composition Variations in Treated Brass Samples with Raman Confocal Microscope	
MRF Instrument	**Raman Confocal Microscope**	**Days requested:** 3
Access Route	Direct Access	**Previous GP Number:** NO
Science Areas	Materials	**DOI:** -
Sponsored Grant	None	**Sponsor:** -
Grant Title	-	**Grant Number:** -
Start Date	-	**Finish Date:** -
Similar Submission?	-	
Industrial Links	RGF SRL	

Non-Technical Abstract

Brass-treated accessories are vital for leather goods and footwear, offering both functionality (like zippers and closures) and aesthetic appeal (such as decorative elements on bags and shoes). Ensuring these components remain durable and free from corrosion is essential. Therefore, research must focus on understanding oxidation to improve manufacturing and surface treatments, guaranteeing the long-term durability and appearance of brass accessories. Understanding the chemical composition on the surface of the brass accessories after corrosion will lead to significant improvements in the development of surface protective treatments and will contribute to the development of more robust and durable fashion and luxury items.

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY
MRF Instrument **Raman Confocal Microscope** **Days Requested:** 3
Special requirements:

SAMPLE

Material	Brass	-	-
Formula	Cu, Zn	-	-
Forms	Solid		
Volume	4 cc		
Weight	10 g		
Container or substrate	No special need	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	RT - K	-	-
Pressure Range	Atmospheric pressure - mbar	-	-
Magnetic field range	No - T	-	-
Standard equipment	-	-	-
Special equipment	No special equipment needed	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	No	-	-
Special equip. reqs	No	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	No	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Advanced Analysis of Surface and Composition Variations in Treated Brass Samples with Raman Confocal Microscope

1. Background and context

Brass-treated accessories are essential in the leather goods and footwear industries due to their dual functional and aesthetic purposes. They serve practical roles as zippers, closures, and handle elements, while also enhancing the visual appeal of the finished products through decorative pieces on bags, charms, and plaques on shoes. It is crucial that these components remain durable and maintain their appearance without corrosion over time. Thus, research and development must focus on understanding surface composition and modification arising from oxidation, in order to optimize manufacturing processes and surface treatments, therefore ensuring long-term durability and functionality of brass accessories themselves and on the finished products, like belts, bags and shoes.

This proposal therefore aims to investigate the surface composition of samples that show corrosive alterations on the surface using Raman Confocal Microscopy, providing detailed elemental and surface information that could be correlated with the mechanisms of corrosion that have occurred on the surface. The fashion and luxury industries could than take proactive measures to treat or modify the product process and the surface finishing applied on the articles, thereby enhancing the durability and quality of the final finished products.

2. Proposed experiment

This proposal is part of a broader study to investigate the modification of the surface composition of treated brass articles after the built-up of oxidation phenomena that gave alteration of the aspect of the surface. The study involves several instrumental techniques: a) RAMAN Spectroscopy; b) X-ray Photoelectron Spectroscopy (XPS); c) Scanning electron microscopy.

RAMAN confocal microscopy investigation would give a great contribution to this study since it is a powerful tool for analysing the surface, providing detailed information on the chemical composition, distribution of compounds, and structural characteristics of the surface. Here are the key types of data that can be obtained: 1) identification of specific compounds present on the accessories surface, including residual oils and other processing chemicals coming from the production process; 2) spatial distribution: mapping the distribution of identified substances across the surface to understand their uniformity or concentration in specific areas to see how these substances are distributed; 3) correlating the presence and concentration of specific substances with the observed corrosive effects in order to identify potential sources of corrosion.

Moreover, thanks to the non-destructive nature of the technique, it reserves the samples for possible further investigations with other instruments.

By conducting these analyses, we will obtain essential data for understanding the corrosive effects that led to alterations on the surface of the brass accessories, useful for research and development teams to improve production processes and surface finishing ultimately leading to improved quality and durability of the accessories.

3. Summary of previous experimental proposals or characterisation

Historically, the durability of brass accessories for the fashion and luxury markets has been constrained by a reliance on empirical methods and trial and error. This approach lacks the detailed insight and predictive capability that sophisticated analytical tools like RAMAN confocal microscopy for the investigation of corrosive phenomena can provide. Moreover, access to such advanced analysis and the requisite expertise is often limited. Although some literature provides a foundational understanding of the surface composition occurring after corrosion for brass articles used for different fields and applications, [1-2], a detailed exploration of the altered surface of the treated brass articles used in the fashion and luxury market for leathergoods and footwear after the occurring of corrosion, has not been conducted extensively so far. This

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research aims to fill that critical knowledge gap, to provide a correlation between the surface-specific elements found with the investigation, enabling to provide then recommendations for production methods and surface treatments that could mitigate the corrosive effects on the surface of metal accessories for fashion and luxury markets.

4. Justification of experimental time requested

As detailed in section 2, RAMAN confocal microscopy is a pivotal tool for this experiment due to its unique capabilities.

To achieve a comprehensive surface analysis of the brass samples, we request machine time for the Raman Confocal microscopy. This will enable us to obtain detailed elemental and surface information, which can be correlated with the corrosion mechanism. We request 3 days of experimental time to analyse 15 samples coming from 3 accessories batches: we collected 3 types of batches of brass accessories; for each batch we will then collect five samples for investigating the effects of the corrosion within the same batch. Single spectra on the surfaces will be initially acquired, both using the 532 nm and the 785 nm laser, in order to define the optimal conditions of measurement. After these preliminary measurements, maps will be acquired on extended areas of the specimens. The analysis of the composition will be performed by a "component analysis", using the database included in the software

This schedule ensures efficient use of the RAMAN confocal microscope, providing detailed insights into the surface composition and chemistry of the corrosion. By understanding these phenomena, we aim to improve the production processes and finishing treatments, in order to enhance the longevity and performance of brass accessories in the fashion and luxury industries.

[1] RAMAN MICRO-SPECTROSCOPIC INVESTIGATION OF CORROSION PRODUCTS. Diaa Atta, Saleh Ahmedc, Mohamed Abdelbar. Egyptian Journal of Chemistry. Vol. 65, No. SI13B pp. 1333 - 1345 (2022)
[2] Initial oxidation of brass induced by humidified air. Ping Qiu*, Christofer Leygraf. Applied Surface Science. 258 (2011) 1235–1241.

Experiment Proposal

Experiment number GP2024108

Principal investigator	Professor Gabriele Gentile, University of Rome Tor Vergata, ITALY
Co-investigator (*)	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Carla Andreani, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Multi-instrumental characterization of Oniscidean isopods to establish the nature and function of their cuticle and tubercles: Raman measurements
MRF Instrument	**Raman Confocal Microscope** — **Days requested:** 3
Access Route	Direct Access — **Previous GP Number:** No
Science Areas	Biology and Bio-materials, Physics — **DOI:** -
Sponsored Grant	None — **Sponsor:** -
Grant Title	- — **Grant Number:** -
Start Date	- — **Finish Date:** -
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

Andronicus is a genus of oniscidean isopods that includes less than a dozen species, most of which are confined to caves in the Italian Pre-Alps. Andronicus offers the unique opportunity to investigate the nature of the cuticle in cave (A. brentanus) and non-cave-dwelling species (A. dentiger), within the same evolutionary lineage, characterizing mineralization in the different species. The proposed investigation will also extend to including tubercles on the surface of the body, the nature of which, currently unknown, could be of a sensorial nature. Preliminary observations would suggest the presence of a possible innervation at the base of these tubercles, which if confirmed, would prove their nature and function. For the characterization on the mineralization of the cuticle and tubercles we envisage to use EDX, FT-IR, Raman, and X-ray diffraction, whereas the morphology characterization will be done by SEM, TEM and nano-XCT. Here, this proposal is focussed on the Raman analysis.

Publications

Vittori, M. et al., Arthropod Struct Dev. 46 (2016), pp. 96-107.
Gentile, G. and Allegrucci, G., International Journal of Speleology 26 (1997), pp. 47-61.
Neues, F. et al., Cryst. Eng. Comm. 9 (2007), pp. 1245-1251.

ISIS neutron and muon source — **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY		
MRF Instrument	**Raman Confocal Microscope**	**Days Requested:** 3	
Special requirements:			

SAMPLE

Material	Oniscidean isopod	-	-
Formula	Organic material, Calcite	-	-
Forms	Solid		
Volume	0.03 cc		
Weight	1-2 g		
Container or substrate	-	-	-
Storage Requirements	Freezer (-20C)	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	-	-

Multi-instrumental characterization of Oniscidean isopods to establish the nature and function of their cuticle and tubercles: Raman measurements

1. Background and Context

Among Crustaceans, oniscidean isopods are uniquely adapted to terrestrial life, exhibiting strongly mineralized cuticles. Onisciceans include several species adapted to the caves. Among the most important evolutionary adaptations found in troglobitic oniscideans (i.e. bound to cave environments, from which they cannot escape due to strict ecological and physiological constraints) are the thinning of the cuticle with a reduce layer of calcite, although calcium carbonate is present in the exocuticle and the endocuticle [1]. Additionally, other adaptations include the lengthening of the appendages, the loss of the eyes, the development of sensory systems alternative to sight such as hygrosensors and chemosensors, usually located in different areas of the body. *Androniscus* (Fig. 1) is a genus of oniscidean isopods that includes less than a dozen species, most of which are confined to caves in the Italian Pre-Alps. Among these, *Androniscus dentiger* is the one that shows the least constraints, being present even in the most superficial layers of the soil in non-cave environments and showing a wide geographical distribution [2]. Indeed, by combining atomic absorption spectroscopy, thermogravimetry and X-ray diffraction, the composition of cuticles in several isopods has been analyzed [3-4]. The use of high-resolution Raman microscopy enabled the determination of the distribution of different mineral phases in the tergal cuticles of some rollers, clingers, and runners [5,6].

Fig. 1 *Androniscus dentiger* (a) and *Androniscus brentanus* (b). Contrary to the second, the first is not troglobite, is pigmented, has thick cuticle, and shows a prominent single-ommatidium eye (arrow).

Androniscus offers the unique opportunity to investigate the nature of the cuticle in cave (*A. brentanus* and more) and non-cave-dwelling species (*A. dentiger*), within the same evolutionary lineage, characterizing mineralization in the different species. The proposed investigation will also extend to including tubercles (tricorns) on the surface of the body, the nature of which, currently unknown, could be of a sensorial nature. Some preliminary observations would suggest the presence of a possible innervation at the base of these tubercles, which if confirmed, would prove their nature and function. For the characterization on the mineralization of the cuticle and tubercles in the two different species (*A. brentanus and A. dentiger*) we plan to use Energy Dispersive X-ray Analysis (SEM-EDX), Fourier-transform infrared spectroscopy (FT-IR), Raman spectroscopy and X-ray diffraction, whereas the morphology characterization will be done by means of electron microscopy techniques (SEM, TEM) and X-ray nano tomography. The benefit of using a multi-instrumental approach would allow us not only to cross compared results to verify their consistency, but also to investigate the degree of resorption/failure to develop the eye in these isopods species, allowing us to observe the presence of vestigial or residual structures, such as for example the presence of an optic nerve, in the absence of the ommatidium (eyeball).

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2. Proposed experiment

In this specific proposal we aim to use the Raman Confocal Microscope instrument available at the CSGI - University of Florence Unit for assessing the degree of mineralization of the cuticle and tubercles on a n. 6 *Androniscus brentanus and n. 6 Androniscus dentiger* isopods samples. Results of this experiment will be cross compared to verify consistency with data obtained by separate proposals where we request FT-IR, SEM-EDX, TEM, XRD and nano XCT measurements on the same set of samples.

3. Justification of experimental time requested

Each of the n. 12 samples of the two Oniscidean isopod species (n. 6 *Androniscus brentanus and n.6 Androniscus dentiger)* will be washed for 1–2 s in double distilled water to remove tissue saline at the surface and then for 2–5 s in 100% methanol to remove water. Specimens will be air dried and stored at −20 °C until its use on the instrument. For the Raman measurement, microtome-polished cuticle and tubercles surfaces will be measured and results compared with pure calcite and amorphous calcium carbonate reference samples. We envisage, after discussion with the instrument scientist, to measure n. 4 samples per day on the instrument. Hence, we request a total of 3 days of instrument time including sample preparation, set-up and calibration time.

References

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[3] Neues, F., Ziegler, A. & Epple, M. (2007) The composition of the mineralized cuticle in marine and terrestrial isopods: a comparative study Cryst. Eng. Comm. 9: 1245-1251.
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[6] Štrus, J., Žnidaršič, N., Hild, S. & Ziegler, A. (2008) Microscopic anatomy and mineral composition of cuticle in amphibious isopods Ligia italica and Titanethes albus (Crustacea: Isopoda) A. Aretz, B. Hermanns-Sachweh, J. Mayer (Eds.), EMC 2008: Life Sciences, Springer Verlag, Berlin:185-186.
[7] Hornung, E. (2011). Evolutionary adaptation of oniscidean isopods to terrestrial life: Structural-physiological-behavioural aspects. Terrestrial Arthropod Reviews. 4: 95-130.

Experiment Proposal

Experiment number GP2024119

Principal investigator	Dr Ivano Aglietto, GrapheneUP SE, CZECH_REPUBLIC
Co-investigator	Dr Gennaro Gentile, IPCB CNR, ITALY
Co-investigator	Dr Marino Lavorgna, CNR, ITALY
Co-investigator (*)	Professor Massimo Bonini, CSGI - University of Florence, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	RAMAN confocal microscopy characterization of innovative graphene-based inks for multifunctional applications
MRF Instrument	**Raman Confocal Microscope**
Days requested:	3
Access Route	Direct Access
Previous GP Number:	-
Science Areas	Chemistry, Engineering, Materials
DOI:	-
Sponsored Grant	None
Sponsor:	-
Grant Title	-
Grant Number:	-
Start Date	-
Finish Date:	-
Similar Submission?	-
Industrial Links	Graphene UP
Non-Technical Abstract	The aim of this proposal is to study, using the RAMAN confocal microscope available at CSGI - University of Florence Unit, the correlation between innovative Few Layers Graphene (FLGs), hybrid fillers consisting of graphene few layers modified with metal, inorganic or carbon particles, the filler spatial distribution, and the processing parameters related to screen-printing deposition technologies. The objective is to investigate how the chemical modification of FLGs, obtained directly during the process of graphene exfoliation starting from graphite, may affect the spatial distribution because of the different technologies adopted for the processing of the composites. In separate experiments SAXS WAXD, SEM and TEM characterization of the inks will be performed.
Publications	-

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E-platform: No

Instruments	
Access Route	
Science Areas	
Sponsored Grant	
Grant Title	
Start Date	
Similar Submission?	
Industrial Links	
Days Requested:	
Previous RB Number:	
DOI:	
Sponsor:	
Grant Number:	
Finish Date:	

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Sample record sheet

Principal contact Professor Massimo Bonini, CSGI - University of Florence, ITALY
MRF Instrument **Raman Confocal Microscope** **Days Requested:** 3
Special requirements:

SAMPLE

Material	FLG based inks (4 samples)	Fillers for FLG based inks (4 samples)	-
Formula	C	C	-
Forms	Solid	Solid	
Volume	1 cc	1 cc	
Weight	1000 mg	1000 mg	
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	- K	-
Pressure Range	- mbar	- mbar	-
Magnetic field range	- T	- T	-
Standard equipment	None	None	-
Special equipment	-	-	-

SAFETY

Prep lab needed	No	No	-
Sample Prep Hazards	NO	NO	-
Special equip. reqs	NO	NO	-
Sensitivity to air	No	No	-
Sensitivity to vapour	No	No	-
Experiment Hazards	NO	NO	-
Equipment Hazards	-	-	-
Biological hazards	NO	NO	-
Radioactive Hazards	NO	NO	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	-

RAMAN confocal microscopy characterization of innovative graphene-based hybrid fillers and their inks for multifunctional applications

Background and Context

Flexible and portable electronic devices and energy storage equipment made from conductive nanomaterials using printing or inks deposition technology have garnered significant attention due to their low-cost, high-throughput, and eco-friendly manufacturing processes over the past few decades. Electrically conductive nanostructured inks have emerged as promising candidates for designing flexible electronics because of their cost-effective synthesis methods and compatibility with current manufacturing processes.

In particular, the development of highly concentrated conductive ink using graphene powders as a raw material is seen as a promising direction. Most high-concentration graphene conductive inks are prepared from low-concentration graphene dispersions containing polymers as stabilizers. However, the solvents used in these dispersions often do not meet the requirements of the printing methods. Among various printing technologies, screen printing shows great promise for industrial-scale production due to its ability to print thick patterns with low sheet resistance (RS) on a wide range of substrates.

To realize applications in graphene-based flexible electronics, further improvement in graphene ink formulation and the development of simple, efficient post-treatment processes for printed patterns are needed. Polymers used to prevent graphene agglomeration in dispersions through steric hindrance can also adjust the rheological properties, storage performance of inks, and the flexibility and adhesion of printed patterns to substrates. New innovative fillers, based on graphene functionalized with metals or other nanoparticles, may be useful for realizing conductive inks with additional properties such as thermal, magnetic, electromagnetic shielding, optical, and catalytic properties.

Therefore, the aim of this proposal is to study, using the instrument suite of IM@IT, the correlation between innovative Few Layers Graphene (FLGs), hybrid fillers consisting of graphene few layers modified with metal, inorganic or carbon particles, the filler spatial distribution, and the processing parameters related to screen-printing deposition technologies. Hybrid fillers may be realized by combining FLGs with pristine metals or inorganic particles, but they can also be prepared by direct synthesis of hybrid graphene structures. A process has been developed to cover the graphene layers with carbon nanotubes and also conductive metal fillers during the direct non-oxidative exfoliation of graphite in gas phase. This process allows to create hybrid structures of few-layer graphene with other fillers without the need of a post-functionalization process.

The objective is to investigate how the chemical modification of FLGs, obtained directly during the process of graphene exfoliation starting from graphite, may affect the morphology of filler as well as the spatial distribution of filler within the inks because of the different technologies adopted for the processing of the composites.

2. Proposed experiment

The characterization of the fillers and inks will be performed as follows.

RAMAN confocal microscopy, available at the CSGI - University of Florence Unit, will provide chemical information about the modified FLGs and their interface interactions with the main components of inks.

In separate experiment proposals, SAXS/WAXD, available at IPCB CNR Unit, will be performed on the fillers and inks and will allow to evaluate the orientation of the filler and its aggregation,

contributing to the enhanced properties of the resulting inks. Furthermore, TEM and SEM characterization of the fillers and inks, performed at the IPCB CNR Unit, will offer insights into the assembling of nanoplatelets and spatial filler distribution.

3. Summary of previous experimental proposals or characterisation

No previous experiments have been carried out on these samples

4. Justification of experimental time requested

We have requested the RAMAN confocal microscope available at the CSGI - University of Florence Unit to characterize 8 samples (4fillers+4inks) obtained by modified FLG with metal nanoparticles, inorganic nanoparticles, multiwalled carbon nanotubes (MWCNTs) and single wall carbon nanotubes (SWCNTs). Non modified FLG will be also characterized by comparison. Therefore, a total of 8 samples will be analysed. After discussion with the instrument scientist, we request 3 days of RAMAN confocal microscope access, for a fully and thorough characterization of the materials. The foreseen beam time accounts for the optimization of the instrumental conditions (selection of the most effective wavelength and power of the laser) and for the data collection on the samples, including collection of maps. The samples will be prepared by deposition hybrid fillers and inks on glasses and/or silicon wafer.

References

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Experiment Proposal

Experiment number GP2024132

Principal investigator (*)	Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY
Co-investigator	Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Stripping of surface treatment from ABS articles investigation with confocal RAMAN microscopy
MRF Instrument	**Raman Confocal Microscope** — **Days requested:** 1
Access Route	Direct Access — **Previous GP Number:** NO
Science Areas	Materials — **DOI:** -
Sponsored Grant	None — **Sponsor:** -
Grant Title	- — **Grant Number:** -
Start Date	- — **Finish Date:** -
Similar Submission?	-
Industrial Links	RGF SRL

Non-Technical Abstract

In the fashion and luxury markets, ABS (Acrylonitrile Butadiene Styrene) components are chosen for their lightweight and cost-effective properties. These components undergo a galvanization process to achieve a metal-like appearance, such as that of brass. This provides the aesthetic qualities of metal accessories at a reduced cost and weight, which is particularly important for large decorative elements on shoes or bags. If made from brass, these elements could be too heavy and potentially damage the final product. With increasing emphasis on sustainability, the industry is focused on recycling defective or scrap products to support circular production. The challenge is to effectively separate the metal coatings from the ABS and recover both materials, ensuring minimal impact on the environment while maintaining product quality. By conducting this study, we will obtain essential data for understanding the surface modifications occurring on the ABS after removal of the various layers.

Publications -

ISIS neutron and muon source — **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY		
MRF Instrument	**Raman Confocal Microscope**	**Days Requested:** 1	
Special requirements:			

SAMPLE

Material	ABS	-	-
Formula	(C8H8·C4H6·C3H3N)n	-	-
Forms	Solid		
Volume	4 cc		
Weight	10 g		
Container or substrate	No special need	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	RT - K	-	-
Pressure Range	Atmospheric pressure - mbar	-	-
Magnetic field range	No - T	-	-
Standard equipment	-	-	-
Special equipment	No special equipment needed	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	No	-	-
Special equip. reqs	No	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	No	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Stripping of surface treatment from ABS articles investigation with confocal RAMAN microscopy

1. Background and context

In the fashion and luxury markets, ABS (Acrylonitrile Butadiene Styrene) components are sometimes used due to their lightweight properties. These components undergo a specific electroplating process that allows for plastic galvanization. The resulting accessories are aesthetically identical to their counterparts made from base metals, such as brass, but offer the advantages of being much more cost-effective and lighter. This is particularly important for large decorative elements on shoes or bags, which would be too heavy if made from brass and could potentially damage the final product.
Given the increasing focus on sustainability, manufacturing industries have been striving to recover and recycle as many defective or scrap products as possible to promote production circularity. For accessories made from materials as diverse as ABS and electroplated metal layers, developing an effective method to separate the metals from the ABS substrate and recover both materials presents a challenge [1, 2].
The goal of this analysis is to verify the effectiveness of removing various electroplated layers from ABS and to characterise the resulting "bare" ABS to understand how the stripping process has affected it.

2. Proposed experiment

Until now, the production of accessories for leather goods has primarily focused on items made from base metals like brass. For the recovery of various metal components, conventional methods of metal stripping and refining have been employed. Recently, however, there has been an increase in the use of base materials such as ABS (Acrylonitrile Butadiene Styrene) in accessory manufacturing. This shift offers significant advantages in terms of cost reduction and lighter weight for accessories. However, it also introduces challenges related to recycling: accessories made with ABS require a specialised process to remove the metal layers from the surface galvanization without damaging the underlying ABS, ensuring it remains suitable for recycling.
For this study, we will use a combination of three techniques: 1) RAMAN confocal microscopy; 2) Field Emission Scanning Electron Microscopy (FESEM) and 3) Fourier Transform Infrared Spectroscopy (FT-IR). RAMAN confocal microscopy investigation would give a great contribution to this study since it is a powerful and versatile analytical technique that allows for the non-destructive, high-resolution chemical analysis of ABS (Acrylonitrile Butadiene Styrene) samples. With confocal Raman microscopy, several important aspects of ABS can be studied in detail.
Each of the three components has distinct Raman signatures, allowing for a detailed analysis of the material's composition and phase distribution.
Here are the key types of data that can be obtained by analysing the surface of the ABS after the removal of the electroplated layers:

1) Chemical Composition Mapping: Confocal Raman identifies the spatial distribution of ABS monomers, creating detailed chemical maps to better understand material properties;
2) Phase Separation and Microstructure: It detects and maps different phases, analyzing the microstructure and the morphology, which influence the possibility of being electroplated again after the stripping procedure;

3) Degradation and Oxidation Studies: Raman detects early degradation, such as butadiene oxidation, through new peaks like carbonyl groups, enabling detection of affected areas after stripping procedure.

Moreover, thanks to the non-destructive nature of the technique, it reserves the samples for possible further investigations with other instruments.
By conducting these analyses, we will obtain essential data for understanding not only the effectiveness of removing various electroplated layers from ABS, bust, most importantly, to ensure if the surface of ABS has undergone some degradation phenomena after removal of the electroplated layers, thus affecting the recyclability of the ABS components.

3. Summary of previous experimental proposals or characterisation

The use of ABS as base material for fashion accessories introduces new challenges, particularly concerning recycling. Accessories made from ABS must undergo a specialised treatment process to effectively remove the metal layers from the surface galvanization without damaging the ABS substrate. This is crucial to ensure that the ABS remains suitable for recycling. Currently, there is a lack of comprehensive data and studies that characterise these materials and their recycling processes. As such, this study aims to fill this gap by thoroughly analysing the stripping process and evaluating the suitability of the recovered ABS for recycling. By doing so, it seeks to provide valuable insights into the recycling of ABS-based accessories and contribute to more sustainable manufacturing practices.

4. Justification of experimental time requested

As detailed in section 2, RAMAN confocal microscopy is a pivotal tool for this experiment due to its unique capabilities.
We request 1 day of experimental time to analyse 5 samples. We will analyse the surface (also in terms of the morphology) of the ABS samples after the stripping procedure, in order to assess the integrity of the surface or the presence of degradation and oxidation phenomena.
These analysis with RAMAN confocal microscope will provide detailed insights into the surface composition and the morphology of the stripped samples. In this way, we aim to assess the integrity of the ABS surface after the removal of the electropated layers, in order to confirm if the ABS could be reused and recycled, in order to enhance the performance in terms of circularity of this kind of accessories in the fashion and luxury industries.

[1] Jae Sik Seo, Ho Tak Jeon, Tae Hee Han, Peeling mechanism of interlocked interface between etched acrylonitrile-butadiene-styrene and electroplated metal layer, Surfaces and Interfaces, Volume 26, 2021, 101337.
[2] Ran Tao, Lujain Fatta, Ruslan Melentiev, Amit K. Tevtia, Gilles Lubineau, Contributions of chemical interactions and mechanical interlocking for the adhesion of electroplated copper to ABS in the Cr(VI) etching process, International Journal of Adhesion and Adhesives, Volume 126, 2023, 103450.

Experiment Proposal

Experiment number GP2024158

Principal investigator	Dr Monica Carosi, Università Roma Tre, ITALY
Co-investigator	Dr Federica Spani, Università Campus Bio-Medico di Roma, ITALY
Co-investigator (*)	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Multi-instrumental characterization of primate baculum and baubellum bone tissues to support functional hypotheses: Raman measurement case
MRF Instrument	**Raman Confocal Microscope** — **Days requested:** 1
Access Route	Direct Access — **Previous GP Number:** No
Science Areas	Biology and Bio-materials, Physics — **DOI:** -
Sponsored Grant	None — **Sponsor:** -
Grant Title	- — **Grant Number:** -
Start Date	- — **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	To better understand the function of primate bacula and baubella bone tissues, we aim to study the micro-architecture of the bone focussed for the first time on characteristics related to either observed shapes and physical-chemical features of this tissue. Key aspects include trabecular density and orientation, bone mineral composition, and the distribution of minerals along the bones. The proposed study will involve and multi-instrumental characterization of different bones which will allow us to fine reconstruct a digital twin of these tissues, and for open exploring image-based finite element analysis to assess the mechanical forces involved during copulation. Here, this proposal is focussed on the Raman analysis.
Publications	Spani F, Morigi MP, Bettuzzi M, Scalici M, Carosi M, PLoS ONE 15(1): e0228131. Spani, F., Morigi, M., Bettuzzi, M. et al., Sci Rep 11 (2021), 11245. Reznikov, N., Bilton, M., Lari, L., Stevens, M. M. & Kröger,Science 360 (2018), eaao2189.

ISIS neutron and muon source — **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
MRF Instrument	**Raman Confocal Microscope** — **Days Requested:** 1
Special requirements:	

SAMPLE

Material	Primate bone tissue	-	-
Formula	Ca10(PO4)6(OH)2	-	-
Forms	Solid		
Volume	0.3 cc		
Weight	0.5 g		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	-	-

Multi-instrumental characterization of primate baculum and baubellum bone tissues to support functional hypotheses: Raman measurement case

1. Background and Context

Inside the external genitals of some placental mammals, including Primates, genital bones are present in one or both sexes: the *baculum* (penile bone; pl. *bacula*) and the *baubellum* (clitoral bone; pl. *baubella*). *Bacula* are common in most primate species, whereas *baubella* are rare. Both bones occur in some species of Hominoidea (the human evolutionary lineage), but not in humans. Although homologous, *baubellum* is only present in species where males have a *baculum,* whereas species with *bacula* may lack *baubella*. Various functions have been proposed for the *baculum* (none for the *baubellum*), however only one is supported by correlational data: baculum supports erection and prevents urethral collapse, aiding sperm transport in species with prolonged copulations and high levels of sexual competition. *In fact, baculum* length positively correlates with copulation duration. Recent studies published the most comprehensive dataset on primate *bacula* and *baubella* occurrence, collecting data from primary literature and samples from fresh cadavers and museum specimens (Natural History Museum La Specola in Italy, the American Museum of Natural History in New York, and the National Museum of Natural History in Washington, DC). Using 3D high-resolution, non-invasive micro-Computed Tomography and a new landmark-free shape analysis (the *alpha*-shape technique), these studies identified three distinct internal and external morphologies in primate *bacula* for the first time.

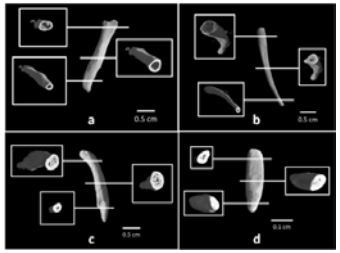

Fig. 1 *3D virtual volumes of 4 different types of primate genital bones, three bacula and one baubellum. For each type, internal structures and cross sections of epiphyses and diaphysis are shown. A: totally hollow structure (Chlorocebus aethiops). B: hollow epiphyses and solid diaphysis with few channels (Otolemur crassicaudatus). C: totally solid structure in both epiphyses and diaphysis with a network of Haversian channels (Papio cynocephalus). D: totally solid structure of baubellum (Sapajus apella) with some Haversian channels*

To better understand the function of primate bacula, the micro-architecture of baculum bone tissue should be investigated focusing for the first time on characteristics related to either observed shapes and mechanical forces exerted on bacula during copulation. Key aspects include trabecular density and orientation, bone mineral composition, and the distribution of minerals along the bones. Potential sex-based differences in these characteristics will aid in interpreting the results. Hence, the proposed study will involve and multi-instrumental approach as follows:

- trabecular density will be assessed by means of quantitative computed tomography (QCT) at different length scale and photon energy which will enable us reconstructing the 3-D bone geometry and volumetric bone mineral density (vBMD);
- trabecular orientation of the bone tissue will be studied in the bulk of the sample by small-angle X-ray scattering (SAXS) to measure crystal shape, their average crystal thickness and their crystal orientation;

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- the structure of bone mineral will be assessed by means of X-ray diffraction (XRD) which is considered as the gold standard for this type of measurement;
- for the compositional characterization of the bone tissue we aim to use nuclear magnetic resonance (NMR) which is uses the responses of isotopes to an external magnetic field to generate compositional information about the sample being scanned, and results will be verified for consistency checks with Raman spectroscopy, Fourier transform infrared spectroscopy (FTIR), X-ray Fluorescence (XRF) spectroscopy, and Energy Dispersive X-ray Analysis (SEM-EDX) data that are used here to determine the chemical and molecular signature of the sample.

2. Proposed experiment

In this specific proposal we aim to use the Raman Confocal Microscope instrument available at the CSGI - University of Florence Unit to perform Raman spectroscopy measurements on n. 5 samples (n. 3 baculum bone tissue and n. 2 baubella bone tissue) to determine the chemical signature of a sample. The technique relies on the characteristic fingerprints of molecular bond vibrations from a material's constituents. When coupled to a microscope and an array detector (as in this specific case) the technique can be used in an imaging mode to assess material composition in a spatially resolved fashion. In bone, Raman spectroscopy (and even FT-IR) can differentiate the molecular signals of the organic matrix components (collagen, proteoglycans, lipids, etc.) from the signals arising from the constituents of the hydroxyapatite (phosphate, carbonate). Hence, results of this experiment will be cross compared to verify consistency with FTIR data measured on the same set of samples with the FT-IR Nexus instrument available at the CSGI - University of Florence Unit, and requested by means of a separate proposal.

3. Justification of experimental time requested

Each of the n. 5 samples will be measured in different region of interest of the sample to establish if any disuniformity is present in the material composition. Hence, after discussion with the instrument scientist, we request 1 days of instrument time including sample preparation, set-up and calibration time.

References

[1] Reznikov, N., Bilton, M., Lari, L., Stevens, M. M. & Kröger, R. Fractal-like hierarchical organization of bone begins at the nanoscale. Science 360, eaao2189 (2018).
[2] Glimcher, M. J. Bone: Nature of the Calcium Phosphate Crystals and Cellular, Structural, and Physical Chemical Mechanisms in Their Formation.*Rev. Mineral. Geochem.*64, 223–282 (2006).
[3] Delmas, P. D., Tracy, R. P., Riggs, B. L. & Mann, K. G. Identification of the noncollagenous proteins of bovine bone by two-dimensional gel electrophoresis. *Calcif. Tissue Int.***36**, 308–316 (1984).
[4] Schultz NG, Lough-Stevens M, Abreu E, Orr T, Dean MD. The Baculum was Gained and Lost Multiple Times during Mammalian Evolution. Integr Comp Biol. 2016 Oct;56(4):644-56. doi: 10.1093/icb/icw034. Epub 2016 Jun 1. PMID: 27252214; PMCID: PMC6080509.
[5] Spani, F., Morigi, M., Bettuzzi, M. et al. The ultimate database to (re)set the evolutionary history of primate genital bones. Sci Rep 11, 11245 (2021). https://doi.org/10.1038/s41598-021-90787-2

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Experiment Proposal

Experiment number GP2024164

Principal investigator (*)	Dr Francesco Saliu, Universita&039; Milano Bicocca, ITALY	
Co-investigator	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY	
Co-investigator		
Co-investigator		
Co-investigator		
Co-investigator		
Co-investigator		
Co-investigator		
Co-investigator		
Experiment title	"Dyes of Valor: Uncovering the Colorful History of Alpine Military Uniforms	
MRF Instrument	**Raman Confocal Microscope**	**Days requested:** 3
Access Route	Direct Access	**Previous GP Number:** -
Science Areas	Cultural Heritage	**DOI:** -
Sponsored Grant	None	**Sponsor:** -
Grant Title	-	**Grant Number:** -
Start Date	-	**Finish Date:** -
Similar Submission?	-	
Industrial Links	AICTC (associazione italiana di chimica tessile e coloristica)	
Non-Technical Abstract	This project aims to investigate the colorants used in vintage Alpine military uniforms through micro-Raman spectroscopy. Historical textiles provide valuable insights into past materials and techniques, reflecting cultural practices and technological advancements. Analyzing the dyes in these uniforms can enhance our understanding of their preservation needs and historical context. By establishing a reference library of identified colorants, the study will identify specific dyes and their chemical structures, including any degradation that may have occurred over time. Various samples from different uniforms will be collected for a systematic analysis. Preliminary assessments may involve visual inspections, while micro-Raman will serve as the primary method for identifying chemical compositions. Additional characterization techniques, such as FTIR and mass spectrometry, may complement the findings. We request 3 days of micro-Raman instrument time to analyze approximately 10 samples, fac	
Publications	-	

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Sample record sheet

Principal contact	Dr Francesco Saliu, Universita&039; Milano Bicocca, ITALY		
MRF Instrument	**Raman Confocal Microscope**	**Days Requested:** 3	
Special requirements:			
	SAMPLE		
Material	Cellulose, PET, PE	-	-
Formula	-	-	-
Forms	Solid		
Volume	cc		
Weight	500 g		
Container or substrate	-	-	-
Storage Requirements	-	-	-
	SAMPLE ENVIROMENT		
Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-
	SAFETY		
Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Dyes of Valor: Uncovering the Colorful History of Alpine Military Uniforms"

Background and Context

Historical textiles, such as military uniforms, offer valuable insights into the materials and techniques used in the past. The analysis of old Alpine military uniforms can reveal information about the dyes and colorants employed, which reflect the technological advancements and cultural practices of their time. Understanding these materials contributes to the fields of textile conservation and historical research. Recent studies have shown the importance of using advanced analytical techniques to identify and characterize the chemical compositions of historical dyes. This project aims to apply micro-Raman spectroscopy to analyze the colorants in vintage Alpine military uniforms, providing a clearer picture of their composition and the dyeing processes used.

Proposed Experiment

Micro-Raman spectroscopy will be utilized to investigate the colorants present in the old military uniforms. The goal is to identify the specific dyes used in the fabrics, focusing on their chemical structure and any degradation that may have occurred over time. A range of samples from different uniforms will be collected, and a systematic approach will be taken to establish a reference library of the identified colorants. The data obtained will help in understanding the historical context of these materials and their preservation needs. Additional characterization techniques, such as FTIR and mass spectrometry, may also be employed to complement the findings.

Summary of Previous Instrument Time or Characterization

Preliminary analyses of the fabric samples may include visual inspections and basic color assessments. The micro-Raman analysis will be the primary method for identifying the chemical composition of the dyes. Reference materials corresponding to known historical dyes will be used to create a comparative dataset.

Justification of Instrument Time Request

We request 3 days of micro-Raman instrument time to analyze a statistically significant number of fabric samples from the military uniforms. Approximately 3 hours will be allocated for experimental setup, with each measurement taking about 1.5 hours, allowing for the characterization of approximately 10 samples across the 3 requested days.

Experiment Proposal

Experiment number GP2024166

Principal investigator	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator (*)	Dr Mattia Gaboardi, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Adriana De Lucia, Arterra Bioscience S.p.A., ITALY
Co-investigator	Dr assunta Tortora, Arterra Bioscience SpA, ITALY
Co-investigator	Dr Mario Campana, Science and Technology Facility Council, UNITED_KINGDOM
Co-investigator	Professor Massimo Bonini, CSGI - University of Florence, ITALY
Co-investigator	Professor Francesca Ridi, University of Florence & CSGI, ITALY
Co-investigator	Professor Silvia Licoccia, University of Rome Tor Vergata, ITALY
Co-investigator	
Experiment title	Characterisation of lipid multilayers mimicking the outer skin layers by Confocal Raman Microscopy
MRF Instrument	**Raman Confocal Microscope**
Days requested:	4
Access Route	Direct Access
Previous GP Number:	-
Science Areas	Biology and Bio-materials
DOI:	-
Sponsored Grant	None
Sponsor:	-
Grant Title	-
Grant Number:	-
Start Date	-
Finish Date:	-
Similar Submission?	-
Industrial Links	Arterra Bioscience

Non-Technical Abstract

We are aiming to study lipid layers that mimic the stratum corneum (SC), the outermost layer of human skin, using Confocal Raman Microscopy, complementing our neutron reflectivity data. SC serves as a primary barrier, preventing water loss and protecting against harmful substances. The way molecules penetrate this layer is key to improving transdermal delivery in cosmetics and medical devices. We will compare three methods for creating skin-like lipid layers: spin-coating, spray-coating, and the more advanced Langmuir-Blodgett/Langmuir-Schaefer technique. The latter allows for precise control over layer composition, potentially leading to more accurate skin models. Uniformity and robustness of these layers will be assessed with a major focus on tracking how a plant-based polypeptide diffuses through them, thus providing insights into how similar compounds penetrate real skin. Depth profiles will be complemented by Raman mapping to shed light on both penetration and distribution.

Publications

Sun, H., et al; J. Colloid interface Sci., 2019, 536, 598-608.
Yang et al., Talanta, 2024, 270, 125559.
Sagle, et al. ACS Appl. Mater. Interfaces 2019, 11, 36, 33442–33451.

Instruments	**SURF**	**Days Requested:**	4
Access Route	Direct Access	**Previous RB Number:**	
Science Areas		**DOI:**	
Sponsored Grant	None	**Sponsor:**	
Grant Title	-	**Grant Number:**	
Start Date	-	**Finish Date:**	
Similar Submission?			
Industrial Links	Arterra Bioscience		

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Sample record sheet

Principal contact	Dr Mattia Gaboardi, University of Rome Tor Vergata, ITALY		
MRF Instrument	**Raman Confocal Microscope**	**Days Requested:**	4
Special requirements:			

SAMPLE

Material	polypeptide layers on Si substrate	-	-
Formula	polypeptide layers	-	-
Forms	Solid		
Volume	cc		
Weight	mg		
Container or substrate	Silicon substrate	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	298 - 298 K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	No	-	-
Sample Prep Hazards	no	-	-
Special equip. reqs	no	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	no	-	-
Equipment Hazards	-	-	-
Biological hazards	no	-	-
Radioactive Hazards	no	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	-	-

Characterisation of lipid multilayers mimicking the outer skin layers by Confocal Raman Microscopy

1. Background and Context

This proposal complements another study where lipid multilayers mimicking the outer skin layers will be investigated by Neutron reflectivity. The skin can be considered as the interface between the human body and the environment. Its main role is to form a barrier against various chemical and biological hazards, as well as to prevent water loss and regulate body temperature [1]. Its thickness can be up to 4 mm and it is composed of several layers, each playing a pivotal function. The outermost layer of the skin, the stratum corneum (SC), plays a paramount role in maintaining the water balance and, as a result, forms the main barrier against the diffusion of substances across the skin [2]. This relatively rigid layer consists of several layers of heavily keratinised corneocytes, which are surrounded by a complex multi-lamellar lipid matrix filling the extracellular space. Passive transport of active molecules across the SC takes place via passive diffusion across the lipid matrix, therefore a deep understanding of these complex diffusion processes is key to the successful delivery of active molecules across the skin using topical applications.

Amongst several studies on the interaction between various compounds with complex lipid structures (*e.g.*, [3]), due to the peculiar composition of the SC, only a few are relevant to skin models. The lipid matrix is in fact composed of ceramides (CER), free fatty acids (FFAs), and cholesterol (CHOL), approximately in equimolar mixture. Phospholipids, which are the most relevant lipids in cell membranes and probably the most studied, can only be found in traces in the SC. CER are the lipids that show the most variability, particularly within the head group region: this can greatly affect their packing and their activity as barrier.

Arterra Bioscience is a company based in Naples, Italy, that develops novel technologies and innovative solutions starting from locally sourced natural raw materials. Arterra Bioscience has a strong interest in transdermal delivery both in cosmetics and particularly in medical devices, as well as obtaining a deep and detailed understanding of diffusion processes across the SC, which would greatly enhance the ability to develop targeted devices. Together with Arterra Bioscience we aim to develop better *in-vitro* models of the SC that could be used, not only to shed light on these processes, but also as tools for rapid *in-vitro* screening. For this reason, it is essential to study how the preparation of these models affects the structure of the deposited lipid matrix.

2. Summary of previous experimental proposals or characterisation

Surface sensitive techniques are required for this task, and neutron reflectivity offers the ability to resolve complex lipid structure at the solid/liquid and solid/air interface to the required level of resolution (*cfr.* Fig. 1), also using deuterium labelling (both in the lipid and in the diffusible molecules) which enables the study the diffusion process in fine details. In the past, skin models were deposited onto solid substrates using spin-coating [4]. There are also several reports of neutron diffraction studies from air/solid systems [5,6], where the SC-mimicking structure was deposited by spraying, followed by evaporation and temperature annealing. With adequate control, these procedures can lead to a thick layer with relatively uniform coverage. However, the main drawback is the lack of control over uniformity and particularly the H/D distribution across the interface, therefore these methodologies have not yet seen a broad use from the scientific community. By using more complex techniques such as Langmuir-Blodgett / Langmuir-Schaefer (LB-LS) we can deposit repetitive lipid layers onto a solid substrate and accurately control the composition of each layer. This way, we can build a complex multi-layer system and still control its composition by depositing layers of known composition. Preliminary work was performed on the Surf reflectometer (at the ISIS neutron spallation source, UK) as a proof-of-principle.

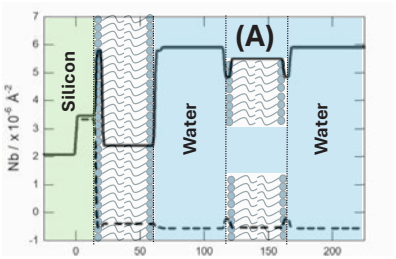

Fig. 1: neutron reflectivity reconstruction of the layers profile at the artificial skin interfacial structure.

3. Proposed experiment

Given the limited time available at a neutron source, the effective use of neutron reflectivity requires previous screening of samples with a suitable technique that allows fast and reliable assessment of surface homogeneity. To this end, Scanning Confocal Raman Microscopy represents a reliable option, especially when samples are supported on SERS-active supports to increase the sensitivity: in fact, Confocal Raman Microscopy has already been used to investigate the composition and structure analysis of different depths in the stratum corneum [7] as well as to study supported lipid bilayers [8], especially with the help of lab-prepared or commercial SERS-active supports. The aim of this experiment is to compare existing skin models obtained from spin-coating and spray-coating with our upgraded LB-LS methodology and, most importantly, follow the diffusive behaviour of a target plant-based polypeptide from an aqueous solution. First and foremost, through this experiment we expect to obtain vital information on the robustness of the different proposed skin models. Additionally, we aim to better understand the distribution of the polypeptide across these lipid multilayers to assess their penetration, ultimately linked to bodily uptake.

4. Justification of experimental time requested

To this end, we request 4 days on the Raman Confocal Microscope instrument at the CGSI – University of Florence for mapping the surface (and depth profile) of 4 samples, each prepared by the 3 different techniques and supported onto 2 different supports. Given the time required to identify the optimal laser conditions (wavelength, power, aperture, and confocality) and the time to acquire one map, after discussions with the local contact, we believe that the time requested is appropriate.

References

1. Gilaberte Y. *et al*., Nanoscience in Dermatology, **2016**, 1-14.
2. Menon G.K. *et al*.; *Int J. Pharm.*, **2012**, 435, 3-9.
3. Qian S. *et al*.; *Langmuir*, **2020**, 36, 15189-15211.
4. Sun, H. *et al*; J. Colloid interface Sci., 2019, 536, 598-608.
5. Groen D. *et al*., *Biophys J.*, **2011**, 100, 1481-1489.
6. Badhe Y. *et al*. *BBA-Biomembranes*, **2022**, 1864, 184007.
7. Yang *et al*., Talanta, **2024**, 270, 125559.
8. Sagle *et al*. ACS Appl. Mater. Interfaces **2019**, 11, 36, 33442–33451.

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Experiment Proposal

Experiment number GP2024039

Principal investigator	Professor Domenico Lo Vetro, Università di Firenze, ITALY
Co-investigator (*)	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Alessandro Cianchi, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Exploring environmental dynamics in ancient remains before and after the last glacial maximum using SAXS and WAXS
MRF Instrument	**SAXS GISAXS**
Days requested:	3
Access Route	Direct Access
Previous GP Number:	No
Science Areas	Biology and Bio-materials, Environment
DOI:	No
Sponsored Grant	None
Sponsor:	-
Grant Title	-
Grant Number:	-
Start Date	-
Finish Date:	-
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

Our aim is to study environmental ancient sediments coming from sediments extracted in the Romito cave (Cosenza, Italy) before and after the Last Glacial Maximum by multi-instrumental approach. The characterization of these samples will assess first the mineralogy composition of the sediment by means of X-ray diffraction and Small/Wide Angle X-ray scattering measurements. Complementary neutron diffraction data on the same set of samples will be measured at the INES beamline, ISIS neutron and muon source (UK). The minerals in each sediment sample will be quantified using Rietveld refinement of X-rays and neutron diffraction data and cross- compared to verify consistency. Hence, we aim here to request access to the SAXS GISAXS instrument operating at the CSGI-Unit of IM@IT.

Publications

Berto et al. 2022. Archaeological and Anthropological Sciences. Vol. 14, article N. 127, (2022)

López-García et al. 2014. Palaeogeography, Palaeoclimatology, Palaeoecology, Vol 251, Issues 3–4, Pages 500-526

Scorrano et al. 2022. Communications Biology, Vol 5, Article N 1262 (2022)

Instruments	**INES**	**Days Requested:**	3
Access Route	Direct Access	**Previous RB Number:**	No
Science Areas		**DOI:**	No
Sponsored Grant	None	**Sponsor:**	
Grant Title	-	**Grant Number:**	
Start Date	-	**Finish Date:**	
Similar Submission?			
Industrial Links			

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Sample record sheet

Principal contact	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY		
MRF Instrument	**SAXS GISAXS**	**Days Requested:**	3
Special requirements:			

SAMPLE

Material	Sediments/remains	Sediments/remains	Sediments/remains
Formula	organic (collagen) and sand/limestone mixtures	organic (collagen) and sand/limestone mixtures	organic (collagen) and sand/limestone mixtures
Forms	Solid	Solid	Solid
Volume	4-10 cc	4-10 cc	4-10 cc
Weight	2-10 g	2-10 g	2-10 g
Container or substrate	Sterile tube or aluminum foil	Sterile tube or aluminum foil	Sterile tube or aluminum foil
Storage Requirements	Freezer (-20C)	Freezer (-20C)	Freezer (-20C)

SAMPLE ENVIROMENT

Temperature Range	273 - 320 K	273 - 320 K	273 - 320 K
Pressure Range	1000 - 1010 mbar	1000 - 1010 mbar	1000 - 1010 mbar
Magnetic field range	- T	- T	- T
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	Yes	Yes
Sample Prep Hazards	No	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	No	No
Sensitivity to vapour	No	No	No
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	No	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	Returned to user by instrument scientist (when inactive)	Returned to user by instrument scientist (when inactive)

Exploring environmental dynamics in ancient remains before and after the last glacial maximum using SAXS and WAXS

Background and Context

Ancient environmental DNA (aDNA) refers to genetic material obtained from environmental samples such as soil, sediment, ice and water, which is thousands or millions of years old [1]. The study of ancient DNA is fascinating because it allows us to reconstruct past ecosystems, understand evolutionary processes and trace the impacts of climate and environmental changes on biodiversity over the millennia. This field is particularly timely, as highlighted by a recent publication in Nature detailing the oldest DNA ever recovered from the environment: 2-million-year-old samples that allowed researchers to reconstruct the ecosystem in Greenland [1].

The broader relevance of ancient DNA research lies in its ability to shed light on the complex interactions between climate, environment and living organisms over geological time scales. By understanding past ecosystems, we gain insights into species resilience and vulnerability, which can guide current biodiversity conservation strategies. This proposal aims to characterize samples coming from sediments extracted in the Romito cave (Cosenza, Italy, see Figure 1) [2, 3] before and after the Last Glacial Maximum (LGM) by multi-instrumental approach. The reason of this is twofold. The LGM, when ice sheets were at their maximum extent, was a period of significant climatic and environmental shifts with profound impacts on global ecosystems and human populations. In an era marked by rapid climate change, insights from the LGM can inform our understanding of how ecosystems and species, including humans, responded to extreme climatic conditions. From the other, the Romito cave is one of the most significant Upper Palaeolithic archaeological sites on the Italian peninsula with a well-dated stratigraphy spanning from ~24,000 to 6,000 years before present (BP) (Figure 1d).

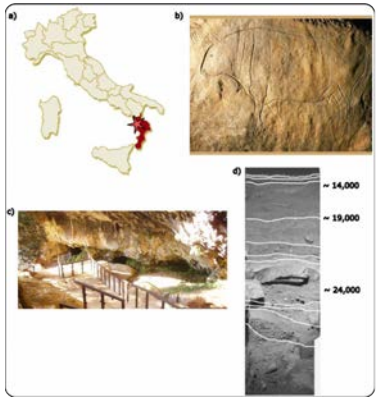

Figure 1. a) Location of Romito cave; b) rock art (Bos primigenius) in the rock-shelter outside the cave; c) cave entrance d) general stratigraphic sequence modified from Blockley et al. (2018).

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To this end, we will study two samples from the oldest layer (pre-LGM, ~24000 years BP) of the sediment, two samples from the LGM (~19000 years BP), and two samples post-LGM (~14000 years BP). The characterization of these samples will be assessing first the mineralogy composition of the sediment by means of X-ray diffraction (Multipurpose X-ray Diffractometer instrument, CNR-ICMATE Unit) and Small/Wide Angle X-ray Scattering (SAXS GISAXS instrument, CSGI-Unit) measurements. Complementary neutron diffraction data on the same set of samples will be measured at the INES beamline, ISIS neutron and muon source (UK). The minerals in each sediment sample will be quantified using Rietveld refinement of X-rays and neutron diffraction data and cross-compared to verify consistency. Later, the presence of organic compounds [4] will be inferred by Fourier-transform infrared spectroscopy measurement on the FT-IR Nicolet instrument available at the Rome Tor Vergata Unit. To follow we will perform shotgun sequencing of aDNA, using DNA Sequencing NGS of Rome Tor Vergata Unit and finally retrieve all the proteins in the remains using Mass Spectrometer 2 at the University of Milano Bicocca Unit which will be useful to support the DNA data [5].

Proposed experiment

In this experiment we aim to perform Small/Wide Angle X-ray Scattering measurements on the SAXS GISAXS instrument of the CSGI-Unit to study the mineralogy composition of n. 6 samples coming from sediments extracted in the Romito cave. Two samples have been extracted by sediments in the oldest layer (pre-LGM, ~24000 years BP), n. 2 samples from the LGM (~19000 years BP), and two samples post-LGM (~14000 years BP). Results of this experiment will be compared with X-ray diffraction measurements performed on the same set of samples with the Multipurpose X-ray Diffractometer instrument (CNR-ICMATE Unit) and with complementary neutron diffraction data still on the same samples collected at the INES beamline, ISIS neutron and muon source (UK). The minerals in each sediment sample will be quantified using Rietveld refinement of X-rays and neutron diffraction data and cross-compared to verify consistency.

Justification of experimental time requested

Each of the n. 6 sample will be enclosed in a sterile tube with about 2 g (4 cc) of sediment, and it will be maintained at -20 °C temperature to preserve aDNA during the measurements. We envisage, after discussion with the instrument scientist, to measure n. 2 samples per day on the instrument. Hence, we request a total of 3 days of instrument time including set-up and calibration time.

References

[1] Kjær et al., Nature **612** (2022), p. 283–291.
[2] Blockley et al. 2018. Quaternary Science Reviews, 184: 5-25.
[3] Craig et al., 2010. Journal of Archaeological Science, 37: 2504-2512.
[4] Scorrano et al., 2015. Annals of Human Biology, 42: 10-19.
[5] Scorrano et al. 2022. Communications Biology, 5: 1262.

Experiment Proposal

Experiment number GP2024050

Principal investigator	Dr Pier Francesco Fabbri, Museo e Istituto Fiorentino di Preistoria, ITALY
Co-investigator (*)	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Carla Andreani, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Alessandro Cianchi, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Ancient DNA analysis of a Neolithic human tooth from eastern Sicily with SAXS GISAXS: insights and implications

MRF Instrument	**SAXS GISAXS**	**Days requested:**	1
Access Route	Direct Access	**Previous GP Number:**	No
Science Areas	Biology and Bio-materials, Cultural Heritage	**DOI:**	No
Sponsored Grant	None	**Sponsor:**	-
Grant Title	-	**Grant Number:**	-
Start Date	-	**Finish Date:**	-
Similar Submission?	-		
Industrial Links	-		

Non-Technical Abstract

This is the first of three IM@IT proposals which aim to analyse with multilevel techniques a tooth belonging to the first Neolithic individual (dated back between 5210-4840 BC) buried at Rocchicella Paliké (CT) in eastern Sicily. We will firstly perform non-destructive X-ray computed tomography (XCT) by XRD TOMOGRAPHY and X-ray diffraction by SAXS GISAXS. 3D rendering and reconstrued XCT scan & X-ray diffraction will be compared and complemented by neutron tomography & diffraction data which will be requested at the IMAT@ISIS and INES@ISIS beamlines. The elemental composition of the sample will be quantified by Rietveld refinement of X-rays data acquired with XRD TOMOGRAPHY and SAXS GISAXS and compared & complemented with neutron diffraction and tomography data. To follow, a destructive shotgun sequencing of ancient DNA (aDNA) will be performed on the tooth, using the DNA Sequencing NGS. Hence, we aim here to request access to the Small/Wide angle with the SAXS GISAXS of IM@IT.

Publications

Lonoce et al. 2023. Journal of Archaeological Science 155, 105790
Viva et al. 2023. Archaeological and Anthropological Sciences 15:193
Vincenti et al. 2023. American Journal of Biological Anthropology 183: e24911.

Instruments	**IMAT**	**Days Requested:**	1
Access Route	Direct Access	**Previous RB Number:**	No
Science Areas		**DOI:**	No
Sponsored Grant	None	**Sponsor:**	
Grant Title	-	**Grant Number:**	
Start Date	-	**Finish Date:**	
Similar Submission?			
Industrial Links			

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Sample record sheet

Principal contact	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY		
MRF Instrument	**SAXS GISAXS**	**Days Requested:**	1
Special requirements:			

SAMPLE

Material	intact tooth (enamel, dentine and cementum) about 2x3x1 cm^3	-	-
Formula	Not known	-	-
Forms	Solid		
Volume	6-8 cc		
Weight	200-500 mg		
Container or substrate	standard INES holder	-	-
Storage Requirements	plastic box	-	-

SAMPLE ENVIROMENT

Temperature Range	273 - 320 K	-	-
Pressure Range	1000 - 1010 mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	No	-	-
Special equip. reqs	No	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	No	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	-	-

Ancient DNA analysis of a Neolithic human tooth from eastern Sicily SAXS GISAXS: insights and implications

1. Background and Context

The Neolithic period, marking the transition from hunter-gatherer societies to agricultural communities, represents one of the most transformative periods in human history (Barker et al. 2015). This shift, often referred to as the Neolithic Revolution, began around 10,000 years ago in the Near East and gradually spread across Europe and the Mediterranean. During the Neolithic transition, farming communities originated in the Middle East and then started to expand into new territories (Hofmanová et al., 2016). These groups spread through Anatolia and the Balkans (Lazaridis et al., 2014; Mathieson et al., 2018), progressively admixing with local hunter-gatherers (Lipson et al., 2017). There are profound differences in the spatiotemporal patterns of Neolithization across Europe, with significant shifts in genetic ancestry varying by region (Allentoft et al., 2024). This genetic transition was particularly extensive in southern Europe, especially in Italy. In this framework east Sicily, strategically located in the central Mediterranean, offers a unique vantage point for studying the diffusion of Neolithic culture and technology. It holds the potential to shed light on the genetic diversity of early Neolithic farmers, their origins, and their genetic legacy. The peopling of Sicily from the Upper Palaeolithic to the Mesolithic has already shown a peculiar pattern involving migrations and partial replacements of local populations (Mathieson et al., 2018; Catalano et al., 2020; Yu et al., 2022; Scorrano et al., 2022).

This project aims to delve deeper into understanding whether the arrival of Neolithic communities in this area involved complex interactions between incoming agriculturalists and indigenous Mesolithic hunter-gatherers by analysing the ancient DNA (aDNA) of the first Neolithic individual from the site Rocchicella Paliké (CT) in eastern Sicily, dated back to the years 5210-4840 BC (calibrated) (LTL12788A) in East Sicily (Figure 1). These interactions likely facilitated the exchange of ideas, technologies, and genetic material, leading to the establishment of early farming villages.

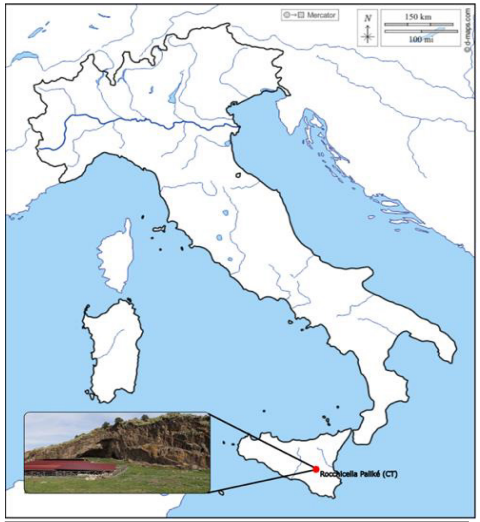

Figure 1: localization of the site.

To characterise the human tooth, we will firstly perform non-destructive X-ray computed tomography (XCT) by XRD TOMOGRAPHY (at the CNR-IPCB Unit), Small/Wide angle X-ray Scattering (SAXS GISAXS instrument, CSGI-Unit), and neutron tomography and diffraction [at the IMAT and INES beamlines, ISIS (UK)]. The elemental composition of the sample will be quantified through Rietveld refinement of X-rays data acquired with XRD TOMOGRAPHY and SAXS GISAXS, and neutron diffraction data, using INES, and neutron tomography, using IMAT, will be cross compared to verify consistency. XCT scan will be complemented with neutron tomography data, which is more sensitive to detect organic-like material. After this analysis a destructive shotgun sequencing of aDNA will be performed on the tooth, using the DNA Sequencing NGS of Rome Tor Vergata Unit.

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2. Proposed experiment

In this experiment we aim to perform a non-destructive Small/Wide angle X-ray Scattering (on the human tooth belonging to the first Neolithic individual buried at Rocchicella Paliké (CT) in eastern Sicily, using the X-ray scattering instrument (SAXS GISAXS) operating at the CSGI-Unit of IM@IT. The elemental composition of the sample will be quantified by Rietveld refinement of X-rays data acquired with SAXS GISAXS and compared with neutron diffraction data collected at the INES@ISIS beamline. Furthermore, on the same sample will be performed a XCT scan and CT reconstructed data will be compared and complemented by neutron tomography measured at the IMAT@ISIS beamline. The characterisation of the sample will end by performing a destructive shotgun sequencing of aDNA of the tooth which will be assessed at the DNA Sequencing NGS instrument, operating at the IM@IT' Unit-University of Rome Tor Vergata.

4. Justification of experimental time requested

We propose to perform Small/Wide angle X-ray Scattering measurements with SAXS GISAXS of the intact human tooth with dimension of 20 mm x 30 mm x 10 mm. Our aim is to collect data of the whole assembly of enamel-dentine-cementum components and compare the results with similar-size modern tooth samples from previous results available in the literature. Hence after discussion with the instrument scientist, we request **1 days** of instrument time including set-up and calibration time.

References

Allentoft et al. Nature 625, 301–311 (2024).
Barker, G. & Goucher, C. (Cambridge Univ. Press, 2015).
Catalano et al. Quaternary International 537, 24-32 (2020).
Hofmanová, Z. et al. Proc. Natl Acad. Sci. 113, 6886–6891 (2016).
Lazaridis, I. et al. Nature 513, 409–413 (2014).
Lipson, M. et al. Nature 551, 368–372 (2017).
Mathieson, I. et al. Nature 555, 197 (2018).
Scorrano et al. Communications Biology 5, 1262 (2022)
Yu et al. iScience 25, 104244 (2022)

Experiment Proposal

Experiment number GP2024052

Principal investigator	Dr Anna Prioriello, University of Rome Tor Vergata, ITALY
Co-investigator (*)	Dr Laura Fazi, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Pietro Morales, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Roberto Senesi, University of Rome Tor Vergata, ITALY
Co-investigator	
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Co-investigator	
Co-investigator	
Experiment title	Training on the use of SAXS-GISAX to investigate Polyethylene-Single Wall Carbon Nanotubes composite
Training MRF	**SAXS GISAXS**
Access Route	Direct Access
Science Areas	Materials
Sponsored Grant	None
Grant Title	-
Start Date	-
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	Composite materials based self-grafted carbon nanotubes (CNTs) in polymers have a variety of applications ranging from biomedical to aerospace. Their study is often based on dynamical, mechanical, electrochemical and surface probes. However, it remains difficult to assess the degree of penetration of CNT bundles within the polymer and the type of the interaction at the interface. In this context, we propose a training access to perform Small-angle x-ray scattering (SAXS) to assess the applicability of the technique in the investigation of composite materials based self-grafted CNTs in polyethilene. In particular, the aim of the training is to understand the technical requirements for sample preparations, and the procedures for running the measurement as well as for the analysis of the collected data. The training will broaden the portfolio of techniques to study this family of samples, and will also allow strengthening of the collaboration within units of the IM@IT infrastructure.
Publications	-

Days requested: 2
Previous GP Number: -
DOI: -
Sponsor: -
Grant Number: -
Finish Date: -

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E-platform: No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Dr Laura Fazi, University of Rome Tor Vergata, ITALY
Training Instrument **SAXS GISAXS** **Days Requested:** 2
Special requirements:

SAMPLE

Material	-	-	-
Formula	-	-	-
Forms			
Volume			
Weight			
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	-	-	-
Pressure Range	-	-	-
Magnetic field range	-	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	-	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	-	-	-
Sensitivity to vapour	-	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	-	-	-

Training on the use of SAXS-GISAX to investigate Polyethylene-Single Wall Carbon Nanotubes composite

1. Background and Context

Conductive stretchable composites are of great interest because of their wide-ranging potential application. They are however extremely complex super-molecular systems, whose detailed mechanical and electrical properties depend on the fabrication method and are not easily established. Combining the electrical conductivity, the robustness and the elasticity of Single Wall Carbon Nanotubes (SWCNT) “as grown” bundles, with the viscoelastic properties of polymer films, such as their stretchability and mouldability, a promising category of composite materials, particularly useful in the biomedical field, have been obtained. Such combination may also be cheap to obtain and versatile in use, due to the self-grating properties of SWCNT on most polymeric substrates, which even allows for the accomplishment of micropatterned devices. For example, by self-grafting SWCNT bundles onto the fairly plastic and mouldable poly-ethylene (PE), we achieved closely spaced microelectrode arrays to monitor, and possibly control, the cortical brain activity of laboratory model animals [1]. On the other hand, a SWCNT/poly-dimethyl-siloxane (PDMS) self-assembled composite, which is much more elastic, allowed the realization of an artificial bladder prototype.
The interface properties between polymer and nanotube bundles have been investigated, since it is understood that the properties of composite materials depend on the nanotubes grafting into the polymeric matrices. Preliminary information about the composite material morphology and conformation have been obtained by Scanning Electron Microscopy and confocal micro-Raman spectroscopy; while the mechanical and electrical properties of the composite have been investigated by measurements of stress vs. strain, of resistance vs. strain and of current vs voltage applied to thin film stripes of SWCNT/polymer composites [2]. Our preparatory experiments reveal a deeper penetration of SWCNT into the polymer bulk for thermosetting elastomer substrates with respect to previous investigation on thermoplastic substrates; the former showing diffusion of the SWCNT limited to few micrometers, while the latter allowing much deeper diffusion. Confocal Micro-Raman spectroscopy shows that SWCNT have drifted into the polymer matrices, even if the limited spatial resolution does not allow detection of single ropes or small coils of nanotubes.
Therefore, additional details are required on the distribution of nanotubes into the polymer bulk and the polymer chains organization within and around the nanotube bundles. Following previous investigations from the literature on analogous specimens [3-5], we believe that the SAXS- GISAXS technique could give us a more detailed insight into the structure of the composite.

2. Proposed Training

In the present training proposal, we wish to use SWCNT-PE sample, in which the nanotubes are self-grafted onto the polymer, which were already characterized by SEM and micro-Raman Spectroscopy. The aim of this proposal is understanding the possibilities offered by this technique and its limit in the study of our composite materials; we would like to use our composite specimens to tackle the understanding of structure of the intertwining polymer chains with carbon nanotube bundles. A further investigation that may partially addressed by this technique is the gradient of CNT concentration into the polymer bulk. We expect with this training activity to acquire skills on the SAXS-GISAXS technique, starting from the precautions to be taken in the realization of the samples

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to continue with the choice of the optimal parameters for the measurement and to end with the analysis of the data returned by the measurement itself.

3. Summary of previous training proposals

This kind of sample was analysed by SEM and micro-Raman spectroscopy at UTOV Unit of ISIS@MACH Italia, obtaining a preliminary information on the gradient of penetration in the bulk and the morphology of the CNT superficial layer as mentioned in the Background and Context paragraph.

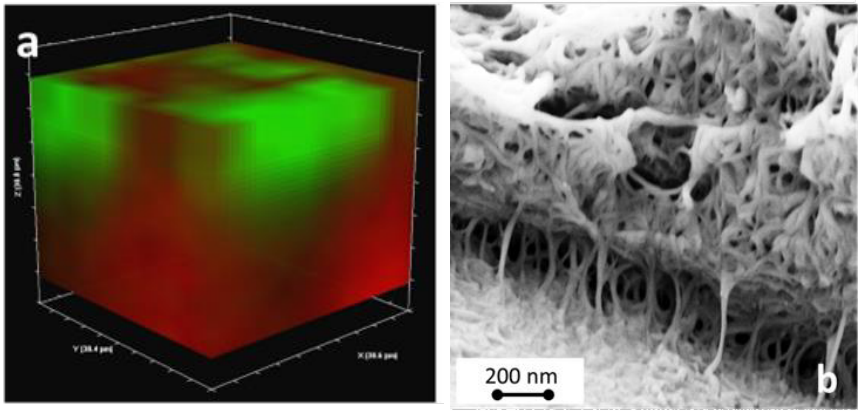

Figure1: (a) 3D Confocal Raman characterization of a thin layer of SWCNT deposited on a PE substrate, where SWCNT are identified in green and MD-PE in red; and (b) SEM characterization of SWCNT/PE composite material, which shows the polymer soaking and wrapping both the CNT ropes and the CNT layer. This was partially separated from the polymer film surface by the CNT layer lifting obtained by immersion of the sample in liquid nitrogen.

4. Justification of experimental proposals request

The request is for 2 days of the CSGI Unit - University of Florence SAXS-GISAXS instrument time for the collection of statistically significant data to characterize the interaction between the polymer chains and the nanotubes. In particular, the PE-SWCNT composite sample will be measured by SAXS-GISAXS scans using parameters depending on the sample. Hence, we request 2 days of instrument time including the training, sample preparation, set-up and calibration time.

References

[1] L. Pavone, et al.; Journal of Neural Engineering. 2020 Jul 3;17(3):036032.
[2] L. Fazi, et al. , Molecules 2023 Feb 13; 28(4):1764.
[3] Chatterjee, T. (2007). Advanced Materials, 19(22), 3850-3853.
[4] Amoroso, L., (2020). Polymer, 201, 122587.
[5] Wurm, A., (2014). Polymer, 55(9), 2220-2232.

Experiment Proposal

Experiment number GP2024071

Principal investigator	Dr Valentina Nigro, ENEA, ITALY
Co-investigator (*)	Dr BARBARA RUZICKA, ISC CNR, ITALY
Co-investigator	Dr Roberta Angelini, National Research Council
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Study of morphological changes of soft responsive microgels at solid interfaces using SAXS GISAXS
Training MRF	**SAXS GISAXS** — **Days requested:** 2
Access Route	Direct Access — **Previous GP Number:** -
Science Areas	Physics — **DOI:** -
Sponsored Grant	None — **Sponsor:** -
Grant Title	- — **Grant Number:** -
Start Date	- — **Finish Date:** -
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

Microgel-formed films are gaining increasing interest for applications in soft nanotechnology and as model systems for studying nanoscale structuring of soft colloids at solid interfaces. Understanding the structure-property relationship of microgel films is crucial for novel strategies in sensing and biosensing, tissue engineering or as temperature-responsive cell surfaces.
GISAXS measurements are essential for investigating the influence of surface confinement on the internal structure and lateral ordering of adsorbed microgels at the solid-liquid interface. A specific training on the SAXS GISAXS instrument is required to test the feasibility on microgels of different sizes and composition spin-coated on various substrates. Details at different length scales will be achieved through comparison with AFM measurements on the AFM BIO instrument, requested through a separate Training Proposal. Additionally, this proposal will strengthen the cooperation between ENEA and ISIS@MACH ITALIA.

Publications -

ISIS neutron and muon source — **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr BARBARA RUZICKA, ISC CNR, ITALY		
Training Instrument	**SAXS GISAXS**	**Days Requested:** 2	
Special requirements:			
	SAMPLE		
Material	-	-	-
Formula	-	-	-
Forms			
Volume			
Weight			
Container or substrate	-	-	-
Storage Requirements	-	-	-
	SAMPLE ENVIROMENT		
Temperature Range	-	-	-
Pressure Range	-	-	-
Magnetic field range	-	-	-
Standard equipment	-	-	-
Special equipment	-	-	-
	SAFETY		
Prep lab needed	-	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	-	-	-
Sensitivity to vapour	-	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	-	-	-

Study of morphological changes of soft responsive microgels at solid interfaces using SAXS GISAXS

1. Background and Context

Microgel-formed films are gaining interest in the scientific community due to their potential technological applications and as model systems for studying self-assembly and phase behaviours at interfaces [1]. Recent research has primarily focused on microgels at liquid-liquid or air-liquid interfaces [2], overlooking behaviours emerging from their adsorption to solid surfaces. The possibility to arrange microgels in 2D arrays inspires their use as smart coatings in soft nanotechnology utilizing their stimuli-responsive behaviour.

Thermo-responsive microgels based on poly(N-isopropylacrilamide) (PNIPAM) are especially promising for their application in sensing and biosensing, as functional tissue in regenerative medicine or even for drug release, thanks to their Volume Phase Transition Temperature (VPTT) at 32°C. In the last years, our group has widely investigated their bulk properties through Dynamic Light Scattering (DLS), rheology, calorimetry, Small-Angle Neutron Scattering (SANS), Small Angle X-ray Scattering (SAXS), Raman spectroscopy, X-ray Photon Correlation Spectroscopy (XPCS), transmission electron microscopy and electrophoretic measurements [3]. Recently, PNIPAM microgel films have been proposed as smart coatings for solid-state radiation detectors for radiobiological experiments on cell cultures (BIOTRACK project, Regione Lazio [4]). Exploiting their thermo-responsiveness, PNIPAM microgel films can be used for fabricating cell surfaces able to modulate cell attachment/detachment with temperature [5]. We have therefore investigated the optical and morphological properties of PNIPAM microgels spin-coated on solid substrates through UV-Vis-NIR spectroscopy, wettability measurements and Atomic Force Microscopy (AFM) [6]. In particular, AFM measurements in the dry state revealed the formation of densely packed layers with particle arrangements depending on microgel size, particle softness, packing density and growth conditions (Fig.1).

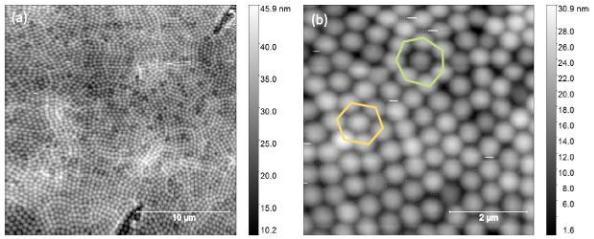

Fig. 1. AFM images on (a) 24×24 µm² and (b) 6×6 µm² area of a film of PNIPAM microgels on glass.

In this framework, application of surface sensitive techniques like Grazing Incidence Small-Angle X-ray Scattering (GISAXS) is of great interest for morphological and structural studies at the solid-liquid interface.

Using the combination of AFM and GISAXS, a detailed characterisation at different length scales will be achieved, providing information on the influence of surface confinement on the internal structure and lateral ordering of adsorbed microgels.

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Initial characterisation of the microgels after adsorption will be performed through the AFM BIO instrument, required through a separate Training Proposal, while bulk properties as hydrodynamic radius, inter-particle distances and characteristic sizes of intra-particle inhomogeneities, have been investigated through SANS measurements previously performed at the ISIS Neutron Spallation Source. This will enable a direct comparison between particle parameters in bulk and in adsorbed state.

2. Proposed Training

Training on the SAXS GISAXS instrument at CSGI-University of Florence is required to evaluate feasibility of GISAXS measurements on microgel films in a wide Q-range. Feasibility of GISAXS measurements should be tested for microgels of different sizes spin-coated on various solid substrates (glass, silicon, silica, etc.) at temperatures below and above the VPTT. While the bulk properties of PNIPAM microgels were widely investigated, GISAXS measurements are needed to probe the structure at solid-liquid interface to understand the influence of the surface confinement on the responsivity of microgel films. GISAXS measurements will be combined with in-liquid AFM through the AFM BIO instrument, and compared with results from DLS, SANS and SAXS on aqueous suspension of PNIPAM microgels. Training is requested for a maximum of two researchers and will be carried out by MRF staff.

3. Summary of previous training proposals

No previous training proposals have been submitted.

4. Justification of training proposal request

SAXS GISAXS instrument is requested for structural characterisation of adsorbed microgels on various substrates at temperatures in the range T=20-50°C. Training is required for grazing incidence SAXS mode in a Q-range enabling the investigation of the lateral behaviour and the internal structure of the adsorbed microgels.

Considering the following breakdown, 2 full days of training are requested:

- Sample handling + basic operations to set-up the instrument = 1day
- Optimization of measurement conditions + data analysis = 1 day

[1] Shaulli, X. et al. ACS Nano 17 (3) (2023) 2067-2078.
[2] Vialetto J. et al., ACS Nano 15 (8) (2021) 13105-13117.
[3] Nigro, V. et al., Polymers 13 (2021) 1353.
[4] https://www.biotrack.enea.it/it/
[5] Sanzari, I. et al., Sci. Rep. 10 (2020) 6126.
[6] Nigro, V. et al., Colloids Surf. A: Physicochem. Eng. Asp. 674 (2023) 131918.

Experiment Proposal

Experiment number GP2024072

Principal investigator	Professor Fabiana Arduini, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Vincenzo Mazzaracchio, University of Rome "Tor Vergata", ITALY
Co-investigator (*)	Professor Massimo Bonini, CSGI - University of Florence, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	SAXS characterisation of inks based on carbon black/Prussian Blue nanocomposites for paper-based electrochemical (bio)sensors

MRF Instrument	**SAXS GISAXS**	**Days requested:**	2
Access Route	Direct Access	**Previous GP Number:**	No
Science Areas	Chemistry	**DOI:**	-
Sponsored Grant	Yes	**Sponsor:**	Other
Grant Title	Lazio Innova Venture and Scientifica Venture Capital	**Grant Number:**	CI Fast
Start Date	01/01/2024	**Finish Date:**	01/07/2025
Similar Submission?	-		
Industrial Links	SENSE4MED		

Non-Technical Abstract

Electrochemical paper-based devices have opened a new route in the analytical science sector, having a huge impact at the academic level as well as in the industrial sector. The development of electrochemical devices with nanomaterials, including carbon black, iridium oxide, and Prussian blue nanoparticles, requires surface and dispersion characterization for obtaining accurate nanomaterial-functionalized paper-based electrochemical devices. The several characterizations that will be carried out thanks to the instrumentation within ISIS@MACH ITALIA will foster the development of electrochemical paper-based devices to carry with national and European projects in which the applicant (Prof. Fabiana Arduini) is the coordinator. In this framework we are proposing a multi-technique approach to investigate nanocomposites made of carbon black and Prussian Blue nanostructures, where SAXS uniqueness relies on the possibility to investigate the nanostructures size, shape and assembly in the inks.

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Professor Massimo Bonini, CSGI - University of Florence, ITALY		
MRF Instrument	**SAXS GISAXS**	**Days Requested:**	2
Special requirements:			

SAMPLE

Material	Water, dimethylformamide, black carbon, Iron, nitrogen	carbon black	-
Formula	carbon black prussian blue nanoparticles	carbon black	-
Forms	Liquid	Liquid	
Volume	1 ml	10 ml	
Weight	1 g	10 g	
Container or substrate	vial, to be trasferred into quartz capillaries, available at the facility	vial	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	298 - K	298 - K	-
Pressure Range	1 - mbar	1 - mbar	-
Magnetic field range	- T	- T	-
Standard equipment	-	None	-
Special equipment	nothing	no	-

SAFETY

Prep lab needed	Yes	Yes	-
Sample Prep Hazards	no	no	-
Special equip. reqs	no	no	-
Sensitivity to air	No	No	-
Sensitivity to vapour	No	No	-
Experiment Hazards	no	no	-
Equipment Hazards	-	-	-
Biological hazards	no	no	-
Radioactive Hazards	no	no	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	-

SAXS characterisation of inks based on carbon black/Prussian Blue nanocomposites for paper-based electrochemical (bio)sensors

1. Background and Context

As reported by the applicant in the recent review entitled "Electrochemical paper-based devices: When the simple replacement of the support to print ecodesigned electrodes radically improves the features of the electrochemical devices" published in Current Opinion in Electrochemistry (Q1) SI: Emerging Opinions (2022) [1]: "*Paper-based electrochemical (bio)sensors have emerged as highly attractive analytical devices for their superior sustainable features, such as avoiding the use of polyester as support and the reduction of waste, being incinerated after use. However, paper-based electrochemical (bio)sensors have recently demonstrated further advantages, including the simple combination with vertical microfluidics and their use as a reservoir to deliver smart electrochemical (bio)sensors able to i) contain the reagents, ii) preconcentrate the target analyte, and iii) synthesize the nanomaterials inside the paper network. Furthermore, these devices have demonstrated their ability to overcome the limitations of the other printed electrochemical sensors in the measurement of entirely liquid samples by detecting the target analyte in the aerosol phase or solid sample, without the additional sampling system. These achievements highlight their valuable and varied advantages in the sensing sector*". Electrochemical paper-based devices have opened a new route in the analytical science sector with a huge impact at the academic level as well as in the industrial sector, since the relevant articles of Prof. Henry in United States [2, 3] and the applicant in Europe [4, 5]. From industrial point of view, the spin-off company SENSE4MED, Department of Chemical Science and Technologies, University of Rome Tor Vergata, in which the applicant is the CEO, has received an investment of 510 KEuro for the developing a paper-based electrochemical sensor for cystic fibrosis diagnosis [6], from the academic point of view, the applicant is the coordinator of the PRIN2022 SMARTMASK4CF with the aim to develop a facemask functionalized with paper-based devices for precision medicine in cystic fibrosis [7]. Additionally, the paper-based devices are creating a new challenge in organ on the chip field, considering the Pathfinder Open Horizon Europe project Phoenix-OoC (2024-2027) which has the overriding goal to create an organ chip on paper and in which the applicant is the European coordinator [8].
In this framework we are proposing a multi-technique approach to investigate nanocomposites made of carbon black and Prussian Blue nanostructures, where Small Angle Scattering of X-rays (SAXS), Transmission Electron Microscopy (TEM) and Scanning Electron Microscopy (SEM) are combined to get a full picture of the system.

2. Proposed experiment

Given the relevance of the self-assembly of nanostructures in inks, especially in terms of potential aggregation and its effect on the casted material, we propose here to characterise the dispersions in water:dimethylformamide mixture (1:1 vol/vol) of carbon black and Prussian Blue nanostructures using the Small Angle Scattering of X-rays instrument (SAXS) located at the CSGI Florence Unit of IM@IT. We propose to investigate 5 dispersions, in addition to the reference dispersions (i.e., dispersions of the single components).
This proposal is part of a multi-technique investigation, complemented by a separate proposal to characterize the nanocomposites by TEM at the IPCB-CNR Unit in Naples and, once the nanocomposites are deposited, to verify the quality of the casted materials by FE-SEM (where no metallization is needed) at the CSGI Florence Unit. SAXS uniqueness in this framework relies on the possibility to fit the results according to structural models mimicking the dispersion of nanostructures in a solvent (such as the dispersion of single objects or the formation of fractal-like aggregates), with a statistic relevance that cannot be matched by microscopy techniques.

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3. Summary of previous experimental proposals or characterisation

In previous experiments we have characterised carbon black nanostructures by means of TEM analysis [9], but we have not yet performed any TEM analysis of the carbon black-Prussian Blue nanoparticle composite used in paper-based electrochemical devices. SAXS has not been used in the past on these samples: nevertheless, the strong difference in the X-ray scattering length density of carbon black and Prussian Blue suggests the possibility to effectively detect the formation of composites rather than the presence of separate objects. Furthermore, the complementary use of TEM will strongly help in the definition of a structural model for the fitting of SAXS data.

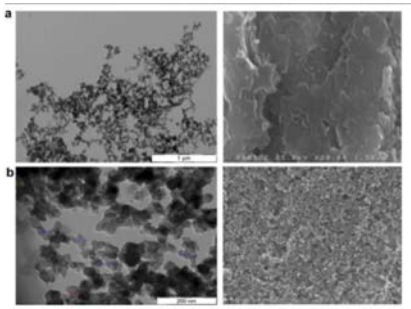

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Fig. 1. (A) TEM images of the CB material (a and b). (B) SEM imagines of bare SPE (a) and CB-SPE (b).

4. Justification of experimental time requested

Experimental time and conditions have been discussed together with the local contact. The 5 different dispersions (in addition to the 2 references) will be contained into capillaries, also thanks to their stability against sedimentation. SAXS curves will be acquired by taking advantage of two different sample-to-detector distances, in order to cover the entire Q range available. Different concentrations will be investigated to optimize signal-to-noise ratio. Taking into account the sample preparation, the acquisition time and the time needed for the modelling of the results, we are requesting 2 days of instrument time.

References

[1] Arduini, F., 2022. Current Opinion in Electrochemistry, **35**, p.101090.
[2] Aryal, P., Hefner, C., Martinez, B. and Henry, C.S., 2024. Microfluidics in environmental analysis: advancements, challenges, and prospects for rapid and efficient monitoring. Lab on a Chip.
[3] Ozer, T., McMahon, C. and Henry, C.S., 2020. Annual Review of Analytical Chemistry, **13**(1), pp.85-109.
[4] Cinti, S., Minotti, C., Moscone, D., Palleschi, G. and Arduini, F., 2017. Biosensors and Bioelectronics, **93**, pp.46-51.
[5] Cinti, S., Talarico, D., Palleschi, G., Moscone, D. and Arduini, F., 2016. Analytica chimica acta, 919, pp.78-84.
[6] https://www.lazioinnova.it/news/innova-venture-e-scientifica-venture-capital-investono-in-sense4med/
[7] https://stc.uniroma2.it/ricerca/progetti-di-ricerca/
[8] https://phoenixooc.com
[9] Arduini, F., Amine, A., Majorani, C., Di Giorgio, F., De Felicis, D., Cataldo, F., Moscone, D. and Palleschi, G., 2010. *Electrochemistry communications*, ***12***(3), pp.346-350

Experiment Proposal

Experiment number GP2024138

Principal investigator	Professor Charles Cockell, University of Edinburgh, UNITED_KINGDOM
Co-investigator	Dr Jens Holtvoeth, Teesside University, UNITED_KINGDOM
Co-investigator (*)	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Carla Andreani, University of Rome Tor Vergata, ITALY
Co-investigator	Miss Julia Puputti, Boulby Underground Laboratory STFC, UNITED_KINGDOM
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Biogeochemical Multi-Instrumental Study of Rocks and Microbial Biomass in Zechstein Salt Deposits of Boulby using SAXS GISAXS
MRF Instrument	**SAXS GISAXS** **Days requested:** 3
Access Route	Direct Access **Previous GP Number:** No
Science Areas	Biology and Bio-materials, Chemistry, Environment, Materials **DOI:** -
Sponsored Grant	Yes **Sponsor:** CNR
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	Our aim is to investigate the potential for preserving biological material in ancient salt deposits, with a focus on the Zechstein salt deposits in Boulby Mine (UK), offering insights into the environmental conditions of the Zechstein Sea ~250 million years ago. The study employs a multi-instrumental approach, combining non-destructive and destructive analyses to correlate biomolecule presence with mineral phases and elemental compositions. By analyzing a range of biomarkers the research aims to create a detailed biogeochemical fingerprint of fossil microbial biomass. This research contributes to astrobiology and our understanding of ancient terrestrial environments. We propose a multi-instrumental approach involving by combining non-destructive and destructive analyses exploiting the analytical instrumental suite at IM@IT and ISIS facilities. In this specific proposal we use SAXS GSAX small/wide angle X-ray Scattering to highligth mesoscopic organic and inorganic structures.
Publications	Cockell, C. et al. (2020) Astrobiology 20, 864-877. Gasparri, R. et al (2022) J. of Breath Research 16.4, 046008

Instruments	**INES** **Days Requested:** 2
Access Route	Direct Access **Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	Yes **Sponsor:** CNR
Grant Title	- **Grant Number:**
Start Date	- **Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY		
MRF Instrument	**SAXS GISAXS**	**Days Requested:** 3	
Special requirements:			
	SAMPLE		
Material	slabs of salt	-	-
Formula	salt (NaCl) sediment and organic phase	-	-
Forms	Solid		
Volume	100-150 cc		
Weight	100-150 g		
Container or substrate	-	-	-
Storage Requirements	-	-	-
	SAMPLE ENVIROMENT		
Temperature Range	273 - 320 K	-	-
Pressure Range	1000 - 1010 mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-
	SAFETY		
Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	Yes	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Biogeochemical Multi-Instrumental Study of Rocks and Microbial Biomass in Zechstein Salt Deposits of Boulby using SAXS GISAXS

1. Background and Context

Can the remains of biological material be preserved in salts many hundreds of millions of years old and what signatures can be preserved? To answer these questions, we must be able to probe the chemical and physical conditions at small scales in ancient salt deposits to understand the geological context of biomolecular preservation. We aim to produce a biogeochemical and molecular fingerprint of fossil microbial biomass and inorganic rock samples in the Zechstein salt deposits of Boulby Mine, North Yorkshire, UK through analysing biomarkers, specifically, alkyl lipids (alkanes, fatty acids and alcohols, steroids), glycerol dialkyl glycerol tetraethers (GDGTs), ancient DNA (aDNA) and proteins. This research is timely as the outcomes will help to interpret Raman spectroscopy data produced from the same material. Raman spectroscopy will be one of the analytical tools aboard the next generation Mars rovers. Martian evaporites are prime targets in the search for extra-terrestrial life since the last places where microbial life could have existed on Mars would have been the evaporating oceans. In addition to astrobiology, outcomes are expected to contribute insights into late Permian hydrology and paleoecology. Biomarker distributions in the salt at Boulby mine and particularly in backfilled desiccation cracks can provide information on the environmental conditions in and around the Zechstein Sea ~250 million years ago. The site represents a shallow near shore setting of the Zechstein Basin, with exposure of the evaporite surfaces during sea-level low stand. The presence of biomarkers originating from both microbes and plants in the Boulby salt has already been demonstrated [1]. Upscaling of the extraction procedure and an improved extraction protocol is expected to produce sufficient material for compound-specific carbon and hydrogen isotope analyses ($d^{13}C$, $d^{2}H$) of leaf wax-derived compounds, providing information on continental plant types and aridity. In this context the knowledge of the exact spatial distribution and inorganic chemical composition of organic matter in the salt on the atomic scale would help much more targeted analyses. For example, leaf waxes may be associated to different material and specific sites compared to microbial membrane lipids. Due to the age of the samples, a good understanding of the exact spatial distribution of the organic matter and its association the inorganic phases of the salt would greatly support targeted analyses. This would allow us to select specific sub-samples with higher organic yields for biomarker investigations and compound-specific isotope analyses, in particular. In addition to targeting the analysis, the physical and chemical context of the biomolecules (or even a lack of them) provides essential information to explain how physical and chemical conditions influence the fate of biomolecules and their potential or long-term preservation over geological time scales. For example, is the presence of any putative biomarkers associated with specific elements or mineral phases? We can answer this by correlating the presence of biomolecules with mineral phases and elemental composition determining using small/wide angle X-ray scattering by means of SAXS GISAXS; hard X-ray Fluorescence (2D/3D XRF), using RETINA and Multipurpose X-ray diffraction; and neutron diffraction combined with tomography, using respectively INES and IMAT neutron beamlines. Does the oxidation state of elements, for example iron, influence the chemical environment and thus the presence and preservability/stability of biosignatures in the salt over time? We will answer this by correlating the presence of any biosignatures to the elemental oxidation state in the same location at the relevant scales using mineralogical information. Therefore, we propose a multi-instrumental approach involving a combination of non-destructive and destructive analyses exploiting the analytical instrumental suite at IM@IT and ISIS facilities. All analyses will be using the same sample materials, first, for non-destructive and then destructive methods, to maximise the complementary character of the resulting data sets. On one hand, for the non-destructive analyses of the samples, we use the analytical suite of instruments of IM@IT and ISIS facilities indicated. The minerals in each salt sample will be quantified using combined Rietveld refinement of X-rays and neutron diffraction data, using laser ablation. On the other hand, due to the very low yields of the organic phase and its fine dispersal in the salt, the samples to be analysed will consist of small slabs of salt of 100-150g, representing two distinct types of material: relatively pure evaporite material and desiccation crack backfill material. The pure evaporite material will be mainly sodium chloride, containing lenses of clay-rich material and isolated potassium chloride crystals. It is expected to include an organic phase originating predominantly from extremophile biomass and some eolian terrigenous input. The slightly darker coloured material from the desiccation cracks, on the other hand, is expected to include higher proportions of terrigenous organic particles and fragments of biofilm from the salt surface that were resuspended and washed into the cracks during rising water level. For the (destructive) analysis of the biomarkers, originating from microorganisms, terrestrial vegetation or processing-related contamination, like alkanes, alcohols, and ketones we propose to use Gas Chromatography – Ion Mobility Spectrometer (GC-IMS), steranes and GDGTs will be analysed by normal-phase UHPLC at Bristol University; for the aDNA analysis, using DNA sequencing NGS, and for the analysis of both fatty acids and ancient proteins using the Mass Spectrometer 2. Genetic, lipid, and proteomic and volatilomic data will then be cross compared to verify their consistency with the possible extremophile species identified. This research is embedded in a wider collaborative attempt to understand extremophile ecology through a comparison with lipid, proteins and DNA data of modern microbes living on the salt surfaces and in brines in Boulby mine. We aim to see if microbial communities adapt to changes in brine salinity and/or ion composition (chloride vs. sulphate, sodium vs. potassium) either by individual species changing their cell membrane properties or by shifts in species distribution. This is a collaboration with Teesside University, the UKRI-STFC Underground Lab. Boulby, the UK Centre for Astrobiology at Edinburgh University, NASA Jet Propulsion Lab, Bristol University, the University of Bern, University of Rome Tor Vergata, the IM@IT and ISIS facilities. It is supported by the Seedcorn Funding scheme of Teesside University to produce pilot data for a larger proposal to fund PhD projects at Teesside and Edinburgh Universities.

2. Proposed experiment using SAXS GISAXS

In this proposal we plan to perform non-destructive small/wide angle X-ray scattering analysis of rock specimens. The crystal shape, average crystal thickness and crystal orientation of crystal domains within the samples will be addressed by means of SAXS GISAXS data (this proposal) and compared with analysis of XRD and neutron diffraction (INES@ISIS) data (in distinct proposals) which will be performed on the same set of samples. Furthermore, the chemical fingerprint of the sample will be measured by hard X-ray Fluorescence (2D/3D XRF) using RETINA, and by T-PGAA on IMAT@ISIS beamline. Additionally, wide-angle information from SAXS GISAXS and laser ablation EA will be compared with biomarker data obtained from destructive analyses of the sample using GC-IMS; for DNA analysis, using DNA sequencing NGS, and for both fatty acids and ancient proteins, using the Mass Spectrometer 2. From this multilevel analysis we aim to get deep understanding of the elemental speciation.

3. Justification of experimental time requested

Salt specimens are small slabs of salt of 100-150g, representing two distinct types of material will be measured in grazing incident geometry using SAXS GISAXS in the Q range 0.0002-3.1 $Å^{-1}$. Mesoscopic organic and inorganic structures will be detected and analysed to assess particle distribution, organization and orientation at the samples' surface. We propose to analyse a duplicate sample of each type of material: relatively pure evaporite material and desiccation crack backfill material, and we envisage a total of **3 days** including set up and change-over.

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Experiment Proposal

Experiment number GP2024155

Principal investigator	Dr Monica Carosi, Università Roma Tre, ITALY
Co-investigator	Dr Federica Spani, Università Campus Bio-Medico di Roma, ITALY
Co-investigator (*)	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Multi-instrumental characterization of primate baculum and baubellum bone tissues to support functional hypotheses: SAXS measurement case
MRF Instrument	**SAXS GISAXS** **Days requested:** 3
Access Route	Direct Access **Previous GP Number:** No
Science Areas	Biology and Bio-materials, Physics **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	To better understand the function of primate bacula and baubella bone tissues, we aim to study the micro-architecture of the bone focussed for the first time on characteristics related to either observed shapes and physical-chemical features of this tissue. Key aspects include trabecular density and orientation, bone mineral composition, and the distribution of minerals along the bones. The proposed study will involve and multi-instrumental characterization of different bones which will allow us to fine reconstruct a digital twin of these tissues, and for open exploring image-based finite element analysis to assess the mechanical forces involved during copulation. Here, this proposal is focussed on the SAXS analysis.
Publications	Spani F, Morigi MP, Bettuzzi M, Scalici M, Carosi M, PLoS ONE 15(1): e0228131. Spani, F., Morigi, M., Bettuzzi, M. et al., Sci Rep 11 (2021), 11245. Reznikov, N., Bilton, M., Lari, L., Stevens, M. M. & Kröger,Science 360 (2018), eaao2189.

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY		
MRF Instrument	**SAXS GISAXS**	**Days Requested:** 3	
Special requirements:			

SAMPLE

Material	Primate bone tissue	-	-
Formula	Ca10(PO4)6(OH)2	-	-
Forms	Solid		
Volume	0.3 cc		
Weight	0.5 g		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	-	-

Multi-instrumental characterization of primate baculum and baubellum bone tissues to support functional hypotheses: SAXS measurement case

1. Background and Context

Inside the external genitals of some placental mammals, including Primates, genital bones are present in one or both sexes: the *baculum* (penile bone; pl. *bacula*) and the *baubellum* (clitoral bone; pl. *baubella*). *Bacula* are common in most primate species, whereas *baubella* are rare. Both bones occur in some species of Hominoidea (the human evolutionary lineage), but not in humans. Although homologous, *baubellum* is only present in species where males have a *baculum,* whereas species with *bacula* may lack *baubella*. Various functions have been proposed for the *baculum* (none for the *baubellum*), however only one is supported by correlational data: baculum supports erection and prevents urethral collapse, aiding sperm transport in species with prolonged copulations and high levels of sexual competition. *In fact, baculum* length positively correlates with copulation duration. Recent studies published the most comprehensive dataset on primate *bacula* and *baubella* occurrence, collecting data from primary literature and samples from fresh cadavers and museum specimens (Natural History Museum La Specola in Italy, the American Museum of Natural History in New York, and the National Museum of Natural History in Washington, DC). Using 3D high-resolution, non-invasive micro-Computed Tomography and a new landmark-free shape analysis (the *alpha*-shape technique), these studies identified three distinct internal and external morphologies in primate *bacula* for the first time.

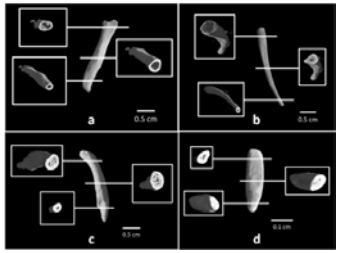

Fig. 1 *3D virtual volumes of 4 different types of primate genital bones, three bacula and one baubellum. For each type, internal structures and cross sections of epiphyses and diaphysis are shown. A: totally hollow structure (Chlorocebus aethiops). B: hollow epiphyses and solid diaphysis with few channels (Otolemur crassicaudatus). C: totally solid structure in both epiphyses and diaphysis with a network of Haversian channels (Papio cynocephalus). D: totally solid structure of baubellum (Sapajus apella) with some Haversian channels*

To better understand the function of primate bacula, the micro-architecture of baculum bone tissue should be investigated focusing for the first time on characteristics related to either observed shapes and mechanical forces exerted on bacula during copulation. Key aspects include trabecular density and orientation, bone mineral composition, and the distribution of minerals along the bones. Potential sex-based differences in these characteristics will aid in interpreting the results. Hence, the proposed study will involve and multi-instrumental approach as follows:

- trabecular density will be assessed by means of quantitative computed tomography (QCT) at different length scale and photon energy which will enable us reconstructing the 3-D bone geometry and volumetric bone mineral density (vBMD);
- trabecular orientation of the bone tissue will be studied in the bulk of the sample by small-angle X-ray scattering (SAXS) to measure crystal shape, their average crystal thickness and their crystal orientation;

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- the structure of bone mineral will be assessed by means of X-ray diffraction (XRD) which is considered as the gold standard for this type of measurement;
- for the compositional characterization of the bone tissue we aim to use nuclear magnetic resonance (NMR) which is uses the responses of isotopes to an external magnetic field to generate compositional information about the sample being scanned, and results will be verified for consistency checks with Raman spectroscopy, Fourier transform infrared spectroscopy (FTIR), X-ray Fluorescence (XRF) spectroscopy, and Energy Dispersive X-ray Analysis (SEM-EDX) data that are used here to determine the chemical and molecular signature of the sample.

2. Proposed experiment

In this specific proposal we aim to use the SAXS GISAXS instrument available at the CSGI - University of Florence Unit to perform small-angle X-ray scattering measurement that will allow us measuring the crystal shape, average crystal thickness and crystal orientation on a n. 3 baculum bone tissue and n. 2 baubella bone tissue. Results of this experiment will be cross compared to verify consistency with XRD data measured on the same set of samples with the Multipurpose X-ray Diffractometer instrument available at the CNR-ICMATE Unit, and requested by means of a separate proposal.

3. Justification of experimental time requested

Each of the n. 5 samples will be fixed to prevent any sample movement on the instrument sample stage and irradiated with X-rays produced by a microsource on a copper anode (λ = 0.15405 nm) in the range of scattering vector from 1.5 nm^{-1} to around 31 nm^{-1}. Acquisition time of 1800 s is envisaged for each sample on different region of interest. Hence, after discussion with the instrument scientist, we request 3 days of instrument time including sample preparation, set-up and calibration time.

References

[1] Reznikov, N., Bilton, M., Lari, L., Stevens, M. M. & Kröger, R. Fractal-like hierarchical organization of bone begins at the nanoscale. Science 360, eaao2189 (2018).
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Experiment Proposal

Experiment number GP2024165

Principal investigator	Dr NAVED MALEK, S V NATIONAL INSTITUTE OF TECHNOLOGY, INDIA
Co-investigator (*)	Professor Jitendra Mata, ANSTO, AUSTRALIA
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Probing Structural Dynamics of Deep Eutectic Solvents Confined within the Pores of Bio-Metal-Organic Frameworks Using SAXS under Extreme Conditions

MRF Instrument	**SAXS GISAXS**	**Days requested:**	3
Access Route	Direct Access	**Previous GP Number:**	NO
Science Areas	Biology and Bio-materials, Chemistry, Materials	**DOI:**	-
Sponsored Grant	None	**Sponsor:**	-
Grant Title	-	**Grant Number:**	-
Start Date	-	**Finish Date:**	-
Similar Submission?	-		
Industrial Links	-		

Non-Technical Abstract

This research explores how Deep Eutectic Solvents (DES) behave when confined within the pores of metal-organic frameworks (MOFs), focusing on structural dynamics under freezing conditions. MOFs are porous materials useful for storing gases and catalyzing chemical reactions, while DES can be designed as eco-friendly, renewable substances with versatile properties. By combining them, we aim to develop hybrid materials with enhanced efficiency for applications like gas storage, catalysis, and pollution control. Using advanced techniques like SAXS, we wish to reveal how confinement affects the liquids' behavior, helping to create sustainable, high-performance materials for industrial use.

Publications

NA
NA
NA

ISIS neutron and muon source

E-platform: No

Instruments	
Access Route	
Science Areas	
Sponsored Grant	
Grant Title	
Start Date	
Similar Submission?	
Industrial Links	

Days Requested:	
Previous RB Number:	
DOI:	
Sponsor:	
Grant Number:	
Finish Date:	

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Science and Technology Facilities Council
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Sample record sheet

Principal contact	Professor Jitendra Mata, ANSTO, AUSTRALIA		
MRF Instrument	**SAXS GISAXS**	**Days Requested:**	3
Special requirements:			

SAMPLE

Material	Bio-DES@MOF	DES-BG@MIP-200	-
Formula	DES-BE@MIP-200	DES-BG@MIP-200	-
Forms	Friable powder	Friable powder	
Volume	cc	cc	
Weight	2 g	2 g	
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	253.15 - 310.15 K	253.15 - 310.15 K	-
Pressure Range	- mbar	- mbar	-
Magnetic field range	- T	- T	-
Standard equipment	Water Bath, Sample Changer	Water Bath, Sample Changer	-
Special equipment	Sample changer , -20°C - +120°C; Lab Space	NA	-

SAFETY

Prep lab needed	Yes	Yes	-
Sample Prep Hazards	NA	NA	-
Special equip. reqs	Inert Atmosphere	Inert Atmosphere	-
Sensitivity to air	No	No	-
Sensitivity to vapour	Yes	Yes	-
Experiment Hazards	NA	NA	-
Equipment Hazards	-	-	-
Biological hazards	NA	NA	-
Radioactive Hazards	NA	NA	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Removed By User	Removed By User	-

Probing Structural Dynamics of Deep Eutectic Solvents Confined within the Pores of Bio-Metal-Organic Frameworks Using SAXS under Extreme Conditions

1. Background and Context

The structural and thermodynamic properties of bulk fluids are well understood, but when fluids are confined within the pores of porous solids, surface-fluid interactions and limited volume can significantly alter their behavior. In nanoscale pores, factors such as pore size, interconnectivity, and surface chemistry further impact fluid properties, making the study of confined fluids a challenging and evolving field. Studying the confinement of the fluids like Deep Eutectic Solvents (DES) within the pores of metal organic framework (MOF) is critical for applications like gas sequestration, oil recovery, hydrogen storage, catalysis, supercapacitors and drug delivery to name a few. Bio-DESs derived from biocompatible molecules and synthesized using tailorable chemistries hold an advantage because of their excellent cytocompatibility and commercial value. Incorporating Bio-DES into high-surface-area porous materials like MOFs could enhance the performance of Bio-DES as well as MOFs and reduce material usage. However, understanding Bio-DES behavior within pores is essential, as pore blockage or full saturation can negate these advantages. Current methods often only assess pore openings, so techniques that explore the bulk pore structure are needed. Small-angle X-ray scattering (SAXS) is effective for studying nanoporous materials and confined fluids. Unlike gas adsorption methods, SAXS can probe the bulk structure, including inaccessible pores. We propose using SAXS and USAXS to study pore filling and Bio-DES organization within nanopores of MOFs with varying pore sizes.

2. Proposed experiment

We wish to run at least 24 samples (4 Samples each Bio-DES (Betaine: Glycerol (BG), and Betaine:Ethylene glycol (BE) concentration and 16 samples each on temperature change (-20,-10, +20 and 37 ^{0}C) based on samples listed in methodology section) using SAXS, USAXS and GISAXS (from 2 x 10^{-5} to 0.31 nm^{-1}). The samples will be in powdered/SEMI SOLID (GEL) form and will be mounted in 1-mm-thick sealed demountable cells). Xenocs XEUSS 3.0 system operating in SAXS (Small Angle X-ray Scattering) and USAXS (Ultra SAXS), are required as only SAXS can probe the length scales (1-100 nm) of interest by accessing the high contrast between the solvent (D_2O) and the hydrocarbon chains in the microstructure of nanoporous materials and the behavior of confined phases within pores. For the contrast-matching, samples will be immersed in an appropriate H_2O/D_2O mixture. The “wet” samples will be allowed to

come to equilibrium for 4-6 h before measurement. Absolute values of the intensity, I(Q), will be obtained by correcting the raw data for sample transmission, scattering of the empty cell, and instrumental background; To collect the full matrix of samples, we kindly request an allocation of 3 days (including time for standard measurements and instrument set-up/calibration) on the Xenocs XEUSS 3.0 system.

3. Summary of previous experimental proposals or characterisation

Figure 1a confirms the crystallinity of bio-MOF (MIP-202) through PXRD measurements. Figures 1b and 1c show that the diffraction peaks of MIP-202 appear in the same positions for both BE@MIP-202 and BG@MIP-202, confirming that the MOF structure remains intact after DES confinement.[1,2] Low-temperature Gonio XRD reveals that significant diffraction peaks are still visible at -40°C, indicating no structural changes before freezing. However, at -80°C, some peaks disappear due to phase transitions, as supported by DSC data. FTIR spectra (Fig 1 d,e) show shifts in the -OH and -CO peaks, indicating strong hydrogen bonding between DES and MIP-202. This confirms that DES is confined within the MOF pores without damaging the structure. DSC analysis (Fig 1 f,g) reveals no crystallization between -140°C and 150°C, and the glass transition temperatures (Tg) of BE and BG are -83°C and -69°C, respectively. This highlights the antifreezing properties of DES@Bio-MOF, attributed to the strong hydrogen bonding that disrupts the typical crystalline lattice formation at low temperatures.[2-3]

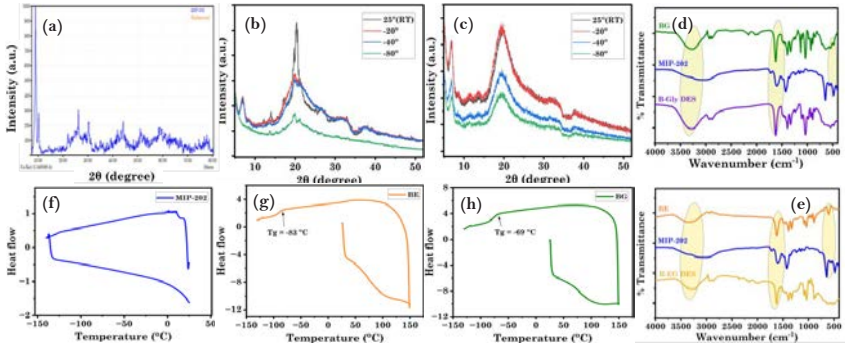

Figure 1: PXRD, DSC and FTIR data of (a,d,e,f) MIP-202, (b, d,g) BE @MIP-202 and (c, e,h) BG@MIP-202

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3. RSC Pharmaceutics 2024, 1, 317-332. https://doi.org/10.1039/D3PM00088E.

SAXS WAXD

Experiment Proposal

Experiment number GP2024069

Principal investigator	Miss Paola Amazio, Next Technology Tecnotessile, ITALY
Co-investigator (*)	Dr Marino Lavorgna, CNR, ITALY
Co-investigator	Dr Gennaro Gentile, IPCB CNR, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	SAXS/WAXD characterization of EoL fibers, and recycled non-wovens and compounds
MRF Instrument	**SAXS WAXD**
Days requested:	3
Access Route	Direct Access
Previous GP Number:	-
Science Areas	Environment, Materials
DOI:	-
Sponsored Grant	None
Sponsor:	-
Grant Title	-
Grant Number:	-
Start Date	-
Finish Date:	-
Similar Submission?	-
Industrial Links	Next Technology Tecnotessile (NTT)

Non-Technical Abstract

Novel technologies for enhancing biobased plastic waste recycling are of high interest for industries. In this frame, activities aimed at recycling bio-based fabrics are in-going at Next technology Tecnotessile. After a pretreatment step of washing and drying, aimed to the removal of residues and impurities, and after a following shredding step, mechanical recycling of textile waste stream are carried out. The aim is to obtain staple fibre by carding action. These staple fibres are used to produce non-woven as up-cycling strategy or are reprocessed to obtain new recycled compounds suitable for the melt spinning process. The recovered fibers, and the produced non-wovens and compounds need a structural characterization in order to verify the effect of the crystallinity the fibers on the properties of recycled non-wovens and compounds. In a separate experiment proposal, the same materials will be characterized by SEM.

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Marino Lavorgna, CNR, ITALY		
MRF Instrument	**SAXS WAXD**	**Days Requested:**	3
Special requirements:			

SAMPLE

Material	3 EoL bio-based fibers	3 recycled non-wovens	3 Recycled compounds
Formula	Poly(lactic acid)	Poly(lactic acid)	-
Forms	Solid	Solid	
Volume	1 cc	1 cc	cc
Weight	1 g	1 g	mg
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	298 - K	298 - K	298 - K
Pressure Range	1000 - mbar	1000 - mbar	1000 - mbar
Magnetic field range	- T	- T	- T
Standard equipment	None	None	-
Special equipment	N/A	N/A	N/A

SAFETY

Prep lab needed	Yes	Yes	Yes
Sample Prep Hazards	no	no	no
Special equip. reqs	no	no	no
Sensitivity to air	No	No	No
Sensitivity to vapour	No	No	No
Experiment Hazards	no	no	no
Equipment Hazards	-	-	-
Biological hazards	no	no	no
Radioactive Hazards	no	no	no
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	Disposed by IS

SAXS/WAXD characterization of EoL fibers, and recycled non-wovens and compounds

1. Background and Context

Even if biobased plastics are still present in the market only with a very small share (< 1%), a dramatic growth is expected in the next years. But the mere replacement of fossil counterparts does not solve the emergence of plastic pollution. Indeed, despite considerable efforts being made around the recycling of bioplastics, reclaiming plants are not adapted to for separate streams of bioplastics, so the technique is not optimized for the future increase in production and sale.

In this context, Next Technology Tecnotessile (NTT) wants to apply the circular (bio)economy concept, developing suitable recycling streams for bioplastics (BP's) to improve waste management efficiency throughout Europe. It is a hierarchical challenge, from the collection of the bioplastic waste, up to the upcycling and validation of the final recycled end-products.

The new value chain will imply sorting, conditioning and valorising three types of waste streams from the packaging, agriculture and textile industries into three end-products, targeting to reach at least the same quality and functionality than the original grades.

In particular, Next Technology Tecnotessile (NTT) will focus on the textile waste stream. As cornerstone targets for maximizing project's impact, the upscaling of the recycling processes will be integrated in pilot plants of actual industrial recycling lines currently operating in waste management companies, focus on bioplastics for which recycling processes are still not in place, excluding bio-based analogues ("drop-ins").

In particular, novel technologies for enhancing biobased plastic waste recycling are under development. After a pretreatment step of washing and drying, aimed to the removal of residues and impurities, and after a shredding step, mechanical recycling of textile waste stream is carried out. The aim is to obtain staple fibre by carding action. These staple fibres are then used to produce non-woven as up-cycling strategy or are processed to obtain new recycled compounds suitable for the melt spinning process. The fibres and the recycled polymer need to be characterized using SAXS/WAXD in order to verify the effect of the crystallinity of EoL fibers on the properties of non-wovens and compounds.

2. Proposed experiment

SAXS/WAXD will be used for examining the cristallinity of recycled fibers as well as the cristallinity of non-wovens and compounds.

In a separate experiment proposal, the same materials will be characterized by SEM to evaluate the effect of the morphology of recycled materials on the properties on non-wovens and recycled compounds.

3. Summary of previous experimental proposals or characterisation

No previous experiments have been carried out on these samples

4. Justification of experimental time requested

We have requested the SAXS/WAXD equipment available at IPCB CNR to evaluate the crystallinity of 9 samples:

3 recycled bio-fibers constituted by poly(lactic acid) (PLA), differing for fiber length and diameter

3 non-vovens produced from recycled PLA fibers

3 compounds produced from recycled PLA fibers.

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SAXS/WAXD analysis will be performed at suitable conditions useful to evaluate the structural characteristics of the EoL and recycled materials. After discussion with the instrument scientist, we request 3 days of SAXS/WAXD access, for a fully and thorough structural characterization of the materials. The foreseen beam time accounts set up and for the data collection on the samples.

References

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Effect of Temperature on Extrusion Process on Mechanical Properties of Recycled Polylactic Acid Filaments
(2024) AIP Conference Proceedings, 2991 (1), art. no. 040035
DOI: 10.1063/5.0198559

Nomadolo N., Mtibe A., Ofosu O., Mekoa C., Letwaba J., Muniyasamy S.
The Effect of Mechanical Recycling on the Thermal, Mechanical, and Chemical Properties of Poly (Butylene Adipate-Co-Terephthalate) (PBAT), Poly (Butylene Succinate) (PBS), Poly (Lactic Acid) (PLA), PBAT-PBS Blend and PBAT-TPS Biocomposite
(2024) Journal of Polymers and the Environment, 32 (6), pp. 2644 - 2659
DOI: 10.1007/s10924-023-03151-y

Ramos-Hernández T., Robledo-Ortíz J.R., Martín del Campo A.S., Rodrigue D., Cano A., Pérez-Fonseca A.A.
Mechanical recycling of poly(lactic acid)/agave fiber biocomposites
(2024) Journal of Reinforced Plastics and Composites
DOI: 10.1177/07316844241253905

Bavaliya K.J., Vala N.S., Raj M., Raj L.
A review on biodegradable composites based on poly (lactic acid) with various bio fibers
(2024) Chemical Papers, 78 (5), pp. 2695 - 2728
DOI: 10.1007/s11696-023-03298-x

Experiment Proposal

Experiment number GP2024098

Principal investigator	Dr Chimenti Stefano, CROMOGENIA UNIT, SPAIN
Co-investigator	Dr Gennaro Gentile, IPCB CNR, ITALY
Co-investigator (*)	Dr Marino Lavorgna, CNR, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Structural characterization of hydrophobic/oleophobic coatings by SAXS WAXD
MRF Instrument	**SAXS WAXD**
Days requested:	3
Access Route	Direct Access
Previous GP Number:	no
Science Areas	Chemistry, Engineering, Materials
DOI:	-
Sponsored Grant	None
Sponsor:	-
Grant Title	-
Grant Number:	-
Start Date	-
Finish Date:	-
Similar Submission?	-
Industrial Links	Cromogenia

Non-Technical Abstract

Water-dispersed polymer nanoparticles, obtained by radical emulsion polymerization, are key ingredients in many technological and industrial products, such as coatings, paints, adhesives, sealants, paper and textile additives, drug delivery systems and cosmetics. Cromogenia UNITS, in this context, is actively working in the research and development of fluorine-free polymer based nanoparticles capable of providing the hydrophobic/oleophobic effect to natural fabrics, such as cotton. For the tailoring of the developed products, a deep structural characterization by SAXS/WAXD of coatings obtained by applying nanoparticles dispersions onto cotton fabrics is needed. In separate experiment proposals, nanoparticles and coatings applied onto fabrics will be characterized by TEM, SEM&C-AFM and XRD tomography.

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Science and Technology Facilities Council
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Sample record sheet

Principal contact	Dr Marino Lavorgna, CNR, ITALY		
MRF Instrument	**SAXS WAXD**	**Days Requested:**	3
Special requirements:			

SAMPLE

Material	6 water dispersed polymer nanoparticles, differing for their composition (polymer and inorganic nanoadditives), applied as coatings onto cotton fabrics	-	-
Formula	polymer coatings onto cotton fabrics	-	-
Forms	Solid		
Volume	2 cc		
Weight	2 g		
Container or substrate	vial	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	298 - K	-	-
Pressure Range	1000 - mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	N/A	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	no	-	-
Special equip. reqs	no	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	no	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	no	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Structural characterization of hydrophobic/oleophobic coatings by SAXS WAXD

Background and Context

Water-dispersed polymer nanoparticles, obtained by radical emulsion polymerization, are key ingredients in many technological and industrial products, such as coatings, paints, adhesives, sealants, paper and textile additives, drug delivery systems and cosmetics.

In the field of the textile industry, for example, some technical properties of fibres, such as repellency to water or other substances, do not meet the needs required of garments during their useful life. To give the repellent effect, a finishing treatment must be applied to the fabric. Currently, fluorinated polymers in aqueous dispersion make it possible to obtain a water and oil repellent finish in the various substrates on which they are applied. Hence their wide use in many sectors and most companies that are dedicated to the marketing of products for textile finishing, constantly find themselves developing new products with new properties and substantial improvements that they give to fabrics in general.

However, in recent years ECHA has placed traditional fluorinated resins under scrutiny as they often contain long-chain perfluorocarbons (PFCs), which are known to persist in the environment and potentially have adverse effects on human health and wildlife. Consequently, research has been oriented towards the development of alternatives with similar hydrophobic and oleophobic properties but with a lower environmental impact given the absence of fluorinated monomers.

Cromogenia UNITS, in this context, is actively working in the research and development of polymers in aqueous dispersion as alternatives to fluorinated polymers which are capable of providing the same repellency to water and good repellency to oils.

Although homogeneous particles consisting of one or single components can be used in many textile applications, multiphase polymer particles (comprising several monomers and/or nanofillers) are preferred, since they synergistically combine various physical and chemical properties of different materials. As a result, a wider range of properties can be achieved, e.g. fast drying hardness and/or high hydrophobicity in water-based systems.

The final performance of multiphase particles depends critically on their morphology and chemical composition. Both morphology and chemical composition are the result of complex kinetic and thermodynamic processes that occur during polymerization.

For this reason, the structural characterization of the developed polymer nanoparticles, applied as coatings onto a cotton fabric, is of fundamental importance to tailor the properties of the synthesized materials and their performances.

2. Proposed experiment

The structural characterization of the coatings obtained by applying the nanoparticles onto cotton substrates will be performed by using the SAXS/WAXD facility available at IPCB-CNR.

In separate experiment proposals, the nanoparticles will be characterized by the TEM-FEI available at IPCB-CNR Unit. Moreover, the nanoparticles and the coated cotton substrates will be characterized by SEM&C-AFM and optical profiler, available at Tor Vergata Unit, and the coatings will be analysed by XRD tomography available at the IPCB CNR Unit, to investigate the distribution of the nanoparticles and the coating structure on fabrics.

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3. Summary of previous experimental proposals or characterisation

No previous experiments have been carried out on these samples

4. Justification of experimental time requested

We have requested the SAXS/WAXD equipment available at the IPCB CNR Unit to evaluate the morphology of the 6 types of coatings obtained by applying water dispersed polymer based nanoparticles, differing for their composition (polymer matrix, inorganic additives) onto cotton substrates.

Therefore, a total of 6 samples will be analysed. After discussion with the instrument scientist, we request 3 days of SAXS/WAXD access, for a fully and thorough structural characterization of the materials. The foreseen beam time accounts set up and for the data collection on the samples.

References

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Kausar A.
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(2019) Superhydrophobic Polymer Coatings: Fundamentals, Design, Fabrication, and Applications, pp. 77 - 90
DOI: 10.1016/B978-0-12-816671-0.00005-9

Attia N.F., Moussa M., Sheta A.M.F., Taha R., Gamal H.
Effect of different nanoparticles based coating on the performance of textile properties
(2017) Progress in Organic Coatings, 104, pp. 72 - 80
DOI: 10.1016/j.porgcoat.2016.12.007

Experiment Proposal

Experiment number GP2024118

Principal investigator	Dr Ivano Aglietto, GrapheneUP SE, CZECH_REPUBLIC
Co-investigator	Dr Gennaro Gentile, IPCB CNR, ITALY
Co-investigator (*)	Dr Marino Lavorgna, CNR, ITALY
Co-investigator	Professor Massimo Bonini, CSGI - University of Florence, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	SAXS WAXD characterization of innovative graphene-based inks for multifunctional applications
MRF Instrument	**SAXS WAXD**
Days requested:	3
Access Route	Direct Access
Previous GP Number:	-
Science Areas	Chemistry, Engineering, Materials
DOI:	-
Sponsored Grant	None
Sponsor:	-
Grant Title	-
Grant Number:	-
Start Date	-
Finish Date:	-
Similar Submission?	-
Industrial Links	Graphene UP

Non-Technical Abstract

The aim of this proposal is to study, using the SAXS WAXD equipment available at IPCB CNR Unit, the correlation between innovative Few Layers Graphene (FLGs), hybrid fillers consisting of graphene few layers modified with metal, inorganic or carbon particles, the filler spatial distribution, and the processing parameters related to screen-printing deposition technologies. The objective is to investigate how the chemical modification of FLGs, obtained directly during the process of graphene exfoliation starting from graphite, may affect the spatial distribution because of the different technologies adopted for the processing of the composites. In separate experiments RAMAN confocal microscopy, SEM and TEM characterization of the inks will be performed.

Publications -

ISIS neutron and muon source

E-platform: No

Instruments	
Days Requested:	
Access Route	
Previous RB Number:	
Science Areas	
DOI:	
Sponsored Grant	
Sponsor:	
Grant Title	
Grant Number:	
Start Date	
Finish Date:	
Similar Submission?	
Industrial Links	

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ISIS Neutron and Muon Source

Sample record sheet

Principal contact	Dr Marino Lavorgna, CNR, ITALY
MRF Instrument	**SAXS WAXD**
Days Requested:	3
Special requirements:	

SAMPLE

Material	FLG based inks (4 samples)	Fillers for FLG based inks (4 samples)	-
Formula	C	C	-
Forms	Solid	Solid	
Volume	1 cc	1 cc	
Weight	1000 mg	1000 mg	
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	- K	-
Pressure Range	- mbar	- mbar	-
Magnetic field range	- T	- T	-
Standard equipment	None	None	-
Special equipment	-	-	-

SAFETY

Prep lab needed	No	No	-
Sample Prep Hazards	NO	NO	-
Special equip. reqs	NO	NO	-
Sensitivity to air	No	No	-
Sensitivity to vapour	No	No	-
Experiment Hazards	NO	NO	-
Equipment Hazards	-	-	-
Biological hazards	NO	NO	-
Radioactive Hazards	NO	NO	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	-

SAXS WAXD characterization of innovative graphene-based hybrid fillers and their inks for multifunctional applications

Background and Context

Flexible and portable electronic devices and energy storage equipment made from conductive nanomaterials using printing or inks deposition technology have garnered significant attention due to their low-cost, high-throughput, and eco-friendly manufacturing processes over the past few decades. Electrical conductive nanostructured inks have emerged as promising candidates for designing flexible electronics because of their cost-effective synthesis methods and compatibility with current manufacturing processes.

In particular, the development of highly concentrated conductive ink using graphene powders as a raw material is seen as a promising direction. Most high-concentration graphene conductive inks are prepared from low-concentration graphene dispersions containing polymers as stabilizers. However, the solvents used in these dispersions often do not meet the requirements of the printing methods. Among various printing technologies, screen printing shows great promise for industrial-scale production due to its ability to print thick patterns with low sheet resistance (RS) on a wide range of substrates.

To realize applications in graphene-based flexible electronics, further improvement in graphene ink formulation and the development of simple, efficient post-treatment processes for printed patterns are needed. Polymers used to prevent graphene agglomeration in dispersions through steric hindrance can also adjust the rheological properties, storage performance of inks, and the flexibility and adhesion of printed patterns to substrates. New innovative fillers, based on graphene functionalized with metals or other nanoparticles, may be useful for realizing conductive inks with additional properties such as thermal, magnetic, electromagnetic shielding, optical, and catalytic properties.

Therefore, the aim of this proposal is to study, using the instrument suite of IM@IT, the correlation between innovative Few Layers Graphene (FLGs), hybrid fillers consisting of graphene few layers modified with metal, inorganic or carbon particles, the filler spatial distribution, and the processing parameters related to screen-printing deposition technologies. Hybrid fillers may be realized by combining FLGs with pristine metals or inorganic particles, but they can also be prepared by direct synthesis of hybrid graphene structures. In particular a process has been developed to cover the graphene layers with carbon nanotubes and also conductive metal fillers during the direct non-oxidative exfoliation of graphite in gas phase. This process allows to create hybrid structures of few-layer graphene with other fillers without the need of a post-functionalization process.

The objective is to investigate how the chemical modification of FLGs, obtained directly during the process of graphene exfoliation starting from graphite, may affect the morphology of filler as well as the spatial distribution of filler within the inks because of the different technologies adopted for the processing of the composites.

2. Proposed experiment

The characterization of the inks will be performed as follows.

SAXS/WAXD, available at IPCB CNR Unit, will be performed on the hybrid FLGs fillers and inks and will allow to evaluate the structure and orientation of the filler and its aggregation, contributing to the enhanced properties of the resulting inks.

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In separate experiment proposals, RAMAN confocal microscopy, available at the CSGI - University of Florence Unit, will provide chemical information about the modified FLGs and their interface interactions with the main components of inks. Furthermore, TEM and SEM characterization of the hybrid fillers and inks, performed at the IPCB CNR Unit, will offer insights into the morphology and assembling of nanoplatelets and spatial filler distribution.

3. Summary of previous experimental proposals or characterisation

No previous experiments have been carried out on these samples

4. Justification of experimental time requested

We have requested the SAXS WAXD equipment available at the IPCB CNR Unit to characterize 8 samples (4fillers+4inks) obtained by modified FLG with metal nanoparticles, inorganic nanoparticles, multiwalled carbon nanotubes (MWCNTs) and single wall carbon nanotubes (SWCNTs). Non additivated FLG will be also characterized by comparison.

Therefore, a total of 8 samples will be analysed. After discussion with the instrument scientist, we request 3 days of SAXS WAXD access, for a fully and thorough characterization of the materials. The foreseen beam time accounts set up and for the data collection on the samples.

References

S. Tkachev, M. Monteiro, J. Santos, E. Placidi, M. Ben Hassine, P. Marques, P. Ferreira, P. Alpuim, A. Capasso
Environmentally Friendly Graphene Inks for Touch Screen Sensors
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T. Sang Tran, N. Kumar Dutta, N. Roy Choudhury,
Graphene inks for printed flexible electronics: Graphene dispersions, ink formulations, printing techniques and applications,
(2018) Advances in Colloid and Interface Science, 261, 41-61
https://doi.org/10.1016/j.cis.2018.09.003

K. Parvez, R. Worsley, A. Alieva, A. Felten, C. Casiraghi
Water-based and inkjet printable inks made by electrochemically exfoliated graphene,
(2019) Carbon, 149, 213-221
https://doi.org/10.1016/j.carbon.2019.04.047

SEM FEI

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Experiment Proposal

Experiment number GP2024068

Principal investigator	Miss Paola Amazio, Next Technology Tecnotessile, ITALY
Co-investigator	Professor Carla Andreani, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Marino Lavorgna, CNR, ITALY
Co-investigator (*)	Dr Gennaro Gentile, IPCB CNR, ITALY
Co-investigator	Professor Alessandro Cianchi, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	SEM characterization of EoL fibers, and recycled non-wovens and compounds
MRF Instrument	**SEM FEI** **Days requested:** 2
Access Route	Direct Access **Previous GP Number:** -
Science Areas	Environment, Materials **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	Next Technology Tecnotessile (NTT)

Non-Technical Abstract

Novel technologies for enhancing biobased plastic waste recycling are of high interest for industries. In this frame, activities aimed at recycling bio-based fabrics are in-going at Next technology Tecnotessile. After a pretreatment step of washing and drying, aimed to the removal of residues and impurities, and after a following shredding step, mechanical recycling of textile waste stream are carried out. The aim is to obtain staple fibre by carding action. These staple fibres are used to produce non-woven as up-cycling strategy or are reprocessed to obtain new recycled compounds suitable for the melt spinning process. The recovered fibers, and the produced non-wovens and compounds need a morphological characterization in order to verify the effect of the length, the surface morphology of the fibers on the recycled non-wovens and compounds. In a separate experiment proposal, the same materials will be characterized by XRD.

Publications -

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Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Gennaro Gentile, IPCB CNR, ITALY
MRF Instrument	**SEM FEI** **Days Requested:** 2
Special requirements:	

SAMPLE

Material	3 EoL bio-based fibers	3 recycled non-wovens	3 Recycled compounds
Formula	Poly(lactic acid)	Poly(lactic acid)	-
Forms	Solid	Solid	
Volume	1 cc	1 cc	cc
Weight	1 g	1 g	mg
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	298 - K	298 - K	298 - K
Pressure Range	1000 - mbar	1000 - mbar	1000 - mbar
Magnetic field range	- T	- T	- T
Standard equipment	None	None	-
Special equipment	N/A	N/A	N/A

SAFETY

Prep lab needed	Yes	Yes	Yes
Sample Prep Hazards	no	no	no
Special equip. reqs	no	no	no
Sensitivity to air	No	No	No
Sensitivity to vapour	No	No	No
Experiment Hazards	no	no	no
Equipment Hazards	-	-	-
Biological hazards	no	no	no
Radioactive Hazards	no	no	no
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	Disposed by IS

SEM characterization of EoL fibers, and recycled non-wovens and compounds

1. Background and Context

Even if biobased plastics are still present in the market only with a very small share (< 1%), a dramatic growth is expected in the next years. But the mere replacement of fossil counterparts does not solve the emergence of plastic pollution. Indeed, despite considerable efforts being made around the recycling of bioplastics, reclaiming plants are not adapted to for separate streams of bioplastics, so the technique is not optimized for the future increase in production and sale.

In this context, Next Technology Tecnotessile (NTT) wants to apply the circular (bio)economy concept, developing suitable recycling streams for bioplastics (BP's) to improve waste management efficiency throughout Europe. It is a hierarchical challenge, from the collection of the bioplastic waste, up to the upcycling and validation of the final recycled end-products.

The new value chain will imply sorting, conditioning and valorising three types of waste streams from the packaging, agriculture and textile industries into three end-products, targeting to reach at least the same quality and functionality than the original grades.

In particular, Next Technology Tecnotessile (NTT) will focus on the textile waste stream. As cornerstone targets for maximizing project's impact, the upscaling of the recycling processes will be integrated in pilot plants of actual industrial recycling lines currently operating in waste management companies, focus on bioplastics for which recycling processes are still not in place, excluding bio-based analogues ("drop-ins").

In particular, novel technologies for enhancing biobased plastic waste recycling are under development. After a pretreatment step of washing and drying, aimed to the removal of residues and impurities, and after a shredding step, mechanical recycling of textile waste stream is carried out. The aim is to obtain staple fibre by carding action. These staple fibres are then used to produce non-woven as up-cycling strategy or are processed to obtain new recycled compounds suitable for the melt spinning process. The fibres and the recycled polymer need to be characterized (using scanning electron microscopy, SEM) in order to verify the effect of the length and surface morphology on the properties of non-wovens and compounds.

2. Proposed experiment

SEM will be used for examining the length and surface morphology of recycled fibers as well as the morphology of non-wovens and compounds. For non-wovens, the morphology of the recycled materials will be investigated and results will be considered and compared with those obtained by mechanical analysis, carried out at NTT premises. For compounds, SEM analysis will be used to evaluate the presence of contaminants that would possibly preclude the melt spinning processing of the recycled materials.

In a separate experiment proposal, the same materials will be characterized by SAXS/WAXD to evaluate the effect of the crystallinity of recycled materials on the structure and properties on non-wovens and recycled compounds.

3. Summary of previous experimental proposals or characterisation

No previous experiments have been carried out on these samples

4. Justification of experimental time requested

We have requested the SEM FEI equipment available at IPCB CNR to evaluate the morphology of the 9 samples:

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3 recycled bio-fibers constituted by poly(lactic acid) (PLA), differing for fiber length and diameter
3 non-vovens produced from recycled PLA fibers
3 compounds produced from recycled PLA fibers.
For SEM analysis, samples will be mounted on aluminium stubs and sputter coated.
SEM analysis will be performed at suitable conditions useful to evaluate the morphology of the materials at different scale lengths. After discussion with the instrument scientist, we request 2 days of SEM FEI access, for a fully and thorough morphological characterization of the materials. The foreseen beam time accounts set up and for the data collection on the samples.

References

Pamilih B.J., Suryanto H., Aminnudin, Putra A.F., Maulana J.
Effect of Temperature on Extrusion Process on Mechanical Properties of Recycled Polylactic Acid Filaments
(2024) AIP Conference Proceedings, 2991 (1), art. no. 040035
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Nomadolo N., Mtibe A., Ofosu O., Mekoa C., Letwaba J., Muniyasamy S.
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Mechanical recycling of poly(lactic acid)/agave fiber biocomposites
(2024) Journal of Reinforced Plastics and Composites
DOI: 10.1177/07316844241253905

Bavaliya K.J., Vala N.S., Raj M., Raj L.
A review on biodegradable composites based on poly (lactic acid) with various bio fibers
(2024) Chemical Papers, 78 (5), pp. 2695 - 2728
DOI: 10.1007/s11696-023-03298-x

Experiment Proposal

Experiment number GP2024106

Principal investigator	Professor Gabriele Gentile, University of Rome Tor Vergata, ITALY
Co-investigator (*)	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Carla Andreani, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Multi-instrumental characterization of Oniscidean isopods for establish the nature and function of their cuticle and tubercles: SEM-EDX measurements
MRF Instrument	**SEM FEI** — **Days requested:** 4
Access Route	Direct Access — **Previous GP Number:** No
Science Areas	Biology and Bio-materials, Physics — **DOI:** -
Sponsored Grant	None — **Sponsor:** -
Grant Title	- — **Grant Number:** -
Start Date	- — **Finish Date:** -
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

Andronicus is a genus of oniscidean isopods that includes less than a dozen species, most of which are confined to caves in the Italian Pre-Alps. Andronicus offers the unique opportunity to investigate the nature of the cuticle in cave (A. brentanus) and non-cave-dwelling species (A. dentiger), within the same evolutionary lineage, characterizing mineralization in the different species. The proposed investigation will also extend to including tubercles on the surface of the body, the nature of which, currently unknown, could be of a sensorial nature. Preliminary observations would suggest the presence of a possible innervation at the base of these tubercles, which if confirmed, would prove their nature and function. For the characterization on the mineralization of the cuticle and tubercles we envisage to use EDX, FT-IR, Raman, and X-ray diffraction, whereas the morphology characterization will be done by SEM, TEM and nano-XCT. Here, this proposal is focussed on the SEM-EDX analysis.

Publications

Vittori, M. et al., Arthropod Struct Dev. 46 (2016), pp. 96-107.
Gentile, G. and Allegrucci, G., International Journal of Speleology 26 (1997), pp. 47-61.
Neues, F. et al., Cryst. Eng. Comm. 9 (2007), pp. 1245-1251.

ISIS neutron and muon source — **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
MRF Instrument	**SEM FEI** — **Days Requested:** 4
Special requirements:	

SAMPLE

Material	Oniscidean isopod	-	-
Formula	Organic material, Calcite	-	-
Forms	Solid		
Volume	0.03 cc		
Weight	1-2 g		
Container or substrate	-	-	-
Storage Requirements	Freezer (-20C)	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	-	-

Multi-instrumental characterization of Oniscidean isopods to establish the nature and function of their cuticle and tubercles: SEM-EDX measurements

1. Background and Context

Among Crustaceans, oniscidean isopods are uniquely adapted to terrestrial life, exhibiting strongly mineralized cuticles. Oniscideans include several species adapted to the caves. Among the most important evolutionary adaptations found in troglobitic oniscideans (i.e. bound to cave environments, from which they cannot escape due to strict ecological and physiological constraints) are the thinning of the cuticle with a reduce layer of calcite, although calcium carbonate is present in the exocuticle and the endocuticle [1]. Additionally, other adaptations include the lengthening of the appendages, the loss of the eyes, the development of sensory systems alternative to sight such as hygrosensors and chemosensors, usually located in different areas of the body. *Androniscus* (Fig. 1) is a genus of oniscidean isopods that includes less than a dozen species, most of which are confined to caves in the Italian Pre-Alps. Among these, *Androniscus dentiger* is the one that shows the least constraints, being present even in the most superficial layers of the soil in non-cave environments and showing a wide geographical distribution [2]. Indeed, by combining atomic absorption spectroscopy, thermogravimetry and X-ray diffraction, the composition of cuticles in several isopods has been analyzed [3-4]. The use of high-resolution Raman microscopy enabled the determination of the distribution of different mineral phases in the tergal cuticles of some rollers, clingers, and runners [5,6].

Fig. 1 *Androniscus dentiger* (a) and *Androniscus brentanus* (b). Contrary to the second, the first is not troglobite, is pigmented, has thick cuticle, and shows a prominent single-ommatidium eye (arrow).

Androniscus offers the unique opportunity to investigate the nature of the cuticle in cave (*A. brentanus* and more) and non-cave-dwelling species (*A. dentiger*), within the same evolutionary lineage, characterizing mineralization in the different species. The proposed investigation will also extend to including tubercles (tricorns) on the surface of the body, the nature of which, currently unknown, could be of a sensorial nature. Some preliminary observations would suggest the presence of a possible innervation at the base of these tubercles, which if confirmed, would prove their nature and function. For the characterization on the mineralization of the cuticle and tubercles in the two different species (*A. brentanus and A. dentiger*) we plan to use Energy Dispersive X-ray Analysis (SEM-EDX), Fourier-transform infrared spectroscopy (FT-IR), Raman spectroscopy and X-ray diffraction, whereas the morphology characterization will be done by means of electron microscopy techniques (SEM, TEM) and X-ray nano tomography. The benefit of using a multi-instrumental approach would allow us not only to cross compared results to verify their consistency, but also to investigate the degree of resorption/failure to develop the eye in these isopods species, allowing us to observe the presence of vestigial or residual structures, such as for example the presence of an optic nerve, in the absence of the ommatidium (eyeball).

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2. Proposed experiment

In this specific proposal we aim to use the SEM FEI instrument available at the CNR IPCB Unit for assessing the degree of mineralization and the morphology of the cuticle and tubercles on a n. 6 *Androniscus brentanus and n. 6 Androniscus dentiger* isopods samples. Results of this experiment will be cross compared to verify consistency with data obtained by separate proposals where we request FT-IR, Raman spectroscopy, XRD, TEM, and nano-XCT measurements on the same set of samples.

3. Justification of experimental time requested

Each of the n. 12 samples of the two Oniscidean isopod species (n. 6 *Androniscus brentanus and n.6 Androniscus dentiger)* will be washed for 1–2 s in double distilled water to remove tissue saline at the surface and then for 2–5 s in 100% methanol to remove water. Specimens will be air dried and stored at −20 °C until its use on the instrument. For the measurement on the SEM instrument sample will be glued on a Si substrate which avoids any sample motion. We envisage, after discussion with the instrument scientist, to measure n. 3 samples per day on the instrument. Hence, we request a total of 4 days of instrument time including sample preparation, set-up and calibration time.

References

[1] Vittori, M., Tusek-Žnidarič, M. & Štrus, J. (2016) Exoskeletal cuticle of cavernicolous and epigean terrestrial isopods: a review and perspectives. Arthropod Struct Dev. 46(1): 96-107.
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[3] Neues, F., Ziegler, A. & Epple, M. (2007) The composition of the mineralized cuticle in marine and terrestrial isopods: a comparative study Cryst. Eng. Comm. 9: 1245-1251.
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[6] Štrus, J., Žnidaršič, N., Hild, S. & Ziegler, A. (2008) Microscopic anatomy and mineral composition of cuticle in amphibious isopods Ligia italica and Titanethes albus (Crustacea: Isopoda) A. Aretz, B. Hermanns-Sachweh, J. Mayer (Eds.), EMC 2008: Life Sciences, Springer Verlag, Berlin:185-186.
[7] Hornung, E. (2011). Evolutionary adaptation of oniscidean isopods to terrestrial life: Structural-physiological-behavioural aspects. Terrestrial Arthropod Reviews. 4: 95-130.

Experiment Proposal

Experiment number GP2024117

Principal investigator	Dr Ivano Aglietto, GrapheneUP SE, CZECH_REPUBLIC
Co-investigator (*)	Dr Gennaro Gentile, IPCB CNR, ITALY
Co-investigator	Dr Marino Lavorgna, CNR, ITALY
Co-investigator	Professor Massimo Bonini, CSGI - University of Florence, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	SEM characterization of innovative graphene-based inks for multifunctional applications
MRF Instrument	**SEM FEI**
Days requested:	2
Access Route	Direct Access
Previous GP Number:	-
Science Areas	Chemistry, Engineering, Materials
DOI:	-
Sponsored Grant	None
Sponsor:	-
Grant Title	-
Grant Number:	-
Start Date	-
Finish Date:	-
Similar Submission?	-
Industrial Links	Graphene UP
Non-Technical Abstract	The aim of this proposal is to study, using the SEM FEI equipment available at IPCB CNR Unit, the correlation between innovative Few Layers Graphene (FLGs), hybrid fillers consisting of graphene few layers modified with metal, inorganic or carbon particles, the filler spatial distribution, and the processing parameters related to screen-printing deposition technologies. The objective is to investigate how the chemical modification of FLGs, obtained directly during the process of graphene exfoliation starting from graphite, may affect the spatial distribution because of the different technologies adopted for the processing of the composites. In separate experiments RAMAN confocal microscopy, SAXS WAXD and TEM characterization of the inks will be performed.
Publications	-

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E-platform: No

Instruments	
Days Requested:	
Access Route	
Previous RB Number:	
Science Areas	
DOI:	
Sponsored Grant	
Sponsor:	
Grant Title	
Grant Number:	
Start Date	
Finish Date:	
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Gennaro Gentile, IPCB CNR, ITALY
MRF Instrument	**SEM FEI**
Days Requested:	2
Special requirements:	

SAMPLE

Material	FLG based inks (4 samples)	fillers for FLG based inks (4 samples)	-
Formula	C	C	-
Forms	Solid	Solid	
Volume	1 cc	1 cc	
Weight	1000 mg	1000 mg	
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	- K	-
Pressure Range	- mbar	- mbar	-
Magnetic field range	- T	- T	-
Standard equipment	None	None	-
Special equipment	-	-	-

SAFETY

Prep lab needed	No	No	-
Sample Prep Hazards	NO	NO	-
Special equip. reqs	NO	NO	-
Sensitivity to air	No	No	-
Sensitivity to vapour	No	No	-
Experiment Hazards	NO	NO	-
Equipment Hazards	-	-	-
Biological hazards	NO	NO	-
Radioactive Hazards	NO	NO	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	-

SEM characterization of of innovative graphene-based hybrid fillers and their inks for multifunctional applications

Background and Context

Flexible and portable electronic devices and energy storage equipment made from conductive nanomaterials using printing or inks deposition technology have garnered significant attention due to their low-cost, high-throughput, and eco-friendly manufacturing processes over the past few decades. Electrical conductive nanostructured inks have emerged as promising candidates for designing flexible electronics because of their cost-effective synthesis methods and compatibility with current manufacturing processes.

In particular, the development of highly concentrated conductive ink using graphene powders as a raw material is seen as a promising direction. Most high-concentration graphene conductive inks are prepared from low-concentration graphene dispersions containing polymers as stabilizers. However, the solvents used in these dispersions often do not meet the requirements of the printing methods. Among various printing technologies, screen printing shows great promise for industrial-scale production due to its ability to print thick patterns with low sheet resistance (RS) on a wide range of substrates.

To realize applications in graphene-based flexible electronics, further improvement in graphene ink formulation and the development of simple, efficient post-treatment processes for printed patterns are needed. Polymers used to prevent graphene agglomeration in dispersions through steric hindrance can also adjust the rheological properties, storage performance of inks, and the flexibility and adhesion of printed patterns to substrates. New innovative fillers, based on graphene functionalized with metals or other nanoparticles, may be useful for realizing conductive inks with additional properties such as thermal, magnetic, electromagnetic shielding, optical, and catalytic properties.

Therefore, the aim of this proposal is to study, using the instrument suite of IM@IT, the correlation between innovative Few Layers Graphene (FLGs), hybrid fillers consisting of graphene few layers modified with metal, inorganic or carbon particles, the filler spatial distribution, and the processing parameters related to screen-printing deposition technologies. Hybrid fillers may be realized by combining FLGs with pristine metals or inorganic particles, but they can also be prepared by direct synthesis of hybrid graphene structures. In particular a process has been developed to cover the graphene layers with carbon nanotubes and also conductive metal fillers during the direct non-oxidative exfoliation of graphite in gas phase. This process allows to create hybrid structures of few-layer graphene with other fillers without the need of a post-functionalization process.

The objective is to investigate how the chemical modification of FLGs, obtained directly during the process of graphene exfoliation starting from graphite, may affect the morphology of filler as well as the spatial distribution of filler within the inks because of the different technologies adopted for the processing of the composites.

2. Proposed experiment

The characterization of the inks will be performed as follows.

SEM characterization of the fillers and inks, performed at the IPCB CNR Unit, will offer insights into the morphology and assembling of nanoplatelets and spatial filler distribution within the inks. In separate experiment proposals, SAXS/WAXD and TEM, available at IPCB CNR Unit, will be performed on the fillers and inks and will allow to evaluate the orientation of the filler and its

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aggregation, contributing to the enhanced properties of the resulting inks. Furthermore, RAMAN confocal microscopy, available at the CSGI - University of Florence Unit, will provide chemical information about the modified FLGs and their interface interactions with the main components of inks.

3. Summary of previous experimental proposals or characterisation

No previous experiments have been carried out on these samples

4. Justification of experimental time requested

We have requested the SEM FEI available at the IPCB CNR Unit to characterize 8 samples (4fillers+4inks) obtained by modified FLG with metal nanoparticles, inorganic nanoparticles, multiwalled carbon nanotubes (MWCNTs) and single wall carbon nanotubes (SWCNTs). Non additivated FLG will be also characterized by comparison.

Therefore, a total of 2 samples will be analysed. After discussion with the instrument scientist, we request 2 days of SEM FEI access, for a fully and thorough characterization of the materials. The foreseen beam time accounts set up and for the data collection on the samples.

References

S. Tkachev, M. Monteiro, J. Santos, E. Placidi, M. Ben Hassine, P. Marques, P. Ferreira, P. Alpuim, A. Capasso
Environmentally Friendly Graphene Inks for Touch Screen Sensors
(2021) Advanced Functional Materials, 31, 2103287
https://doi.org/10.1002/adfm.202103287

T. Sang Tran, N. Kumar Dutta, N. Roy Choudhury,
Graphene inks for printed flexible electronics: Graphene dispersions, ink formulations, printing techniques and applications,
(2018) Advances in Colloid and Interface Science, 261, 41-61
https://doi.org/10.1016/j.cis.2018.09.003

K. Parvez, R. Worsley, A. Alieva, A. Felten, C. Casiraghi
Water-based and inkjet printable inks made by electrochemically exfoliated graphene,
(2019) Carbon, 149, 213-221
https://doi.org/10.1016/j.carbon.2019.04.047

SEM ZEISS GEMINI

Experiment Proposal

Experiment number GP2024056

Principal investigator (*)	Dr Enrico Perelli Cippo, Consiglio Nazionale delle Ricerche, ITALY
Co-investigator	Dr Stephanie Cancelli, University of Milano-Bicocca, ITALY
Co-investigator	Professor Giuseppe Gorini, Milano-Bicocca University, ITALY
Co-investigator	Mr Federico Caruggi, University of Milano-Bicocca, ITALY
Co-investigator	Professor Gabriele Croci, University of Milano - Bicocca, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Test of radiation hardness and material degradation of boron GEM foils using SEM ZEISS GEMINI
MRF Instrument	**SEM ZEISS GEMINI** **Days requested:** 1
Access Route	Direct Access **Previous GP Number:** No
Science Areas	Engineering, Physics **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

Neutron detectors based on Gas Electron Multiplier (GEM) technology are increasingly used thanks to their features, such as: good spatial resolution (in the order of mm) and good detection efficiency; they can cover large areas with different shapes and they can sustain counting rates up to MHz/cm2. Usually GEMs are coupled with a single boron layer to detect thermal neutrons reaching a detection efficiency in the order of a few percent. To increase this value a new device has been developed between the two IM@IT Units University of Milano Bicocca and ISTP-CNR. The new GEM detector has the innovative boron-GEM (BGEM) foils, standard GEM foils covered on both sides with a B4C layer. The GEM will be tested using SEM ZEISS GEMINI to investigate the boron deposition thickness and composition and SOURIRE to the test the resistance of the BGEM foils under 10^10 n/cm2s neutron flux, both instruments of IM@IT and IMAT beamline to study the uniformity of the boron coating at ISIS Facility.

Publications

Cippo, E.P., et al. : gem-based thermal neutron detector for high counting rate applications. Journal of Instrumentation 10 (2015).

G. Croci, et al. : Gem-based thermal neutron beam monitors for spallation sources. Nuclear Instruments and Methods in Physics Research Section A 732, 217–220 (2013)

S. Cancelli, et al. : Development of a ceramic double thick gem detector for transmission measurements at the vesuvio instrument at isis. Journal of Instrumentation 16 (2021)

Instruments	**IMAT** **Days Requested:** 1
Access Route	Direct Access **Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	None **Sponsor:**
Grant Title	- **Grant Number:**
Start Date	- **Finish Date:**
Similar Submission?	
Industrial Links	

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Science and Technology Facilities Council
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Sample record sheet

Principal contact	Dr Enrico Perelli Cippo, Consiglio Nazionale delle Ricerche, ITALY		
MRF Instrument	**SEM ZEISS GEMINI**	**Days Requested:** 1	
Special requirements:			

SAMPLE

Material	BGEM foil	-	-
Formula	10B4C and Cu	-	-
Forms	Solid		
Volume	cc		
Weight	10 g		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	No	-	-
Sample Prep Hazards	None	-	-
Special equip. reqs	None	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	None	-	-
Equipment Hazards	-	-	-
Biological hazards	None	-	-
Radioactive Hazards	None	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Removed By User	-	-

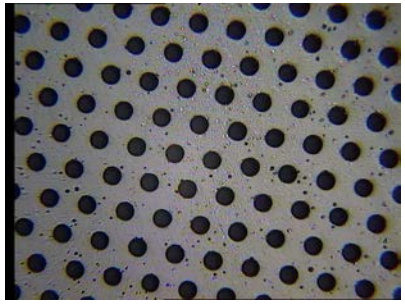
Test of radiation hardness and material degradation of boron GEM foils using SEM ZEISS GEMINI

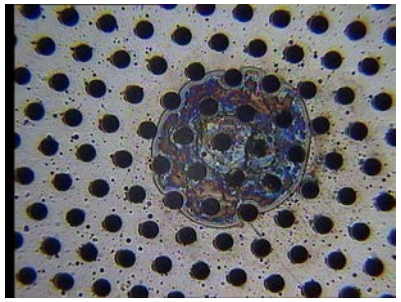
1. Background and Context

Neutron detectors utilizing Gas Electron Multiplier (GEM) technology are gaining popularity due to their notable characteristics, such as high spatial resolution (approximately in the millimeter range) and efficient detection capabilities [1]. These detectors can cover extensive areas in various shapes and can handle counting rates up to MHz/cm². Typically, GEMs are paired with a single boron layer to detect thermal neutrons, achieving a detection efficiency of a few percent [2]. To improve this efficiency, a new device has been developed through collaboration between the IM@IT Units at the University of Milano Bicocca and ISTP-CNR. This advanced GEM detector incorporates innovative boron-GEM (BGEM) foils, which are standard GEM foils coated on both sides with a B4C layer [3]. The final device has six BGEM foils and it has been characterised at the ISIS Facility on the VESUVIO experiment reaching a detection efficiency of 16% at 25 meV.
Thanks to the obtained results, the detector can be used to perform experiment in transmission geometry.
The GEM will be examined using the SEM ZEISS GEMINI to analyse the thickness and composition of the boron deposition, and SOURIRE to test the BGEM foils' resistance under a neutron flux of 10^{10} n/cm²s. Both instruments are part of IM@IT. Additionally, the IMAT beamline at the ISIS Facility will be used to assess the uniformity of the boron coating.
In this proposal our team ask to obtain measurement time at the SEM ZEISS GEMINI.

2. Proposed experiment

Despite of the obtained good results, during the neutron measurements we observed an anomalous behaviour of the detector and after preliminary tests, the GEM response resulted to be different from the response before the ISIS measurements.
Thus, further analysis on the B4C coating is needed to improve the BGEM production process. Preliminary information on the status of coating in selected locations of the foils can be obtained performing just an image. The SEM ZEISS GEMINI instrument allows to obtain it with high spatial resolution (in the order of 1.2 nm) that is necessary to resolve the details of the internal structure (and possible damages like cracks or inhomogeneity) of the B4C layer that is just 1 μm thick.
Significant locations on the GEM foils will be selected in agreement with the Instrument scientist, including both parts of the foil that are in a good condition (image on the left) or visibly damaged for instance by electric discharges (picture on the right).





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4. Justification of experimental time requested

The GEM foil under study is made of a 50 µm kapton layer sandwiched between two 5 µm copper layers. The foil is micro-perforated with a high hole density (50-100 mm2) with a bi-conical shape (external diameter of 70 µm, internal diameter of 50 µm) and the pitch is 140 µm. The BGEM foil is obtained covering the GEM foil with a 1µm thick $^{10}B_4C$ layer on both sides. The $^{10}B_4C$ has been chosen since it has more stable than pure ^{10}B. The BGEM foil has an area of 10x10 cm^2.
The GEM foil to be analysed is just one but the number of slices to be obtained from it and analysed will be discussed with the IS. Excluding specimen preparation, to conduct the experiment, we expect one day of measurements at the SEM instrument. The facility already has the adequate sample holder.

References

1. Sauli, F.: The gas electron multiplier (gem): Operating principles and applications. Nuclear Instruments and Methods in Physics Research, Section A: Accelerators, Spectrometers, Detectors and Associated Equipment 805, 2–24 (2016). https://doi.org/10.1016/j.nima.2015.07.060.
2. Cancelli, S., Muraro, A., Cippo, E.P., Romanelli, G., Abba, A., Chen, Y., Grosso, G., Gorini, G., Hu, Z., Lai, C.-C., Cormack, O.M., Robinson, L., Svensson, P.-O., Tardocchi, M., Hall-Wilton1, R., Xie, Y., Zhijia, S., Zhou, J., Zhou, X., Croci, G.: Development of a ceramic double thick gem detector for transmission measurements at the vesuvio instrument at isis. Journal of Instrumentation 16 (2021). https://doi.org/10.1088/1748-0221/16/06/P06003.
3. Muraro, A., Claps, G., Croci, G., Lai, C.C., Oliveira, R.D., Altieri, S., Cancelli, S., Gorini, G., Hall-Wilton, R., H¨oglund, C., Cippo, E.P., Robinson, L., Svensson, P., Murtas, F.: Mbgem: a stack of borated GEM detector for high efficiency thermal neutron detection. European Physical Journal Plus 136, 1–14 (2021). https://doi.org/10.1140/epjp/s13360-021-01707-2.

Experiment Proposal

Experiment number GP2024121

Principal investigator (*) Dr Ilaria Negri, Università Cattolica del Sacro Cuore, ITALY
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator

Experiment title	Identification and characterization of the size and shape of biodegradable microplastics in contaminated honey bees through SEM/EDX	
MRF Instrument	**SEM ZEISS GEMINI**	**Days requested:** 3
Access Route	Direct Access	**Previous GP Number:** No
Science Areas	Biology and Bio-materials, Environment, Materials	**DOI:** -
Sponsored Grant	None	**Sponsor:** -
Grant Title	-	**Grant Number:** -
Start Date	-	**Finish Date:** -
Similar Submission?	-	
Industrial Links	-	

Non-Technical Abstract

Honeybees are key pollinators of crops and wild plants and provide humans with various products. This insect is also a well-known bioindicator of environmental pollution, including micro- and nano-plastics (MNPs), i.e. small plastic particles originating from the degradation of plastics. Plastic pollution may also harm the health of bees by ingestion of contaminated diet and/or by skin contact. While ecotoxicological studies have almost exclusively focused on conventional plastics, little information is available on the environmental toxicity of biodegradable MNPs. The present proposal aims to investigate the impact of oral and contact exposure of honeybees to biodegradable MNPs and the potential contribution of bees to the dispersal of such MNPs through the food chain, where they may bioaccumulate and bio-magnify in ecosystem top predators.

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Dr Ilaria Negri, Università Cattolica del Sacro Cuore, ITALY
MRF Instrument **SEM ZEISS GEMINI** **Days Requested:** 3
Special requirements:

SAMPLE

Material	PBAT (Polybutylene Adipate Terephthalate) - based biodegradable plastic	Insect (honeybees) bodies	Insect (honeybees) guts
Formula	C20H30O10	-	-
Forms	Friable powder	Solid	
Volume	cc	cc	cc
Weight	100 mg	10 mg	1 mg
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	- K	- K
Pressure Range	- mbar	- mbar	- mbar
Magnetic field range	- T	- T	- T
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	Yes	Yes
Sample Prep Hazards	No	No	No
Special equip. reqs	No	No	No
Sensitivity to air	No	No	No
Sensitivity to vapour	No	No	No
Experiment Hazards	No	No	No
Equipment Hazards	-	-	-
Biological hazards	No	No	No
Radioactive Hazards	No	No	No
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	Disposed by IS

MRF Proposal: identification and characterization of the size and shape of biodegradable microplastics in contaminated honey bees through SEM/EDX

1. Background and Context

Micro- and nano-plastics (MNPs), i.e. small plastic particles originating from the degradation of plastics, are ubiquitous contaminants in aquatic and terrestrial environments, able to pose significant risks to human and ecosystem health. MNPs are particularly mobile and may easily reach small organisms, especially invertebrates, which may ingest or transport them contributing to their dispersion through ecological interactions. Honeybees are of great importance as key pollinators in terrestrial ecosystems, for the production of honey and beeswax, and for biomedicine. Honeybees are highly sensitive to pollutants, and their health can be affected by various environmental contaminants, including pesticides, heavy metals, and airborne particulate matter. Plastic pollution may also harm the health of bees and the ecosystem services they provide. Indeed, MNPs can disrupt the bees' digestive systems and affect the gut microbiota, leading to health issues and weakening the insects' immunity system. Furthermore, previous works demonstrated that during the foraging activity honeybees can collect airborne particulate matter, including plastic fragments which may be trapped by the hairy body of the insects (Negri et al., 2015; Edo et al. 2021). Microplastics can therefore enter the food web through predation, leading to bioaccumulation and further ecological impacts affecting not only bees but also other species within the ecosystem.

Understanding these interactions is therefore important for assessing the broader environmental impact of microplastics, especially biodegradable ones whose effects on human health and the environment are less known. Indeed, while ecotoxicological studies of MNPs have almost exclusively focused on conventional plastics, little information is available on the environmental toxicity of biodegradable plastic fragments. Moreover, knowledge gaps exist with regard to the fate of biodegradable MNPs in the environment and mechanisms of transportation.

The present proposal aims to investigate the impact of the exposure of honeybees to biodegradable MNPs and the contribution of honeybees in MNPs dispersal. SEM/EDX will be used to assess the size, morphology and purity of the plastic fragments before and after the exposure.

This proposal falls within the frame of the project Minagris (MIcro- and NAno-plastics in AGRIcultural Soils) funded by the European Union's Horizon 2020 Programme for Research & Innovation under Grant Agreement number: 101000407. The aim of the project Minagris is to address the environmental impacts of MNPs used in agricultural systems, particularly focusing on their effects on soil health, biodiversity, and ecosystem services.

2. Proposed experiment

Honeybees will be exposed orally and by contact according to OECD guidelines for the testing of chemicals (OECD 1998a, b) to MNPs made of PBAT (Polybutylene Adipate Terephthalate), a common biodegradable plastic made from renewable resources, such as corn starch and sugarcane. Oral exposure will be carried out for 48h during which the bees will be exposed to an artificial diet contaminated with the biodegradable MNPs (OECD 1998a). Then, the gut of bees will be dissected and the gut content analysed to detect the presence of the plastic fragments.

The aims of the experiment are to verify if: i) the MNPs are ingested by bees and excreted with the faeces; ii) if modifications in the size and morphology occur during oral exposure; iii) if oral exposure to the MNPs can induce lethal or sublethal effects on bees (e.g. behavioural abnormalities).

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Exposure by contact will be carried out with anesthetized bees (OECD 1998b). After 48h from the treatment, the body of the bees will be analysed to detect the presence of the plastic fragments.

The aim of the experiment is to verify: i) if the MNPs are actively removed and/or manipulated by the bees through the typical cleaning behaviour and if modifications in the size and morphology of MNPs occur after manipulation; ii) if the contact exposure to the MNPs can induce lethal or sublethal effects on bees (e.g. behavioural abnormalities).

These data will expand our knowledge about the biotic transportation routes of biodegradable plastic fragments, contributing to their aerial dispersal and increased risk of MNPs to enter into the food web through predation, where they may bioaccumulate and bio-magnify in ecosystem top predators. In addition, data on the impact of PBAT MNPs on the bee health will be collected.

The ISIS@MACH ITALIA SEM/EDX ZEISS Gemini will allow to characterise the size and morphology of the MNPs prior and after the experiments.

In particular, SEM/EDX ZEISS Gemini is the unique instrument able to provide the following information: i) size down to the nanoscale of the plastic fragments (provided by SEM); ii) details on the morphology and topography of the sample's surface (provided by Secondary Electron Imaging); iii) presence/absence of inorganic contaminants in the PBAT fragments (provided by Backscattered Electron Imaging and EDX).

4. Justification of experimental time requested

For the current proposal we require 3 working days to be used as follow:

Day 1:

- Sample preparation: mounting of MNPs (about 10 μg x 5 replicates), insects' guts (3 guts from plastic-treated bees and 3 guts from control bees), and insects' bodies (3 bodies from plastic-treated bees and 3 bodies from control bees) onto SEM stubs, and Chromium or carbon coating.
- SEM observations of MNPs to measure the size and characterise their morphology, topography, etc.

Day 2:

- SEM observations of the 6 dissected guts of bees (treated and control) to check for the presence of MNPs which will be characterised by size, morphology, and topography

Day 3:

- SEM observations of the 6 bodies of bees (treated and control) to check for the presence of MNPs which will be characterised by size, morphology, and topography.

References

- Edo C, Fernandez-Alba AR, Vejsnæs F, van der Steen JJM, Fernandez-Pinas F, Rosal R (2021). Honeybees as active samplers for microplastics. Sci. Total Environ. 767, 144481.
- Negri I, Mavris C, Di Prisco G, Caprio E, Pellecchia M (2015). Honey Bees (*Apis mellifera*, L.) as Active Samplers of Airborne Particulate Matter. PLoS ONE 10(7): e0132491.
- OECD (1998a), *Test No. 213: Honeybees, Acute Oral Toxicity Test*, OECD Guidelines for the Testing of Chemicals, Section 2, OECD Publishing, Paris.
- OECD (1998b), *Test No. 214: Honeybees, Acute Contact Toxicity Test*, OECD Guidelines for the Testing of Chemicals, Section 2, OECD Publishing, Paris.

Experiment Proposal

Experiment number GP2024135

Principal investigator	Professor Alessandro Colombo, Politecnico di Milano, ITALY
Co-investigator (*)	Dr Stephanie Cancelli, University of Milano-Bicocca, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Fiber fractography of climbing ropes
MRF Instrument	**SEM ZEISS GEMINI**
Access Route	Direct Access
Science Areas	Engineering
Sponsored Grant	None
Grant Title	-
Start Date	-
Similar Submission?	-
Industrial Links	-

Days requested: 2
Previous GP Number: No
DOI: -
Sponsor: -
Grant Number: -
Finish Date: -

Non-Technical Abstract

European standards require climbing ropes to undergo a breaking test known EN 892, which is meant to simulate the conditions that bring a rope to rupture during typical climbing accidents. Recent findings however have shown that the mechanisms through which rope fibers break are diverse and involve both shear between fibers and the carabiner or rock, and heat and energy transfer between the fibers. The resulting rupture mechanics may be significantly different in a realistic scenario and in the EN 892 test (or other tests, such as the UIAA 101 test). We aim to investigate the fiber fractography of different models of climbing ropes in the EN 892, UIAA 101 and realistic scenario tests, to provide a more solid baseline against which to compare the results of alternative, and potentially more realistic standardized testing protocols.

Publications -

ISIS neutron and muon source

E-platform: No

Instruments	
Access Route	
Science Areas	
Sponsored Grant	
Grant Title	
Start Date	
Similar Submission?	
Industrial Links	

Days Requested:
Previous RB Number:
DOI:
Sponsor:
Grant Number:
Finish Date:

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Sample record sheet

Principal contact Dr Stephanie Cancelli, University of Milano-Bicocca, ITALY
MRF Instrument **SEM ZEISS GEMINI** **Days Requested:** 2
Special requirements:

SAMPLE

Material	Polyamide	Polyamide	Polyamide
Formula	-	-	-
Forms	Solid	Solid	Solid
Volume	cc	cc	cc
Weight	mg	mg	mg
Container or substrate	glass	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	- K	- K
Pressure Range	- mbar	- mbar	- mbar
Magnetic field range	- T	- T	- T
Standard equipment	-	None	None
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	Yes	Yes
Sample Prep Hazards	None	None	None
Special equip. reqs	-	None	None
Sensitivity to air	No	No	No
Sensitivity to vapour	No	No	No
Experiment Hazards	None	None	None
Equipment Hazards	-	-	-
Biological hazards	None	None	None
Radioactive Hazards	None	None	None
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Removed By User	Removed By User	Removed By User

Fiber fractography of climbing ropes with the SEM ZEISS GEMINI instrument

1. Background and Context

In recent years, climbing has experienced a surge in popularity, becoming one of the fastest-growing sports worldwide. This rise can be attributed to several factors, including its debut as an Olympic sport in 2021 and the increasing accessibility of climbing gyms in urban areas.
One of the main components in the set of safety devices used in sport climbing is the rope, which allows the climber to climb the wall while being belayed by the partner.
European norms mandate several tests to verify the resistance of ropes to falls, and rope manufacturers as well as mountaineering associations are continuously researching novel testing protocols specifically designed to simulate rope breakage on rocks, as this is the most common scenario [1].
A recent work [2] has identified 4 different mechanisms involved in the rope failure: lateral pressure with high stress, lateral pressure with low stress concentration, high-speed tensile break and melted and fused fibre ends. The failure mechanisms are recognizable through SEM imaging by the geometry of the broken rope fibres. These 4 failure mechanism appear with different frequency through the rope structure depending on the fall dynamics, as it was observed in ropes subject to different recently proposed testing protocols [2]. The geometric distribution of failure mechanisms through the rope diameter during normative lab tests, such as the EN 892 [3] or the UIAA 101 standard [4] has however been only partially characterized. Moreover, little data is available regarding the failure mechanisms of ropes coming from real-world failures. A more complete characterization of both the failure mechanisms appearing through the rope diameter in normative tests, and in real-world failures, may guide the improvement of the normative tests.
In this proposal our team, which includes researchers from the University of Milano-Bicocca, Politecnico di Milano, as well as Centro Studi Materiali e Tecniche, ask to obtain measurement time at the SEM ZEISS GEMINI at the Milano-Bicocca University Unit, which is part of ISIS@MACH ITALIA.

2. Proposed experiment

We propose to analyse three sets of fibres. Figures 1 and 2 show two different sets of fibres (core and sheath) of ropes broken with EN 892 and UIAA 101 test protocols.

Figure 1 Core (white fibre) and sheath (yellow fibre) of a climbing rope broken with the EN 892 protocol.

Figure 2 Core (white fibre) and sheath (yellow fibre) of a climbing rope broken with the UIAA 101 protocol.

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We want to analyse the fibre ends of fibres taken at different depths along the rope fracture with a SEM analysis to map the failure mechanisms obtained both in EN 892 or UIAA 101 tests, and in real-world failures.
All the fibres to be analysed are made of the same material (polyamide).
The SEM ZEISS GEMINI of ISIS@MACH ITALIA is necessary to obtain accurate spatial resolution images of the single fibre.
The SEM images will be provided as per the agreement with the Instrument Scientist of SEM ZEISS GEMINI.

3. Summary of previous instrument time or characterisation

This is the first time that this experiment is being conducted by University of Milano-Bicocca, Politecnico di Milano and Centro Studi Materiali e Tecniche.

4. Justification of experimental time requested

We propose to analyse 3 samples (EN 892 test, UIAA 101 test, 2 real-world failures (same sample)) and each sample is made of several fibres taken from different depths through the rope failure surface. Since every sample needs to be cover with metal, one day is required to perform almost 2 samples; thus we request two days of experiment.

References

[1] Leal, A.A., Stämpfli, R. and Hufenus, R. (2017) “On the analysis of cut resistance in polymer-based climbing ropes: New testing methodology and resulting modes of failure,” Polymer Testing, 62, pp. 254–262. Available at: https://doi.org/10.1016/j.polymertesting.2017.07.004.

[2] Sedláček, D. and Stöhr, A. (2024) “Cut resistance of climbing ropes - A comparative analysis of existing measurement methods and a simulated accident,” Engineering Failure Analysis, 157. Available at: https://doi.org/10.1016/j.engfailanal.2023.107906.

[3] https://krok.biz/info/file_download/131/BS_EN_892_2012.pdf

[4] https://theuiaa.org/documents/safety-standards/101_UIAA_Ropes_V9_2019.pdf

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Experiment Proposal

Experiment number GP2024149

Principal investigator	Dr Mara Limonta, CNRS, FRANCE
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Geochemical composition and microfeatures of single detrital biotite and carbonate grains in the provenance study of the Bengal Fan turbidites (IODP Expedition 354).
MRF Instrument	**SEM ZEISS GEMINI** **Days requested:** 3
Access Route	Direct Access **Previous GP Number:** No
Science Areas	Environment **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	Sedimentary provenance analysis aims at unraveling the origin and processes that generated any given sediment, tracing back its evolution from the sink to its sources. Traditional approaches rely on the investigation of bulk composition based on petrography, geochemistry or heavy mineral assemblages. In addition, single-grain geochemical and geochronological methods may be also exploited to further assess the evolution and provenance of sedimentary systems. In this project the analysis of chemical composition and surface microfeatures of single detrital grains of biotite and carbonate in Bengal Fan turbidites using SEM ZEISS GEMINI, allow to discriminate their provenance from different source rocks and to better reconstruct the evolution of the Himalayas during the last 18 Ma, and to detect diagenetic processes. Single grain analysis allows to pinpoint sediment sources and to highlight sediment mixing from specific sources enhancing provenance resolution with respect to bulk approaches
Publications	-

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Dr Mara Limonta, CNRS, FRANCE
MRF Instrument **SEM ZEISS GEMINI** **Days Requested:** 3
Special requirements:

SAMPLE

Material	Biotite grains from Manaslu Leucogranite	Biotite grains from Greater Himalaya gneiss	Carbonates of Bengal fan turbidites
Formula	-	-	-
Forms	Solid	Solid	Solid
Volume	cc	cc	cc
Weight	mg	mg	mg
Container or substrate	plastic box	plastic box	plastic box
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	- K	- K
Pressure Range	- mbar	- mbar	- mbar
Magnetic field range	- T	- T	- T
Standard equipment	None	None	None
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	Yes	Yes
Sample Prep Hazards	No	No	No
Special equip. reqs	No	No	No
Sensitivity to air	No	No	No
Sensitivity to vapour	No	No	No
Experiment Hazards	No	No	No
Equipment Hazards	-	-	-
Biological hazards	No	No	No
Radioactive Hazards	No	No	No
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Removed By User	Removed By User	Removed By User

Geochemical composition and microfeatures of single detrital biotite and carbonate grains in the provenance study of the Bengal Fan turbidites (IODP Expedition 354).

1. Background and Context

Sedimentary provenance analysis aims at unraveling the origin and processes that generated any given sediment, tracing back its evolution from the sink to its sources.
In the last 30 years, modern sediments of the Ganga-Brahmaputra-Bengal sedimentary system, have been studied using traditional approaches (e.g. petrography, mineralogy and geochemistry) to calculate sediment budgets and erosion rates, to quantify physical and chemical processes during transport and deposition and to reconstruct the evolution of the Himalayas, defining the interrelationships between tectonic activity and climate [1-3].
Despite that, some relevant aspects of weathering and evolution of the Himalayas are still subject of debate. The main goal of my current post-doctoral research at the CNRS-CRPG is to better constrain provenance of Bengal Fan turbidites, to reconstruct the evolution of the Himalayan belt and to quantify weathering conditions in the Himalayan system from Miocene to the present by implementing single grain geochemical/isotopic analysis on detrital species that are commonly found in the Bengal Fan detrital record (IODP Exp. 354) and by increasing accuracy of provenance determination.
The main goal of this project is to define single-grain geochemical signatures of detrital biotites and carbonates and to characterize their surface microfeatures with primary application in source-to-sink studies. It will allow to exploit their chemical composition and their morphologies as a provenance and diagenesis tracer, respectively. This method will be tested on Bengal Fan turbidites.

2. Proposed experiment and previous characterization

- Major elements of single biotite grains from different Himalayan source rocks (Manaslu leucogranites *versus* Greater Himalaya gneiss) will allow to define their chemical fingerprint. In-house Raman library for biotites will be developed combining Raman peaks position and Fe/Fe+Mg concentration as revealed by SEM-EDS analysis on the same spot. The feasibility of this approach was already tested on a few biotite grains from the leucogranite and from gneiss (Fig.1). Single grain fingerprint is expected to provide new information on the sources eroded in the Himalayan belt and to highlight sediment mixing from specific sources enhancing provenance resolution with respect to bulk approaches.
- Coating, dissolution microfeatures and recrystallization of secondary mineral on carbonate grains surface allow to recognize diagenetic carbonates and distinguish them from marine and detrital carbonates. Oxygen and carbon isotopic composition of bulk carbonates from Bengal Fan turbidites show mixing of carbonate grains of different sources (marine, diagenetic and detrital) and we miss part of the information that is carried by each detrital grain (Fig.2). Isotopic fingerprint of single detrital carbonate grains could not be measured using the mass spectrometer coupled with a gas chromatograph in fine sand and silt grain-size[4]. SEM ZEISS GEMINI allows me to analyze major elements of single grains of biotite and to recognize different microfeatures on the carbonate grains surface with the EBSD detector. Thanks to its high resolution of 1.2 nm it will be possible to analyze morphologies not only sand-sized grains (63-2000 μm) but also on silt-sized grains (4-63 μm) which characterize an important part of the Bengal fan turbidites.

3. Justification of experimental time requested

Analysis of the samples will be performed in three days as described in the table below. For each sample ~100 grains will be analyzed.

	sample n°	Days
Analysis of single biotite and carbonate grains by SEM ZEISS GEMINI		
Preparation of samples n° 1-2-3 (carbon coating)		
Major elements and microfeatures of biotites of Manaslu leucogranite sample	1	
Major elements and microfeatures of biotites of gneiss sample from Greater Himalaya	2	
Major elements and microfeatures of carbonates of Bengal Fan turbidite	3	
Data interpretation		

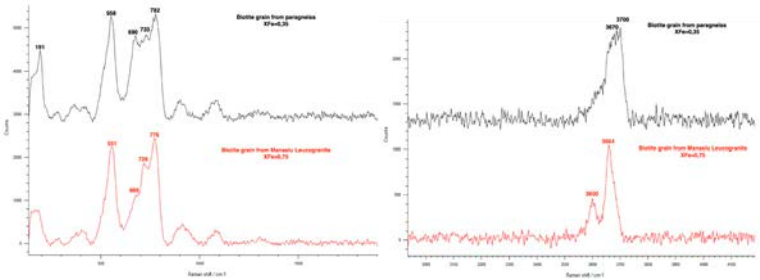

Fig. 1 Raman spectra of biotites of Manaslu leucogranites and gneiss formation (Limonta, unpublished).

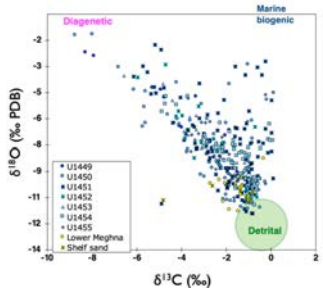

Fig 2. C and O isotopic compositions of carbonates of Bengal fan turbidites (France-Lanord, unpublished).

REFERENCES

[1] Galy, A., and France-Lanord, C. "Higher erosion rates in the Himalaya: Geochemical constraints on riverine fluxes." *Geology* 29.1 (2001): 23-26.
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[3] France-Lanord, C., et al. "Bengal fan." Proceedings of the International Ocean Discovery Program 354 (2016).
[4] Limonta, Europlanet TA 2022 report “Isotopic composition of single detrital carbonate grains in the source-to-sink study of the Bengal Fan record”.

Experiment Proposal

Experiment number GP2024160

Principal investigator	Dr Stefania Zappia, Consiglio Nazionale delle Ricerche, ITALY
Co-investigator (*)	Dr Anna Maria Ferretti, CNR SCITEC, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	SEM and SEM EDX analysis of Metal NP supported Over Microporous Polymelamine Framework for the CO2 absorption and reduction

MRF Instrument	**SEM ZEISS GEMINI**	**Days requested:**	1
Access Route	Direct Access	**Previous GP Number:**	-
Science Areas	Energy, Materials	**DOI:**	-
Sponsored Grant	None	**Sponsor:**	-
Grant Title	-	**Grant Number:**	-
Start Date	-	**Finish Date:**	-

Similar Submission? We present a proposal for the 13CNMR solid state analysis of the melamine based POP

Industrial Links -

Non-Technical Abstract

Porous organic polymers (POPs) have long been considered as prime candidates for CO2) capture, separation, and conversion. Their permanent porosity, structural tunability, stability and relatively low cost are key factors in such considerations. Whereas heteroatom-rich microporous networks have been actively exploited to boost the CO2 affinity of POPs, recently, the focus has shifted to engineering the pore environment, resulting in a new generation of highly microporous POPs rich in heteroatoms and featuring abundant catalytic sites for the capture and conversion of CO2 into value-added products. We have just tested the capability of the POP, obtained by the coupling between melamine and benzene-1,3,5-tricarboxaldehyde, to absorb CO2 and generate Re@POP derivative, that show a catalytic activity for (CO2RR). We synthesized a new family of melamine based POP changing the aldehydic components in order to see if changing the size and the density of the pore the CO2 absorption capability

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Science and Technology Facilities Council
ISIS Neutron and Muon Source

Sample record sheet

Principal contact	Dr Anna Maria Ferretti, CNR SCITEC, ITALY		
MRF Instrument	**SEM ZEISS GEMINI**	**Days Requested:**	1
Special requirements:			

SAMPLE

Material	Microporous Polymelamine Framework (POP)	Microporous Polymelamine Framework (POP)@PtNP (Platinum nanoparticles)	-
Formula	Melamine and aldehyde	Melamine and aldehyde, Pt	-
Forms	Friable powder	Friable powder	
Volume	cc	cc	
Weight	5-10 mg	mg	
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	- K	-
Pressure Range	- mbar	- mbar	-
Magnetic field range	- T	- T	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	Yes	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	No	-
Sensitivity to vapour	No	No	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	Returned to user by instrument scientist (when inactive)	-

1. Background and Context

Porous organic polymers (POPs) have long been considered as prime candidates for CO_2) capture, separation, and conversion. Their permanent porosity, structural tunability, stability and relatively low cost are key factors in such considerations. Whereas heteroatom-rich microporous networks have been actively exploited to boost the CO_2 affinity of POPs, recently, the focus has shifted to engineering the pore environment, resulting in a new generation of highly microporous POPs rich in heteroatoms and featuring abundant catalytic sites for the capture and conversion of CO_2 into value-added products. We have just tested the capability of the POP, obtained by the coupling between melamine and benzene-1,3,5-tricarboxaldehyde, to absorb CO_2 and generate Re@POP derivative, that show a catalytic activity for (CO2RR). We synthesized a new family of melamine based POP changing the aldehydic components in order to see if changing the size and the density of the pore the CO_2 absorption capability increase. Moreover, we plan to coordinate the POP with metal nanoparticles (MNPs) using NPs that have a good catalytic activity for the CO2 reduction like Pt, Ag but also Ni to test them as catalysts for the CO_2 reduction simultaneously.

In order to get a good characterization of the POP and POP@MNP from the morphological and chemical point of view SEM images and SEM-EDX analysis are necessary

2. Proposed experiment

We want to see the POP and POP@MNP morphology and also the MNP distribution so we plan to take SEM micrographs of the POP with and without the MNPs.

3. Summary of previous experimental proposals or characterization

This is the first proposal submitted at ISIS@MACH, anyway previous analysis are reported in Zappia S. et al. Polymers 2022, 14, 5472

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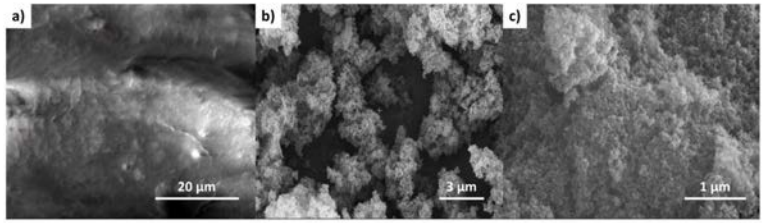

Figure 3. SEM images of **POP2** taken at different magnification, **(a)** 20 µm, **(b)** 3 µm, and **(c)** 1 µm.

4. Justification of experimental time requested

We would like to test just 2 samples so we estimated that one day is enough.

SEM ZEISS SIGMA

Experiment Proposal

Experiment number GP2024065

Principal investigator (*) Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator

Experiment title	Training on Scanning electron Microscopy with SEM ZEISS SIGMA		
Training MRF	**SEM ZEISS SIGMA**	**Days requested:**	2
Access Route	Direct Access	**Previous GP Number:**	GP2024026 (experimental proposal)
Science Areas	Materials	**DOI:**	-
Sponsored Grant	None	**Sponsor:**	-
Grant Title	-	**Grant Number:**	-
Start Date	-	**Finish Date:**	-
Similar Submission?	-		
Industrial Links	RGF SRL		

Non-Technical Abstract

Consorzio Physis SRL S.B. is a benefit company dedicated to the production of high-quality metallic components and surface treatments for fashion and luxury. We aim to enhance product quality and longevity by understanding surface composition, defects imaging and analysis, and mapping of surface elements. We are interested in the SEM ZEISS instrument at the CSGI Unit, which can provide valuable insights into surface morphology and composition, offering advantages for examining surface defects and characterizing metallic artifacts. We seek training on the SEM ZEISS instrument, focusing on Scanning Electron Microscopy principles and practical applications. The knowledge obtained from this training would be valuable for the submission of future experimental proposals.

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Science and Technology Facilities Council
ISIS Neutron and Muon Source

Sample record sheet

Principal contact Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY
Training Instrument **SEM ZEISS SIGMA** **Days Requested:** 2
Special requirements:

	SAMPLE		
Material	-	-	-
Formula	-	-	-
Forms			
Volume			
Weight			
Container or substrate	-	-	-
Storage Requirements	-	-	-
	SAMPLE ENVIROMENT		
Temperature Range	-	-	-
Pressure Range	-	-	-
Magnetic field range	-	-	-
Standard equipment	-	-	-
Special equipment	-	-	-
	SAFETY		
Prep lab needed	-	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	-	-	-
Sensitivity to vapour	-	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	-	-	-

Training on Scanning electron Microscopy with SEM ZEISS SIGMA

1. Background and context

Consorzio Physis SRL S.B. is actively engaged in the production of fashion accessories, encompassing the manufacturing of metallic accessories and surface treatments, as well as supplying chemicals for these treatments. As part of our ongoing efforts to enhance our technical capabilities and address critical challenges related to the quality and longevity of our products, we seek to deepen our understanding of surface defects analysis, surface elemental composition, and element mapping on surfaces.

To this end, we are particularly interested in the SEM ZEISS instrument at CSGI Unit, which performs Scanning Electron Microscopy (SEM) measurements and Energy Dispersive Analysis of X-rays (EDX) using a Field Emission source. This advanced tool allows the observation of samples, including non-conductive ones, without requiring a metallization step. Understanding and investigating surface alterations, defects, and non-uniformities in the metallic components of our products is crucial for us. We believe that gaining proficiency in using this advanced tool will significantly benefit our research and development processes. Additionally, direct training of industrial personnel would provide a competitive advantage to the Italian industry and could lead to future experimental proposals at this instrument.

2. Proposed training

Consorzio Physis SRL S.B. collects producers of metallic fashion accessories and handles surface treatments and related chemicals. To address quality and longevity challenges, we aim to deepen our understanding of surface defects, elemental composition, and element mapping. We are particularly interested in training on the SEM ZEISS instrument at CSGI Unit for its advanced Scanning Electron Microscopy (SEM) capabilities. Mastering this tool will greatly benefit our research and development efforts by enhancing our ability to analyze and understand surface alterations and defects in our products and could lead to future experimental proposal.

SEM is a sophisticated surface analysis technique that offers crucial advantages for examining surface defects and characterizing the surface composition of metallic artifacts. It allows for the precise identification and analysis of defects such as cracks, porosity, and inclusions. Additionally, energy-dispersive X-ray spectroscopy (EDS) integrated with SEM enables the determination of the chemical composition of materials, providing essential information to understand the causes of defects and improve manufacturing processes. Specific training on the SEM instrument is fundamental to optimizing our team's analytical capabilities and ensuring the quality and reliability of the metallic products we produce. It also offers opportunities to improve production processes and coatings, thereby enhancing the longevity of our products.

The Zeiss Sigma scanning electron microscope with a field-emission source at the CSGI Unit is equipped with a Field Emission Source, a GEMINI column and In-Lens detector. This setup allows the acquisition of high-resolution images of both conducting and non-conducting samples, which is crucial since metallic items in the fashion industry can be conductive (e.g., galvanized items) or non-conductive (e.g., varnished items coated with lacquers or cataphoretic layers). The microscope is equipped with X-ray detectors (EDS), backscattered electrons (BSE), and secondary electrons (SE). The X-ray detection system, in addition to conventional X-ray analysis capabilities, produces high-resolution maps of electron emissions, allowing for elemental mapping on the surface. This is of great interest for understanding the surface properties, modifications, and appearance of items in footwear and leather goods finished products.

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3. Summary of previous training proposals

No previous training proposal has been presented. However, previous analysis performed at the instrument (ref. GP2024026) showed that this technique offers valuable insights on surface composition, defects imaging and elemental mapping, giving strong inputs for the research and development team to improve production processes and coating to ensure long-lasting products. An example is given here below, in Figure 1.

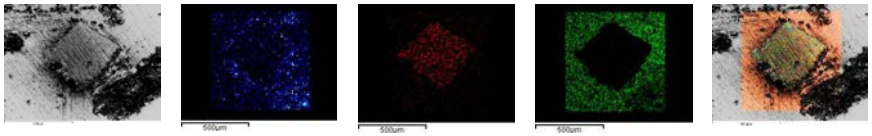

Figure 1. Secondary electron image of an inclusion, together with Cu, Fe, Zn elemental analysis maps highlighting the difference in composition between the matrix and the inclusion. The image on the right represents a cameo map where the colour coding corresponds to the energy of the emitted X-rays.

4. Justification of training proposal request

Due to the abovementioned advantages that a deep knowledge in this technique would improve our technological development, we request a training on the instrument to gain knowledge in the technique and on the analytical capabilities of the instrument.

Therefore, we request 2 days of training, as discussed and agreed with the instrument scientists. Specifically, our objectives for the training course would include:

1. Learning the principles of SEM (Scanning Electron Microscopy).
2. Learning the differences between the available detectors: energy dispersion (EDS), backscattering (BSE) and secondary electrons (in- Lens and conventional SE).
3. Preparation of the samples (combined with sample stage motions for the measurement of cross-section surfaces).
4. Use of the instrument software for data acquisition, mapping, elemental analysis and reporting.
5. Instrumental session to apply the technique on some samples.
6. Hands-on sessions for the trainees.

We believe that this training will empower us to perform more accurate and detailed analyses, ultimately leading to improved quality and durability of the fashion accessories and the knowledge on the analytical capabilities of the instrument would be valuable for the submission of future experimental proposals.

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Experiment Proposal

Experiment number GP2024074

Principal investigator	Professor Fabiana Arduini, University of Rome Tor Vergata, ITALY	
Co-investigator (*)	Dr Vincenzo Mazzaracchio, University of Rome "Tor Vergata", ITALY	
Co-investigator	Professor Massimo Bonini, CSGI - University of Florence, ITALY	
Co-investigator		
Co-investigator		
Co-investigator		
Co-investigator		
Co-investigator		
Co-investigator		
Experiment title	FE-SEM characterisation of paper-based electrochemical (bio)sensors based on carbon black/Prussian Blue nanocomposites inks	
MRF Instrument	**SEM ZEISS SIGMA**	**Days requested:** 1
Access Route	Direct Access	**Previous GP Number:** No
Science Areas	Chemistry	**DOI:** -
Sponsored Grant	Yes	**Sponsor:** Other
Grant Title	Lazio Innova Venture and Scientifica Venture Capital	**Grant Number:** CI Fast
Start Date	01/01/2024	**Finish Date:** 01/07/2025
Similar Submission?	-	
Industrial Links	SENSE4MED	

Non-Technical Abstract

Electrochemical paper-based devices have opened a new route in the analytical science sector, having a huge impact at the academic level as well as in the industrial sector. The development of electrochemical devices with nanomaterials, including carbon black, iridium oxide, and Prussian blue nanoparticles, requires surface and dispersion characterization for obtaining accurate nanomaterial-functionalized paper-based electrochemical devices. The several characterizations that will be carried out thanks to the instrumentation within ISIS@MACH ITALIA will foster the development of electrochemical paper-based devices to carry with national and European projects in which the applicant (Prof. Fabiana Arduini) is the coordinator. This proposal is part of a multi-technique investigation: the importance of FE-SEM relies on the possibility to directly image the drop-casted materials without the need for a metallisation step.

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Science and Technology Facilities Council
ISIS Neutron and Muon Source

Sample record sheet

Principal contact	Dr Vincenzo Mazzaracchio, University of Rome "Tor Vergata", ITALY		
MRF Instrument	**SEM ZEISS SIGMA**	**Days Requested:** 1	
Special requirements:			
	SAMPLE		
Material	paper, black carbon, Iron, nitrogen	carbon black	-
Formula	carbon black prussian blue nanoparticles paper (CHNFe)	carbon black	-
Forms	Solid	Liquid	
Volume	10 ml	1 cc	
Weight	10 g	1 g	
Container or substrate	paped based electrodes to be imaged as they are	paper based film	-
Storage Requirements	-	-	-
	SAMPLE ENVIROMENT		
Temperature Range	298 - K	298 - K	-
Pressure Range	1 - mbar	1 - mbar	-
Magnetic field range	- T	- T	-
Standard equipment	-	None	-
Special equipment	nothing	no	-
	SAFETY		
Prep lab needed	Yes	Yes	-
Sample Prep Hazards	no	no	-
Special equip. reqs	no	no	-
Sensitivity to air	No	No	-
Sensitivity to vapour	No	No	-
Experiment Hazards	no	no	-
Equipment Hazards	-	-	-
Biological hazards	no	no	-
Radioactive Hazards	no	no	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	-

FE-SEM characterisation of paper-based electrochemical (bio)sensors based on carbon black/Prussian Blue nanocomposites inks

1. Background and Context

As reported by the applicant in the recent review entitled "Electrochemical paper-based devices: When the simple replacement of the support to print ecodesigned electrodes radically improves the features of the electrochemical devices" published in Current Opinion in Electrochemistry (Q1) SI: Emerging Opinions (2022) [1]: "*Paper-based electrochemical (bio)sensors have emerged as highly attractive analytical devices for their superior sustainable features, such as avoiding the use of polyester as support and the reduction of waste, being incinerated after use. However, paper-based electrochemical (bio)sensors have recently demonstrated further advantages, including the simple combination with vertical microfluidics and their use as a reservoir to deliver smart electrochemical (bio)sensors able to i) contain the reagents, ii) preconcentrate the target analyte, and iii) synthesize the nanomaterials inside the paper network. Furthermore, these devices have demonstrated their ability to overcome the limitations of the other printed electrochemical sensors in the measurement of entirely liquid samples by detecting the target analyte in the aerosol phase or solid sample, without the additional sampling system. These achievements highlight their valuable and varied advantages in the sensing sector*". Electrochemical paper-based devices have opened a new route in the analytical science sector with a huge impact at the academic level as well as in the industrial sector, since the relevant articles of Prof. Henry in United States [2, 3] and the applicant in Europe [4, 5]. From industrial point of view, the spin-off company SENSE4MED, Department of Chemical Science and Technologies, University of Rome Tor Vergata, in which the applicant is the CEO, has received an investment of 510 KEuro for the developing a paper-based electrochemical sensor for cystic fibrosis diagnosis [6], from the academic point of view, the applicant is the coordinator of the PRIN2022 SMARTMASK4CF with the aim to develop a facemask functionalized with paper-based devices for precision medicine in cystic fibrosis [7]. Additionally, the paper-based devices are creating a new challenge in organ on the chip field, considering the Pathfinder Open Horizon Europe project Phoenix-OoC (2024-2027) which has the overriding goal to create an organ chip on paper and in which the applicant is the European coordinator [8].
In this framework we are proposing a multi-technique approach to investigate nanocomposites made of carbon black and Prussian Blue nanostructures, where Scanning Electron Microscopy (SEM), Small Angle Scattering of X-rays (SAXS) and Transmission Electron Microscopy (TEM) are combined to get a full picture of the system.

2. Proposed experiment

Given the relevance of the self-assembly of nanostructures in inks, especially in terms of potential aggregation and its effect on the casted material, we propose here to characterise the electrodes prepared by drop-casting on paper of dispersions in water:dimethylformamide ratio (1:1 v/v) of carbon black and Prussian Blue nanoparticles using the FE-SEM (where no metallization is needed) located at the CSGI Florence Unit of IM@IT. The modified electrode size is around 3 mm, making it very well-suited for being investigated by SEM. In particular, the use of a Field Emission microscope allows for the direct analysis of non-metallised samples. This proposal is part of a multi-technique investigation, complemented by a separate proposal to characterize the nanocomposites by TEM at the IPCB-CNR Unit in Naples and, their dispersions by SAXS at the CSGI Florence Unit. The importance of SEM in this investigation relies on the possibility to directly image the drop-casted materials. In particular, the use of a SEM equipped with a Field Emission Gun allows for the imaging of the proposed samples without the need of a metallisation step.

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3. Summary of previous experimental proposals or characterisation

In previous experiments we have characterised carbon black nanostructures by means of TEM analysis [9], but we have not yet performed any SEM carbon black-Prussian Blue nanoparticle composite used in paper-based electrochemical devices. In particular, the possibility of investigating the paper-based electrodes without the need for a metallisation step allows for the in-depth analysis of the samples.

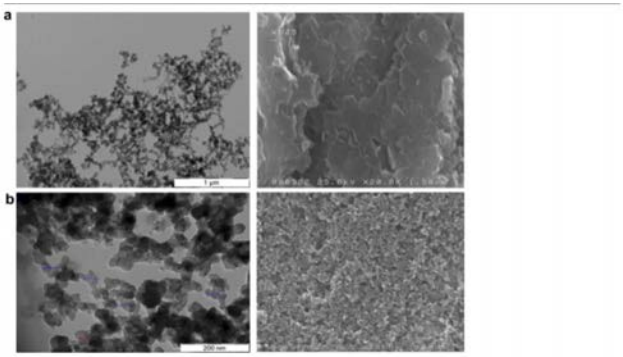

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Fig. 1. (A) TEM images of the CB material (a and b). (B) SEM imagines of bare SPE (a) and CB-SPE (b).

4. Justification of experimental time requested

This section has been discussed together with the local contact of the instrument. Considering the number of modified electrodes (5) to be investigated (as well as the 2 references), we propose one day of instrument time. Samples will be directly mounted on aluminium stubs with the help of conductive double-sided tape.

References

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[8] https://phoenixooc.com
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Experiment Proposal

Experiment number GP2024102

Principal investigator (*) Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator

Experiment title	Steel alloys behaviours to corrosion testing for metal accessories quality control with SEM ZEISS SIGMA		
MRF Instrument	**SEM ZEISS SIGMA**	**Days requested:**	2
Access Route	Direct Access	**Previous GP Number:**	NO
Science Areas	Materials	**DOI:**	-
Sponsored Grant	None	**Sponsor:**	-
Grant Title	-	**Grant Number:**	-
Start Date	-	**Finish Date:**	-
Similar Submission?	-		
Industrial Links	RGF SRL		

Non-Technical Abstract

In luxury leather goods like handbags, metal components are generally made of brass. However, for certain parts such as thin-section plates, brass may lack the necessary mechanical strength, leading to the use of steel instead. Traditionally, steel alloys are chosen for their low cost and ease of machining. Ensuring these components remain durable and free from corrosion is essential. Therefore, research must focus on understanding oxidation to improve the choice of the base materials in the manufacturing process, guaranteeing the long-term durability and appearance of the final leathergoods where the steel articles are mounted (like bags). Understanding the chemical composition on the surface of the steel components after accelerates corrosion testing will lead to significant improvements in the choice of steel base materials and this will contribute to the development of more robust and durable fashion and luxury items.

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY		
MRF Instrument	**SEM ZEISS SIGMA**	**Days Requested:**	2
Special requirements:			

SAMPLE

Material	Steel	-	-
Formula	Fe	-	-
Forms	Solid		
Volume	4 cc		
Weight	10 g		
Container or substrate	No special need	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	RT - K	-	-
Pressure Range	Atmospheric pressure - mbar	-	-
Magnetic field range	No - T	-	-
Standard equipment	-	-	-
Special equipment	No special equipment needed	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	No	-	-
Special equip. reqs	No	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	No	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Steel alloys behaviours to corrosion testing for metal accessories quality control with SEM ZEISS SIGMA

1. Background and context

In luxury leather goods like handbags, metal components are generally made of brass. However, for certain parts, such as thin-section plates, brass may lack the necessary mechanical strength, leading to the use of steel instead. Traditionally, spring steel alloys are often chosen for their low cost and ease of machining, rather than the more expensive stainless (inox) steels.
To ensure the long-term durability of both the steel accessory and the entire leather item, it's important to assess the longevity of the steel components. Consequently, tests exposing these accessories to corrosive environments have been developed to verify their durability over time. These tests assist in making informed decisions regarding steel alloys, considering factors such as resistance, cost, and workability.
The primary difference between commonly used steel alloys, such as spring steel and stainless steel, lies in their mechanical properties and corrosion resistance. Spring steel is more affordable and offers high tensile strength, making it suitable for components subjected to mechanical stress. However, it has low corrosion resistance. In contrast, stainless steel provides excellent corrosion resistance and durability, but at a higher cost and with more challenging workability.
To determine the optimal choice in terms of cost and performance, a comparative evaluation of corrosion resistance is necessary to ensure the longevity of accessories mounted on leather goods. Understanding the long-term behaviour of more economical spring steels compared to stainless steel is crucial for making informed decisions in product design and material selection.

2. Proposed experiment

This proposal is part of a broader study to investigate the modification of the surface composition of steel articles after the exposure of the samples to corrosive atmospheres tests. The study involves the following instrumental techniques: a) Field Emission Scanning electron microscopy (FESEM) and b) X-ray Photoelectron Spectroscopy (XPS). Electron Microscopy analysis with the instrument ZEISS SIGMA would give a great contribution to this study since it is an advanced tool for surface analysis: in fact, FESEM is instrumental in this experiment for its: a) high-resolution imaging, crucial for identifying defects coming from corrosion and understanding their morphology; b) elemental analysis, enabled by energy-dispersive X-ray spectroscopy (EDS), to determine elemental composition, crucial for analysing surface composition and modifications after corrosion; c) non-destructive nature, preserving sample integrity for analysis with multiple techniques; d) high brilliance of the images thanks to the Field Emission Source.
By conducting these analyses, we will obtain essential data for understanding the corrosive effects that led to alterations on the surface of the steel articles, useful for research and development teams to improve alloy type choice during design and costing of the products, ultimately leading to improved quality and durability of the final products (typically, luxury leather bags).

3. Summary of previous experimental proposals or characterisation

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Historically, the durability of accessories in the fashion and luxury markets has been constrained by a reliance on empirical methods and trial and error. This approach lacks the detailed insight and predictive capability that sophisticated analytical tools like SEM analysis for the investigation of corrosive phenomena can provide. Access to such advanced analysis and the requisite expertise is often limited. Although some literature provides a foundational understanding of corrosion in steel articles across various fields and applications, [1-2], a detailed exploration of altered surfaces in the context of leather goods has not been extensively conducted. This research aims to fill that critical knowledge gap by correlating surface-specific elements identified through investigation. The goal is to offer recommendations for steel alloy selection and potential surface treatments that could mitigate corrosive effects.

4. Justification of experimental time requested

As detailed in section 2, Scanning Electron Spectroscopy (SEM) is a pivotal tool for this experiment due to its unique capabilities.
To achieve a comprehensive surface analysis of the two sets of steel samples, we request machine time for the ZEISS SIGMA at CSGI Florence Unit to perform the analysis. This will enable us to obtain detailed elemental and surface imaging information, which can be correlated with the corrosion mechanism. We request 2 days of experimental time to analyse 6 samples coming from 2 different alloys batches. Both the morphology We will study the difference between two different steel alloys and the samples realised with such alloys, after been subjected to accelerated corrosion trough exposure of corrosive atmosphere, will then be analysed with SEM in order to highlight the surface morphology and composition on the surface and compare the results between the two steel alloys used. For each batch we will collect three samples (for a total of 6 samples to be analysed) for investigating the homogeneity of the effects of the exposure to corrosive atmospheres within the same batch.
The first day will be dedicated to the imaging and EDS mapping of all the 6 samples. The second day will focus on high resolution imaging and mapping on regions of interest selected from a preliminary review of the collected data to identify key patterns, anomalies, and areas requiring deeper investigation. This step is crucial for effective time management and prioritising detailed analyses.
This schedule ensures efficient use of the ZEISS SIGMA, providing detailed insights into the surface composition and morphology of the corrosion. By understanding these phenomena, we aim to improve the choice of base materials in the production processes and finishing treatments, in order to enhance the longevity and performance of steel components in the fashion and luxury industries.

[1] Shi, Wn., Yang, Sf. & Li, Js. Effect of nonmetallic inclusions on localized corrosion of spring steel. *Int J Miner Metall Mater* **28**, 390–397 (2021).

[2] Shiqiang Chen, Y. Frank Cheng, Gerrit Voordouw, A comparative study of corrosion of 316L stainless steel in biotic and abiotic sulfide environments, International biodeterioration & Biodegradation, Volume 120, 2017, Pages 91-96

Experiment Proposal

Experiment number GP2024103

Principal investigator (*) Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator

Experiment title	Stripping of surface treatment from ABS articles investigation with SEM ZEISS SIGMA		
MRF Instrument	**SEM ZEISS SIGMA**	**Days requested:**	2
Access Route	Direct Access	**Previous GP Number:**	NO
Science Areas	Materials	**DOI:**	-
Sponsored Grant	None	**Sponsor:**	-
Grant Title	-	**Grant Number:**	-
Start Date	-	**Finish Date:**	-
Similar Submission?	-		
Industrial Links	RGF SRL		

Non-Technical Abstract

In the fashion and luxury markets, ABS (Acrylonitrile Butadiene Styrene) components are chosen for their lightweight and cost-effective properties. These components undergo a galvanization process to achieve a metal-like appearance, such as that of brass. This provides the aesthetic qualities of metal accessories at a reduced cost and weight, which is particularly important for large decorative elements on shoes or bags. If made from brass, these elements could be too heavy and potentially damage the final product. With increasing emphasis on sustainability, the industry is focused on recycling defective or scrap products to support circular production. The challenge is to effectively separate the metal coatings from the ABS and recover both materials, ensuring minimal impact on the environment while maintaining product quality. By conducting this study, we will obtain essential data for understanding the surface modifications occurring on the ABS after removal of the various layers.

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Science and Technology Facilities Council
ISIS Neutron and Muon Source

Sample record sheet

Principal contact	Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY		
MRF Instrument	**SEM ZEISS SIGMA**	**Days Requested:**	2
Special requirements:			

SAMPLE

Material	ABS	-	-
Formula	(C8H8·C4H6·C3H3N)n	-	-
Forms	Solid		
Volume	4 cc		
Weight	10 g		
Container or substrate	No special need	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	RT - K	-	-
Pressure Range	Atmospheric pressure - mbar	-	-
Magnetic field range	No - T	-	-
Standard equipment	-	-	-
Special equipment	No special equipment needed	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	No	-	-
Special equip. reqs	No	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	No	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Stripping of surface treatment from ABS articles investigation with SEM ZEISS SIGMA

1. Background and context

In the fashion and luxury markets, ABS (Acrylonitrile Butadiene Styrene) components are sometimes used due to their lightweight properties. These components undergo a specific electroplating process that allows for plastic galvanization. The resulting accessories are aesthetically identical to their counterparts made from base metals, such as brass, but offer the advantages of being much more cost-effective and lighter. This is particularly important for large decorative elements on shoes or bags, which would be too heavy if made from brass and could potentially damage the final product.
Given the increasing focus on sustainability, manufacturing industries have been striving to recover and recycle as many defective or scrap products as possible to promote production circularity. For accessories made from materials as diverse as ABS and electroplated metal layers, developing an effective method to separate the metals from the ABS substrate and recover both materials presents a challenge [1, 2].
The goal of this analysis is to verify the effectiveness of removing various electroplated layers from ABS and to characterise the resulting "bare" ABS to understand how the stripping process has affected it.

2. Proposed experiment

Until now, the production of accessories for leather goods has primarily focused on items made from base metals like brass. For the recovery of various metal components, conventional methods of metal stripping and refining have been employed. Recently, however, there has been an increase in the use of base materials such as ABS (Acrylonitrile Butadiene Styrene) in accessory manufacturing. This shift offers significant advantages in terms of cost reduction and lighter weight for accessories. However, it also introduces challenges related to recycling: accessories made with ABS require a specialised process to remove the metal layers from the surface galvanization without damaging the underlying ABS, ensuring it remains suitable for recycling.
For this study, we will use a combination of two techniques: 1) Field Emission Scanning Electron Microscopy (FESEM) and 2) Fourier Transform Infrared Spectroscopy (FT-IR). SEM will allow us to study the metallic composition of the galvanic coatings and analyse the surface composition after each stripping step, examining the elemental components left on the surface by the process as well as the surface morphology. FT-IR will be used to analyse the ABS and identify any alterations in the base material after the stripping process.
The ZEISS SIGMA Field Emission Scanning Electron Microscope will greatly contribute to this study, as it is an advanced tool for surface analysis. FESEM is particularly instrumental in this experiment due to its specific features: a) elemental analysis, enabled by energy-dispersive X-ray spectroscopy (EDS), to determine the elemental composition, crucial for analysing surface composition and residual metals after stripping steps; b) high-resolution imaging, crucial for identifying defects from the stripping process and any damage to the surface; c) non-destructive nature, preserving sample integrity for analysis with multiple techniques; d) no need to apply a metal coating to make the sample conductive.

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By conducting these analyses, we will obtain essential data for understanding the effectiveness of removing various electroplated layers from ABS, ultimately improving the stripping process and ensuring the recyclability of all components.

3. Summary of previous experimental proposals or characterisation

The use of ABS as base material for fashion accessories introduces new challenges, particularly concerning recycling. Accessories made from ABS must undergo a specialised treatment process to effectively remove the metal layers from the surface galvanization without damaging the ABS substrate. This is crucial to ensure that the ABS remains suitable for recycling. Currently, there is a lack of comprehensive data and studies that characterise these materials and their recycling processes. As such, this study aims to fill this gap by thoroughly analysing the stripping process and evaluating the suitability of the recovered ABS for recycling. By doing so, it seeks to provide valuable insights into the recycling of ABS-based accessories and contribute to more sustainable manufacturing practices.

4. Justification of experimental time requested

As detailed in section 2, Scanning Electron Spectroscopy (SEM) is a pivotal tool for this experiment due to its unique capabilities.
We request 2 days of experimental time to analyse 5 samples. We will compare the plated ABS samples before the stripping procedure and after the stripping steps, in order to analyse the progression of the removal procedure.
As agreed with the instrument scientist, the first day will be dedicated to the imaging and EDS mapping of all the samples. The second day will focus on high resolution imaging and mapping on regions of interest selected from a preliminary review of the collected data to identify key patterns, anomalies, and areas requiring deeper investigation. This step is crucial for effective time management and prioritising detailed analyses.
This schedule ensures efficient use of the ZEISS SIGMA, providing detailed insights into the surface composition and morphology of the treated samples. By understanding these phenomena, we aim to improve the stripping process of the galvanic layers from ABS base materials, allowing the ABS to be reused and recyclable, in order to enhance the performance in terms of circularity of this kind of accessories in the fashion and luxury industries.

[1] Jae Sik Seo, Ho Tak Jeon, Tae Hee Han, Peeling mechanism of interlocked interface between etched acrylonitrile-butadiene-styrene and electroplated metal layer, Surfaces and Interfaces, Volume 26, 2021, 101337.
[2] Ran Tao, Lujain Fatta, Ruslan Melentiev, Amit K. Tevtia, Gilles Lubineau, Contributions of chemical interactions and mechanical interlocking for the adhesion of electroplated copper to ABS in the Cr(VI) etching process, International Journal of Adhesion and Adhesives, Volume 126, 2023, 103450.

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Experiment Proposal

Experiment number GP2024104

Principal investigator (*) Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY
Co-investigator
Co-investigator
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Co-investigator
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Co-investigator
Co-investigator
Co-investigator

Experiment title	Wire bending and adhesion investigation with SEM ZEISS (Scanning Electron Microscopy)		
MRF Instrument	**SEM ZEISS SIGMA**	**Days requested:**	2
Access Route	Direct Access	**Previous GP Number:**	NO
Science Areas	Materials	**DOI:**	-
Sponsored Grant	None	**Sponsor:**	-
Grant Title	-	**Grant Number:**	-
Start Date	-	**Finish Date:**	-
Similar Submission?	-		
Industrial Links	RGF SRL		

Non-Technical Abstract

In the fashion industry, rings of various sizes and finishes are widely used for structural and decorative purposes, such as connecting multiple decorative elements, like charms on leather bags. Given the size requirements for these charms, the rings are produced by bending brass wires to the desired dimensions. These rings are then subjected to surface finishing treatments that are both protective (to prevent brass corrosion) and decorative (to meet the aesthetic demands of design offices). To achieve diverse aesthetic needs, painting is often employed, allowing for a wide range of colours. However, painting can mask structural defects like cracks caused by bending. Such cracks pose a risk as they can lead to the fracturing of the outer paint layer over time, causing product oxidation and impacting the final product (the bag). This study aims to analyse internal cracks and potential fissures/voids/bubbles at the brass-paint interface to optimize production processes and improve paint

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY
MRF Instrument **SEM ZEISS SIGMA** **Days Requested:** 2
Special requirements:

SAMPLE

Material	Brass	-	-
Formula	Cu, Zn	-	-
Forms	Solid		
Volume	4 cc		
Weight	10 g		
Container or substrate	No special need	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	RT - K	-	-
Pressure Range	Atmospheric pressure - mbar	-	-
Magnetic field range	No - T	-	-
Standard equipment	-	-	-
Special equipment	No special equipment needed	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	No	-	-
Special equip. reqs	No	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	No	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Wire bending and adhesion investigation with SEM ZEISS (Scanning Electron Microscopy) (CSGI)

1. Background and context

In the fashion industry, rings of various sizes and finishes are widely used for structural purposes, such as connecting multiple decorative elements, like charms on leather bags, as well as decorative purposes, as aesthetical connection elements on leathergoods. For the production of these rings for charms, the required dimensions are achieved through wire bending of brass wires. These rings are then subjected to surface finishing treatments that serve both protective and decorative purposes. The protective coatings prevent brass corrosion, while the decorative finishes meet the aesthetic demands of design offices. To cater to diverse aesthetic needs, painting is often employed, offering a wide range of colours. However, painting can conceal structural defects such as cracks caused by the bending process. These cracks pose a risk as they can lead to the fracturing of the outer paint layer over time, resulting in oxidation of the product, which impacts the final quality of the item, such as a bag.

2. Proposed experiment

This proposal is part of a dual campaign, integrating findings from Scanning Electron Microscopy (SEM) with XRD Tomography, to understand the microstructural defects in bended brass wire rings and their interaction with varnish coating, applied as surface treatment.
The objective of this study is to conduct a morphological and compositional surface analysis using Scanning Electron Microscopy in order to analyse the surface of the samples looking for surface defects in the material, which are not visible to the naked eye, as well as any potential surface cracking or failure of the paint layer that could lead to corrosion phenomena. This analysis will help optimise production processes and potentially improve the formulations of the paints used.
Additionally, the study aims to compare the formation of cracks and surface modifications between raw brass rings (without surface painting) and painted rings. By conducting a comparative analysis between two sets of samples—rings subjected to repeated bending in both raw brass and painted brass—we aim to better understand the impact of opening and closing during assembly on structural integrity and surface modification.
This proposal aims to provide comprehensive instruction on the utilisation of Scanning Electron Microscopy, a crucial tool for non-destructive testing and detailed surface analysis.
We therefore request experimental time on the SEM ZEISS to conduct non-destructive measurements on brass rings manufactured through wire bending. These rings undergo a surface painting treatment and are then subject to further deformation during the assembly process on finished products, such as attaching charms to bags. During this assembly process, the rings are opened to insert the charms and then closed again. This repeated bending may cause cracks within the brass and issues with the adhesion between the brass base and the paint layer (i.e. starting of micro-flaking on the surface). Currently, these assembled pieces are inspected visually and such microscopic defects are therefore not detected; however, over time, they may exhibit corrosion phenomena due to the aforementioned defects.
Therefore, it is crucial to conduct this surface analysis using SEM to detect any defect on the surface, which is not visible to the naked eye, as well as any potential delamination or flaking of the paint layer, which could result in further development of corrosion phenomena. SEM is fundamental in this experiment for its:
a) high-resolution imaging, crucial for identifying surface defects and understanding their morphology;
b) elemental analysis, to determine elemental composition, crucial for analysing surface treatments and detecting inclusions that could interfere with good adhesion of the subsequent varnish;
c) non-destructive nature, preserving sample integrity for analysis with multiple techniques. We aim to conduct a comparative analysis between two sets of samples: rings subjected to repeated bending in both raw brass and painted brass;
d) the high brilliance of the Field Emission source which makes it possible to image samples, including those coated by varnish, without the need of a metallisation step.
The experiment would be carried out at CSGI, with whom we are already in contact and agreed on the proposed experiment. The Zeiss Sigma scanning electron microscope with a field-emission source at the CSGI Unit is in fact equipped with a Field Emission Source, a GEMINI column and In-Lens detector. This setup allows the acquisition of high-resolution images of both conducting and non-conducting samples, which is crucial to perform the analysis on both conductive samples (brass base material) and non-conductive (varnished items coated with lacquer). The microscope is equipped with X-ray detectors (EDS), backscattered electrons (BSE), and secondary electrons (SE). The X-ray detection system, in addition to conventional X-ray analysis capabilities, produces high-resolution maps of electron emissions, allowing for elemental mapping on the surface. This is of great interest for understanding the surface properties and modifications.

3. Summary of previous experimental proposals

The link between the wire bending of brass, varnish application and cracking formation upon opening and closing of the rings during assembly of the final product has traditionally relied on a trials and errors approach, supplemented by empirical knowledge. This method, while offering some insights, lacks the precision and predictability afforded by scientific analysis. Small and medium-sized enterprises (SMEs) in the fashion sector, in particular, encounter obstacles due to limited access to advanced analytical methods like Scanning Electron Microscopy and the necessary expertise to interpret data effectively.
Scientific literature provides findings that Scanning Electron Microscopy is an essential tool for the investigation of defects and failures in brass [1-2] but a detailed and comprehensive study on brass articles manufactured with different technologies for fashion and luxury accessories has not been performed so far. This proposal seeks to fill that knowledge gap through focused research, in order to investigate the effects of widely used production processes such as wire bending that would be essential for production technological improvement.

4. Justification of experimental proposals request

As detailed in section 2, Scanning Electron Microscopy is a crucial tool for this experiment due to its unique capabilities. The first day will be dedicated to the analysis of the brass base material rings, in order to have reference measurements on the surface of the wire upon bending; the second day will focus on the varnished sample and to data analysis and comparison.
In detail, we request 2 days of experimental time to analyse 8 samples: 4 samples with no varnish finishing, in order to have a reference measurement of the surface of the brass by itself, without surface varnish, to compare the surface appearance and modifications before opening and closing and after the opening and closing procedure; the other 4 samples will be produced with the same wire bending procedure and will also have the final surface varnish coating, allowing to perform the same set of measures and to compare the results: on the rings as produced and after the “opening and closing” procedure for the final assembly of the finished product. This number ensures comparison between the behaviour upon mechanical stress on the base material itself and with the varnish coating, helping in detecting cracks formation, delamination and flacking phenomena between brass and varnish. This structured approach allows for thorough examination while maintaining a strict timeline.

[1] Pantazopoulos, G. "A review of defects and failures in brass rods and related components." Practical Failure Analysis 3.4 (2003): 14-22.
[2] Liu, Wei, et al. "Component Analysis of Defects in Secondary Special Brass Alloy." TMS 2020 149th Annual Meeting & Exhibition Supplemental Proceedings. Springer International Publishing, 2020.

Experiment Proposal

Experiment number GP2024169

Principal investigator	Professor Claudio Goletti, University of Rome Tor Vergata, ITALY
Co-investigator (*)	Professor Massimo Bonini, CSGI - University of Florence, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Online training on multiple instrumentation for the Students of the GREENANO Erasmus Mundus Joint Master (EMJM) on Materials Science: event #2 organized by CSGI Unit

Training MRF	**SEM ZEISS SIGMA**	**Days requested:**	1
Access Route	Direct Access	**Previous GP Number:**	NO
Science Areas	Materials	**DOI:**	No
Sponsored Grant	None	**Sponsor:**	-
Grant Title	-	**Grant Number:**	-
Start Date	-	**Finish Date:**	-
Similar Submission?	-		
Industrial Links	-		

Non-Technical Abstract

This proposal is part of a series of three online events designed to provide specialized training. We propose an online training program specifically for students enrolled in the GREENANO Erasmus Mundus Joint Master (EMJM) in Materials Science. The training focuses on state-of-the-art instrumentation essential for research in materials science, helping students develop skills critical for careers in both academia and industry. Over three events, students will be introduced to advanced tools such as imaging, spectroscopy, and material characterization, provided by three ISIS@MACH ITALIA units and ISIS UK. The program includes demonstrations of real-world case studies and offers students a comprehensive overview of preparing and analyzing samples. The goal is to enhance the students' research capabilities before they begin their practical research stages. The proposed training could serve as a foundation for future users of these facilities and inspire additional training events.

Publications -

Instruments	**INES**	**Days Requested:**	1
Access Route	Direct Access	**Previous RB Number:**	No
Science Areas		**DOI:**	No
Sponsored Grant	None	**Sponsor:**	
Grant Title	-	**Grant Number:**	
Start Date	-	**Finish Date:**	
Similar Submission?			
Industrial Links			

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Science and Technology Facilities Council
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Sample record sheet

Principal contact	Professor Massimo Bonini, CSGI - University of Florence, ITALY		
Training Instrument	**SEM ZEISS SIGMA**	**Days Requested:**	1
Special requirements:			

SAMPLE

Material	-	-	-
Formula	-	-	-
Forms			
Volume			
Weight			
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	-	-	-
Pressure Range	-	-	-
Magnetic field range	-	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	-	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	-	-	-
Sensitivity to vapour	-	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	-	-	-

Online training on multiple instrumentation for the Students of the GREENANO Erasmus Mundus Joint Master (EMJM) on Materials Science: event #2 organized by CSGI-UNIFI Unit

1. Background and Context

The field of materials science is rapidly evolving, with advancements impacting various sectors, including technology, healthcare, and environmental science. Understanding and utilizing state-of-the-art instrumentation is crucial for young researchers to push the boundaries of innovation. The proposed training events are timely as they align with the increasing need for specialized knowledge in high-tech instrumentation, which is essential for cutting-edge research and development. The proposed training is essential for different reasons:

- skill development: introducing students to advanced instrumentation early in their careers will enhance their research capabilities and improve the quality of their experimental work.
- industry relevance: as industry increasingly relies on advanced materials, having a workforce skilled in using high-tech instruments is critical.
- research enhancement: knowledge of these instruments can significantly boost the research output and innovation potential of participating students.

This training proposal facilitates mobility and high-quality education for students across Europe, as the participants will be students enrolled in the **GREENANO Erasmus Mundus Joint Master (EMJM) on Materials Science**.

Figure 1. Partners and pillars of the GREENANO Erasmus Mundus Joint Master (EMJM) on Materials Science (as described at this link).

2. Proposed Training

The proposed training consists of three online events where state-of-the-art instrumentation relevant in the field of materials science will be introduced to the participants through selected case studies. The training participants will be students enrolled in a European master's programme, which involves studying at different universities each semester. The primary goal of "GREENANO" is to foster a new generation of engineers, researchers, and sustainability managers, equipping them to confront the major challenges posed by green and digital transitions.

The University of Rome Tor Vergata is one of the Universities involved and one of the proposers (Prof. Claudio Goletti) is the responsible for the programme. During the first semester (when the proposed training will be held) the students will be hosted by the Université de Lorraine. Helding the event online allows for maximizing the number of students allowed to participate, as well as their training before they are required to perform their own research: in fact, these students are expected to perform a research stage within the Masters. In this framework, the instruments available at ISIS@MACH ITALIA units offer a unique collection of tools for Materials Science, including advanced imaging, spectroscopy, and material characterization tools. The training will be conducted by academic staff and researchers from the following ISIS@MACH ITALIA units:

- University of Rome Tor Vergata Unit (Event #1);
- CSGI - University of Florence Unit (Event #2);
- University of Milano Bicocca Unit (Event #3).

Personnel from the INES beamline at the ISIS UK will also be involved, to present the instrumentation and opportunities available at the facility.

3. Summary of previous training proposals

This is the first proposal for such training events. Therefore, there are no previous training outcomes to summarize. However, we anticipate that this initial training will lay a strong foundation for future, more advanced training sessions.

4. Justification of Training Proposal Request

We ask for three days in total, 1 per each unit involved. The training will be conducted over three separate online events, each lasting approximately 8 hours. During the training, local scientists will demonstrate the use of the instruments online. The time will be allocated as follows:

- Introduction to the portfolio of instruments available at the CSGI-UNIFI unit (2 hours), with a special focus on the SEM ZEISS Sigma instrument.
- Case study #1: description of a scientific case study carried out at the CSGI-UNIFI unit, with detailed description on how to set up and prepare samples and instruments for the experiment, as well as the analysis of the results (1 hour).
- Case study #2: description of a scientific case study carried out at the CSGI-UNIFI unit, with detailed description on how to set up and prepare samples and instruments for the experiment, as well as the analysis of the results (1 hour).
- Description of the INES beamline at ISIS Facility: each event will present different science cases from t from the portfolio of analysis of materials for application in engineering, cultural heritage, health and earth sciences (2 hours).
- Questions and Answers session (2 hours).

This structure ensures comprehensive coverage of all essential aspects of planning measurements and using the instruments. The timing is designed to maximize learning while being mindful of the online format's limitations.

SEM&C-AFM & correlative AFM

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Experiment Proposal

Experiment number GP2024043

Principal investigator	Professor Alessandro Cianchi, University of Rome Tor Vergata, ITALY
Co-investigator (*)	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Mario Galletti, Universitá di Roma Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Study of TArget Resistance to damaGE and contamination (TARGET) by means SEM and profilometry measurements

MRF Instrument	**SEM&C-AFM with Optical Profiler**	**Days requested:**	3
Access Route	Direct Access	**Previous GP Number:**	-
Science Areas	Materials, Physics, Technique Development	**DOI:**	-
Sponsored Grant	None	**Sponsor:**	-
Grant Title	-	**Grant Number:**	-
Start Date	-	**Finish Date:**	-
Similar Submission?	-		
Industrial Links	-		

Non-Technical Abstract

Our aim is to study the damages produced by high-intensity laser, photon and particle beams on targets currently used as diagnostics at particle accelerators. The characterization of both a particle beams damaged sample and a pristine one used as a reference will be done at the surface level by means of scanning electron microscopy and optical profilometry which it will allow verify the consistency of the results. We also plan to assess eventual implantation of different ion species that develops after radiation bombardment of the target diagnostic by means of energy dispersive spectroscopy (EDS) microanalysis and X-ray Fluorescence spectroscopy, whereas bulk damage of the sample will be assessed by X-ray computed tomography (XCT) which will allow us reconstructing in 3D the entire sample with micrometric spatial resolution in one shot. Hence, we aim here to request access to the SEM instrument (SEM&C-AFM with Optical Profiler) operating at the Roma Tor Vergata Unit of IM@IT.

Publications

Gschwendtner, Edda, and Patric Muggli. "Plasma wakefield accelerators." Nature Reviews Physics 1.4 (2019): 246-248.

Rule, D. W. "Transition radiation diagnostics for intense charged particle beams."NIM in Phys. Research B: Beam Interactions with Materials and Atoms 24 (1987): 901-904.

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E-platform: No

Instruments	
Access Route	
Science Areas	
Sponsored Grant	
Grant Title	
Start Date	
Similar Submission?	
Industrial Links	

Days Requested:	
Previous RB Number:	
DOI:	
Sponsor:	
Grant Number:	
Finish Date:	

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Sample record sheet

Principal contact	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY		
MRF Instrument	**SEM&C-AFM with Optical Profiler**	**Days Requested:**	3
Special requirements:			

SAMPLE

Material	Silicon crystal Aluminium film	-	-
Formula	-	-	-
Forms	Solid		
Volume	0,03 cc		
Weight	1000 mg		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	No	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	-	-

Study of TArget Resistance to damaGE and contamination (TARGET) by means SEM and profilometry measurements

Background and Context

Material survival in a hostile environment is a fundamental requirement for beam instrumentation in a particle accelerator. Intercepting diagnostics is the most critical part, as particle beams continuously hit it. A new generation of particle accelerators is developing, based on the interaction of strong laser (>10^{18} W/cm^2) with plasma or particle beam, to boost the particle energy or the photon energy via inverse Compton scattering [1].

In a photon-electron collider, such as one used in a gamma-ray source, electron and high-intensity photon beams can hit the diagnostic. View screens, e.g., small targets that emit light via optical transition radiation [2], when struck by particles or reflect photons, are widely used to image the photon and electron beam. This proposal investigates the damages produced by high-intensity laser and photon beams on such diagnostics and identifies the critical aspect of the current design.

These screens are usually realized with a bulk substrate of crystalline Silicon, about 300 μm thick, with an aluminium deposition of several hundreds of micrometers to enhance the reflectivity.

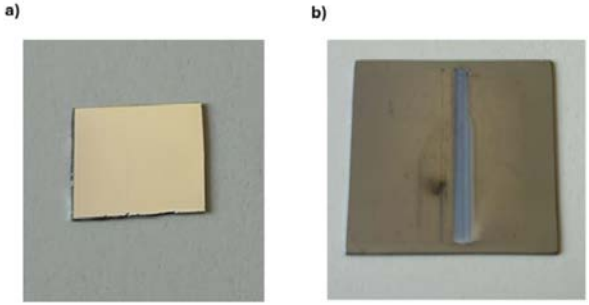

Figure 1: Diagnostic target of 10 mm x 10 mm area, and of 300 µm thickness used at particle accelerator to image the beam profile. a) Pristine target never been exposed to any particle or laser beam radiation damage. b) Target sample with evident damage done after particle radiation.

High-intensity electron beams can deposit relevant amounts of energy, leading to possible surface damage. High-intensity laser beams can accidentally hit the screen during the alignment process, also giving surface or bulk damage. Both electron and laser beams can also hit the vacuum pipe or other device inside the vacuum, producing evaporation of ions that can have enough energy to be implanted in the screen material. To better understand the damage on such diagnostic targets, we propose a multi-instrumental approach study. From one end, we aim to assess the damage occurring at the surface of the target by means of scanning electron microscopy and optical profilometry. Eventual craters, cracks and defects produced by particle beams on the sample will be measured by SEM and optical microscopy to verify the outcomes. This can be done by means of the SEM&C-AFM with Optical Profiler instrument available at the Roma Tor Vergata Unit of ISIS@MACH ITALIA. We also plan to assess eventual implantation of different ion species that develops after radiation

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bombardment of the target diagnostic by means of energy dispersive spectroscopy (EDS) microanalysis that is an ancillary equipment available on the same instrument. For the characterization of the bulk damage of the sample we plan to use X-ray computed tomography (XCT) which will allow us reconstructing in 3D the entire sample with micrometric spatial resolution in one shot. Defects detected on the sample by XCT data can be compared with results obtained by SEM and optical microscopy data. To do that, we aim to perform XCT scans of the two samples at the RETINA instrument available at the POLIMI-National Medium Range Facilities Unit. The choice of this instrument potentially opens the possibility to do X-ray Fluorescence (XRF) spectroscopy measurements, which we can use here as a second investigation tool for studying the ion implantation on the surface of the sample already assessed by SEM-EDS data. The expected results, in terms of morphological study, surface contamination, and structure damage, can offer important insight into the choice of our diagnostic target material, the production system, and the long-term survival in a harsh environment.

Proposed experiment

In this experiment we aim to perform scanning electron microscopy measurements on the SEM&C-AFM with Optical Profiler (Roma Tor Vergata Unit) to study the surface damage of one irradiated sample (Fig 1b) which will be compared to the reference sample (Fig 1a) that has not be exposed to any particle or laser beam. Furthermore, on the irradiated sample we plan to do energy dispersive spectroscopy (EDS) microanalysis to study the presence of eventual implanted ions on the surface and compared to the outcomes of the not irradiated sample. Such SEM-EDS results will be compared to verify consistency with data obtained by XRF measurements performed on the same set of samples with the RETINA instrument available at the POLIMI-National Medium Range Facilities Unit. Also, the presence of eventual defects detected by SEM measurements can be verified by XCT data still collected at the RETINA instrument.

Justification of experimental time requested

The radiation damaged and reference target diagnostic samples have dimensions of about 10mm x 10mm and thickness of about 300 μm. We aim to acquire SEM data and optical profilometry on these samples using a field of view and magnification which depends on the size of the damage. We predict to cover a large portion of the surface of the sample with n. 15 image per sample. Hence, after discussion with the instrument scientist, we request 3 days of SEM instrument time including set-up and calibration time.

References

[1] Gschwendtner, Edda, and Patric Muggli. "Plasma wakefield accelerators." *Nature Reviews Physics* 1.4 (2019): 246-248.

[2] Rule, D. W. "Transition radiation diagnostics for intense charged particle beams." *Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms* 24 (1987): 901-904.

Experiment Proposal

Experiment number GP2024048

Principal investigator (*) Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator

Experiment title	Investigating the Corrosive Impact of Chrome-Tanned Semi aniline Calf Leather using SEM with correlative EDX		
MRF Instrument	**SEM&C-AFM with Optical Profiler**	**Days requested:**	3
Access Route	Direct Access	**Previous GP Number:**	NO
Science Areas	Materials	**DOI:**	-
Sponsored Grant	None	**Sponsor:**	-
Grant Title	-	**Grant Number:**	-
Start Date	-	**Finish Date:**	-
Similar Submission?	-		
Industrial Links	LBS LUXURY BRANDS SERVICES SRL		
Non-Technical Abstract	The aim of this study is to systematically investigate the composition of semi aniline chrome-tanned calf leather and to identify correlations between adsorbed substances on the leather surface and its aggressiveness towards metal accessories. By understanding these relationships, we can develop better methods for predicting and mitigating corrosion, thereby improving the quality and durability of leather products in the fashion and luxury industries. Understanding the chemical composition of semi aniline chrome-tanned calf leather will lead to significant improvements in product quality ad in particular this research will provide a scientific basis for better quality control practices and contribute to the development of more robust and durable fashion items.		
Publications	-		

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY
MRF Instrument **SEM&C-AFM with Optical Profiler** **Days Requested:** 3
Special requirements:

SAMPLE			
Material	Chrome-Tanned Semi aniline Calf Leather	-	-
Formula	Chrome-Tanned Semi aniline Calf Leather	-	-
Forms	Solid		
Volume	4 cc		
Weight	10 g		
Container or substrate	No special need	-	-
Storage Requirements	-	-	-
SAMPLE ENVIROMENT			
Temperature Range	RT - K	-	-
Pressure Range	Atmospheric pressure - mbar	-	-
Magnetic field range	No - T	-	-
Standard equipment	-	-	-
Special equipment	No special equipment needed	-	-
SAFETY			
Prep lab needed	Yes	-	-
Sample Prep Hazards	No	-	-
Special equip. reqs	No	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	No	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Investigating the Corrosive Impact of Chrome-Tanned Semi aniline Calf Leather using SEM with correlative EDX

1. Background and context

Semi aniline chrome-tanned calf leather is a highly valued material in the fashion and luxury industries, commonly used for products such as handbags, belts, and footwear. Ensuring the quality and longevity of these products is crucial, particularly in preventing corrosive processes that can damage metallic accessories like buckles, zippers, and decorative elements. The leather can release tanning substances that may react with metals, leading to corrosion and tarnishing, which compromises the aesthetic and functional integrity of the final products.

To assess the corrosive potential of leathers, quality control laboratories typically perform simulated corrosion tests using a reference sample. The extent of oxidation on the sample, after exposure to the leather, is evaluated and the leather is classified on a scale of aggressiveness from 1 to 5 (1 = highly aggressive, 5 = non-aggressive). However, beyond this empirical test, there has been no systematic study to investigate the underlying causes of oxidation and the specific substances responsible for the corrosive effects on metal accessories.

Although some literature provides a foundational understanding of surface structure of leather [1], a detailed investigation, especially regarding the correlation of the structure and elemental distribution on the surface with aggressiveness and corrosion properties toward metal accessories, is notably absent. This research aims to fill that critical knowledge gap: this study is in fact expected to reveal specific chemical components in the leather that are responsible for initiating or accelerating corrosion in metal accessories, such as chromium and chlorine. By identifying these elements and their distribution on the surface of leather, the fashion and luxury industries can take proactive measures to treat or modify leather to reduce its corrosive impact, thereby enhancing the durability and quality of leather products.

2. Proposed experiment

This proposal is part of a broader study to investigate the underlying causes of oxidation and the specific substances responsible for the corrosive effects on metal accessories by Semi aniline chrome-tanned calf leather. The study involves several instrumental techniques: a) SEM-EDX; b) RAMAN Spectroscopy and profilometry; c) FT-IR Spectroscopy; d) Tomography; e) XPS and ISS.

SEM (Scanning Electron Microscopy) is a pivotal tool for this experiment due to its unique capabilities.

By employing the SEM Tescan Vega instrument, we can therefore obtain comprehensive data on the chemical and structural properties of the surface of semi aniline chrome-tanned calf leather. The use of SEM with both secondary electron and backscattered electron probes provides significant advantages for studying leather samples: we could obtain high-resolution topographical images and detailed compositional maps, essential for understanding surface composition and elemental distribution. Such comprehensive analysis is invaluable for understanding and mitigating the corrosive effects that these leathers can have on metallic accessories, ultimately leading to improved quality and durability of leather products in the fashion and luxury industries.

In particular, the “SEM&C-AFM with Optical Profiler” is a powerful tool for analysing the surface composition of chrome-tanned semi aniline calf leather because the analysis will provide detailed information on the chemical composition, distribution of elements, and structural characteristics of the leather. Here are the key types of data that can be obtained: 1) identification of specific elements present on the leather surface, such as chromium and chlorine compounds, that are crucial data for understanding the tanning process and

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potential corrosion issues; 2) spatial distribution: mapping the distribution of identified elements across the leather surface to understand their uniformity or concentration in specific areas to see how these elements are distributed within the surface layers of the leather; 3) structural analysis on the morphology of the leather surface, that would come in contact with the metal accessories in the final leather finished good.

3. Summary of previous experimental proposals or characterisation

Historically, the understanding of the aggressiveness of leather toward metal accessories has been constrained by a reliance on empirical methods and trial and error. This approach lacks the detailed insight and predictive capability that sophisticated analytical tools like Scanning Electron Microscopy can provide. In particular, access to such advanced analysis and the requisite expertise is often limited. Although some literature provides a foundational understanding of the surface structure of tanned leather as reported in section 1, a detailed exploration regarding the surface composition and element distribution and their correlation with aggressiveness and corrosion properties toward metal accessories, is notably absent. This research aims to fill that critical knowledge gap in order to provide a correlation between chemical composition and spatial distribution of tanning agents and other compounds and the observed corrosive properties when in contact with metal accessories.

4. Justification of experimental time requested

As detailed in section 2, Scanning Electron Microscopy is a pivotal tool for this experiment due to its unique capabilities. **We request 3 days of experimental time on the SEM&C-AFM with Optical Profiler to analyse 15 samples** coming from 5 leather batches: we collected 5 types of batches of semi aniline chrome-tanned calf leather with high level of aggressiveness (level 1 to level 2, with reference to the empirical scale where (1 = highly aggressive, 5 = non-aggressive) as determined by the currently used corrosion test as detailed in section 1. For each batch we will then collect three samples for investigating the spatial homogeneity of the composition. This number ensures diverse representation from different batches of treatment conditions. The samples will then be analysed by using both secondary electron and backscattered electron detectors, in order to obtain high-resolution topographical images and detailed compositional maps.

This dual approach enables a comprehensive understanding of the surface characteristics and elemental distribution, which are critical in identifying the presence and distribution on the surface of potentially corrosive elements.

References

[1] Yolanda S. Hedberg, Carola Lidén, Inger Odnevall Wallinder, Correlation between bulk- and surface chemistry of Cr-tanned leather and the release of Cr(III) and Cr(VI), Journal of Hazardous Materials, Volume 280, 2014, Pages 654-661.

Experiment Proposal

Experiment number GP2024055

Principal investigator	Dr Fabio Battisti, Cecom Srl, ITALY
Co-investigator (*)	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Silver diffusion in Glydcop grain boundaries for ultra high vacuum applications
MRF Instrument	**SEM&C-AFM with Optical Profiler**
Days requested:	3
Access Route	Direct Access
Previous GP Number:	-
Science Areas	Materials
DOI:	-
Sponsored Grant	None
Sponsor:	-
Grant Title	-
Grant Number:	-
Start Date	-
Finish Date:	-
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

Slits, important components at synchrotron facilities such as the ESRF, are required to stop the undesirable X-ray going to beamlines and need to be manufactured according to the approved technical standards for the construction of Ultra High Vacuum Stainless steel, GLIDCOP and OFHC copper. Glidcops are copper-based alloys mixed primarily with small amounts of aluminium oxide ceramic particles that block dislocation creeps. The brazing of Glidcop is usually carried out with brazing filler metals such as silver. The main problem related to the brazing of Glidcop is the excessive diffusion of the silver-based filler along the grain boundaries of Glidcop. To assess this issue, we propose an experimental characterization using SEM-EDX available at the Tor Vergata unit of IM@IT. In particular, SEM will allow to focus on microscopic regions along the grain boundaries, and the concurrent EDX will allow the determination of the magnitude of silver diffusion over the sample surface.

Publications -

ISIS neutron and muon source	**E-platform:** No
Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY		
MRF Instrument	**SEM&C-AFM with Optical Profiler**	**Days Requested:**	3
Special requirements:			

SAMPLE

Material	Copper + GlydCop	-	-
Formula	Cu + Al + O + Ag	-	-
Forms	Solid		
Volume	10 cc		
Weight	80 g		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	300 - 300 K	-	-
Pressure Range	0 - 1000 mbar	-	-
Magnetic field range	0 - 0 T	-	-
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

1. Background and Context

The storage ring of the European Synchrotron Research Facility (ESRF) – in a sense, the source of X-rays at the facility – is 844 meters in circumference, consist in 64 dipoles magnets, which bend the electron beam on its roughly circular orbit and 64 straight sections with quadrupoles magnets to focus the electron beam down to a very small cross section. Half of these straight sections contain “Insertion Devices” sources of intense radiation generally constructed from arrays of permanent magnets. X-rays are extracted tangentially from the storage ring at the end of dipole magnets, but most of the X-rays amounting to 1MW power are stopped by water cooled collimators. Periodically the storage ring is filled with electrons from an injector consisting of a booster synchrotron (300 m in circumference) and a 200 MeV linear accelerator. The “Slits” are important parts of front ends: they are necessary to stop the undesirable X-ray going to the Beam line. These pieces must be manufactured according to the approved technical standards for the construction of Ultra High Vacuum Stainless steel, GLIDCOP and OFHC copper already installed at ESRF.

Glidcop is a family of copper-based metal matrix composite (MMC) alloys mixed primarily with small amounts of aluminium oxide ceramic particles. The aluminium oxide particles block dislocation creep, which retards recrystallization and prevents grain growth; thus preserving the metal's strength at high temperatures. They also protect the metal against radiation damage. On the other hand, they exclude the possibly of heat treatment or hot working of the worked parts [1-3].

CECOM s.r.l., a precision machine shop experienced in the field of ultra-high vacuum, was involved in the fabrication of a slit whose components consist of stainless steel and copper Glidcop components. Slits require a vacuum seal to ensure the uninterrupted passage of particles within them. To do this, CECOM has decided to make vacuum furnace brazes to make the Glidcop assembly. The brazing of Glidcop is usually carried out with the brazing filler metals that are commonly used to join plain copper (i.e., gold- and silver-based braze alloys). The brazing of copper generally leads to a grain coarsening, due to the grain growth that occurs during the brazing process. Glidcop does not suffer from this problem, because of its fine structure and reduced recrystallization at the brazing temperatures. **The main problem related to the brazing of Glidcop is the excessive diffusion of the silver-based filler along the grain boundaries of Glidcop.** The electroplating of Glidcop with copper or nickel prior to brazing is used as a common method to prevent and solve this issue. Copper plating is usually carried out in a copper cyanide solution; the cyanide-copper bath has the aim of facilitating the quality of the braze joints by preventing diffusion of the braze alloy into the Glidcop base material. On the other hand, the Cu-plating process introduces additional steps in the manufacturing process, since additional control stages are needed to ensure blister-free plating. Nickel plating is obtained through more complicated processes, which are based on a Watts bath or electroplating.

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2. Proposed experiment

To assess the magnitude of the silver-based filler diffusion along the grain boundaries of Glidcop, CECOM has decided to make specimens with geometries similar to that of finished components, experimenting with brazing Glidcop components in the following versions:

- Glidcop-Glidcop
- Glidcop nickel plated-Glidcop nickel plated
- Glidcop copper-plated-Glidcop copper-plated
- Glidcop coppered-Stainless Steel

For each of these samples/combinations, we propose to run Scanning Electron Microscopy complemented with Energy-Dispersive X-ray Spectroscopy (SEM-EDS) using the medium range facility of the IM@IT Unit of the university of Rome Tor Vergata. The elemental maps obtained with EDS, superimposed to the images of the sample morphology obtained with SEM, are expected to provide a clear evidence silver diffusion along the boundaries, as well as the magnitude of this phenomenon.

3. Justification of experimental time requested

To perform SEM-EDS experiments on 4 different samples (Glidcop-Glidcop; Glidcop nickel plated-Glidcop nickel plated; Glidcop copper-plated-Glidcop copper-plated; and Glidcop coppered-Stainless Steel) we request 3 days of instrument time on the SEM&C-AFM with Optical Profiler to be used as follows: 2 hours of acquisition for three regions along the boundary of each of the four samples (2 hours x 3 measurements x 4 samples = 24 hours = 3 days).

References

[1] Edwards DJ, Anderson KR, Garner FA, Hamilton ML, Stubbins JF, Kumar AS. Irradiation performance of oxide dispersion strengthened copper alloys to 150 dpa at 415 C. Journal of nuclear materials. 1992 Sep 1;191:416-20.

[2] Singh BN, Eldrup M, Toft P, Edwards DJ. Effects of heat treatments and neutron irradiation on the physical and mechanical properties of copper alloys at 100 deg. C.

[3] Simos N. BNL Irradiation Damage Studies of the Metal Matrix Composite Mo-GR Considered for High Luminosity LHC Collimator Upgrade. Brookhaven National Lab.(BNL), Upton, NY (United States); 2016 Feb 1.

Experiment Proposal

Experiment number GP2024058

Principal investigator	Miss Virginia Pietrosanti, University of Rome Tor Vergata, ITALY
Co-investigator (*)	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Roberta Mancini, Thales Alenia Space, ITALY
Co-investigator	Mr Stefano Francola, Thales Alenia Space, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	How optical and electron microscopies can help investigating radiation-damaged electronic devices
Training MRF	**SEM&C-AFM with Optical Profiler** **Days requested:** 2
Access Route	Direct Access **Previous GP Number:** -
Science Areas	Engineering, ICT **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	Thales Alenia Space Italia
Non-Technical Abstract	Radiation characterization of commercial equipment and apparatus is a relevant topic in the research field known as “radiation hardness assurance”. This topic is particularly relevant for aerospace applications, where the radiation is composed of a very wide spectrum of ionizing and non-ionizing particles. In this framework, advanced optical and electronic microscopies, such as optical profilometry and scanning electron microscopy, can provide the right combination of spatial scales, resolution and non-invasive nature to assess the presence and morphology of a hardware rupture, thus possibly enabling its explanation in terms of radiation-device interaction, and therefore possibly leading to an improvement of the device itself. Here, we propose a training assess to the Scanning Electron Microscopy (SEM) and Optical Profilometry to clarify the capabilities of these techniques and instrumentation to tackle the problem of single event effects in COTS and other electronic devices.
Publications	-

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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ISIS Neutron and Muon Source

Sample record sheet

Principal contact Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Training Instrument **SEM&C-AFM with Optical Profiler** **Days Requested:** 2
Special requirements:

		SAMPLE	
Material	-	-	-
Formula	-	-	-
Forms			
Volume			
Weight			
Container or substrate	-	-	-
Storage Requirements	-	-	-
		SAMPLE ENVIROMENT	
Temperature Range	-	-	-
Pressure Range	-	-	-
Magnetic field range	-	-	-
Standard equipment	-	-	-
Special equipment	-	-	-
		SAFETY	
Prep lab needed	-	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	-	-	-
Sensitivity to vapour	-	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	-	-	-

How optical and electron microscopies can help investigating radiation-damaged electronic devices

1. Background and Context

Radiation characterization of commercial equipment and apparatus is a relevant topic in the research field known as “radiation hardness assurance”. This topic is particularly relevant for aerospace applications, where the radiation is composed of a very wide spectrum of ionizing and non-ionizing particles including photons, electrons, protons, heavy ions, as well as neutrons as abundant secondary radiation. The main goal is to assess the reliability of commercial off-the-shelf (COTS) equipment in aerospace applications, where an electronic device is subject to complex radiation fields. The complexity of the results of this kind of characterization requires a dedicated and robust research approach, so as to assure the possible applicability in industrialization of such devices, which is very attractive for the Market.

In most of the cases, the sensitivity of a given device cannot be derived from the sensitivity of its elementary components, i.e., in terms of simpler electronic parts, but needs to be evaluated as a whole object. This is generally done by monitoring current and voltage input and output of a device while irradiated with a given radiation field (protons, photons, electrons, neutrons). While irradiated, the device can stop working because of an error at the software level (soft error), which can be corrected by rebooting or power-cycling the device. However, in some cases the damage is permanent in nature, as it affects the hardware itself, e.g., because of burnout effects due to single interaction events (Single Event Burnouts, SEBs) [1,2]. In such cases, the reason for a device stopping working can be more easily traced back to one of its electronic components, therefore providing the opportunity to make the device, as a whole, more resistant to cosmic radiation. However, such ruptures can happen at the nano-to-microscopic level, at several depths with respect to the surface of the component and are generally hard to detect with standard laboratory microscopies.

In this framework, advanced optical and electronic microscopies, such as optical profilometry and scanning electron microscopy, can provide the right combination of spatial scales, resolution and non-invasive nature to assess the presence and morphology of a hardware rupture, thus possibly enabling its explanation in terms of radiation-device interaction, and therefore possibly leading to an improvement of the device itself.

2. Proposed Training

Here, we propose a training assess to the Scanning Electron Microscopy (SEM) and Optical Profilometry available at the IM@IT Unit at the University of Tor Vergata. The aim of the training is to clarify the capabilities of these techniques and instrumentation to tackle the problem of single event effects in COTS and, more in general, electronic devices for space applications. The training is thought for 2 people from Thales Alenia Space, including a PhD student with a thesis about COTS damages related to nuclear irradiation. The training will be carried out by the MRF staff of the instrument, also included amongst the proposers of this training proposal.

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3. Summary of previous training proposals

The trainees have experience with the technological and engineering aspects of COTS irradiations and failures due to single event effects, while this training proposal would provide the possibility to look at the topic from a point of view more related to materials science and condensed-matter physics.

4. Justification of experimental proposals request

We request 2 days of beamtime on the SEM&C-AFM with Optical Profiler, to be used as 1 day of training on the TESCAN VEGA Scanning Electron Microscope, and 1 day of training on the optical profiling microscope Z20 by ZETA.

References

[1] Fred W. Sexton, Destructive Single-Event Effects in Semiconductor Devices and ICs, IEEE TRANSACTIONS ON NUCLEAR SCIENCE, VOL. 50, NO. 3, JUNE 2003 603

[2] M.T.M. Littlefair, et al., Single event burnout sensitivity of SiC and Si, 2022 Semicond. Sci. Technol. 37 065013

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Experiment Proposal

Experiment number GP2024060

Principal investigator	Dr Francesco Basoli, Università Campus Bio-Medico di Roma, ITALY
Co-investigator (*)	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Roberto Senesi, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Continuation of Morphological characterisation and compositional analysis of SiCa based Bioglasses using SEM-EDX
MRF Instrument	**SEM&C-AFM with Optical Profiler**
Days requested:	3
Access Route	Direct Access
Previous GP Number:	2024006
Science Areas	Materials, Medicine
DOI:	-
Sponsored Grant	None
Sponsor:	-
Grant Title	-
Grant Number:	-
Start Date	-
Finish Date:	-
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

The main objective of the study is the production of bioglass nanoparticles with bioactive, osteoconductive and antimicrobial functions to be used as coatings for titanium grafts. These bioactive glasses will have composition within the ternary system silicon, calcium, phosphorus, formulations already proven to promote the formation of hydroxyapatite, and stimulate rapid regeneration and new bone tissue formation. Following a preliminary characterization using SEM-EDX, deficit in the concentration of Ca and P were observed in several samples. In order to identify the correct synthesis routes to maintain a stoichiometric level of the final elements similar, if not identical to those of the starting formulations, or in any case such as to achieve the doping level necessary to activate the sought osteogenetic properties, we propose a continuation characterization on a newly prepared set of samples.

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
MRF Instrument	**SEM&C-AFM with Optical Profiler** **Days Requested:** 3
Special requirements:	

SAMPLE

Material	BioactiveGlasses nanoparticles	-	-
Formula	SiCa / SiCaSr / SiCaZn / SiCaGa	-	-
Forms	Solid		
Volume	1 cc		
Weight	10 mg		
Container or substrate	Sample holder	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	300K - K	-	-
Pressure Range	1 atm / High vacuum - mbar	-	-
Magnetic field range	None - T	-	-
Standard equipment	-	-	-
Special equipment	none	-	-

SAFETY

Prep lab needed	No	-	-
Sample Prep Hazards	No	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	None	-	-
Radioactive Hazards	None	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Removed By User	-	-

1. Background and Context

The research activities that will be carried out are part of a broader project which specifically aims to validate an innovative platform that allows the creation of a patient-specific path, which includes the different phases (assessment, treatment, and rehabilitation) which articulate the execution of a surgical procedure optimized for osteotomy (in the specific case, tibial osteotomy). This platform must prove capable of quantifying functional needs, minimizing the invasiveness of the operation and monitoring the rehabilitation process, thus drastically reducing recovery times and complications associated with the operation itself. As part of this activity, medical titanium grafts with a trabecular structure will be created, which can be completely integrated into the patient's bone structure. This result cannot be obtained with current technological knowledge, as current prostheses allow only superficial osseointegration, without involving the presence of vascularization and subsequent formation of bone tissue within the metal structure. These will be subsequently coated to increase osteointegration by bioactive glass nanoparticles, the latter produced using the sol-gel technique, with which particles of regular and nanometric shape can be created, but with a low percentage of dopant elements, or by grinding from melt, which usually gives rise to irregularly shaped micrometer-sized particles. The main objective of the study is the production of bioglass nanoparticles with bioactive, osteoconductive and antimicrobial functions to be used as coatings for titanium grafts[1]. These bioactive glasses will have composition within the ternary system silicon, calcium, phosphorus, formulations already proven to promote the formation of hydroxyapatite, and stimulate rapid regeneration and new bone tissue formation. New formulations will be proposed with appropriately selected doping elements to confer improved characteristics in terms of osteoconduction and bacteriostatic properties, according to the project requests. Of primary importance will be the fabrication of spherical nanoparticles with high dimensional homogeneity. A methodology for the coating of bioactive grafts will also be developed. The creation of these nanoparticles requires an extensive characterization campaign of the bioglasses created both to control their morphology and to quantitatively define the quantity of dopants included. For this reason, the possibility of using electron microscopy (SEM) systems coupled with Energy Dispersive X-ray spectroscopy (EDX) microanalysis is of fundamental importance. The advantage of the proposed approach is due on the one hand to the use of highly doped glasses with formulations capable of increasing osteogenesis (through the use of dopants such as strontium, potassium, magnesium and manganese), but above all in the obtaining of nanoparticles with a size and shape that allow uniform coverage of the surface of the titanium graft, speeding up the processes of osteointegration and osteogenesis. Therefore, a correct morphological characterization is of fundamental importance in order to identify the correct synthesis routes to have a spherical geometry of the nanoparticles produced. At the same time, the nanoparticles created will have to maintain a stoichiometric level of the final elements similar, if not identical to those of the starting formulations or in any case such as to achieve the doping level necessary to activate the sought osteogenetic properties.

2. Summary of previous characterizations

SEM-EDX measurements (GP2024006) were performed on a series of Si/Ca/X samples (X=Ga ,Sr, Zn, P) with the Tescan Vega SEM-EDX at the Tor Vergata Unit. A relatively low-current beam of 30 pA was used to minimize charging effects with an accelerating voltage of 20 kV. EDX analyses were also performed at 20 keV with beam currents of 3 nA. In general, most of the samples showed stoichiometries in agreement with what expected from the preparation procedure yet generally showing a systematic deficit in the amount of Ca, possibly related to sample washes before measurement. In contrast, two types of P-based samples (SiCaP and SiP) did not show a sufficient amount of phosphorous.

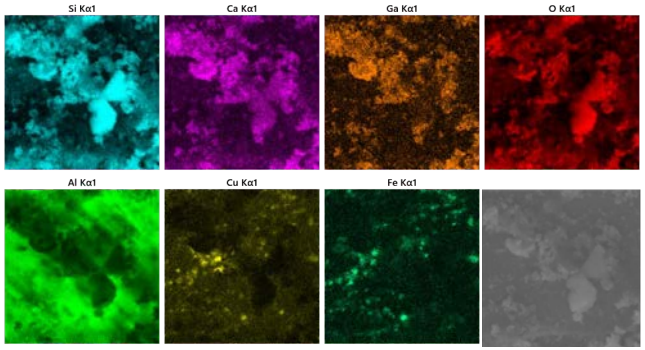

Figure 1. SEM-EDX measurements from access GP2024006, showing the elemental analysis on a bioglass of the type 80% Si : 15% Ca : 5% Ga (elements from sample and stub).

3. Proposed experiment

We propose a continuation proposal (previous access: GP2024006) to use the SEM&C-AFM with Optical Profiler MRF at the Tor Vergata Unit for the characterization of newly prepared nanoparticles[3], particularly those containing potassium, to assess the following information: morphology and shape of the nanoparticles, size of the nanoparticles, elemental composition of the nanoparticles. Additional tests to check the Ca content of SiCa / SiCaSr / SiCaZn / SiCaGa will be performed as well. The comparison of the results from a given set of samples will allow to identify the correct synthesis routes to have a spherical geometry of the nanoparticles produced and the right stoichiometry.

4. Justification of experimental time requested

Considering the need to acquire both SEM images and elemental maps with EDX from a set of 6 different samples, we request 3 days of instrument time on the “SEM with correlative AFM” instrument, to be used as follows: about half a day per sample, to obtain a good statistics and accurate results of the elemental composition, for a total of 6 samples.

4. References

1. S.L. Greasley “Controlling particle size in the Stöber process and incorporation of calcium” J. Colloid Interface Sci. 469 (2016)
2. Rainer et al. “Fabrication of bioactive glass–ceramic foams mimicking human bone portions for regenerative medicine” Acta Biomaterialia 4 (2008) 362–369
3. G.M. Luz, J.F. Mano, Preparation and characterization of bioactive glass nanoparticles prepared by sol–gel for biomedical applications, Nanotechnology. 22 (2011) 494014.

Experiment Proposal

Experiment number GP2024066

Principal investigator (*) Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator

Experiment title	Advanced Analysis of Surface and Composition Variations in Treated Brass Samples with Scanning Electron microscopy (SEM) and Profilometry	
MRF Instrument	**SEM&C-AFM with Optical Profiler**	**Days requested:** 3
Access Route	Direct Access	**Previous GP Number:** NO
Science Areas	Materials	**DOI:** -
Sponsored Grant	None	**Sponsor:** -
Grant Title	-	**Grant Number:** -
Start Date	-	**Finish Date:** -
Similar Submission?	-	
Industrial Links	RGF SRL	

Non-Technical Abstract

Brass-treated accessories are vital for leather goods and footwear, offering both functionality (like zippers and closures) and aesthetic appeal (such as decorative elements on bags and shoes). Ensuring these components remain durable and free from corrosion is essential. Therefore, research must focus on understanding oxidation to improve manufacturing and surface treatments, guaranteeing the long-term durability and appearance of brass accessories. Understanding the chemical composition on the surface of the brass accessories after corrosion will lead to significant improvements in the development of surface protective treatments and will contribute to the development of more robust and durable fashion and luxury items.

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY
MRF Instrument **SEM&C-AFM with Optical Profiler** **Days Requested:** 3
Special requirements:

SAMPLE

Material	Brass	-	-
Formula	Cu, Zn	-	-
Forms	Solid		
Volume	4 cc		
Weight	10 g		
Container or substrate	No special need	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	RT - K	-	-
Pressure Range	Atmospheric pressure - mbar	-	-
Magnetic field range	No - T	-	-
Standard equipment	-	-	-
Special equipment	No special equipment needed	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	No	-	-
Special equip. reqs	No	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	No	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Advanced Analysis of Surface and Composition Variations in Treated Brass Samples with Scanning Electron microscopy (SEM) and Profilometry

1. Background and context

Brass-treated accessories are essential in the leather goods and footwear industries due to their dual functional and aesthetic purposes. They serve practical roles as zippers, closures, and handle elements, while also enhancing the visual appeal of the finished products through decorative pieces on bags, charms, and plaques on shoes. It is crucial that these components remain durable and maintain their appearance without corrosion over time. Thus, research and development must focus on understanding surface composition and modification arising from oxidation, in order to optimize manufacturing processes and surface treatments, therefore ensuring long-term durability and functionality of brass accessories themselves and on the finished products, like belts, bags and shoes.

This proposal therefore aims to investigate the surface composition of samples that show corrosive alterations on the surface using SEM (Scanning Electron Microscopy) with the profilometer as ancillary equipment, providing detailed elemental and surface information that could be correlated with the mechanisms of corrosion that have occurred on the surface. The fashion and luxury industries could than take proactive measures to treat or modify the product process and the surface finishing applied on the articles, thereby enhancing the durability and quality of the final finished products.

2. Proposed experiment

This proposal is part of a broader study to investigate the modification of the surface composition of treated brass articles after the built-up of oxidation phenomena that gave alteration of the aspect of the surface. The study involves several instrumental techniques: a) Scanning electron microscopy (SEM) + profilometry; b) X-ray Photoelectron Spectroscopy (XPS); c) RAMAN Spectroscopy.

Investigation with SEM would give a great contribution to this study since it is an advanced tool for surface analysis, particularly useful for its features: a) high-resolution imaging, crucial for identifying effects of corrosion and understanding their morphology; b) elemental analysis, enabled by energy-dispersive X-ray spectroscopy (EDS), to determine elemental composition in the affected areas; c) non-destructive nature, preserving sample integrity for analysis with multiple techniques.

In particular the analysis will provide valuable insights on the following aspects:

1. Surface Composition: SEM is highly sensitive to the surface composition of materials, providing detailed information about the elements present on the surface. This surface-specific data is critical for identifying products arising from corrosive phenomena.

2. Elemental Quantification: SEM allows for the precise quantification of elemental concentrations on the metallic surface. By understanding the abundance of specific elements, we can assess the effects of the corrosion processes.

Moreover, the Z20 Profilometer by ZETA INSTRUMENTS is an essential ancillary tool for this experiment. In fact, the Z20 Profilometer can provide precise measurements of the surface's topography, including its roughness, texture, and any surface irregularities. These measurements are critical for correlating the physical surface properties with the chemical composition data obtained from the SEM analysis: 1) Surface Roughness: we will obtain data on the roughness and texture of the altered surface; 2) Topographical Mapping: High-resolution maps of the accessories surface, showing the distribution of peaks, valleys, and other features will help understand their correlation with observed corrosion properties.

By conducting these analyses, we will obtain essential data for understanding the corrosive effects that led to alterations on the surface of the brass articles, useful for research and development teams to improve

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production processes and surface finishing ultimately leading to improved quality and durability of the accessories.

3. Summary of previous experimental proposals or characterisation

Historically, the durability of brass accessories for the fashion and luxury markets has been constrained by a reliance on empirical methods and trial and error. This approach lacks the detailed insight and predictive capability that sophisticated analytical tools like SEM investigation and profilometry for the investigation of corrosive phenomena can provide. In particular, access to such advanced analysis and the requisite expertise is often limited. Although some literature provides a foundational understanding of the surface composition occurring after corrosion for brass articles used for different fields and applications, [1], a detailed exploration of the altered surface of the treated brass articles used in the fashion and luxury market for leathergoods and footwear after the occurring of corrosion, has not been conducted extensively so far. This research aims to fill that critical knowledge gap, to provide a correlation between the surface-specific elements found with the investigation, enabling to provide then recommendations for production methods and surface treatments that could mitigate the corrosive effects on the surface of metal accessories for fashion and luxury markets.

4. Justification of experimental time requested

As detailed in section 2, scanning electron microscopy and profilometry are pivotal tools for this experiment due to their unique capabilities.

To achieve a comprehensive analysis of the brass samples, we request machine time for the Scanning Electron Microscope and the Profilometer as ancillary equipment. This will enable us to obtain detailed elemental and surface information, together with profile analysis, which can be correlated with the corrosion mechanism. We request 3 days of experimental time to analyse 15 samples coming from 3 accessories batches: we collected 3 types of batches of brass accessories; for each batch we will then collect five samples for investigating the effects of the corrosion within the same batch.

This schedule ensures efficient use of the SEM and profilometer instruments, providing detailed insights into the surface composition and chemistry of the corrosion. By understanding these phenomena, we aim to improve the production processes and finishing treatments, in order to enhance the longevity and performance of brass accessories in the fashion and luxury industries.

[1] Initial oxidation of brass induced by humidified air. Ping Qiu, Christofer Leygraf. Ping Qiu∗, Christofer Leygraf

Experiment Proposal

Experiment number GP2024087

Principal investigator	Professor Romolo Loreto, University of Naples Orientale, ITALY
Co-investigator (*)	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Archaeometric study of iron ingots from the North Arabian oasis of Dumat al-Jandal using SEM-EDX

MRF Instrument	**SEM&C-AFM with Optical Profiler**	**Days requested:**	2
Access Route	Direct Access	**Previous GP Number:**	-
Science Areas	Cultural Heritage	**DOI:**	-
Sponsored Grant	None	**Sponsor:**	-
Grant Title	-	**Grant Number:**	-
Start Date	-	**Finish Date:**	-
Similar Submission?	-		
Industrial Links	-		

Non-Technical Abstract

Dumat al-Jandal stands as one of the main archaeological sites in the Kingdom of Saudi Arabia, currently on the UNESCO Tentative List. In the Nabataean-Roman era, and until the advent of Islam, the oasis was the hub of a caravan trade involving the Arabian oases, the Nabataean kingdom and Roman Imperial Arabia. Therefore, a complex picture of the development of the phases of ancient trade can be sketched out. The archaeometric study of ingots can thus contribute, for the first time, to shedding light on new dynamics of trade, particularly iron products (if only iron is involved).

We propose a morphological and elemental characterization of a set of iron ingots from the North Arabian oasis of Dumat al-Jandal using the “SEM with correlative AFM” instrument, featuring both scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX). In a separate proposal, we plan to use neutron tomography and activation analysis to obtain bulk information on sample composition.

Publications -

Instruments	**IMAT**	**Days Requested:**	2
Access Route	Direct Access	**Previous RB Number:**	
Science Areas		**DOI:**	
Sponsored Grant	None	**Sponsor:**	
Grant Title	-	**Grant Number:**	
Start Date	-	**Finish Date:**	
Similar Submission?			
Industrial Links			

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Sample record sheet

Principal contact	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY		
MRF Instrument	**SEM&C-AFM with Optical Profiler**	**Days Requested:**	2
Special requirements:			

SAMPLE

Material	iron ingots	-	-
Formula	Iron based	-	-
Forms	Solid		
Volume	20 cc		
Weight	50 g		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	300 - 300 K	-	-
Pressure Range	1000 - 1000 mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

1. Background and Context

Dumat al-Jandal stands as one of the main archaeological sites in the Kingdom of Saudi Arabia, currently on the UNESCO Tentative List and the subject of excavations and restorations by the Italian Archaeological Mission in Saudi Arabia (headed by R. Loreto). The site's archaeological landscape presents an extensive archaeological park composed of the medieval-era settlement that insists on the pre-Islamic era stratifications (Byzantine, Nabateoroman and Neo-Assyrian phases), the funerary area and an imposing fortified caravan station from the Roman era.

Known in 7th century Assyrian sources as Adummatu and in 1st century BC-2nd century AD Nabataean-Roman sources as Dumah/Dumatha, it is one of the main North-Arabian oases that emerged in historical times as caravan centres along the routes between the Levant, Mesopotamia and the South-Arabian kingdoms of pre-Islamic Yemen. Thus, together with the other oases of Tayma and Dadan, Dumat al-Jandal represents one of the main key sites in North-Arabia, an ecological niche in an environment, that of the 1st millennium BC - 1st millennium AD, which was arid.

In the Nabataean-Roman era, and until the advent of Islam, the oasis was the hub of a caravan trade involving the Arabian oases, the Nabataean kingdom and Roman Imperial Arabia. Fifteen years of Italian-Saudi research are contributing not only to the historical rediscovery of an oasis whose occupation is unbroken from the 8th century B.C. to the present day, but also make it possible to better recognise the oasis' commercial role thanks to surveys conducted in the North Arabian region of Jawf, for the recognition of the passage routes of ancient caravans.

At the current state of research, the material culture, especially ceramics and examples of terracotta figurines, show close trade contacts with Assyria (7th-7th c. BC), the Tayma oasis (6th c. BC) and Nabatene or Arabian Province from 105-106 AD. The latter period is characterised by a particular abundance of materials, such as painted ceramics produced in Petra and exported to the Near East, or fictile productions (statuary) and monumental architecture. Thus, an initial and complex picture of the development of the phases of ancient trade can be sketched out. This proposal is well suited to the state of the art as it would allow us to examine the earliest examples of iron ingots traded in North Arabia. Excavations of the Nabataean settlement have revealed, exceptionally, a 'treasure trove' of ingots preserved within a private dwelling located at the foot of the acropolis, thus in a privileged urban sector.

2. Proposed experiment

The archaeometric study of ingots can thus contribute, for the first time, to shedding light on new dynamics of trade, particularly iron products (if only iron is involved). This first research question would be followed by others equally refined: is it iron imported from distant or local areas? Is it transported or locally produced ingots? Are the ingots found in their place of use or are they destined for a later destination? If local, can these ingots prove the existence of a local iron craft? In case of previous datasets from other contexts can we define the chronology and date for this kind of ingots? To answer these questions, we propose a morphological and elemental characterization of a set of iron ingots from the North Arabian oasis of Dumat al-Jandal **combining surface and bulk probes.** The first **surface characterization** using the “SEM&C-AFM with Optical Profiler” instrument, featuring both scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX). The morphological characterization will provide information on the manufacturing practices and original use of the artefacts, while the elemental analysis, providing information on the additional elements and impurities on the ingots supposedly mainly containing iron, could provide information on the origin of the metallic ores or the commercial routes of this ancient civilization. Both pieces of information will be available concurrently using the SEM-EDX capabilities of the MRF instrument selected.

In addition to the SEM-EDX proposal, we plan to submit a related proposal at the ISIS Neutron and Muon Source to obtain a neutron tomography of some of these artefacts using the IMAT beamline, **to obtain bulk information on the samples**. The artefacts, mainly containing iron and relatively thick, are not suitable for most X-ray tomography instrument, while neutrons would provide an optimal and non-invasive way to study their internal composition. **Neutron Activation Analysis on the irradiated samples will be run at ISIS to obtain information on the elemental composition in the bulk.**

3. Justification of experimental time requested

We request 2 days of instrument time of the “SEM&C-AFM with Optical Profiler” instrument to be used as follows: Up to 4 hours per sample of measurements, for a total of up to 6 samples.

Fig. 1. Top: The core of the ancient oasis of Dumah/Dumatha. To the left the acropolis, t the right the medieval village resting on the Nabataean phase. Bottom: Ingots found in a domestic unit dated to between the I cent. BCE – II cent. CE. Average diameter 8cm. Both complete and in fragmentary status

References

R. Loreto. 2017. Alle origini degli Arabi. Un viaggio nell’archeologia dell’Arabia Saudita. Mondadori Education.

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Experiment Proposal

Experiment number GP2024090

Principal investigator	Dr Leonardo Baldassarre, L.B. Servizi per le Aziende S.r.l., ITALY
Co-investigator (*)	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Co-investigator	Ms Margherita Simoni, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Roberto Senesi, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	SEM-EDX characterization of polymeric dosimeters for fast neutrons
MRF Instrument	**SEM&C-AFM with Optical Profiler** **Days requested:** 4
Access Route	Direct Access **Previous GP Number:** -
Science Areas	Materials, Medicine **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	LB Servizi

Non-Technical Abstract

CR39 is a commonly used dosimeter composed of a polycarbonate polymer that can break when a high-energy particle interacts with the nuclei in the polymeric chain. In particular, in the case of fast neutrons, hydrogen atoms from the dosimeter polymer or from the surrounding can be ejected and break the polymer chain leaving defects as small tracks that can be highlighted and enlarged by chemical etching with sodium hydroxide chemical baths. After the etching process, optical microscopes and transmission scanners are used to detect the number of tracks left, their size and shape. As neutrons are highly penetrating particles, one needs to design strategies to make the interaction of the neutron with the dosimeter polymer as probable as possible. We propose to perform SEM-EDX measurements on exposed CR39s to characterize the distribution in shape and size of the defects created after the etching procedure, to test new geometries increasing CR3 efficiency to neutrons.

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
MRF Instrument	**SEM&C-AFM with Optical Profiler** **Days Requested:** 4
Special requirements:	

SAMPLE

Material	CR39	-	-
Formula	polycarbonate	-	-
Forms	Solid		
Volume	0.1 cc		
Weight	1 g		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	300 - 300 K	-	-
Pressure Range	1000 - 1000 mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

1. Background and Context

CR39 is a commonly used solid-state nuclear-track detector for dosimetry [1,2]. It is composed of poly(diethyleneglycol bis[allylcarbonate]), a polycarbonate polymer that can break when a high-energy particle interacts with the nuclei in the polymeric chain. In particular, in the case of fast neutrons, hydrogen atoms from the dosimeter polymer or from the surrounding can be ejected and break the polymer chain leaving defects as small tracks that can be highlighted and enlarged by chemical etching with sodium hydroxide chemical baths.

As neutrons are highly penetrating particles, one needs to design strategies to make the interaction of the neutron with the dosimeter polymer as probable as possible. Two main strategies can be envisaged, both combining the actual dosimeter with an additional layer of diffusive/scattering material. The two strategies are based on positioning such layer, also of polymeric nature (e.g. polymethyl methacrylate), either in front of the dosimeter (i.e., between it and the radiation source) or behind it (i.e., beyond it with respect to the radiation source). In the first case, some neutrons will likely interact with the polymer ejecting high-energy protons which, subsequently, will create tracks in the CR39. In the latter case, some neutrons that passed through the dosimeter without interacting will be bounced back, thus increasing the probability for them to create tracks during a second passage through the CR39.

After the etching process, optical microscopes and transmission scanners are used to detect the number of tracks left, their size and shape. In general, neutrons are expected to create spherical tracks, while alpha particles, mostly related to the decay of environmental radon, are expected to cause more elliptical tracks. There is an instrumental and practical limitation to the size of the tracks for which a shape analysis can be done and, therefore, for which a dosimetry reading is possible. In this context, to make the dosimetry assessment procedure more robust, it would be important to assess the shape and number of tracks at spatial scales smaller than the instrumental limits. These pieces of information need to be determined experimentally on the specific type of dosimeter used, as they may change depending on the material and geometry employed during exposure to radiation.

2. Proposed experiment

We propose to perform SEM-EDX measurements, combined with optical profilometry measurements, on a series of CR39 dosimeters previously irradiated with an epithermal-fast neutron beam in three different configurations:

1. with no diffusive/scattering material associated to the dosimeter
2. with a diffusive/scattering material positioned in front of the dosimeter
3. with a diffusive/scattering material positioned behind the dosimeter

Scanning Electron Microscopy measurements will allow the morphological characterization of etched tracks at high magnification, while concurrent Energy-Dispersive X-ray spectroscopy will allow the detection of residual sodium carbonate compounds on the surface of the dosimeters after etching. The different composition in sodium carbonates with respect to the polymeric material is expected to provide a high contrast in the measurement of the samples, providing relevant information related to the distribution in size and shape of the tracks formed on the dosimeter.

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3. Summary of previous experimental proposals or characterisation

The samples to be investigated in the proposed experiment were recently irradiated at the VESUVIO spectrometer with a mixed flux of epithermal and fast neutrons for durations of irradiation between 10 and 60 minutes. The VESUVIO beam had a fast neutron component of $5x10^4$ neutrons/s/cm2 with energies above 10 MeV. At present, dosimetry samples are being processed for standard dose reading, implying chemical etching and optical image analysis.

4. Justification of experimental time requested

To perform the SEM-EDX measurements we request 4 days of instrument time on the “SEM&C-AFM with Optical Profiler” instrument of ISIS@MACH ITALIA. For each of the three configurations reported in Section 2, we plan to measure 4 samples irradiated for 1 hour at the VESUVIO spectrometer at ISIS Neutron and Muon Source. We expect, given the high number of tracks on these samples, that each sample will require about 3 hours of instrument time. Overall, we request 4 days to measure 12 CR39 samples.

Figure 1. Sets of 4 CR39 dosimeters prepared for neutron irradiation at different irradiation times, in the standard configuration (left), in the configuration with front-positioned diffuser material (centre) and in the configuration with the back-positioned diffuser material (right).

References

[1] Tse, K. C. C., Dragoslav Nikezic, and K. N. Yu. "Comparative studies of etching mechanisms of CR-39 in NaOH/H2O and NaOH/ethanol." Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms 263.1 (2007): 300-305.
[2] Nikezic, Dragoslav, and K. N. Yu. "Formation and growth of tracks in nuclear track materials." Materials Science and Engineering: R: Reports 46.3-5 (2004): 51-123.

Experiment Proposal

Experiment number GP2024099

Principal investigator	Dr Chimenti Stefano, CROMOGENIA UNIT, SPAIN
Co-investigator	Dr Gennaro Gentile, IPCB CNR, ITALY
Co-investigator	Dr Marino Lavorgna, CNR, ITALY
Co-investigator (*)	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Morphological characterization of hydrophobic/oleophobic nanoparticles and coatings by SEM@C-AFM
MRF Instrument	**SEM&C-AFM with Optical Profiler**
Days requested:	3
Access Route	Direct Access
Previous GP Number:	no
Science Areas	Chemistry, Engineering, Materials
DOI:	-
Sponsored Grant	None
Sponsor:	-
Grant Title	-
Grant Number:	-
Start Date	-
Finish Date:	-
Similar Submission?	-
Industrial Links	Cromogenia

Non-Technical Abstract

Water-dispersed polymer nanoparticles, obtained by radical emulsion polymerization, are key ingredients in many technological and industrial products, such as coatings, paints, adhesives, sealants, paper and textile additives, drug delivery systems and cosmetics. Cromogenia UNITS, in this context, is actively working in the research and development of fluorine-free polymer based nanoparticles capable of providing the hydrophobic/oleophobic effect to natural fabrics, such as cotton. For the tailoring of the developed products, a deep morphological characterization of nanoparticles by correlative SEM/AFM is needed. In separate experiment proposals, nanoparticles and coatings applied onto fabrics will be characterized by TEM, SAXS/WAXD and XRD tomography.

Publications -

ISIS neutron and muon source	**E-platform:** No
Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Science and Technology Facilities Council
ISIS Neutron and Muon Source

Sample record sheet

Principal contact	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
MRF Instrument	**SEM&C-AFM with Optical Profiler** **Days Requested:** 3
Special requirements:	

SAMPLE

Material	3 water dispersed polymer nanoparticles, differing for their composition (polymer and inorganic nanoadditives)	3 water dispersed polymer nanoparticles applied onto cotton fabrics	-
Formula	polymer nanoparticles in water dispersion	polymer coatings onto cotton substrates	-
Forms	Liquid	Solid	
Volume	2 cc	2 cc	
Weight	2 g	2 g	
Container or substrate	vial	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	298 - K	298 - K	-
Pressure Range	1000 - mbar	1000 - mbar	-
Magnetic field range	- T	- T	-
Standard equipment	None	None	-
Special equipment	N/A	N/A	-

SAFETY

Prep lab needed	Yes	Yes	-
Sample Prep Hazards	no	no	-
Special equip. reqs	no	Observation after embedding in resin and preparation of ultrathin sections by ultramicrotomy	-
Sensitivity to air	No	No	-
Sensitivity to vapour	No	No	-
Experiment Hazards	no	no	-
Equipment Hazards	-	-	-
Biological hazards	-	no	-
Radioactive Hazards	no	no	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	-

Morphological characterization of hydrophobic/oleophobic nanoparticles and coatings by SEM@C-AFM

Background and Context

Water-dispersed polymer nanoparticles, obtained by radical emulsion polymerization, are key ingredients in many technological and industrial products, such as coatings, paints, adhesives, sealants, paper and textile additives, drug delivery systems and cosmetics.

In the field of the textile industry, for example, some technical properties of fibres, such as repellency to water or other substances, do not meet the needs required of garments during their useful life. To give the repellent effect, a finishing treatment must be applied to the fabric. Currently, fluorinated polymers in aqueous dispersion make it possible to obtain a water and oil repellent finish in the various substrates on which they are applied. Hence their wide use in many sectors and most companies that are dedicated to the marketing of products for textile finishing, constantly find themselves developing new products with new properties and substantial improvements that they give to fabrics in general.

However, in recent years ECHA has placed traditional fluorinated resins under scrutiny as they often contain long-chain perfluorocarbons (PFCs), which are known to persist in the environment and potentially have adverse effects on human health and wildlife. Consequently, research has been oriented towards the development of alternatives with similar hydrophobic and oleophobic properties but with a lower environmental impact given the absence of fluorinated monomers.

Cromogenia UNITS, in this context, is actively working in the research and development of polymers in aqueous dispersion as alternatives to fluorinated polymers which are capable of providing the same repellency to water and good repellency to oils.

Although homogeneous particles consisting of one or single components can be used in many textile applications, multiphase polymer particles (comprising several monomers and/or nanofillers) are preferred, since they synergistically combine various physical and chemical properties of different materials. As a result, a wider range of properties can be achieved, e.g. fast drying hardness and/or high hydrophobicity in water-based systems.

The final performance of multiphase particles depends critically on their morphology and chemical composition. Both morphology and chemical composition are the result of complex kinetic and thermodynamic processes that occur during polymerization.

For this reason, the morphological characterization by SEM/AFM and optical profilometry of the developed polymer nanoparticles and related coatings applied onto substrates of interest (cotton fabrics) is of fundamental importance to tailor the properties of the synthesized materials and their performances.

2. Proposed experiment

The morphological characterization of the polymer nanoparticles and coatings obtained by applying the nanoparticles onto cotton substrates will be performed by using the SEM&C-AFM, available at Tor Vergata Unit.

In separate experiment proposals, the nanoparticles will be characterized by the TEM-FEI available at IPCB-CNR Unit. Moreover, the coated cotton substrates will be characterized by SAXS/WAXD and XRD tomography available at the IPCB CNR Unit, to investigate the distribution of the nanoparticles and the coating structure on fabrics.

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3. Summary of previous experimental proposals or characterisation

No previous experiments have been carried out on these samples

4. Justification of experimental time requested

We have requested the SEM&C-AFM equipment available at the Tor Vergata University Unit to evaluate the morphology of the 3 types of nanoparticles, differing for their composition (polymer matrix, inorganic additives) and coatings obtained by application of the nanoparticle dispersions onto cotton fabrics.

Therefore, a total of 6 samples will be analysed. After discussion with the instrument scientist, we request 3 days of SEM&C-AFM + optical profiler access (2 days for SEM&C-AFM + 1 day for optical profiler analysis), for a fully and thorough morphological characterization of the materials. The foreseen beam time accounts set up and for the data collection on the samples.

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(2016) ACS Nano, 10 (3), pp. 3042 - 3068.
DOI: 10.1021/acsnano.5b08176

Kausar A.
Nanomaterials for design and fabrication of superhydrophobic polymer coating
(2019) Superhydrophobic Polymer Coatings: Fundamentals, Design, Fabrication, and Applications, pp. 77 - 90
DOI: 10.1016/B978-0-12-816671-0.00005-9

Attia N.F., Moussa M., Sheta A.M.F., Taha R., Gamal H.
Effect of different nanoparticles based coating on the performance of textile properties
(2017) Progress in Organic Coatings, 104, pp. 72 - 80
DOI: 10.1016/j.porgcoat.2016.12.007

Experiment Proposal

Experiment number GP2024143

Principal investigator	Dr Valerio Scacco, University of Rome Tor Vergata, ITALY
Co-investigator (*)	
Co-investigator (*)	
Co-investigator (*)	
Co-investigator (*)	
Co-investigator (*)	
Co-investigator (*)	
Co-investigator (*)	
Co-investigator (*)	
Experiment title	Surface characterization of amorphous GaN and GaP thin films deposited by magnetron sputtering
MRF Instrument	**SEM-AFM with Optical Profiler**
Days requested:	2
Access Route	Direct Access
Previous GP Number:	-
Science Areas	Chemistry, Materials, Physics
DOI:	-
Sponsored Grant	None
Sponsor:	-
Grant Title	-
Grant Number:	-
Start Date	-
Finish Date:	-
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

We request machine time to perform an experimental analysis aimed at characterizing thin coatings of Gallium Phosphide (GaP) and Gallium Nitride (GaN), deposited on silicon and fused silica substrates using magnetron sputtering. The analysis will focus on assessing the morphological, topological, and chemical properties of these coatings, which are only a few hundred nanometers thick. Our primary objective is to verify the uniformity of the deposition, ensure the absence of impurities or contamination, and confirm that the deposited material is amorphous rather than crystalline. This can be observed through SEM analysis, as crystal structures would be clearly visible at high magnifications if present. These measurements are crucial for evaluating the quality of the coatings, which are intended for use in gravitational wave interferometry, specifically within the framework of the "Einstein Telescope" experiment.

Publications -

ISIS neutron and muon source

E-platform: No

Instruments	
Days Requested:	
Access Route	
Previous RB Number:	
Science Areas	
DOI:	
Sponsored Grant	
Sponsor:	
Grant Title	
Grant Number:	
Start Date	
Finish Date:	
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact

MRF Instrument **SEM-AFM with Optical Profiler** **Days Requested:** 2

Special requirements:

SAMPLE

Material	GaN coatings on Si and SiO2 substrates	GaP coatings on Si and SiO2 substrates	-
Formula	-	-	-
Forms	Solid	Solid	
Volume	cc	cc	
Weight	mg	mg	
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	- K	-
Pressure Range	- mbar	- mbar	-
Magnetic field range	- T	- T	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	Yes	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	No	-
Sensitivity to vapour	No	No	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	-

Proposal ISIS@MACH ITALIA

Surface characterization of amorphous GaN and GaP thin films deposited by magnetron sputtering

1. Background and Context

Recent years have seen growing interest in advanced coatings for next-generation gravitational wave detectors like the Einstein Telescope [1-3]. The aim is to identify materials that minimize thermal noise, a key factor limiting the sensitivity of these interferometers. Amorphous Gallium Phosphide (GaP) and Gallium Nitride (GaN) have shown promise as mirror coatings due to their favorable optical and thermal properties.

This proposal is part of a broader research program focused on optimizing coatings for gravitational wave detection. The project involves collaboration across several research institutions, supported by internal and external funding for material fabrication and characterization.

2. Proposed Experiment

The aim of this experiment is to perform detailed surface characterization of amorphous GaP and GaN coatings. Amorphous materials are of great interest for next-generation interferometers [4-6], and these materials are being explored for the Einstein Telescope due to their potential to reduce thermal noise.

The coatings must have uniform surface morphology and topology, as surface irregularities could increase thermal noise and reduce interferometer performance.

It is also critical to verify the chemical purity of the coatings, as impurities could degrade their optical and thermal properties. A detailed chemical analysis of the surface is essential to confirm the absence of contaminants.

We propose using the AF-SEM microscope, equipped with an EDX detector for microanalysis. This instrument will provide three essential types of information:

- **Morphological and Topological Characterization:** The AF-SEM will offer high-resolution surface imaging to detect non-uniformities or defects.
- **Chemical Analysis:** The EDX detector will provide elemental analysis, verifying material purity and identifying contaminants.
- **Crystallinity Check:** It's crucial to confirm the coatings are amorphous. The SEM's high magnification will detect any crystalline structures, if present.

These capabilities make the AF-SEM the ideal tool for this experiment.

3. Summary of Previous Experimental Proposals or Characterization

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This experiment is part of a wider experimental campaign conducted at various facilities, although not a direct continuation of prior work at this site. Earlier studies have provided insight into GaN's structural, optical, and mechanical properties, forming the foundation for the proposed surface characterization. Combining these results will help evaluate the coatings' potential for gravitational wave detection.

4. Justification of Experimental Time Requested

We request two days of machine time on the AF-SEM with EDX capabilities to analyze two material types—GaP and GaN—across two to three samples each. The time requested is based on:

- **Surface Uniformity Analysis:** High-resolution imaging will identify any surface irregularities or defects.
- **Chemical Purity:** Elemental analysis using the EDX detector will confirm the absence of contaminants.
- **Amorphous Structure Check:** The crystallinity of the coatings will be examined to verify they are amorphous.

Setup time for each sample will be minimal, ensuring efficient use of the machine during the requested period.

Bibliography

[1] J. Degallaix et al (2014) *Classical and Quantum Gravity*, *31*(18), 185010

[2] R. Birney, Cumming et al (2017) *Classical and Quantum Gravity*, *34*(23), 235012.

[3] K. Craig et al (2019) *Physical Review Letters*, *122*(23), 231102.

[4] M. Granata *et al* (2020) *Class. Quantum Grav.* 37 095004.

[5] J. Steinlechner, & I. W. Martin (2021) *Physical Review D*, *103*(4), 042001.

[6] A. Paolone et al (2022) *Coatings*, *12*(7), 1001.

SOURIRE

Experiment Proposal

Experiment number GP2024057

Principal investigator (*)	Dr Enrico Perelli Cippo, Consiglio Nazionale delle Ricerche, ITALY
Co-investigator	Dr Stephanie Cancelli, University of Milano-Bicocca, ITALY
Co-investigator	Dr Oscar Putignano, CNR, ITALY
Co-investigator	Mr Federico Caruggi, University of Milano-Bicocca, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Test of radiation hardness and material degradation of boron GEM foils at the SOURIRE Neutron Source

MRF Instrument	**SOURIRE**	**Days requested:**	1
Access Route	Direct Access	**Previous GP Number:**	No
Science Areas	Engineering, Physics	**DOI:**	No
Sponsored Grant	None	**Sponsor:**	-
Grant Title	-	**Grant Number:**	-
Start Date	-	**Finish Date:**	-
Similar Submission?	-		
Industrial Links	-		

Non-Technical Abstract

Neutron detectors utilizing Gas Electron Multiplier (GEM) technology are gaining popularity due to their characteristics. GEMs are paired with a single boron layer to detect thermal neutrons, achieving a detection efficiency of a few percent. To improve this efficiency, a new device has been developed and the new detector is coupled with the innovative boron-GEM (BGEM) foils, a standard GEM foils coated on both sides with a B4C layer; the final device has six BGEM foils.
The GEM will be examined using the SOURIRE to test the BGEM foils' resistance under a neutron flux of 10^{10} n/cm²s, and SEM ZEISS GEMINI to analyse the thickness and composition of the boron deposition. Both instruments are part of IM@IT. Additionally, the IMAT beamline at the ISIS Facility will be used to assess the uniformity of the boron coating.
In this proposal our team ask to obtain measurement time at the SOURIRE facility.

Publications

Cancelli, S., et al., Development of a ceramic double thick gem detector for transmission measurements at the vesuvio instrument at isis, JINST, 16 (2021).
Muraro, A., et al., Mbgem: a stack of borated GEM detector for high efficiency thermal neutron detection. EPJ Plus, 1–14 (2021).

Instruments	**IMAT**	**Days Requested:**	1
Access Route	Direct Access	**Previous RB Number:**	No
Science Areas		**DOI:**	No
Sponsored Grant	None	**Sponsor:**	
Grant Title	-	**Grant Number:**	
Start Date	-	**Finish Date:**	
Similar Submission?			
Industrial Links			

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Sample record sheet

Principal contact	Dr Enrico Perelli Cippo, Consiglio Nazionale delle Ricerche, ITALY		
MRF Instrument	**SOURIRE**	**Days Requested:**	1
Special requirements:			

SAMPLE

Material	1 BGEM foil (Cu, Kapton, 10B4C)	-	-
Formula	-	-	-
Forms	Solid		
Volume	cc		
Weight	10 g		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	No	-	-

SAFETY

Prep lab needed	No	-	-
Sample Prep Hazards	No	-	-
Special equip. reqs	None	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	No	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	-	-

Test of radiation hardness and material degradation of boron GEM foils at the SOURIRE facility

1. Background and Context

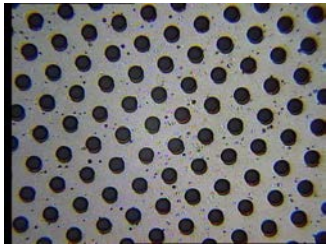
Neutron detectors utilizing Gas Electron Multiplier (GEM) technology are gaining popularity due to their notable characteristics, such as high spatial resolution (approximately in the millimeter range) and efficient detection capabilities [1]. These detectors can cover extensive areas in various shapes and can handle counting rates up to MHz/cm². Typically, GEMs are paired with a single boron layer to detect thermal neutrons, achieving a detection efficiency of a few percent [2]. To improve this efficiency, a new device has been developed through collaboration between the IM@IT Units at the University of Milano Bicocca and ISTP-CNR. This advanced GEM detector incorporates innovative boron-GEM (BGEM) foils, which are standard GEM foils coated on both sides with a B4C layer [3]. The final device has six BGEM foils and it has been characterised at the ISIS Facility on the VESUVIO experiment reaching a detection efficiency of 16% at 25 meV.
Thanks to the obtained results, the detector can be used to perform experiment in transmission geometry.
The GEM will be examined using the SOURIRE to test the BGEM foils' resistance under a neutron flux of 10^{10} n/cm²s, and SEM ZEISS GEMINI to analyse the thickness and composition of the boron deposition. Both instruments are part of IM@IT. Additionally, the IMAT beamline at the ISIS Facility will be used to assess the uniformity of the boron coating.
In this proposal our team ask to obtain measurement time at the SOURIRE facility.

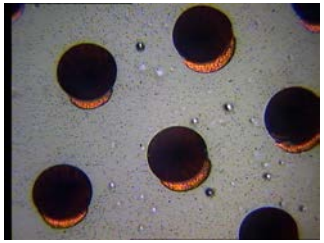
2. Proposed experiment

Despite of the obtained good results, during the neutron measurements we observed an anomalous behaviour of the detector and after preliminary tests, the GEM response resulted to be different from the response before the ISIS measurements.
Thus, further analysis on the B4C coating is needed to improve the BGEM production process. In particular, a neutron source allows bulk measurements into the B4C layer to check its status (e.g. homogeneity of deposition) and it allows also to test the radiation hardness of the foils.
SOUIRIRE is a fast neutron source with a high neutron flux and it can provide information about the radiation hardness after a long exposure of the GEM foils.
The main information we need is the 2D-resolved neutron transmission through the different borated GEM foils. The present proposers have the necessary expertise for the analysis of the raw images.

3. Summary of previous experimental proposals or characterisation

Preliminary tests with optical microscopy have been performed to check the foils status. The images show an example of a GEM foil boron coating and the holes structure. As it is visible from the right picture, the holes of the irradiated sample look damaged; however, no information can be inferred on the status of the B4C layer with this technique.





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4. Justification of experimental time requested

The GEM foil under study is made of a 50 μm kapton layer sandwiched between two 5 μm copper layers. The foil is micro-perforated with a high hole density (50-100 mm2) with a bi-conical shape (external diameter of 70 μm, internal diameter of 50 μm) and the pitch is 140 μm. The BGEM foil is obtained covering the GEM foil with a 1μm thick $^{10}B_4C$ layer on both sides. The $^{10}B_4C$ has been chosen since it has more stable than pure ^{10}B. The BGEM foil has an area of 10x10 cm^2.
Thanks to the high neutron flux even after moderation, SOURIRE allows to quickly test an entire BGEM foil (10x10 cm^2) within a single run of a few minutes irradiation, thus the full set of six significant specimens can be measured in less than one hour. For what is concerning the radiation hardness test, we require to irradiate a single spare gem foil with a full working day of *neutrons*.

References

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Experiment Proposal

Experiment number GP2024062

Principal investigator (*)	Dr Enrico Perelli Cippo, Consiglio Nazionale delle Ricerche, ITALY
Co-investigator	Dr Marco Tardocchi, CNR, ITALY
Co-investigator	Dr Marica Rebai, CNR, ITALY
Co-investigator	Dr Oscar Putignano, CNR, ITALY
Co-investigator	Dr Stephanie Cancelli, University of Milano-Bicocca, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Characterisation of a Diamond detector matrix at the SOURIRE Neutron Source
MRF Instrument	**SOURIRE** **Days requested:** 1
Access Route	Direct Access **Previous GP Number:** -
Science Areas	Engineering, Physics **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

Fast neutron detection is crucial for assessing plasma parameters in fusion machines. Fast neutron spectra with good energy resolution provide insights into plasma temperature, fuel ion ratio, and other quantities.

Solid State Detectors (SSDs) are ideal due to their compact size, low cost, fast response times, low energy resolution, low gamma ray sensitivity, mechanical resilience, and radiation hardness. Diamond SSDs are noted for their 1% energy resolution, simple response function, and MHz counting rate. A new diamond SSD matrix with four 4.5x4.5 mm² diamonds has been developed to test spectroscopic capabilities using SOURIRE. Neutron detection in SSDs relies on electron-hole pairs from neutron interactions with 12C nuclei, focusing on the 12C(n,α)9Be reaction for 14 MeV neutron spectroscopy from Deuterium-Tritium plasma.

Characterization will continue at ISIS@MACH ITALIA for bonding analysis before SOURIRE irradiation. Calibration will be validated at the NILE facility at ISIS.

Publications

Rebai M et al 2016 "Response function of single crystal synthetic diamond detectors to 1-4 MeV neutrons for spectroscopy of D plasmas" Rev. Sci. Instrum. 87 11D823

Muraro A et al 2016 "First neutron spectroscopy measurements with a pixelated diamond detector at JET" Rev. Sci. Instrum. 87 11D833

Cazzaniga C et al 2014 "Single crystal diamond detector measurements of deuterium-deuterium and deuteriumtritium neutrons in Joint European Torus fusion plasmas"

Instruments	**NILE**	**Days Requested:** 1	
Access Route	Direct Access	**Previous RB Number:** No	
Science Areas		**DOI:**	
Sponsored Grant	None	**Sponsor:**	
Grant Title	-	**Grant Number:**	
Start Date	-	**Finish Date:**	
Similar Submission?			
Industrial Links			

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Sample record sheet

Principal contact	Dr Enrico Perelli Cippo, Consiglio Nazionale delle Ricerche, ITALY		
MRF Instrument	**SOURIRE**	**Days Requested:** 1	
Special requirements:			

SAMPLE

Material	Carbon. aluminium	-	-
Formula	-	-	-
Forms	Solid		
Volume	cc		
Weight	100 g		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	No	-	-
Sample Prep Hazards	None	-	-
Special equip. reqs	None	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	None	-	-
Equipment Hazards	-	-	-
Biological hazards	None	-	-
Radioactive Hazards	None	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	-	-

Characterisation of a Diamond detector matrix at the SOURIRE Neutron Source

1. Background and Context

Fast neutron detection is one of the prominent methods for assessing the plasma parameters in fusion machines. Measuring the flux of 14 MeV neutrons on a direct line of sight on the plasma can be used to calculate the number of DT fusion reactions, and thus the fusion power; measuring the fast neutron spectra with sufficient energy resolution can be used to obtain information on the plasma ion temperature, on the fuel ion ratio nD/nT and on other quantities; lastly, measuring the neutron flux inside the breeding blanket can be used to calculate the tritium reaction rate and, therefore, the amount of tritium produced.

Solid State Detectors (SSDs) are very good candidates for the application to fusion devices, since they are compact (a few millimetres across) and have comparatively low costs respect to other neutron sensors, allowing for arrays of detectors to be arranged into tomographic cameras (i.e. along multiple lines of sight). They also feature fast response times, low energy resolutions, low sensitivity to gamma rays, good mechanical resilience and good radiation hardness. Diamond SSDs are widely present in literature, highlighting their strengths (1% energy resolution [1], simple response function and MHz counting rate). Neutron detection in SDD is based on the collection of electron-hole pairs produced by charged particles generated by neutron interaction with 12C carbon nuclei in the detector. The main nuclear reaction channels occurring are: elastic and inelastic scattering 12C(n,n')12C; n-3α reaction (carbon breakup) 12C(n,n')3α (Q_{value}=7.23 MeV) and n-α reaction 12 C(n,α) 9 Be (Q_{value}=5.7 MeV). The latter reaction 12C(n,α)9Be is the selected one for 14 MeV neutron spectroscopy measurements from a Deuterium-Tritium (DT) plasma [2].

A new configuration of diamond detectors has been developed by ISTP-CNR in the form of a matrix of 4 diamonds (4,5x4,5 mm^2). Aim of this proposal is to test the spectroscopic capability of the matrix detector with SOURIRE, making use of the 14 MeV neutron source. Since the present authors are involved into the characterisation of the device, another proposal to another ISIS@MACH ITALIA facility, the confocal microscope 3 instrument, will be submitted in parallel, in order to obtain precise information about the bonding of the diamond crystals before the irradiation with SOURIRE.

The measurements will be validated at the NILE facility at the ISIS Neutron and Muon Source.

2. Proposed experiment

The present authors propose to use the SOURIRE facility to perform a scan of the functionality,
efficiency, response function and energy resolution of a matrix of a Diamond detector prototype.

In particular, the aim of the experiment is the measurement of the energy spectra at different angles, i.e. at different energies of the fast neutrons.

3. Summary of previous experimental proposals or characterisation

The detector has been preliminarily tested with a ^{241}Am source at the ISTP-CNR laboratories to check the detector response and its spectroscopic capability. In particular, ^{241}Am emits α particles with three different energies (5.486, 5.442, 5.388 MeV) with branching ratio (84.5%, 13.06%, 1.62%, respectively).

A dedicated custom electronic chain was used for these measurements; the detector was coupled to a CIVIDEC C6 fast charge preamplifier, which provides a signal with a rise time of 3.5 ns and a shaping time of 25 ns. The preamplifier has a bias current of 25 mA, a gain of 6

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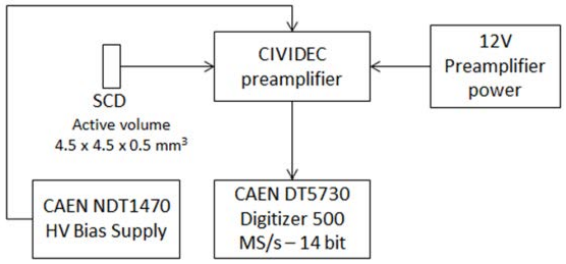
mV fC−1 and a bandwidth of 100 MHz. A CAEN high voltage supply model NDT1470 was used to supply a voltage of +400 V to the detector (reported in the following figure).



3. Justification of experimental time requested

The sample is a detector made of a matrix of Diamond 4,5x4,5 mm^2 each mounted on a PCB and encased in a small volume aluminium casing (less than 5 cm x 5 cm x 3 cm). It is connected to the preamplifier (which is a rigid box measuring less than 15 in length) through a short coaxial cable (no more than few tens of cm).

The effective irradiation time would be in the order of 3 hours for an adequate statistics for the different angular positions proposed Additional time will also be needed to set up the experimental apparatus and to adjust the instrumentation before, between and after the irradiation. Thus, a total time slot of one day is requested for the full experimental activity.

References

1. *D. Rigamonti et al., Neutron spectroscopy measurements of 14 MeV neutrons at unprecedented energy resolution and implications for deuterium–tritium fusion plasma diagnostics, Meas. Sci. Technol. 29 (2018) 045502 (9pp), doi: 10.1088/1361-6501/aaa675*
2. *A. V. Krasilnikov et al., Study of d-t neutron energy spectra at JETusing natural diamond detectors, Nucl. Instrum. Methods Phys. Res. A476 (2002) 500-505.*

Spectrofluorimeter

Experiment Proposal

Experiment number GP2024129

Principal investigator	Dr Barbara Vercelli, Consiglio Nazionale delle Ricerche CNR, ITALY
Co-investigator	Professor Barbara La Ferla, University of Milano Bicocca, ITALY
Co-investigator	Dr Alice Pavan, Università degli Studi di Milano-Bicocca, ITALY
Co-investigator	Professor Maddalena Collini, Università degli Studi di Milano Bicocca, ITALY
Co-investigator (*)	Professor Giuseppe Chirico, Università degli Studi di Milano-Bicocca, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Hydrothermal Sustainable Approaches for the Preparation of Carbon Quantum Dots: an Insight into the Effect of Different Carbon Sources on the Photo-Emission Behavior
MRF Instrument	**Spectrofluorimeter** **Days requested:** 5
Access Route	Direct Access **Previous GP Number:** no
Science Areas	Chemistry, Materials **DOI:** -
Sponsored Grant	Yes **Sponsor:** Other
Grant Title	Multifunctional Compounds for a Multitarget Approach against Neurodegenerative Disorders (MULTIFUN) **Grant Number:** MUR PRIN Project n. 2022N9E847
Start Date	01/10/2023 **Finish Date:** 30/09/2025
Similar Submission?	GP No: GP2024128; PI: Dr. Alice Pavan
Industrial Links	-
Non-Technical Abstract	The present proposal is part of a broader research program aiming to study and develop reliable and sustainable synthesis approaches for preparing Carbon Quantum Dots (CDs) with robust and reproducible properties. We submit a request for an experimental campaign aiming at performing a study of the photo-emission behavior (including PL-emission, PL-quantum yield, and the dependence of the emission wavelength on the excitation one) of a series of CDs samples prepared through the hydrothermal approach employing gallic acid (GA) and citric acid (CA) or both as carbon sources. The main scope is to investigate a possible synergic effect of the two carbon sources on the CDs. In particular, we expect that the CDs obtained from the combination of both carbon sources (GA and CA) will merge the intrinsic properties of the samples prepared employing only one carbon source (GA or CA).
Publications	B. Vercelli et al., Small Structures, recently submitted

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Professor Giuseppe Chirico, Università degli Studi di Milano-Bicocca, ITALY
MRF Instrument	**Spectrofluorimeter** **Days Requested:** 5
Special requirements:	

SAMPLE

Material	Carbon Quantum Dots obtained at 160°C from gallic acid and urea, solvent EtOH (carbon based graphitic structures)	Carbon Quantum Dots obtained at 160°C from gallic acid/citric acid and urea, solvent EtOH (carbon based graphitic structures)	Carbon Quantum Dots obtained at 160°C from citric acid and urea, solvent EtOH (carbon based graphitic structures)
Formula	-	-	-
Forms	Solid	Solid	
Volume	cc	cc	cc
Weight	5 mg	5 mg	5 mg
Container or substrate	vial	vial	vial
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

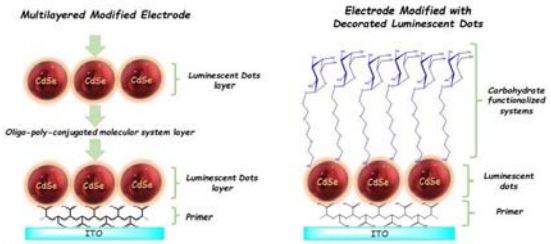
Temperature Range	- K	- K	- K
Pressure Range	- mbar	- mbar	- mbar
Magnetic field range	- T	- T	- T
Standard equipment	None	None	None
Special equipment	no	no	no

SAFETY

Prep lab needed	Yes	Yes	Yes
Sample Prep Hazards	no	no	no
Special equip. reqs	no	no	no
Sensitivity to air	No	No	No
Sensitivity to vapour	No	No	No
Experiment Hazards	no	no	no
Equipment Hazards	-	-	-
Biological hazards	no	no	no
Radioactive Hazards	no	no	no
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	Disposed by IS

1. Background and Context

The group of Electrochemistry and Nanomaterials of the Icmate unit of Milano has a long-time experience in the realization and characterization of self-assembled nano-systems, obtained by the alternation of semiconductor nanocrystals or noble metals clusters and oligo-polyconjugates molecular systems or carbohydrates functionalized systems (Scheme 1), for optoelectronic, photovoltaic and biomedical applications. This is a consolidated blue-sky research activity documented by a series of publications in high-impact ISI journals.

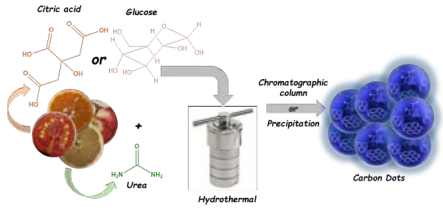


Scheme 1. – Self-assembled Nanosystems

Within this research program and considering the growing request for eco-sustainable, non-toxic nanomaterials, the research studies were recently devoted to the employment of carbon quantum dots (CDs) as a "green" and cheap alternative to noble metal and calcogenide-based dots.

CDs are fluorescent carbon-based nanomaterials, which, since their discovery in 2004, gained grooving interest from the research community, because of their excellent fluorescence properties and surface rich in functionalities, which enables functionalization with a wide range of molecules, including receptors, bio-molecules, molecular semiconductors, etc. Furthermore, they are soluble in water, exhibit an extremely low toxicity, and an excellent biocompatibility useful for real-world biological applications. Their synthesis approaches are simple, sustainable, and could employ cheap and recyclable precursors derived from biomass and agro-industrial waste.

As a first approach, we focused on the development of a sustainable CDs synthesis strategy, which could be reliable and reproducible. We selected the hydrothermal approach owing to its feasibility for large-scale industrial applications, and we employed precursors that could be obtained from agro-industrial waste, like citric acid (CA) or glucose (Glu) as carbon sources and urea as both base and nitrogen sources (Scheme 2).



Scheme 2. – Scheme of hydrothermal preparation of Carbon Quantum Dots

We published two preliminary works dealing with the role played by the nitrogen centers and the thermal post-treatments, respectively, on CDs electrochemical and optical properties[1]. Then we studied the influence of the reaction parameters on CDs properties: we published a work on the issues encountered in the synthesis/purification of red-emitting CDs[2], and we recently submitted a comprehensive work on the influence of the process parameters, particularly temperature, on the properties of CDs[3]. In a further step of our research program, we employed gallic acid (GA) alone and in combination with CA, as carbon source to study a possible synergic effect on the optical and properties of CDs. Preliminary, UV-vis absorption and FT-IR determinations seem to support the hypothesis. In this context, a study of the photo-emission behaviour (including PL-emission, PL-quantum yield, and the dependence of the emission wavelength on the excitation one) is of crucial interest/importance to obtain a comprehensive characterization of the new CDs. In particular, we expect that the CDs obtained from the combination of both carbon sources (GA and CA) will merge the intrinsic properties of the samples prepared employing only one carbon source (GA or CA). Optimistically, we also expect to obtain information about the influence of the temperature process on the photo-emission properties of the CDs prepared employing both GA and CA.

2. Proposed experiment

With the present proposal, we submit the request of a 5 days' experimental campaign finalized at performing a study of the photoemission behaviour (including PL-emission, PL-quantum yield, and the dependence of the emission wavelength on the excitation one) on a series of CDs samples (optimistically 3/4) prepared through the hydrothermal approach employing GA, CA or both as carbon sources. The main scope is to investigate the influence of the carbon source on the photo-emission characteristics of the obtained materials.

The results expected from the proposed experimental campaign are of paramount importance for the development of the research programme briefly described at point 1, because they are expected to support and enforce the preliminary obtained UV-vis and FT-IR results, employing a facility (Spectrofluorimeter), which is not present in the laboratories of the Icmate unit of Milano.

3. Summary of previous experimental proposals or characterisation

We previously submitted the proposal n°GP2024022 for a series of TEM analyses of CDs samples.

4. Justification of experimental time requested

For the development of the present proposal, we selected the ISIS@MACH ITALIA Spectrometer Eclipse, Varian facility at the University of Milano-Bicocca because it meets the experimental requests and is located in the proximity of the Icmate unit of Milano. So, in case of analysis problems or specific sample preparations, it is possible to promptly intervene in the near Icmate laboratories. We planned the photophysical characterization of 3/4 CDs samples for a total of 5 days, including eventual time waste related to possible issues in sample preparation, specific instrument settings, and unexpected problems during measurement execution and signal optimization/collection.

5. References

1. Vercelli B. et al. Elec. Acta, **2021**, 138557, https://doi.org/10.1016/j.electacta.2021.138557; Vercelli B. et al. Molecules **2023**, 28(1), 72; https://doi.org/10.3390/molecules28010072.

2. Vercelli B. et al. Nanomaterials **2023**, 13, 1365; https://doi.org/10.3390/nano13101635.

3. Vercelli B. et al., Small Structures, submitted.

TEM FEI

Experiment Proposal

Experiment number GP2024075

Principal investigator	Professor Fabiana Arduini, University of Rome Tor Vergata, ITALY
Co-investigator (*)	Dr Vincenzo Mazzaracchio, University of Rome "Tor Vergata", ITALY
Co-investigator	Professor Massimo Bonini, CSGI - University of Florence, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	TEM characterisation of inks based on carbon black/Prussian Blue nanocomposites for paper-based electrochemical (bio)sensors

MRF Instrument	**TEM FEI**	**Days requested:**	2
Access Route	Direct Access	**Previous GP Number:**	No
Science Areas	Chemistry	**DOI:**	-
Sponsored Grant	Yes	**Sponsor:**	Other
Grant Title	Lazio Innova Venture and Scientifica Venture Capital	**Grant Number:**	CI Fast
Start Date	01/01/2024	**Finish Date:**	01/07/2025
Similar Submission?	-		
Industrial Links	SENSE4MED		

Non-Technical Abstract

Electrochemical paper-based devices have opened a new route in the analytical science sector, having a huge impact at the academic level as well as in the industrial sector. The development of electrochemical devices with nanomaterials, including carbon black, iridium oxide, and Prussian blue nanoparticles, requires surface and dispersion characterization for obtaining accurate nanomaterial-functionalized paper-based electrochemical devices. The several characterizations that will be carried out thanks to the instrumentation within ISIS@MACH ITALIA will foster the development of electrochemical paper-based devices to carry with national and European projects in which the applicant (Prof. Fabiana Arduini) is the coordinator. In this framework we are proposing a multi-technique approach to investigate nanocomposites made of carbon black and Prussian Blue nanostructures, where Transmission Electron Microscopy (TEM) will be used to obtain high magnification images of the nanostructures.

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Vincenzo Mazzaracchio, University of Rome "Tor Vergata", ITALY		
MRF Instrument	**TEM FEI**	**Days Requested:**	2
Special requirements:			

SAMPLE

Material	Water, dimethylformamide, black carbon, Iron, nitrogen	carbon black	-
Formula	carbon black prussian blue nanoparticles	carbon black	-
Forms	Liquid	Liquid	
Volume	10 ml	10 ml	
Weight	10 g	10 g	
Container or substrate	vial	vial	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	298 - K	298 - K	-
Pressure Range	1 - mbar	1 - mbar	-
Magnetic field range	- T	- T	-
Standard equipment	-	None	-
Special equipment	nothing	no	-

SAFETY

Prep lab needed	Yes	Yes	-
Sample Prep Hazards	no	no	-
Special equip. reqs	no	no	-
Sensitivity to air	No	No	-
Sensitivity to vapour	No	No	-
Experiment Hazards	no	no	-
Equipment Hazards	-	-	-
Biological hazards	no	no	-
Radioactive Hazards	no	no	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	-

TEM characterisation of inks based on carbon black/Prussian Blue nanocomposites for paper-based electrochemical (bio)sensors

1. Background and Context

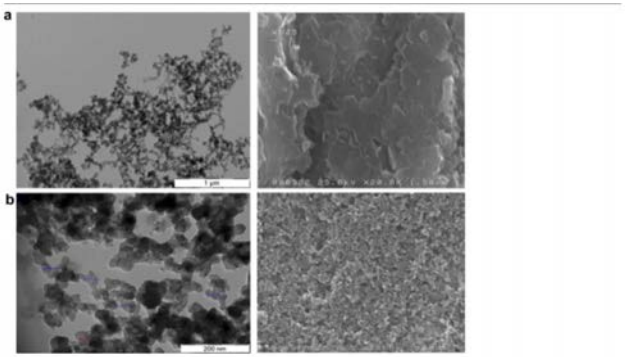
As reported by the applicant in the recent review entitled "Electrochemical paper-based devices: When the simple replacement of the support to print ecodesigned electrodes radically improves the features of the electrochemical devices" published in Current Opinion in Electrochemistry (Q1) SI: Emerging Opinions (2022) [1]: "*Paper-based electrochemical (bio)sensors have emerged as highly attractive analytical devices for their superior sustainable features, such as avoiding the use of polyester as support and the reduction of waste, being incinerated after use. However, paper-based electrochemical (bio)sensors have recently demonstrated further advantages, including the simple combination with vertical microfluidics and their use as a reservoir to deliver smart electrochemical (bio)sensors able to i) contain the reagents, ii) preconcentrate the target analyte, and iii) synthesize the nanomaterials inside the paper network. Furthermore, these devices have demonstrated their ability to overcome the limitations of the other printed electrochemical sensors in the measurement of entirely liquid samples by detecting the target analyte in the aerosol phase or solid sample, without the additional sampling system. These achievements highlight their valuable and varied advantages in the sensing sector*". Electrochemical paper-based devices have opened a new route in the analytical science sector with a huge impact at the academic level as well as in the industrial sector, since the relevant articles of Prof. Henry in United States [2, 3] and the applicant in Europe [4, 5]. From industrial point of view, the spin-off company SENSE4MED, Department of Chemical Science and Technologies, University of Rome Tor Vergata, in which the applicant is the CEO, has received an investment of 510 KEuro for the developing a paper-based electrochemical sensor for cystic fibrosis diagnosis [6], from the academic point of view, the applicant is the coordinator of the PRIN2022 SMARTMASK4CF with the aim to develop a facemask functionalized with paper-based devices for precision medicine in cystic fibrosis [7]. Additionally, the paper-based devices are creating a new challenge in organ on the chip field, considering the Pathfinder Open Horizon Europe project Phoenix-OoC (2024-2027) which has the overriding goal to create an organ chip on paper and in which the applicant is the European coordinator [8].
In this framework we are proposing a multi-technique approach to investigate nanocomposites made of carbon black and Prussian Blue nanostructures, where Transmission Electron Microscopy (TEM), Small Angle Scattering of X-rays (SAXS) and Scanning Electron Microscopy (SEM) are combined to get a full picture of the system.

2. Proposed experiment

Given the importance of both size and shape of the composites towards the electrochemical performances of the paper-based analytical devices, we propose here to characterise carbon black and Prussian Blue nanocomposites using the TEM (FEI) located at the IPCB-CNR Unit of IM@IT in Naples. This proposal is part of a multi-technique investigation, complemented by a separate proposal to characterize the nanoparticles dispersion by Small Angle Scattering of X-rays in the CSGI Florence Unit and, once the nanocomposites are deposited, to verify the quality of the casted materials by FE-SEM (where no metallization is needed) at the CSGI Florence Unit.

3. Summary of previous experimental proposals or characterisation

In previous experiments we have characterised carbon black nanostructures by means of TEM analysis [9], but we have not yet performed any TEM analysis of the carbon black-Prussian Blue nanoparticle composite used in paper-based electrochemical devices. The previous results highlight the feasibility of the proposed study. Furthermore, given the strong difference between the electronic density in carbon black and iron-based Prussian Blue nanostructures, TEM is very well suited for the characterisation of their composites.



Download : Download full-size image

Fig. 1. (A) TEM images of the CB material (a and b). (B) SEM imagines of bare SPE (a) and CB-SPE (b).

4. Justification of experimental time requested

The details of this section have been discussed with the ISIS@MACH Italia team. The composites are dispersed in a water:dimethylformamide mixture (1:1 v/v). Samples will be prepared by deposition of highly diluted dispersions onto TEM grids, followed by drying under the hood. Considering that, in addition to 2 references, 5 different dispersions will be investigated, we are requesting 2 days of instrument time.

References

[1] Arduini, F., 2022. Current Opinion in Electrochemistry, **35**, p.101090.
[2] Aryal, P., Hefner, C., Martinez, B. and Henry, C.S., 2024. Microfluidics in environmental analysis: advancements, challenges, and prospects for rapid and efficient monitoring. Lab on a Chip.
[3] Ozer, T., McMahon, C. and Henry, C.S., 2020. Annual Review of Analytical Chemistry, **13**(1), pp.85-109.
[4] Cinti, S., Minotti, C., Moscone, D., Palleschi, G. and Arduini, F., 2017. Biosensors and Bioelectronics, **93**, pp.46-51.
[5] Cinti, S., Talarico, D., Palleschi, G., Moscone, D. and Arduini, F., 2016. Analytica chimica acta, 919, pp.78-84.
[6] https://www.lazioinnova.it/news/innova-venture-e-scientifica-venture-capital-investono-in-sense4med/
[7] https://stc.uniroma2.it/ricerca/progetti-di-ricerca/
[8] https://phoenixooc.com
[9] Arduini, F., Amine, A., Majorani, C., Di Giorgio, F., De Felicis, D., Cataldo, F., Moscone, D. and Palleschi, G., 2010. *Electrochemistry communications*, ***12***(3), pp.346-350

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Science and Technology Facilities Council
ISIS Neutron and Muon Source

Experiment Proposal

Experiment number GP2024096

Principal investigator	Dr Chimenti Stefano, CROMOGENIA UNIT, SPAIN
Co-investigator (*)	Dr Gennaro Gentile, IPCB CNR, ITALY
Co-investigator	Dr Marino Lavorgna, CNR, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	TEM characterization of water dispersed fluorine-free polymer based nanoparticles
MRF Instrument	**TEM FEI**
Access Route	Direct Access
Science Areas	Chemistry, Engineering, Materials
Sponsored Grant	None
Grant Title	-
Start Date	-
Similar Submission?	-
Industrial Links	Cromogenia

Days requested: 5
Previous GP Number: no
DOI: -
Sponsor: -
Grant Number: -
Finish Date: -

Non-Technical Abstract

Water-dispersed polymer nanoparticles, obtained by radical emulsion polymerization, are key ingredients in many technological and industrial products, such as coatings, paints, adhesives, sealants, paper and textile additives, drug delivery systems and cosmetics. Cromogenia UNITS, in this context, is actively working in the research and development of fluorine-free polymer based nanoparticles capable of providing the hydrophobic/oleophobic effect to natural fabrics, such as cotton. For the tailoring of the developed products, a deep morphological/structural characterization of nanoparticles by TEM is needed. In separate experiment proposals, nanoparticles and coatings applied onto fabrics will be characterized by SEM&C-AFM, SAXS/WAXD and XRD tomography.

Publications -

ISIS neutron and muon source

E-platform: No

Instruments	
Access Route	
Science Areas	
Sponsored Grant	
Grant Title	
Start Date	
Similar Submission?	
Industrial Links	

Days Requested:
Previous RB Number:
DOI:
Sponsor:
Grant Number:
Finish Date:

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Science and Technology Facilities Council
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Sample record sheet

Principal contact Dr Gennaro Gentile, IPCB CNR, ITALY
MRF Instrument **TEM FEI** **Days Requested:** 5
Special requirements:

SAMPLE

Material	6 water dispersed polymer nanoparticles, differing for their composition (polymer and inorganic nanoadditives)	6 water dispersed polymer nanoparticles, differing for their composition (polymer and inorganic nanoadditives)	-
Formula	polymer nanoparticles in water dispersion	polymer nanoparticles in water dispersion	-
Forms	Liquid	Liquid	
Volume	2 cc	2 cc	
Weight	2 g	2 g	
Container or substrate	vial	vial	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	298 - K	298 - K	-
Pressure Range	1000 - mbar	1000 - mbar	-
Magnetic field range	- T	- T	-
Standard equipment	None	None	-
Special equipment	N/A	N/A	-

SAFETY

Prep lab needed	Yes	Yes	-
Sample Prep Hazards	no	no	-
Special equip. reqs	Observation after deposition onto carbon-coated TEM grids	Observation after embedding in resin and preparation of ultrathin sections by ultramicrotomy	-
Sensitivity to air	No	No	-
Sensitivity to vapour	No	No	-
Experiment Hazards	no	no	-
Equipment Hazards	-	-	-
Biological hazards	-	no	-
Radioactive Hazards	no	no	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	-

TEM characterization of water dispersed fluorine-free polymer based nanoparticles

Background and Context

Water-dispersed polymer nanoparticles, obtained by radical emulsion polymerization, are key ingredients in many technological and industrial products, such as coatings, paints, adhesives, sealants, paper and textile additives, drug delivery systems and cosmetics.

In the field of the textile industry, for example, some technical properties of fibres, such as repellency to water or other substances, do not meet the needs required of garments during their useful life. To give the repellent effect, a finishing treatment must be applied to the fabric. Currently, fluorinated polymers in aqueous dispersion make it possible to obtain a water and oil repellent finish in the various substrates on which they are applied. Hence their wide use in many sectors and most companies that are dedicated to the marketing of products for textile finishing, constantly find themselves developing new products with new properties and substantial improvements that they give to fabrics in general.

However, in recent years ECHA has placed traditional fluorinated resins under scrutiny as they often contain long-chain perfluorocarbons (PFCs), which are known to persist in the environment and potentially have adverse effects on human health and wildlife. Consequently, research has been oriented towards the development of alternatives with similar hydrophobic and oleophobic properties but with a lower environmental impact given the absence of fluorinated monomers.

Cromogenia UNITS, in this context, is actively working in the research and development of polymers in aqueous dispersion as alternatives to fluorinated polymers which are capable of providing the same repellency to water and good repellency to oils.

Although homogeneous particles consisting of one or single components can be used in many textile applications, multiphase polymer particles (comprising several monomers and/or nanofillers) are preferred, since they synergistically combine various physical and chemical properties of different materials. As a result, a wider range of properties can be achieved, e.g. fast drying hardness and/or high hydrophobicity in water-based systems.

The final performance of multiphase particles depends critically on their morphology and chemical composition. Both morphology and chemical composition are the result of complex kinetic and thermodynamic processes that occur during polymerization.

For this reason, the morphological and structural characterization by TEM of the developed polymer nanoparticles is of fundamental importance to tailor the properties of the synthesized materials and their performances.

2. Proposed experiment

The morphological characterization of the polymer nanoparticles will be performed by the TEM-FEI available at IPBC CNR. To have an insight on the internal structure of the nanoparticles, in addition to TEM analysis of nanoparticles deposited on TEM grids, ultrathin sections of nanoparticles will be prepared after their embedding in resin and observed by high magnification TEM.

In separate experiment proposals, the same nanoparticles, as well as coatings obtained by applying nanoparticles onto cotton fabrics, will be characterized by SEM-AFM (SEM&C-AFM, available at Tor Vergata Unit). Moreover, the treated substrates will be characterized by SAXS/WAXD and XRD tomography available at the IPCB CNR Unit, to investigate the distribution of the nanoparticles and the coating structure on fabrics.

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3. Summary of previous experimental proposals or characterisation

No previous experiments have been carried out on these samples

4. Justification of experimental time requested

We have requested the TEM FEI equipment available at IPCB CNR to evaluate the morphology of the 6 types of nanoparticles, differing for their composition (polymer matrix, inorganic additives).

TEM analysis will be performed on nanoparticles directly deposited onto TEM grids and on cross-sections of the nanoparticles after their embedding in resins and their preparation into ultrathin sections.

Therefore, a total of 12 samples will be analysed. For 6 samples the preparation facility ultramicrotomy, available at IPCB-CNR, will be used

After discussion with the instrument scientist, we request 5 days of TEM FEI access, for a fully and thorough morphological characterization of the materials. The foreseen beam time accounts set up and for the data collection on the samples.

References

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(2017) Progress in Organic Coatings, 104, pp. 72 - 80
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Experiment Proposal

Experiment number GP2024111

Principal investigator	Professor Gabriele Gentile, University of Rome Tor Vergata, ITALY
Co-investigator (*)	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Carla Andreani, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Multi-instrumental characterization of Oniscidean isopods to establish the nature and function of their cuticle and tubercles: TEM measurements
MRF Instrument	**TEM FEI**
Days requested:	2
Access Route	Direct Access
Previous GP Number:	No
Science Areas	Biology and Bio-materials, Physics
DOI:	-
Sponsored Grant	None
Sponsor:	-
Grant Title	-
Grant Number:	-
Start Date	-
Finish Date:	-
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

Andronicus is a genus of oniscidean isopods that includes less than a dozen species, most of which are confined to caves in the Italian Pre-Alps. Andronicus offers the unique opportunity to investigate the nature of the cuticle in cave (A. brentanus) and non-cave-dwelling species (A. dentiger), within the same evolutionary lineage, characterizing mineralization in the different species. The proposed investigation will also extend to including tubercles on the surface of the body, the nature of which, currently unknown, could be of a sensorial nature. Preliminary observations would suggest the presence of a possible innervation at the base of these tubercles, which if confirmed, would prove their nature and function. For the characterization on the mineralization of the cuticle and tubercles we envisage to use EDX, FT-IR, Raman, and X-ray diffraction, whereas the morphology characterization will be done by SEM, TEM and nano-XCT. Here, this proposal is focussed on the TEM analysis.

Publications

Vittori, M. et al., Arthropod Struct Dev. 46 (2016), pp. 96-107.
Gentile, G. and Allegrucci, G., International Journal of Speleology 26 (1997), pp. 47-61.
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ISIS neutron and muon source — **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY		
MRF Instrument	**TEM FEI**	**Days Requested:**	2
Special requirements:			

SAMPLE

Material	Oniscidean isopod	-	-
Formula	Organic material, Calcite	-	-
Forms	Solid		
Volume	0.03 cc		
Weight	1-2 g		
Container or substrate	-	-	-
Storage Requirements	Freezer (-20C)	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	-	-

Multi-instrumental characterization of Oniscidean isopods to establish the nature and function of their cuticle and tubercles: TEM measurements

1. Background and Context

Among Crustaceans, oniscidean isopods are uniquely adapted to terrestrial life, exhibiting strongly mineralized cuticles. Oniscideans include several species adapted to the caves. Among the most important evolutionary adaptations found in troglobitic oniscideans (i.e. bound to cave environments, from which they cannot escape due to strict ecological and physiological constraints) are the thinning of the cuticle with a reduce layer of calcite, although calcium carbonate is present in the exocuticle and the endocuticle [1]. Additionally, other adaptations include the lengthening of the appendages, the loss of the eyes, the development of sensory systems alternative to sight such as hygrosensors and chemosensors, usually located in different areas of the body. *Androniscus* (Fig. 1) is a genus of oniscidean isopods that includes less than a dozen species, most of which are confined to caves in the Italian Pre-Alps. Among these, *Androniscus dentiger* is the one that shows the least constraints, being present even in the most superficial layers of the soil in non-cave environments and showing a wide geographical distribution [2]. Indeed, by combining atomic absorption spectroscopy, thermogravimetry and X-ray diffraction, the composition of cuticles in several isopods has been analyzed [3-4]. The use of high-resolution Raman microscopy enabled the determination of the distribution of different mineral phases in the tergal cuticles of some rollers, clingers, and runners [5,6].

Fig. 1 *Androniscus dentiger* (a) and *Androniscus brentanus* (b). Contrary to the second, the first is not troglobite, is pigmented, has thick cuticle, and shows a prominent single-ommatidium eye (arrow).

Androniscus offers the unique opportunity to investigate the nature of the cuticle in cave (*A. brentanus* and more) and non-cave-dwelling species (*A. dentiger*), within the same evolutionary lineage, characterizing mineralization in the different species. The proposed investigation will also extend to including tubercles (tricorns) on the surface of the body, the nature of which, currently unknown, could be of a sensorial nature. Some preliminary observations would suggest the presence of a possible innervation at the base of these tubercles, which if confirmed, would prove their nature and function. For the characterization on the mineralization of the cuticle and tubercles in the two different species (*A. brentanus and A. dentiger*) we plan to use Energy Dispersive X-ray Analysis (SEM-EDX), Fourier-transform infrared spectroscopy (FT-IR), Raman spectroscopy and X-ray diffraction, whereas the morphology characterization will be done by means of electron microscopy techniques (SEM, TEM) and X-ray nano tomography. The benefit of using a multi-instrumental approach would allow us not only to cross compared results to verify their consistency, but also to investigate the degree of resorption/failure to develop the eye in these isopods species, allowing us to observe the presence of vestigial or residual structures, such as for example the presence of an optic nerve, in the absence of the ommatidium (eyeball).

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2. Proposed experiment

In this specific proposal we aim to use the TEM FEI instrument available at the CNR IPCB Unit for assessing the morphology of the cuticle and tubercles on a n. 1 *Androniscus brentanus and n. 1 Androniscus dentiger* isopods samples as a proof-of-concept experiment. Results of this test will be cross compared to verify consistency with data obtained by separate proposals where we request FT-IR, Raman spectroscopy, XRD, SEM-EDX, and nano-XCT measurements on the same set of samples.

3. Justification of experimental time requested

Each of the n. 2 samples of the two Oniscidean isopod species (n.1 *Androniscus brentanus and n.1 Androniscus dentiger)* will be washed for 1–2 s in double distilled water to remove tissue saline at the surface and then for 2–5 s in 100% methanol to remove water. Specimens will be air dried and stored at −20 °C until its use on the instrument. For the measurement on the TEM instrument sample will be embedded in Agar 100 resin. Ultrathin sections were contrasted with uranyl acetate and lead citrate. We envisage, after discussion with the instrument scientist, to measure n. 1 samples per day on the instrument. Hence, we request a total of 2 days of instrument time including sample preparation, set-up and calibration time.

References

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[7] Hornung, E. (2011). Evolutionary adaptation of oniscidean isopods to terrestrial life: Structural-physiological-behavioural aspects. Terrestrial Arthropod Reviews. 4: 95-130.

Experiment Proposal

Experiment number GP2024116

Principal investigator	Dr Ivano Aglietto, GrapheneUP SE, CZECH_REPUBLIC
Co-investigator (*)	Dr Gennaro Gentile, IPCB CNR, ITALY
Co-investigator	Dr Marino Lavorgna, CNR, ITALY
Co-investigator	Professor Massimo Bonini, CSGI - University of Florence, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	TEM characterization of innovative graphene-based inks for multifunctional applications
MRF Instrument	**TEM FEI**
Days requested:	3
Access Route	Direct Access
Previous GP Number:	-
Science Areas	Chemistry, Engineering, Materials
DOI:	-
Sponsored Grant	None
Sponsor:	-
Grant Title	-
Grant Number:	-
Start Date	-
Finish Date:	-
Similar Submission?	-
Industrial Links	Graphene UP
Non-Technical Abstract	The aim of this proposal is to study, using the TEM FEI equipment available at IPCB CNR Unit, the correlation between innovative Few Layers Graphene (FLGs), hybrid fillers consisting of graphene few layers modified with metal, inorganic or carbon particles, the filler spatial distribution, and the processing parameters related to screen-printing deposition technologies. The objective is to investigate how the chemical modification of FLGs, obtained directly during the process of graphene exfoliation starting from graphite, may affect the spatial distribution because of the different technologies adopted for the processing of the composites. In separate experiments RAMAN confocal microscopy, SAXS WAXD and SEM characterization of the inks will be performed.
Publications	-

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E-platform: No

Instruments	
Access Route	
Science Areas	
Sponsored Grant	
Grant Title	
Start Date	
Similar Submission?	
Industrial Links	
Days Requested:	
Previous RB Number:	
DOI:	
Sponsor:	
Grant Number:	
Finish Date:	

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Sample record sheet

Principal contact	Dr Gennaro Gentile, IPCB CNR, ITALY
MRF Instrument	**TEM FEI**
Days Requested:	3
Special requirements:	

SAMPLE

Material	FLG based ink (4 samples)	fillers for FLG based inks (4 samples)	-
Formula	C	C	-
Forms	Solid	Solid	
Volume	1 cc	1 cc	
Weight	1000 mg	1000 mg	
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	- K	-
Pressure Range	- mbar	- mbar	-
Magnetic field range	- T	- T	-
Standard equipment	None	None	-
Special equipment	-	-	-

SAFETY

Prep lab needed	No	No	-
Sample Prep Hazards	NO	NO	-
Special equip. reqs	NO	NO	-
Sensitivity to air	No	No	-
Sensitivity to vapour	No	No	-
Experiment Hazards	NO	NO	-
Equipment Hazards	-	-	-
Biological hazards	NO	NO	-
Radioactive Hazards	NO	NO	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	-

TEM characterization of innovative graphene-based hybrid fillers and their inks for multifunctional applications

Background and Context

Flexible and portable electronic devices and energy storage equipment made from conductive nanomaterials using printing or inks deposition technology have garnered significant attention due to their low-cost, high-throughput, and eco-friendly manufacturing processes over the past few decades. Electrical conductive nanostructured inks have emerged as promising candidates for designing flexible electronics because of their cost-effective synthesis methods and compatibility with current manufacturing processes.

In particular, the development of highly concentrated conductive ink using graphene powders as a raw material is seen as a promising direction. Most high-concentration graphene conductive inks are prepared from low-concentration graphene dispersions containing polymers as stabilizers. However, the solvents used in these dispersions often do not meet the requirements of the printing methods. Among various printing technologies, screen printing shows great promise for industrial-scale production due to its ability to print thick patterns with low sheet resistance (RS) on a wide range of substrates.

To realize applications in graphene-based flexible electronics, further improvement in graphene ink formulation and the development of simple, efficient post-treatment processes for printed patterns are needed. Polymers used to prevent graphene agglomeration in dispersions through steric hindrance can also adjust the rheological properties, storage performance of inks, and the flexibility and adhesion of printed patterns to substrates. New innovative fillers, based on graphene functionalized with metals or other nanoparticles, may be useful for realizing conductive inks with additional properties such as thermal, magnetic, electromagnetic shielding, optical, and catalytic properties.

Therefore, the aim of this proposal is to study, using the instrument suite of IM@IT, the correlation between innovative Few Layers Graphene (FLGs), hybrid fillers consisting of graphene few layers modified with metal, inorganic or carbon particles, the filler spatial distribution, and the processing parameters related to screen-printing deposition technologies. Hybrid fillers may be realized by combining FLGs with pristine metals or inorganic particles, but they can also be prepared by direct synthesis of hybrid graphene structures. In particular a process has been developed to cover the graphene layers with carbon nanotubes and also conductive metal fillers during the direct non-oxidative exfoliation of graphite in gas phase. This process allows to create hybrid structures of few-layer graphene with other fillers without the need of a post-functionalization process.

The objective is to investigate how the chemical modification of FLGs, obtained directly during the process of graphene exfoliation starting from graphite, may affect the morphology of filler as well as the spatial distribution of filler within the inks because of the different technologies adopted for the processing of the composites.

2. Proposed experiment

The characterization of the hybrid filler and inks will be performed as follows.

TEM characterization of the hybrid fillers and inks, performed at the IPCB CNR Unit, will offer insights into the filler morphology and assembling of nanoplatelets and spatial filler distribution within the inks.

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In separate experiment proposals, SAXS/WAXD and SEM, available at IPCB CNR Unit, will be performed on the hybrid filler and inks and will allow to evaluate the filler structure and their orientation and aggregation, contributing to the enhanced properties of the resulting inks. Furthermore, RAMAN confocal microscopy, available at the CSGI - University of Florence Unit, will provide chemical information about the modified FLGs and their interface interactions with the main components of inks.

3. Summary of previous experimental proposals or characterisation

No previous experiments have been carried out on these samples

4. Justification of experimental time requested

We have requested the TEM FEI available at the IPCB CNR Unit to characterize 8 samples (4fillers+4inks) obtained by modified FLG with metal nanoparticles, inorganic nanoparticles, multiwalled carbon nanotubes (MWCNTs) and single wall carbon nanotubes (SWCNTs). Non additivated FLG will be also characterized by comparison.

Therefore, a total of 8 samples will be analysed. After discussion with the instrument scientist, we request 3 days of TEM FEI access, for a fully and thorough characterization of the materials. The foreseen beam time accounts set up and for the data collection on the samples.

References

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Water-based and inkjet printable inks made by electrochemically exfoliated graphene,
(2019) Carbon, 149, 213-221
https://doi.org/10.1016/j.carbon.2019.04.047

TEM High Resolution

Experiment Proposal

Experiment number GP2024073

Principal investigator	Dr Umberto Pasqual Laverdura, ENEA, ITALY
Co-investigator (*)	Dr Leonardo Duranti, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Anna Paola Panunzi, Università degli Studi di Roma Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	High resolution TEM characterization of metallic nanoparticles supported on perovskite oxides as electrocatalysts for water splitting
MRF Instrument	**TEM High Resolution** — **Days requested:** 2
Access Route	Direct Access — **Previous GP Number:** -
Science Areas	Energy, Materials — **DOI:** -
Sponsored Grant	None — **Sponsor:** -
Grant Title	- — **Grant Number:** -
Start Date	- — **Finish Date:** -
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

Ni-Fe-containing perovskite oxides are known to be excellent electrocatalysts for oxygen evolution reaction (OER) in alkaline medium. Some of these oxide structures have also shown to easily undergo a structural rearrangement upon high temperature treatment in reducing environment, with metallic Ni and Fe partly segregating to the oxide surface in form of evenly distributed NiFe alloy nanoparticles. This metal segregation is commonly referred to as the exsolution phenomenon, and has the potential to dramatically affect the catalytic activity and the stability of the parent oxide. We propose to employ high-resolution transmission electron microscopy to directly observe the NiFe nanoparticles, their size, shape, and interaction with the crystal planes of the oxide lattice substrate. Moreover, TEM-EDS is crucial to follow the morphological evolution of the exsolved nanoparticles after prolonged OER operation, enabling to link it to the electrocatalytic activity variation.

Publications -

ISIS neutron and muon source — **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Leonardo Duranti, University of Rome Tor Vergata, ITALY
MRF Instrument	**TEM High Resolution** — **Days Requested:** 2
Special requirements:	

SAMPLE

Material	As prepared perovskite oxide	reduced perovskite oxide + metallic nanoparticles	Perovskite oxide reduced + metallic nanoparticles + vulcan carbon
Formula	La0.6Sr0.4Fe0.8Ni0.2O3	La0.6Sr0.4Fe0.8Ni0.2O3 + NiFe	La0.6Sr0.4Fe0.8Ni0.2O3 + NiFe + C
Forms	Friable powder	Friable powder	Friable powder
Volume	cc	cc	cc
Weight	10 mg	10 mg	10 mg
Container or substrate	eppendorf vials	eppendorf vials	eppendorf vials
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	- K	- K
Pressure Range	- mbar	- mbar	- mbar
Magnetic field range	- T	- T	- T
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	Yes	Yes
Sample Prep Hazards	Samples contain Nickel	Samples contain Nickel	Samples contain Nickel
Special equip. reqs	-	-	-
Sensitivity to air	No	No	No
Sensitivity to vapour	No	No	No
Experiment Hazards	samples may show ferromagnetism	samples may show ferromagnetism	samples may show ferromagnetism
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	Returned to user by instrument scientist (when inactive)	Returned to user by instrument scientist (when inactive)

1. Background and Context

Water electrolysis is an environmentally friendly way of producing hydrogen, employing energy derived from renewable sources. Proton exchange membrane water electrolyzers (PEMWE) operating in acidic conditions are the state-of-the-art device, ensuring high current density and high hydrogen purity.

Unfortunately, PEMWE require critical and costly platinum group metal-based electrocatalysts. On the other hand, traditional alkaline water electrolyzers (AWE) suffer from low current density and poor gas purity [1]. Recently developed anion exchange membranes water electrolyzers (AEMWE), allow to obtain high current density and high hydrogen purity with cost-effective electrocatalysts.

The oxygen evolution reaction (OER), occurring at the anode side of the electrolyzer, represents the bottleneck of the overall water splitting process [2]. Expensive and critical oxides RuO_2 and IrO_2 are the state-of-the-art OER electrocatalysts. Among the PGM-free alternatives, Ni and NiFe-containing materials exhibit the best performance. Perovskite oxides have aroused growing interest as these mixed oxides are cost-effective and have an easily tailorable structure to maximize the catalytic activity.

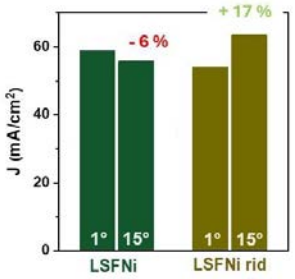

Figure 1- Maximum current density at the end of the 1° and 15° OER cycle using the as prepared LSFNi perovskite and the reduced LSFNi.

We tested $La_{0.6}Sr_{0.4}Fe_{0.8}Ni_{0.2}O_{3-\delta}$ (LSFNi) as OER electrocatalyst before and after high temperature reduction (400 °C in 5%H_2/Ar for 10 h). After reduction, LSFNi undergoes exsolution: some Ni and Fe cations are reduced to their metallic state and they nucleate at the oxide surface in form of uniformly dispersed nanoparticles. After 15 OER cycles the as prepared perovskites revealed a -6% loss, on the other hand, the presence of exsolved nanoparticles enhanced the stability of the electrocatalyst with a +17% current density increase after 15 OER cycles (Figure 1).

Through this proposal we aim at deepening our understanding of the existing link between the electrochemical properties and the morphology of the perovskite surface. This is a pivotal aspect in the research area of our group, the Solid Oxide Research Group at Tor Vergata: the development of innovative, non-critical materials for electrolyzers and fuel cells electrodes. Our research is currently being funded by several national grants, besides a full professor and a research associate, three PhD students and one post-doctoral researcher are involved. Concerning this research topic, fruitful collaborations are on-going with the Italian National Agency for New Technologies, Energy and Sustainable Economic Development ENEA and other Universities.

2. Proposed experiment

Our purpose is to unveil the reason behind the performance decrease/increase observed with the as prepared/exsolved perovskites, respectively. Using the Thermo Scientific Talos F200X STEM energy dispersive X-ray spectroscopy (EDS), available at ISIS@MACH ITALIA, our purpose is to inspect the LSFNi sample in the following conditions:

- as prepared

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- after reduction treatment and NiFe nanoparticles exsolution
- after 1° OER measurement cycle
- after 15° OER measurement cycle

The TEM/S-TEM can highlight the probable formation of a core-shell system in which the exsolved nanoparticle is surrounded by highly active hydroxides or oxo-hydroxides. The EDS will provide information on the nature of such core shell system, that we except to be only few nm thick. The obtained morphological-elemental characterization will be correlated with electrochemical results.

3. Summary of previous experimental proposals or characterisation

Previous characterizations were performed on exsolved perovskite oxide samples, using the field emission SEM LEO SUPRA available among the medium-range facilities of the ISIS@MACH ITALIA laboratories at University of Rome Tor Vergata. Through the FE-SEM morphological analysis the presence of uniformly dispersed nanoparticles was assessed (Figure 2a). It was not possible to accurately assess the actual size of the exsolved metallic particles, estimated around 4-5 nm (Figure 2b).

A high resolution TEM-EDS analysis would disclose further insights on the nature of the nanoparticles, how far they are embedded in the perovskite oxide substrate and whether they arise from preferential crystalline orientations.

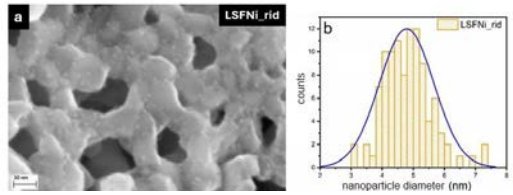

Figure 2 – a) FE-SEM micrograph of LSFNi perovskite oxide surface after high temperature reduction treatment; b) nanoparticle diameter assessment.

4. Justification of experimental time requested

Thermo Scientific Talos F200X STEM energy dispersive X-ray spectroscopy (EDS), available at ISIS@MACH ITALIA is fundamental for our morphological analysis. We plan to measure 4 samples and requested two days of access to the facility, with the purpose of measuring two samples per day. The reason for that is based on previous experience with these samples at the TEM.

References

[1] Hua, Daxing, et al. "Development of anion exchange membrane water electrolysis and the associated challenges: a review." ChemElectroChem 10.1 (2023): e202200999.

[2] Song, Jiajia, et al. "A review on fundamentals for designing oxygen evolution electrocatalysts." Chemical Society Reviews 49.7 (2020): 2196-2214.

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Experiment Proposal

Experiment number GP2024114

Principal investigator	Dr Caterina c/o LENS Credi, Italian National Research Council, ITALY		
Co-investigator (*)	Dr Caterina Dallari, LENS, ITALY		
Co-investigator	Miss Luisa Ponticelli, University of Florence, ITALY		
Co-investigator			
Co-investigator			
Co-investigator			
Co-investigator			
Co-investigator			
Co-investigator			
Experiment title	Advanced Nanoparticle Probes for Early Disease Detection in Biofluids		
MRF Instrument	**TEM High Resolution**	**Days requested:** 4	
Access Route	Direct Access	**Previous GP Number:** -	
Science Areas	Chemistry, Materials, Physics	**DOI:** -	
Sponsored Grant	Yes	**Sponsor:** Other	
Grant Title	I-PHOQS - Integrated Infrastructure Initiative in Photonic and Quantum Science	**Grant Number:** project financed by the EU next generation PNRR action	
Start Date	01/12/2022	**Finish Date:** 30/11/2025	
Similar Submission?	-		
Industrial Links	-		

Non-Technical Abstract

Our research focuses on developing and refining innovative probes designed to selectively detect specific analytes in various biological fluid samples. A key objective is to leverage these probes, particularly metallic nanoparticles, for the optical analysis of biofluids such as saliva, blood, urine, and cerebrospinal fluid. This approach aims to facilitate the early detection of a wide range of diseases. By interacting with biofluids, the nanoparticles act as both signal transducers and enhancers, significantly amplifying the response. This enhanced signal is then processed to extract valuable information about the patient's physiological state, potentially providing insights into disease progression or risk factors well before clinical symptoms arise. This method holds great promise for improving diagnostic accuracy and enabling more proactive and personalized healthcare.

Publications

Ventisette I., et al "Gold-Hydrogel Nanocomposites for High-Resolution Laser-Based 3D Printing of Scaffolds with SERS-Sensing Properties", ACS App Biomat, 2024

Dallari C., et al "Multilayered Bioorthogonal SERS Nanoprobes Selectively Aggregating in Human Fluids" ACS Sensors, 2023

Dallari C., et al "Gold Nanostars Bioconjugation for Selective Targeting and SERS Detection of Biofluids" Nanomaterials, 2021

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E-platform: No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Caterina Dallari, LENS, ITALY		
MRF Instrument	**TEM High Resolution**	**Days Requested:** 4	
Special requirements:			

SAMPLE

Material	Gold nanoparticles of various shape	-	-
Formula	-	-	-
Forms	Liquid		
Volume	0.5 ml		
Weight	0.00025 mg		
Container or substrate	Eppendorf	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	RT - K	-	-
Pressure Range	PT - mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Removed By User	-	-

"Advanced Nanoparticle Probes for Early Disease Detection in Biofluids"

***General Instructions on format of the Experimental Proposals* for a MRF Proposal:**
- ***Keep the top and bottom margins** in this document as **there are***
- *The science case must be **no longer than two A4 pages**,* **figures and references included,** *and font must be Helvetica 12 (MAX)- Helvetica 11,5 (MIN) and* about a total of max **4000 characters (spaces included)**

1. Background and Context

Our research focuses on developing and refining innovative probes designed to selectively detect specific analytes in various biological fluid samples. A key objective is to leverage these probes, particularly metallic nanoparticles, for the optical analysis of biofluids such as saliva, blood, urine, and cerebrospinal fluid. This approach aims to facilitate the early detection of a wide range of diseases. By interacting with biofluids, the nanoparticles act as both signal transducers and enhancers, significantly amplifying the response. This enhanced signal is then processed to extract valuable information about the patient's physiological state, potentially providing insights into disease progression or risk factors well before clinical symptoms arise. This method holds great promise for improving diagnostic accuracy and enabling more proactive and personalized healthcare.

In order to deeply characterise the synthesized nanoparticles, direct measures of the shape and morphology through transmission electron microscopy will be of crucial importance. With this information, we can confirm the successful synthesis and functionalization of the nanoprobes.

Our research programme is supported by the "Integrated infrastructure initiative in photonic and quantum sciences—I-PHOQS" project financed by the EU next generation PNRR action; PRIN 2022b; Twinning for excellence of the Serbian Research Center for quantum biophotonics" – BioQantSense, project funded thanks to the call WIDERA Horizon Europe.

2. Proposed experiment

The synthesized nanoparticles are initially characterized using UV-Visible spectroscopy and Dynamic Light Scattering to assess their plasmonic properties, size and polydispersity, respectively. Zeta potential analysis is then performed to provide insights into the surface charge of the colloidal dispersion. Following these preliminary evaluations, transmission electron microscopy (TEM) and scanning electron microscopy (SEM) are employed to confirm the hypothesized size, shape, and structural features of the fabricated metallic nanosystems. Moreover, EDX measurements are needed to evaluate the chemical composition of the nanoparticles. Results given will be crucial to decide how to proceed with the synthesis and modification of surface chemistry of such nanoparticles.

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Electron microscopy is an exceptionally powerful technique for studying materials at the nanoscale, as it provides valuable information of the material distribution as well as of the chemical composition that would be otherwise impossible to directly characterize.

The idea will be then to analyze the TEM and SEM data by statistically measuring the mean particle size, which will allow to determine the average dimensions. Additionally, the particle shape will be assessed and the actual dimensions will be evaluated to ensure an accurate understanding of the nanoparticle morphology.

4. Justification of experimental time requested

Our objective is to make significant advancements in the nanosystems we are currently developing by achieving better size dispersity and optimizing functionalization strategies. To accomplish this, we plan to implement and test these upgrades almost once a month over the six-month period, requiring a total of **four measurement sessions**. We have requested access to the specific MRFs instruments because they offer the comprehensive suite of high-resolution imaging and analytical capabilities essential for our research. The combination of **SEM**, **TEM**, **HR-TEM**, and **EDX** analyses available at the MRFs facility is critical for accurately evaluating both the morphology and chemical composition of our nanoparticle samples. In particular, we will have 6 nanoparticle samples at different stages of synthesis, which is crucial for understanding the relationship between synthesis parameters and nanoparticle characteristics.

Experiment Proposal

Experiment number GP2024120

Principal investigator (*)	Dr Anna Maria Ferretti, CNR SCITEC, ITALY
Co-investigator	
Co-investigator	Dr Fulvio Bellato, CNR, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Morphological and microanalytical characterization of electronspun porous high-entropy spinel oxide nanofibers containing the following ions: Fe, Co, Ni, Zn, and Nd
MRF Instrument	**TEM High Resolution** — **Days requested:** 3
Access Route	Direct Access — **Previous GP Number:** -
Science Areas	Chemistry, Materials — **DOI:** -
Sponsored Grant	None — **Sponsor:** -
Grant Title	- — **Grant Number:** -
Start Date	- — **Finish Date:** -
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

High entropy materials (HEM) consist of multicomponent solid solutions, where the multicomponents are not homogeneous in the lattice. High-entropy oxides (HEOs) have recently been prepared as nanofibers (NFs) through the electrospinning technique, which is suitable for widespread industrial production. In particular, the High Entropy Spinel Oxide -NFs (HESO-NFs) display rich and diverse magnetic behaviors as a function of the composition and can be used as building blocks of next-generation electromagnetic devices, in magnetic sensors and flexible magnets.

The objective of the requested analysis is to obtain information about the composition and structure of the HESO-NFs to correlate to their magnetic properties.

We plan to characterize 3 samples of HESO-NFs containing (Fe0.2, Co0.2, Ni0.2, Zn0.2, Nd0.2x)3O4 where x= 0.6,1,1.33 performing the following analysis: TEM and HR-TEM images, Electron Diffraction, EDX maps, and/or EDX line scan of 4-5 NFs

Publications -

ISIS neutron and muon source	**E-platform:** No
Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Anna Maria Ferretti, CNR SCITEC, ITALY
MRF Instrument	**TEM High Resolution** — **Days Requested:** 3
Special requirements:	

SAMPLE

Material	High entropy Spinel oxide nanofibers containing Fe,Co,Ni,Zn,Nd,O	High entropy Spinel oxide nanofibers containing Fe,Co,Ni,Zn,Nd,O	High entropy Spinel oxide nanofibers containing Fe,Co,Ni,Zn,Nd,O
Formula	(Fe0.2,Co0.2,Ni0.2,Zn0.2,Nd0.2y)x,O4 x=3,4 y=0.6	(Fe0.2,Co0.2,Ni0.2,Zn0.2,Nd0.2y)x,O4 x=3,4 y=1	(Fe0.2,Co0.2,Ni0.2,Zn0.2,Nd0.2y)x,O4 x=3,4 y=1.3
Forms	Friable powder	Friable powder	Friable powder
Volume	cc	cc	cc
Weight	mg	mg	mg
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	- K	- K
Pressure Range	- mbar	- mbar	- mbar
Magnetic field range	- T	- T	- T
Standard equipment	None	None	None
Special equipment	-	-	-

SAFETY

Prep lab needed	No	Yes	Yes
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	No	No
Sensitivity to vapour	No	Yes	Yes
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	no, there aren't	-	-
Radioactive Hazards	No there aren't	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	Returned to user by instrument scientist (when inactive)	Returned to user by instrument scientist (when inactive)

Morphological and microanalytical characterization of electronspun porous high-entropy spinel oxide nanofibers containing the following ions: Fe, Co, Ni, Zn, and Nd

High entropy materials (HEM) consisting in multicomponent solid solutions, where the components are distributed in not homogeneous way in the lattice. The High Entropy Oxides (HEOs) recently have been prepared in the form of nanofibers (NFs) through the electrospinning technique, which is a cost-effective and an easy-to-use technique, that is suitable for a widespread industrial production of NFs. We focused on the High Entropy Spinel Oxide -NFs (HESO-NFs) because this class of NFs display a rich and diverse magnetic behavior as a function of the composition. The incorporation of multiple metal elements (Ni, Cu, Mn, Co, Zn, Fe, Nd, Cr, etc.) in equimolar ratios leads to a high-entropy state, which enhances their stability and functional properties. From previous works we can assert that the HESO-NFs can be used as building block of next generation electromagnetic device, in magnetic sensor and flexible magnet.
The samples that we plan to study contain $(Fe_{0.2},Co_{0.2},Ni_{0.2},Zn_{0.2},Nd_{0.2y})_xO_4$ where x=3,4 and y = 0.6,1,1.33.

2. Proposed experiment

Analyzes are necessary to obtain information about the structure and composition of the HESO-NFs and to correlate them to their magnetic properties.
We plan to characterize 3 different samples of HESO-NFs containing $(Fe_{0.2}, Co_{0.2}, Ni_{0.2}, Zn_{0.2}, Nd_{0.2y})_x O_4$ where x=3,4 y=0.6,1,1.33 and treated at different calcination temperature. The HESO-NFs are polycrystalline, so we are interested in characterizing them from the structural and from the chemical point of view, identifying crystal structure of the crystals and the elements distribution inside the fibers
Experimental plan for each sample:Morphological study of the NFs,HR-TEM and Electron diffraction, EDX spectrum, EDX map and/or EDX line-scan

3. Summary of previous experimental proposals or characterisation

We have just analyzed similar samples, but with different chemical composition here there is a list of publication regarding similar samples and an example of images/data we suppose to collect.
Adv. Funct. Mater. **2024**, *34*, 2306375
Journal of The Electrochemical Society, **2024,** 171, 060509
Small **2023**, *19*, 2304585
Phys. Chem. Chem. Phys., 2023, 25, 2212–2226

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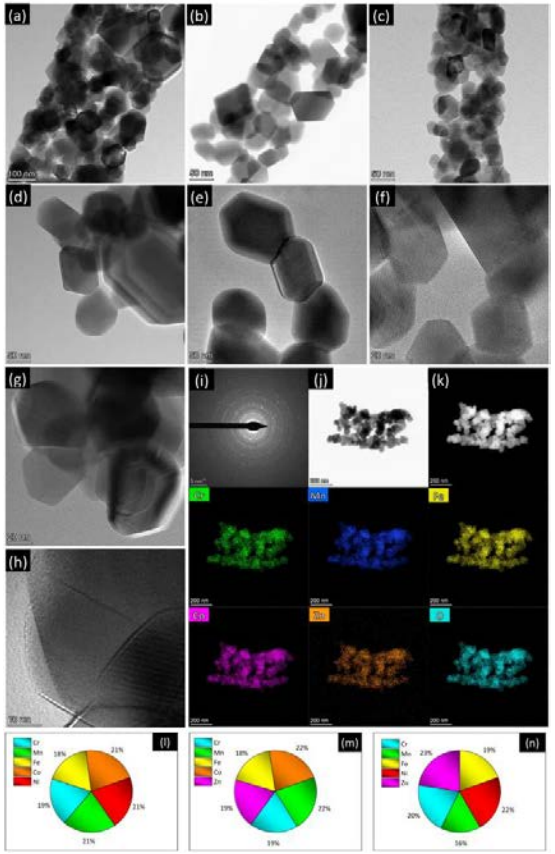

Figure 1 Results of the HRTEM/STEM/EDX analysis on samples (a, d, g, h and l) CoNi, (b, e, i–k and m) CoZn and (c, f and n) NiZn. (a–h) HRTEM images; (i) SAED pattern for (j) an isolated NF, (k) corresponding STEM/EDX elemental maps and (l–n) relative concentrations of the TMs
Phys. Chem. Chem. Phys., 2023, 25, 2212–2226

4. Justification of experimental time requested

The machine time has been estimated considering that the acquisition of compositional maps using EDX might take time.

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Experiment Proposal

Experiment number GP2024137

Principal investigator (*)	Dr Francesca Migliorini, CNR, ITALY	
Co-investigator	Dr SILVANA DE IULIIS, CNR-ICMATE sede di Milano, ITALY	
Co-investigator		
Co-investigator		
Co-investigator		
Co-investigator		
Co-investigator		
Co-investigator		
Co-investigator		
Experiment title	Effect of laser irradiation on soot nanostructure	
MRF Instrument	**TEM High Resolution**	**Days requested:** 2
Access Route	Direct Access	**Previous GP Number:** no
Science Areas	Materials, Technique Development	**DOI:** -
Sponsored Grant	Yes	**Sponsor:** Other
Grant Title	UPcycling SOOT for sustainable nanocomposites-based wearable sensors (UP - SOOT)	**Grant Number:** PRIN2022SEYHXP
Start Date	28/09/2023	**Finish Date:** 27/09/2025
Similar Submission?	-	
Industrial Links	-	

Non-Technical Abstract

The concept of circular economy is generating renewed interest in combustion-generated carbon nanoparticles, which can be transformed from harmful pollutants into valuable resources. These particles possess unique properties that can be effectively utilized and enhanced in various applications. In this context, controlled laser irradiation stands out as an efficient method to modify their physico-chemical properties, enabling the creation of customized structures for specific applications. The main objective of this proposal is to investigate the specific morphological and nanostructural changes induced by laser irradiation, using high-resolution transmission electron microscopy (HRTEM) analysis. The ultimate goal is to establish laser-based heating as a practical method for continuous material processing and synthesis.

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Francesca Migliorini, CNR, ITALY	
MRF Instrument	**TEM High Resolution**	**Days Requested:** 2
Special requirements:		

SAMPLE

Material	carbon, hydrogen, oxygen	carbon, hydrogen, oxygen	carbon, hydrogen, oxygen
Formula	-	-	-
Forms	Solid	Solid	Solid
Volume	cc	cc	cc
Weight	mg	mg	mg
Container or substrate	TEM grid	TEM grid	TEM grid
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	- K	- K
Pressure Range	- mbar	- mbar	- mbar
Magnetic field range	- T	- T	- T
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	Yes	Yes
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	No	No
Sensitivity to vapour	No	No	No
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	Returned to user by instrument scientist (when inactive)	Returned to user by instrument scientist (when inactive)

1. Background and Context

Carbon nanoparticles produced by incomplete combustion processes, commonly known as soot, have attracted considerable interest for both a health and environmental concern and a potential resource in a circular economy. The unique properties of soot, including its high surface-to-volume ratio, excellent electrical conductivity, thermal stability, and versatility in processing, make it a promising candidate for various innovative applications. In the context of the Italian Grant PRIN2017PJ5XXX, we observed that soot can be modified via controlled laser irradiation, which impacts its optical, electronic, and physicochemical properties [1-2]. The findings highlight the necessity for extensive data on the influence of laser treatment on soot morphology and nanostructure. High-resolution transmission electron microscopy (HRTEM) is an essential instrument for analyzing the graphitic structure of carbon nanoparticles. This technique enables accurate measurements of the size, form, and positioning of graphitic edges, which are crucial for evaluating soot molecular organization, and thus assessing the reactivity of carbon of interest in several potential applications.
This knowledge will be crucial for an ongoing Italian Grant (PRIN2022SEYHXP- UPcycling SOOT for sustainable nanocomposites-based wearable sensors), focused on the application of soot for fabrication of sustainable nanocomposites-based wearable sensors for health care monitoring.

2. Proposed Experiment

The proposed experiment aims at investigating the impact of laser exposure on soot nanostructure and, in particular, collect comprehensive data on the morphological and structural changes before and after exposure to a Nd:YAG laser.
For irradiation, two laser fluences, 220 and 400 mJ/cm², are considered, which are expected to induce different thermal effects on the particles, resulting in observable changes in their properties. After irradiation, the particles will be directly collected on a TEM grid for further analysis. For this reason, we request the access to the High Resolution TEM at ISIS@MACH ITALIA. HRTEM is necessary for examining the detailed morphological and nanostructural alterations of particles post-irradiation. Significant differences are expected comparing pristine and irradiated particles, which can be quantified by using advanced tools, as an open-source automatic image processing program for morphological characterization [3] and lattice fringe analysis for a semi-quantitative assessment of the arrangement of soot graphitic layers to measure fringe length, tortuosity, and interlayer spacing [4].

3. Summary of previous experimental proposals or characterisation

Pristine and laser irradiated soot have already been characterized in terms of optical and physicochemical properties, by performing on-line laser extinction measurements and ex-situ FTIR and Raman analysis [1-2]. It was observed that laser irradiation promotes strong variation in soot optical properties and in the chemical structure of the particles, indicating that particles experience intricate modification procedures under irradiation. Hence, the HRTEM analysis suggested in this experiment is fundamental for enhancing the understanding of these phenomena.

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4. Justification of instrument time request

The access to the HR-TEM is requested to resolve detailed morphological and nanostructural properties of the following samples:

1. *Pristine soot;*
2. *Soot irradiated at 220 mJ/cm^2;*
3. *Soot irradiated at 400 mJ/cm^2.*

For statistical analysis we need for each sample:

- *40 images at low resolution for morphological analysis*
- *15 images at high resolution for the nanostructure (about 2000/3000 fringes)*

Therefore, we estimate an instrument time of approximately 2 working days.

[1] F. Migliorini, S. Belmuso, D. Ciniglia, R. Dondè, S. De Iuliis, Applied Physics B 129 (2023) 133.
[2] F. Migliorini, R. Dondè, A. Lucotti, M. Fasoli, M. Tommasini, S. De Iuliis, Journal of Aerosol Science 182 (2024) 106440
[3] T.A. Sipkens, R. Dastanpour, U. Trivanovic, H. Nikookar, S.N. Rogak, Journal of Open Source Software, 9(99) (2024), 6416.
[4] P. Pré, G. Huchet, D. Jeulin, J.-N. Rouzaud , M. Sennour , A. Thorel , Carbon 52 (2013) 239.

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Experiment Proposal

Experiment number GP2024140

Principal investigator (*)	Dr Francesco Rizzo, ENEA, ITALY		
Co-investigator	Dr Laura Piperno, ENEA, ITALY		
Co-investigator			
Co-investigator			
Co-investigator			
Co-investigator			
Co-investigator			
Co-investigator			
Co-investigator			
Experiment title	Innovative strategies for Improving the pinning efficiency of YBCO thin films		
MRF Instrument	**TEM High Resolution**	**Days requested:** 4	
Access Route	Direct Access	**Previous GP Number:** GP2023018	
Science Areas	Energy, Materials, Physics	**DOI:** -	
Sponsored Grant	None	**Sponsor:** -	
Grant Title	-	**Grant Number:** -	
Start Date	-	**Finish Date:** -	
Similar Submission?	-		
Industrial Links	-		

Non-Technical Abstract

The samples object of this proposal are YBa2Cu3O7-x (YBCO) films with nano-engineered pinning
landscapes the transport property performances improvement in the low temperature and high magnetic field regime. These nano-composite YBCO films are obtained by Pulsed Laser Deposition
(PLD) on single crystals combining the functionalization of the substrate with the columnar growth of secondary phases in the film matrix.
This growth occurs on substrates previously decorated with a uniform distribution of CeO2 nanoparticles, homogeneous in diameter
and height. The combined use of the substrate functionalization and of the secondary phase nanoengineering in the YBCO matrix
greatly strengthen the pinning capabilities under applied magnetic field.
The key objective is to improve the understanding of the most suitable defect landscape in terms of pinning centres shape, dimension, density and distribution
in YBCO. An in-depth structural analysis of these films with HR-TEM is therefore required.

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Francesco Rizzo, ENEA, ITALY		
MRF Instrument	**TEM High Resolution**	**Days Requested:** 4	
Special requirements:			

SAMPLE

Material	YBa2Cu3O7-x on SrTiO3 single crystal and metallic substrates	YBa2Cu3O7-x on SrTiO3 single crystal and metallic substrates	YBa2Cu3O7-x on SrTiO3 single crystal and metallic substrates
Formula	YBa2Cu3O7-x	YBa2Cu3O7-x	YBa2Cu3O7-x
Forms	Single crystal	Single crystal	Single crystal
Volume	0.056 cc	0.056 cc	0.056 cc
Weight	10 mg	10 mg	10 mg
Container or substrate	SrTiO3 single crystal, hastelloy substrate	SrTiO3 single crystal, hastelloy substrate	SrTiO3 single crystal, hastelloy substrate
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	275 - 300 K	275 - 300 K	275 - 300 K
Pressure Range	1013 - 1050 mbar	1013 - 1050 mbar	1013 - 1050 mbar
Magnetic field range	0 - 0 T	0 - 0 T	0 - 0 T
Standard equipment	Do Not Know	Do Not Know	None
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	Yes	Yes
Sample Prep Hazards	NO	NO	NO
Special equip. reqs	-	NO	-
Sensitivity to air	No	No	No
Sensitivity to vapour	Yes	Yes	Yes
Experiment Hazards	NO	NO	NO
Equipment Hazards	-	-	-
Biological hazards	NO	NO	NO
Radioactive Hazards	NO	NO	NO
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	Returned to user by instrument scientist (when inactive)	Returned to user by instrument scientist (when inactive)

Title of the proposal: Innovative strategies for Improving the pinning efficiency of YBCO thin films

Acronym of the proposal: INSIGHT

Background and Context

For the last quarter century, the study of pinning centres in superconducting thin films has been a fundamental topic in material science[1–4], with the aim of producing high quality coated-conductors by enhancing the critical current density (J_c) of the superconductor when a magnetic field is applied. J_c is ultimately related to the dissipation due to the onset of magnetic flux vortex motion. In $YBa_2Cu_3O_{7-x}$ (YBCO) films, one of most mature high temperature superconductors for practical applications, artificially formed columnar nano-sized structural defects can act as effective pinning centres for magnetic flux vortices in a wide range of temperature and magnetic field[5] The effect on J_c provided by mixed addition of secondary phases in the matrix of YBCO films grown by pulsed laser deposition (PLD) is well known[5–8] : the interaction between the dopant phases during the film growth stage dramatically changes the final film microstructure with a significant enhancement of J_c. In particular, this effect has been investigated with Ta- and Nb-based double perovskites, $Ba_2Y(Nb/Ta)O_6$ (BYNTO), showing that a variety of defects, with peculiar shapes and dimensions, can be generated and controlled in YBCO nanocomposites depending on the film growth conditions (Fig.1).

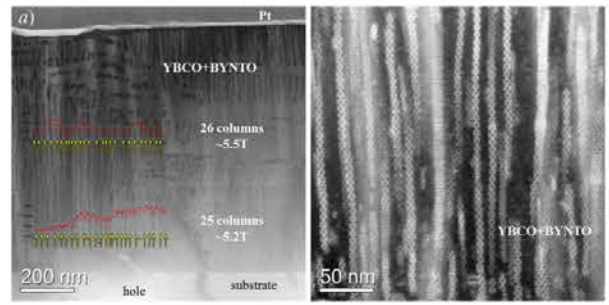

Figure 1 : ABF STEM overview of the film showing the BYNTO columnar structures developed across the entire film thickness. Yellow arrows identify BYNTO columns in three different regions[4]

On the other hand, substrate functionalization has also been recently studied for YBCO films grown by both PLD and chemical solutiond deposition (CSD) [9–11]. In both cases, a large amount of defects was generated in the films, as an effect of the interference of the oxide nanoparticles with the growth process (Fig.2). These defects were able to boost the material properties acting as pinning centres for magnetic flux vortexes. Thus, the combination of these two strategies could open up new microstructural scenarios in YBCO films grown by PLD, possibly achieving a superior vortex pinning capability, especially in the low temperature (*T*) and high applied magnetic field (*B*) regime. Additionally, the structural properties of some of these highly defected YBCO films were further modified through the irradiation with Au ions (250 MeV) with an incident beam direction inclined with respect to the growth direction of the secondary phase. By tuning the irradiation conditions (i.e., energy, fluence and ions beam direction), it was possible to modify the pinning scenario with the introduction of additional columnar defects that are effective in changing the angular behaviour of the critical current density, reducing the anisotropy.

The emerging interest in high temperature superconductor materials for nuclear fusion and high energy physics applications has motivated new research activities on YBCO aimed at the development of YBCO coated conductor for high field and low *T* applications[2,7,12–14]. In perspective of a commercial exploitation of the nuclear fusion energy, YBCO based magnet technology offers a realistic vision for more compact (higher field) and more efficient (higher temperature) fusion devices if compared to the currently available low temperature superconductor Nb-based technology that is intrinsically limited to 4.2 K and to a maximum magnetic field of 13 T. Similarly, YBCO would offer a suitable and feasible opportunity for the development of very high field magnets (> 20 T) for future high energy particle accelerators. However, at low temperature conditions YBCO has yet to be sistematically investigated so and thus there is insufficient understanding of the most suitable defect landscape, neither of the required optimum pinning centres nor of the materials engineering needed to create them.

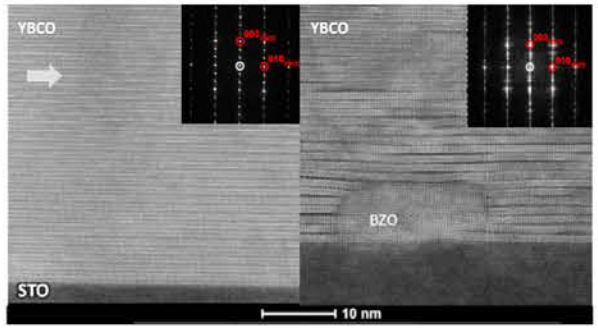

Figure 2 : HAADF images of a YBCO films grown on a $BaZrO_3$ (BZO) decorated template, showing the high density of defects generated around the oxide nanoparticle[8].

Proposed Experiment

A detailed investigation of the microstructure of nanocomposite YBCO films deposited by PLD on decorated substrates is proposed, with and without the further Au ion irradiation process of the YBCO thin films. An extensive high-resolution TEM (HRTEM) analysis is essential to clarify the role of different kind of nanoparticles used in the functionalization of the substrate for promoting or, on the contrary, hindering the expected nanocolumnar growth of the secondary phase in the YBCO film matrix. The possibility to locally explore and compare the film matrix at the different interfaces (i.e. film-substrate, nanoparticles-film, film-secondary phase and nanoparticles-secondary phase) will help in understanding the growth mechanisms of those highly defective structures. In this perspective, the energy dispersive x-ray mapping analysis (EDX) is a fundamental tool to determine the compositions and to elucidate the interaction of nanoparticles and secondary phase during the film growth.

The obtained microstructural information will be coupled with the transport properties, measured as a function of the applied magnetic field intensity and direction and of the temperature, to find the structural origin of the observed enhancement of the pinning capabilities and an explanation for the observed in field/temperature behaviour. In particular, the samples object of this proposal have already been fully characterized in terms of J_c properties in order to be able to clarify the mechanisms of segregation and self-assembly of secondary phases in the presence of the decorated

Experiment Proposal

Experiment number GP2024152

Principal investigator	Dr Mohsin Muhyuddin, University of Milano-Bicocca, ITALY
Co-investigator (*)	Professor CARLO SANTORO, University of Milano-Bicocca, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Influence of pyrolysis conditions on the Mn-incorporated Fe-N-Cs
MRF Instrument	**TEM High Resolution**
Access Route	Direct Access
Science Areas	Chemistry, Energy, Materials
Sponsored Grant	None
Grant Title	-
Start Date	-
Similar Submission?	-
Industrial Links	-

Days requested: 3
Previous GP Number: -
DOI: -
Sponsor: -
Grant Number: -
Finish Date: -

Non-Technical Abstract

Oxygen reduction reaction (ORR) is a cathode-side reaction and is considered as a critical bottleneck in the commercialization of fuel cells. To improve ORR's kinetics, expensive Pt-based electrocatalysts (ECs) are used. Meanwhile, transition metal-nitrogen-carbons (M-N-Cs) with atomically dispersed active sites are emerging as reliable ECs to replace Pt where Fe-N-Cs are of key interest. The limited catalytic activity of Fe-N-Cs can be improved by incorporating a second metal i.e. Mn, however, the pyrolysis remains indispensable to produce such ECs which decides the morphology of the evolved ECs. Therefore, the morphologies of the monometallic and bimetallic Fe-N-Cs as a function of pyrolysis parameters i.e. temperatures and atmosphere and choice of precursors will be analyzed via Thermo Scientific Talos F200X STEM. With HRTEM/STEM and EDS, the Fe and Mn atomic level distribution of active motives, the nature of the metallic nanoparticles and carbon structures will be studied.

Publications -

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E-platform: No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Professor CARLO SANTORO, University of Milano-Bicocca, ITALY
MRF Instrument **TEM High Resolution** **Days Requested:** 3
Special requirements:

SAMPLE

Material	carbons functionalized with iron and/or manganese phethalocynine and porphyrins, pyrolyzed at different temperatures and under different atmospheres	-	-
Formula	-	-	-
Forms	Solid		
Volume	10 cc		
Weight	10 mg		
Container or substrate	Vials	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	Do Not Know	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Influence of pyrolysis conditions on the Mn-incorporated Fe-N-Cs

1. Background and Context

Oxygen reduction reaction (ORR) being a critical bottleneck in the commercialization of fuel cells holds significant importance from the perspective of green energy. The slow ORR kinetics are typically dealt with by scarce and overpriced Pt-based electrocatalysts (ECs). Meanwhile, transition metal-nitrogen-carbons (M-N-Cs) with atomically dispersed active sites are emerging as reliable ECs to replace Pt where Fe-N-Cs are of key interest. However, the catalytic activity and selectivity of Fe-N-Cs are still inferior to Pt. One of the effective ways to improve the performance is to develop bimetallic Fe-N-Cs. For example, Mn addition in the Fe-N-Cs could help in optimizing the electrocatalytic activity. Such monometallic and bimetallic M-N-Cs can be developed by functionalizing the high surface area carbon support i.e. Ketjenblack with metal phthalocyanines (MPc) and porphyrins (MPor) or by mixing metallic salts and N-rich organic precursors where pyrolysis remained indispensable to evolve a robust EC. The pyrolysis parameters i.e. temperature (T) and atmosphere (A) have a marked influence on the morphological attributes (graphitic and amorphous domains, defects and metallic species) of the evolved ECs that ultimately dictate the ORR activity. Therefore, it is essential to decipher the role of metal incorporation in the Fe-N-C electrocatalysts throughout pyrolysis under varying T and A. The role of Fe and Mn on the morphological distribution of M-N_x sites while restricting or controlling the size/shape of metallic nanoparticles (NPs) along with modifications in carbon structures is crucial to understand. Such aspects should be elucidated in the consideration of the nature of precursors used i.e. MPc, Mpor, or Metal salt and pyrolysis parameters.

2. Proposed experiment

Morphologies of the monometallic and bimetallic Fe-N-Cs with varying pyrolysis T (400, 600 & 800 ºC) and A (Ar and Ar/H_2) will be analysed via Thermo Scientific Talos F200X STEM. With STEM and EDS, the Fe and Mn atomic level distribution of active motives and the nature of the metallic NPs will be studied. Using HRTEM lattice fringes graphitic defects and Mn/Fe species will be revealed whereas the HAADF-STEM mode along with EDS mapping will help in understanding the dispersion of evolved species as a function of the nature of precursor used, pyrolysis T and A. Moreover, SEAD patterns can provide crystallographic details of moieties.

3. Summary of previous experimental proposals or characterisation

Using HR-TEM and HAADF-STEM, influence of pyrolysis parameters in the development of Fe-N-Cs with Mn incorporations will be studied. Recently, we have observed the influence of pyrolysis T and A on the evolution of site structures of FePc-derived ECs via advanced characterizations like HRTEM and in-situ/ex-situ XAS [1]. It was found that the higher temperatures cause the formation and growth of NPs whereas the nature of the pyrolysis atmosphere decides the morphology of the evolved NPs. For instance, the formation of core-shell NPs under Ar/H_2 and Fe_3O_4 NPs under Ar (Fig.1)[1].

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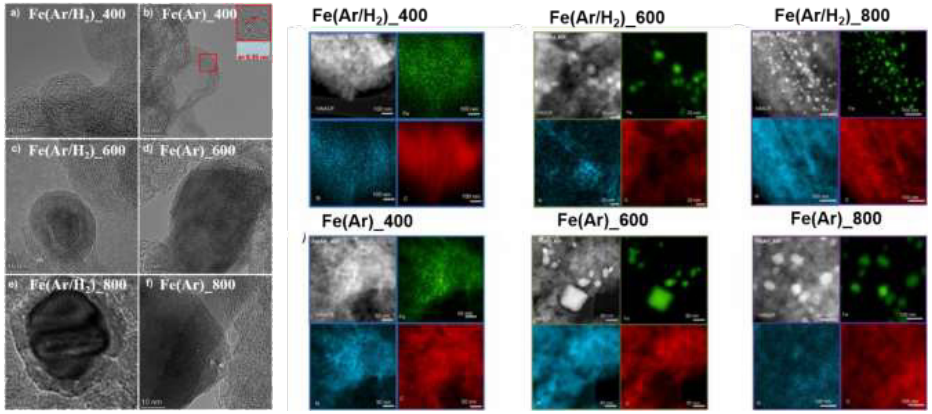

Fig. 1[1]

4. Justification of experimental time requested

We request the use of F200X STEM to understand the role of Mn incorporation in the structural and morphological evolution of Fe-N-Cs during pyrolysis. Comparisons among factors affecting the properties of final ECs such as the precursor choice i.e. MPc or Mpor, pyrolysis T and A will be made. 10 samples will be analysed in 3 days, requiring 3.5 hours for preparations and 20.5 hours for analysis (15 min for sample insertion and 2 hours for observing each sample)

Ref.

[1] M. Muhyuddin *et al.*, *Applied Catalysis B: Environmental*, vol. 343, p. 123515, Apr. 2024.

Experiment Proposal

Experiment number GP2024162

Principal investigator (*)	Dr Alessio Gabbani, University of Florence, ITALY
Co-investigator	Professor Lorenzo Sorace, Università degli Studi di Firenze, ITALY
Co-investigator	Professor Francesco Pineider, University of Pisa, ITALY
Co-investigator	Miss Giulia Millacci, Università di Pisa, ITALY
Co-investigator	Professor Francesco Di Benedetto, Università degli Studi di Ferrara, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	High Resolution Transmission Electron Microscopy on Yb-doped CsPbX3 perovskite Nanocrystals
MRF Instrument	**TEM High Resolution** — **Days requested:** 4
Access Route	Direct Access — **Previous GP Number:** No
Science Areas	Chemistry, Energy, Materials — **DOI:** -
Sponsored Grant	None — **Sponsor:** -
Grant Title	- — **Grant Number:** -
Start Date	- — **Finish Date:** -
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

Doping Cesium Lead Halide perovskites (LHPs) nanocrystals with Yb3+ cations induces excellent near-infrared luminescence with Quantum Yield approaching 200% due to the quantum cutting phenomenon that is responsible for the emission of 2 NIR photons thorugh the f-f transition of Yb3+ centers for each absorbed UV photon through band gap excitation. This phenomenon can empower novel strategies for solar light harvesting, such as (i) depositing a quantum cutting layer on a Si solar cell and (ii) utilizing luminescent solar concentrators. Nevertheless, the mechanism beyond the quantum cutting phenomenon is still far from being understood. In this project we will study this phenomenon as a function of Yb content and halogen content (Br:Cl ratio) in CsPbX3 nanocrystals. In this context, HRTEM investigation can play a pivotal role in elucidating the Yb incorporation and distribution within the NCs and the corresponding variation of the crystal structure at the single particle level.

Publications -

ISIS neutron and muon source	**E-platform:** No
Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Alessio Gabbani, University of Florence, ITALY		
MRF Instrument	**TEM High Resolution**	**Days Requested:** 4	
Special requirements:			

SAMPLE

Material	toluene dispersions of nanocrystals made of: CsPbCl3, CsPbBr3, CsPbBr1.5Cl1.5, with and without different Yb3+ cation dopants (1, 5, 10% with respect to Pb). The nanocrystals are coated with oleyl amine.	-	-
Formula	CsYbxPb(1-x)Br(1-y)Cly	-	-
Forms	Liquid		
Volume	5 ml		
Weight	mg		
Container or substrate	The liquid dispersions stored in glass vials will be drop casted onto carbon coated TEM grids.	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	295 - 300 K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	carbon coated copper grids	-	-

SAFETY

Prep lab needed	No	-	-
Sample Prep Hazards	No	-	-
Special equip. reqs	No	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	No	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Removed By User	-	-

Science case for ISIS@MACH ITALIA Experimental Proposal

Title: High Resolution Transmission Electron Microscopy on Yb-doped $CsPbX_3$ perovskite Nanocrystals

1. Background

Lead-halide perovskites (LHPs) nanocrystals (NCs) have shown great potential for photovoltaics, optoelectronics and photocatalysis.[1] Doping thin films or NCs of LHPs of formula $CsPbX_3$ (X=Cl, Br or mixed halogens) with Yb^{3+} ions enables narrow near-infrared luminescence due to the 4f–4f transition, with Quantum Yield (QY) approaching 200%.[2,3,4] This peculiar behavior is known as *quantum cutting*, and works as a very efficient down-conversion process, in which electrons excited through bandgap absorption in the UV de-excite from the conduction band through emission of two photons at 980 nm through Yb centers. This phenomenon discloses novel strategies for solar light harvesting, such as (i) depositing a quantum cutting layer on a Si solar cell to boost its efficiency[5] and (ii) employing luminescent solar concentrators.[6] However, despite numerous studies on Yb^{3+}:$CsPbX_3$ aimed at applications, the detailed mechanism beyond quantum cutting is still not completely understood. To this aim, in our project we are exploiting magneto-optics and electron paramagnetic resonance (EPR) spectroscopy, which can address the electronic fine structure of the material as well as the Yb species and defects involved in the process. This would trigger the establishment of precious guidelines for the rational design of highly optimized quantum cutting platforms.

In this proposal, HRTEM will be used to gather structural and morphological information on the NCs that are complementary to the one gathered with the techniques available in our team (standard TEM imaging, ICP-AES, X-ray powder diffraction).

This activity is supported by a PRIN 2022 PNRR national project, entitled “Magnetic field effects in doped kesterites and perovskites for photovoltaics applications” (P20229723Z), which involves partners from University of Pisa, Florence and Ferrara. The materials we propose to investigate with this experiment has been synthesized and preliminarily characterized at the University of Pisa and will be deeply analysed by a research fellow funded by the PRIN 2022 PNRR project.

2. Proposed experiment

We propose to use the unique capabilities of the HRTEM instrument (Thermo Scientific Talos F200X STEM, 200 kV FEG) available at CNR ICCOM (Florence), combining imaging with x-ray Energy Dispersive Spectroscopy (EDS) and electron diffraction (ED). EDS allows us to determine the Yb:Pb ratio (and Br:Cl ratio in mixed halogen NCs) and distribution within the single NCs, as well as its variation within the NCs’ dispersion. Moreover, ED can give information on the crystal phase of the single NCs, which cannot be extracted by the standard powder x-ray diffraction (PXRD) we already used. HRTEM will integrate the characterizations already performed on NCs dispersions or powders, adding single particle resolution of both crystal phase and chemical composition. We will perform EDS and ED on 2-3 NCs per sample. The samples studied in this proposal will be Yb-doped $CsPbX_3$ NCs with variable halogen (X=Br or Cl or mixed compositions) and Yb content (in the range 1-10% with respect to Pb). Both the halogen composition and Yb content are key factors to maximize the QY of NIR emission.

3. Summary of previous experimental proposal and characterizations

Our team already synthesized undoped $CsPbX_3$ NCs with different halogen composition (X=Br, Cl, or Cl:Br 1:1) with tunable optical properties and size of about 10-13 nm (Figure 1a-b). Moreover, we synthesized Yb-doped $CsPbCl_3$, with 1.4 %Yb content (as found from ICP-AES) with intense photoluminescence at 980 nm owing to the f-f transition of Yb^{3+} (Figure 1c-d). Basic structural characterization was performed by PXRD and size and morphology of the NCs were addressed through standard TEM. Advanced characterization was also performed (the data analysis is currently ongoing): MCD and MCPL to study the effect of magnetic field on the excitonic resonance, and EPR spectroscopy to study the Yb^{3+} environment and the presence of defects.

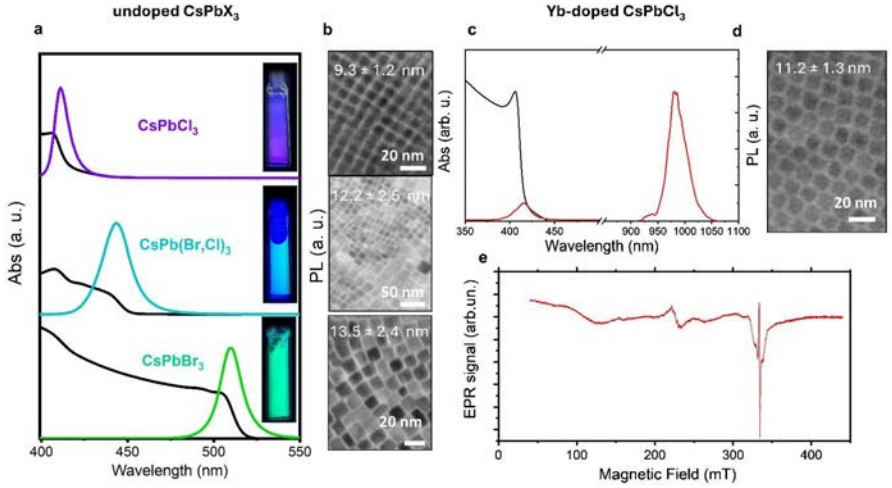

Figure 1: (a) absorption and photoluminescence (PL) spectra of $CsPbCl_3$, $CsPb(BrCl)_3$ and $CsPbBr_3$ NCs (the inset shows pictures of the samples illuminated by UV light) and (b) corresponding TEM images of the samples. (c) Absorption and PL spectrum, (d) TEM image, and (e) EPR spectrum of Yb:doped (1.4% from elemental analysis) $CsPbCl_3$ NCs.

4. Justification of experimental time requested

We plan to investigate 3 NCs samples with the optimal Yb^{3+} content (with maximized PL quantum yield at 980 nm) and different Cl:Br ratio, and other 2 NCs with different Yb^{3+} content and the optimal Cl:Br ratio. The 3 corresponding undoped samples will be also characterized to highlight the differences introduced by the incorporation of Yb on perovskite lattice. More instrumental time will be dedicated to the characterization of the sample with the higher QY. The samples will be prepared by drop drying a toluene dispersion of the NCs onto carbon coated copper grids. The different NCs will have comparable sizes, around 10 (±2) nm. After discussing with the instrument staff, we think that 4 days is a reasonable time for the number of samples and type of characterizations needed in this proposal.

References

[1] Dey, A. *et al.* ACS Nano 2021, 15, 10775−10981; [2] Pan *et al. Nano Lett.* **17**, 8005–8011 (2017); [3] Kroupa, D. M. *et al.*, *ACS Energy Lett.* **3**, 2390–2395 (2018); [4] Milstein, T. J. *et al.*, *Nano Lett.* **18**, 3792–3799 (2018); [5] Zhou, D. *et al.*, *Advanced Materials* **29**, 1704149 (2017); [6] Luo, X. et al., *Nano Lett.* **19**, 338–341 (2019).

TEM JEOL

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Experiment Proposal

Experiment number GP2024150

Principal investigator (*) Dr Anna Maria Ferretti, CNR SCITEC, ITALY
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator

Experiment title	HRTEM,STEM and STEM EDX Noble metal nanoparticles (NPs) supported on 3D metal/metal oxide framework Characterization	
MRF Instrument	**TEM JEOL**	**Days requested:** 3
Access Route	Direct Access	**Previous GP Number:** -
Science Areas	Chemistry, Energy, Materials	**DOI:** -
Sponsored Grant	None	**Sponsor:** -
Grant Title	-	**Grant Number:** -
Start Date	-	**Finish Date:** -
Similar Submission?	-	
Industrial Links	-	

Non-Technical Abstract

The selective oxidation of vicinal diols is one of the most challenging synthetic processes that involve biomass. Glycerol, originating from fatty acids, is a platform molecule for generating glyceric acid and lactic acid through aerobic oxidation under harsh conditions. The supported metal-based nano-catalysts are the best catalysts for these processes. In recent years nanocomposite-based catalysts that combine magnetic susceptors with high hyperthermic efficiency and catalytic moiety, represent a new and promising approach to overcome some typical limitations of catalytic processes by simplifying the reactor structure, improving the process efficiency, and saving energy. Metallic microporous foams functionalized with catalytically active phases are candidates for inductively heated heterogeneous catalysts. Ni-foam, chosen for its good magnetic properties and low cost, was decorated with Au and Pt metal NPs. It is necessary to characterize these samples with HRTEM, HAADF-STEM, and EDX

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Dr Anna Maria Ferretti, CNR SCITEC, ITALY
MRF Instrument **TEM JEOL** **Days Requested:** 3
Special requirements:

SAMPLE

Material	Nichel foam, 3D metal/metal oxide framework with porous size in the range of 500 nm to 2 um decorated with Gold nanoparticles	Nichel foam, 3D metal/metal oxide framework decorated with Platinum nanoparticles	Nichel foam, 3D metal/metal oxide framework decorated with Gold/Platinum Alloy nanoparticles
Formula	Ni,O, Au	Ni, O Pt	Ni, O, Au, Pt
Forms	Friable powder	Friable powder	Friable powder
Volume	cc	cc	cc
Weight	3 mg	3 mg	4 mg
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	- K	- K
Pressure Range	- mbar	- mbar	- mbar
Magnetic field range	- T	- T	- T
Standard equipment	-	None	-
Special equipment	-	-	-

SAFETY

Prep lab needed	No	No	Yes
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	No	No
Sensitivity to vapour	No	No	No
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	no there aren't.	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	Returned to user by instrument scientist (when inactive)	Returned to user by instrument scientist (when inactive)

Noble metal nanoparticles supported on 3D metal/metal oxide framework

1.Background and Context

In recent years, induction heating of magnetic materials using an AC magnetic field has been proposed for catalytic processes, offering several advantages over traditional contact heating reactors. This method allows for remote temperature control with real-time on/off switching, achieving high temperatures rapidly and selectively on the catalyst's surface without heating the reactor walls or reaction medium.

Nanocomposite-based catalysts, which combine a catalytic component and a magnetic susceptor with high hyperthermic efficiency, are promising but challenging. These catalysts are crucial for developing a sustainable green circular economy, particularly for converting large-scale waste into valuable products. Biomass-derived polyols are important renewable sources for synthesizing products like lactic acid, acrolein, glyceric acid, and dihydroxyacetone through catalytic aerobic oxidation reactions, which require high temperatures and pose challenges for catalyst stability.

A good catalytic system must be heterogeneous, selective, stable, recyclable, and energy efficient. A new approach involves using a recyclable nanostructured metal-based catalyst supported on magnetic foam to catalyze aerobic alcohol oxidation via magnetic induction heating. Nickel-based metal foam is chosen for its magnetic properties and low cost. The project aims to control the morphological and structural features of nanoparticles (NPs) on the Ni foam, preventing NP migration, sintering, and leaching during reactions. Various techniques will be used to investigate the effective immobilization, stability, and loading of NPs on Ni foam: ICP-OES, XPS, SEM, HAADF-STEM and HRTEM, EDX, XRD.

This project is supported by PRIN: PROGETTI DI RICERCA DI RILEVANTE INTERESSE NAZIONALE – Bando 2022 Prot. 20225RBM98

MAGnetic Inductive heating of nano-CATalyst onto metal foam as innovative approach for selective aerobic alcohol and polyol oxidation - MAGICAT

2. Proposed experiment

The goal of the proposal is to study the morphological and structural features of the noble metal NPs on the Ni foam, check NPs' migration and sintering after the catalytic reaction. The effective immobilization and the stability of the NPs on Ni foam as well as the NPs' loading

The NP supported on the foam will be investigated by means:

Conventional and High-Resolution TEM, STEM and EDX maps and spectra to highlight

- the NP distribution on the FOAM surface
- crystallinity of the NPs and their interaction with the foam
- noble metal NP composition (Au, Pt, noble metal alloy)

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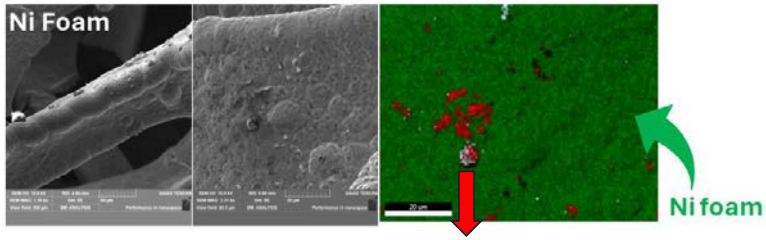
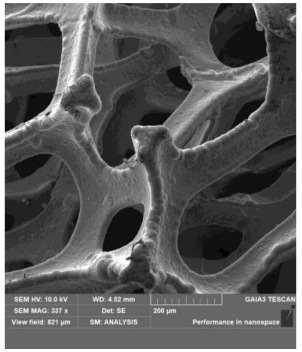
3. Summary of previous experimental proposals or characterization

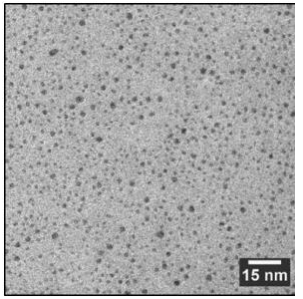
Previous experiment showed that the Ni foam pore size is between 200-650 μm, on the surface of the foam is principally composed by metal nichel, with some spots of Ni_xO.



Commercial Ni Foam
(pore size range 200 – 650 μm)



Pt NPs $d_m \approx 3 \pm 1$ nm



4. Justification of experimental time requested

The machine time has been estimated considering that the acquisition of compositional maps using EDX might takes time, and it will be possible to perform a comparison between fresh and used catalyst during the same session.

Experiment Proposal

Experiment number GP2024163

Principal investigator	Dr Michele Cassetta, Università di Torino, ITALY
Co-investigator (*)	Professor Giancarlo Capitani, University of Milano-Bicocca, ITALY
Co-investigator	Dr Sonia La Felice, Consiglio Nazionale delle Ricerche, ITALY
Co-investigator	Professor NICOLA DALDOSSO, Università di Verona, ITALY
Co-investigator	Dr Daniele Giordano, University of Torino, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Nanocrystals evolution in volcanic glasses
MRF Instrument	**TEM JEOL** — **Days requested:** 5
Access Route	Direct Access — **Previous GP Number:** no
Science Areas	Chemistry, Environment, Materials, Physics — **DOI:** -
Sponsored Grant	Yes — **Sponsor:** Other
Grant Title	A new generation Spectroscopy Tool to monitor rheology and phase transformation prOcesses in volcaNology and cEramic production (STONE) — **Grant Number:** 2022PXHTXM; CUP: D53D23004840006
Start Date	28/09/2023 — **Finish Date:** 28/09/2025
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

This research explores how nanocrystals form in volcanic melts and glasses, affecting volcanic eruptions and industrial applications like glass ceramics. Nanocrystal growth increases the melt's viscosity and influences gas bubble formation, impacting how eruptions unfold. In particular, heating processes govern nanocrystal formation impacting directly properties like strength, elasticity, and heat capacity. Advanced tools like Transmission Electron Microscopy (TEM) will help analyze how nanocrystals interact with bubbles and cracks within our natural volcanic remelted rock samples. By combining TEM with other techniques (Raman, IR, Mössbauer spectroscopies and mechanical tests), we aim to improve methods for predicting volcanic behavior and enhancing industrial materials. Supported by collaborations and prior successful projects, this work seeks to uncover how nanocrystals impact both natural volcanic activity and practical applications in manufacturing.

Publications

none
none
none

ISIS neutron and muon source — **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Professor Giancarlo Capitani, University of Milano-Bicocca, ITALY
MRF Instrument	**TEM JEOL** — **Days Requested:** 5
Special requirements:	

SAMPLE

Material	Natural Silicate Glass	Natural Silicate Glass-Ceramic. It has the same composition but previously thermally treated	-
Formula	average composition i weight %, SiO_2: 49.7, TiO_2: 1.6, Al_2O_3: 16.1, FeO(t): 10.3, MnO: 0.2, MgO: 5.9, CaO: 7.6, Na_2O: 4.0, K_2O: 2.1, P_2O_5: 0.8.	average composition i weight %, SiO_2: 49.7, TiO_2: 1.6, Al_2O_3: 16.1, FeO(t): 10.3, MnO: 0.2, MgO: 5.9, CaO: 7.6, Na_2O: 4.0, K_2O: 2.1, P_2O_5: 0.8.	-
Forms	Solid	Solid	
Volume	17.86 cc	17.24 cc	
Weight	50 g	50 g	
Container or substrate	plastic box	plastic box	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	RT - K	RT - K	-
Pressure Range	1013.25 - mbar	1013.25 - mbar	-
Magnetic field range	- T	- T	-
Standard equipment	None	None	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	Yes	-
Sample Prep Hazards	no	no	-
Special equip. reqs	All the equipment is already available in the TEM laboratory.	All the equipment is already available in the TEM laboratory.	-
Sensitivity to air	No	No	-
Sensitivity to vapour	No	No	-
Experiment Hazards	no	no	-
Equipment Hazards	-	-	-
Biological hazards	no	no	-
Radioactive Hazards	no	no	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	-

MRF Proposal: Nanocrystals evolution in volcanic glasses (*Michele Cassetta*)

1. Background and Context

The development of nano-crystals in melts and glasses represents a current hot-topic in volcanology and ceramics processing. Crystals, in general, increase melt viscosity due their content, whether nanocrystal growth apparently provide a substantial effect in what it determines the formation of SiO_2-Al_2O_3 enriched heterogeneities which, according to previous studies, may increase viscosity of up to 3 orders of magnitude. In addition, the presence of nanoparticles enhances gas bubble nucleation at all scales at all scales. [1–4]. On the other hand, volcanic systems, especially basalts, are valuable for producing glass ceramics with high strength and resistance. Basaltic melts, with lower viscosity, are easier to shape and notably energy-saving and represents a vast reservoir of raw material naturally-prone to applications. In this context, understanding the thermal processes governing nano-crystallization dynamics (nucleation and growth) is crucial, as they have a direct impact on the material's macroscopic properties (viscosity, elasticity, hardness, toughness, heat capacity and thermal conductivity-diffusivity [5–8]).

This proposal integrates seamlessly into a wider research program, which focuses on the chemical heterogeneity and nano-structural dynamics of volcanic materials. Transmission Electron Microscopy (TEM) is essential for characterizing nanocrystals, their interaction with bubbles, cracks, and amorphous regions, all crucial for understanding volcanic glass and melt behaviour. Additionally, TEM-based investigation will extend and integrate Raman spectroscopy approaches to determine viscosity and water content of nano-crystal bearing magmas [9–11].

The research program is well-supported through a combination of partnerships, funding, and collaborations. Our group have strong experiences in Raman and FTIR spectroscopies and high-*T* apparatus. I have successfully secured beam-time as a principal investigator at ELETTRA-Sincrotrone where I conducted advanced in-situ high-*T* studies on volcanic and contributed to projects at the EPOS ELBE Positron Source, to characterize voids in engineered glasses. My research has been bolstered by postdoctoral positions at leading institutions, including the University of Trento, where I managed the PolGla project and University of Torino where I currently handling the PRIN-granted project STONE. Additionally, my supervision of projects and collaborations with students further enriches the scope and success of this program.

2. Proposed experiment

The experiments aim at unravel the two expected mineral phases constituting the solid fraction (magnetite and augite) upon different thermal treatment previously determined by the help of differential scanning calorimetry (DSC) on different volcanic glasses (from basalt to rhyolite), in order to estimate: 1) crystal size distribution; 2) what elements are subtracted from the glass and in which proportion; 3) mapping the nano-scaled chemical heterogeneities; 4) correlate spectroscopic data (Raman and XRD) to the type of nano-solid fraction; 5) understand their role on the final mechanical properties. In order to get access to all the above information, a nanoscale investigation is required. To this regard, transmission electron microscopy (TEM) coupled with energy dispersive spectroscopy (EDS) is the most suitable technique. The same software used to acquire TEM images (Digital Micrograph, Gatan) and chemical data (Aztec, Oxford), will be used also for data analysis and presentation, along with other free software such as ImageJ (NIH).

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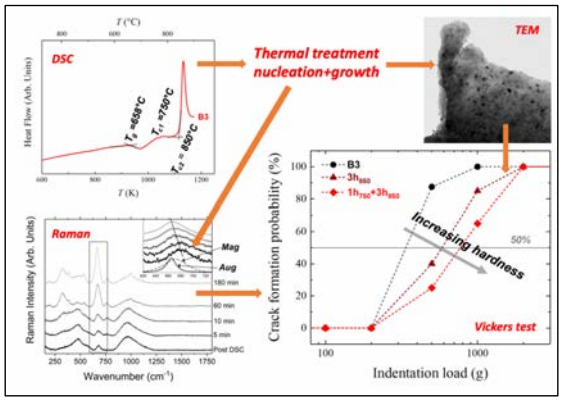

Figure 1. *sketch of the experimental design of our wider research programme:* ***DSC-determination*** *of the crystallization temperature (**$T_{c,n}$**),* ***Raman*** *spectra obtained after different thermal treatment. Finally, the effect of nanocrystal on mechanical properties (**crack formation probability*** *and* ***hardness***) *obtained by Vickers tests.*

The result will lead us to obtain outcomes similar to our study [9], where we combined TEM, DSC and low-frequency micro-Raman spectroscopy to disentangle the effect of nanocrystals in residual glass and modelling its liquid viscosity to fully quantify the viscous flow erupted product of Etna and Calbuco volcanoes.

4. Justification of experimental time requested

Samples for TEM investigations will be prepared as it follows: representative fragments of each bulk sample will be powdered in agate mortar, dispersed in isopropyl alcohol, ultrasonicated and finally few microliters of the suspension will be pipetted on holey carbon films supported by cupper grids. Overall, we estimate to prepare and study 10 samples. Considering the time required for the preparation, insertion of the specimen holder, imaging the nanoparticles and chemical mapping at least three areas in each sample, half a day is required for each sample, and five days for the whole investigation (10 samples).

References

1. A. J. Hornby, et al., Nat. Commun. **15**, 1 (2024).
2. D. Di Genova, et al., Sci. Adv. **6**, 1 (2020).
3. F. Cáceres, et al., Nat. Commun. **15**, (2024).
4. S. Okumura, et al., Commun. Earth Environ. **3**, 1 (2022).
5. L. F. de Lima, et al., Bol. La Soc. Esp. Ceram. y Vidr. 1 (2020).
6. R. Casasola, et al., J. Mater. Sci. **47**, 553 (2012).
7. J. M. Klein, et al., Mater. Sci. Technol. (United Kingdom) **35**, 544 (2019).
8. D. Giordano, et al., Chem. Geol. **560**, 119981 (2021).
9. M. Cassetta, et al., Chem. Geol. **616**, 121241 (2023).
10. D. González-García, et al., Chem. Geol. **567**, (2021).
11. D. Di Genova, et al., Lithos **318–319**, 209 (2018).

X-Ray Diffractometer

Experiment Proposal

Experiment number GP2024100

Principal investigator	Professor Valeria Amendola, Università di Pavia, ITALY
Co-investigator (*)	Professor Angiolina Comotti, Università degli studi di Milano Bicocca, ITALY
Co-investigator	Dr Sonia La Cognata, University of Pavia, ITALY
Co-investigator	Professor Silvia Bracco, University of Milano Bicocca, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Porous organic materials for CO2 separation from gaseous mixtures
MRF Instrument	**X-Ray Diffractometer** — **Days requested:** 5
Access Route	Direct Access — **Previous GP Number:** No
Science Areas	Materials — **DOI:** -
Sponsored Grant	None — **Sponsor:** -
Grant Title	- — **Grant Number:** -
Start Date	- — **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	Selective CO2 sorption is one of the most widely used and effective techniques for separating CO2 from gas mixtures under various conditions. However, to facilitate future industrial applications, it is crucial to develop effective CO2-philic porous sorbents using environmentally friendly procedures. Our research group has recently developed a protocol for synthesizing CO2-philic POPs in water, avoiding the use of catalysts or additives. The proposed X-ray powder diffraction experiment aims to i) investigate the crystallinity and phase purity of the materials synthesized in water, ii) compare the results obtained in i) with those for the same material synthesized in organic solvent, iii) evaluate the effect of the polyamine structure on the properties of the final sorbent, iv) evaluate the effect of CO2 (vs. N2) sorption on the investigated solids. For the future application of our materials, investigations using the ISIS@MACH ITALIA X-ray powder diffractometer are essential.
Publications	-

ISIS neutron and muon source — **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Professor Angiolina Comotti, Università degli studi di Milano Bicocca, ITALY
MRF Instrument	**X-Ray Diffractometer** — **Days Requested:** 5
Special requirements:	

SAMPLE

Material	porous organic polymer (POP-W-s1); starting materials: polyamine-1, triformylphloroglucinol	porous organic polymer (POP-O-s1); starting materials: polyamine-1, triformylphloroglucinol	porous organic polymer (POP-W-s2); starting materials: polyamine-2, triformylphloroglucinol
Formula	C21H21N3O3	C21H21N3O3	C24H27N3O3
Forms	Friable powder	Friable powder	Friable powder
Volume	1 cc	1 cc	1 cc
Weight	150 mg	200 mg	200 mg
Container or substrate	No	No	No
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	200 - 300 K	200 - 300 K	200 - 300 K
Pressure Range	- 1000 mbar	- 1000 mbar	- 1000 mbar
Magnetic field range	- T	- T	- T
Standard equipment	Gas Handdling	Gas Handdling	Gas Handdling
Special equipment	No	No	No

SAFETY

Prep lab needed	No	No	No
Sample Prep Hazards	No	No	No
Special equip. reqs	No	No	No
Sensitivity to air	No	No	No
Sensitivity to vapour	No	No	No
Experiment Hazards	No	No	No
Equipment Hazards	-	-	-
Biological hazards	No	No	No
Radioactive Hazards	No	No	No
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Removed By User	Removed By User	Removed By User

Experiment title: Porous organic materials for CO_2 separation from gaseous mixtures

1. Background and Context

The increasing concentration of greenhouse gases in the atmosphere, primarily CO_2, is a pressing concern today. Developing and implementing new technologies for efficient Carbon Capture has therefore become an imperative task. Various technologies have been developed to remove CO_2 from gas mixtures such as flue gas. In adsorption-based approaches, solid sorbents that can selectively interact with CO_2 through physical interactions are employed. Among the materials that have been explored for this purpose, promising results have been achieved with Porous Organic Polymers (POPs).[1] These materials typically exhibit high porosity, thermal and chemical stability, and can be easily tuned for pore size and functionalization. Most current syntheses of CO_2-philic POPs involve the use of toxic organic solvents, acidic or metal catalysts or other additives, and high reaction temperatures. Therefore, developing environmentally friendly procedures for synthesizing POPs is crucial to facilitate their future industrial applications in CO_2 sorption.[2] While water is a greener solvent alternative for POP synthesis, its efficient utilization is still limited. Our group (Department of Chemistry, University of Pavia) developed a green method for the synthesis of CO_2-philic POPs. The developed protocol not only avoids the use of catalyst or other additional additives, but also directly produces a material with permanent porosity and a high selectivity for CO_2 vs. N_2.

2. Proposed experiment

A deep understanding of the physicochemical properties of new developed materials, especially before and after exposure to the target gas, is fundamental for effective applications in gas sorption and separation. The here proposed experiment aims to: i) investigate the crystallinity and phase purity of the materials synthesized in water, ii) compare the results obtained in i) with those for the same material synthesized in organic solvent, iii) evaluate the effect of the polyamine structure on the properties of the final sorbent, iv) evaluate the effect of CO_2 (vs. N_2) sorption on the investigated solids. For the future application of our materials, investigations using the ISIS@MACH ITALIA X-ray powder diffractometer, equipped with a chamber for performing experiments at variable temperature and presence of gases, are essential.

3. Summary of previous experimental proposals or characterisation

The porous materials have been characterized by elemental and thermogravimetric analyses, FTIR, solid-state NMR. Both FT-IR and ^{1}H-^{13}C CP MAS NMR spectra confirmed the ketoenamine framework. The affinity for CO_2 vs. N_2 has also been tested by measuring the uptakes of both gases at 298 K (1 bar). These adsorption studies pointed out a remarkable difference in adsorption with CO_2 (see the Figure), suggesting a high CO_2/N_2 selectivity in conditions suitable for Vacuum-Swing-Adsorption.

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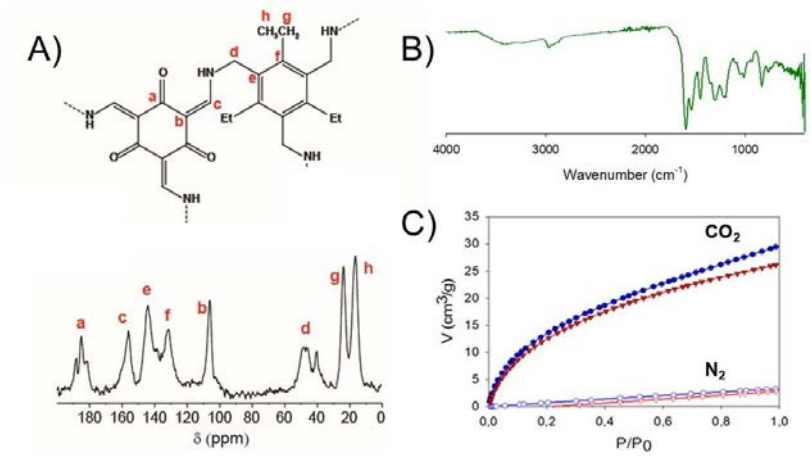

Figure: A) Example of POP synthesized by our group: CP MAS NMR and B) FTIR characterization spectra; C) Adsorption curves at 298 K of CO_2 and N_2 for two ketoenamine POPs (red and blue lines, respectively).

4. Justification of experimental time requested

As previously mentioned, the application of our materials as sorbents for CO_2 from gaseous mixtures requires a thorough characterization using the ISIS@MACH ITALIA diffractometer, which is equipped with a chamber for performing experiments at various temperatures (200–300 K) and in the presence of target gases (CO_2, N_2). The experiment will be conducted over 5 days, focusing on 3 different samples.

All samples will be characterized at different temperatures and in the presence of either CO_2 or N_2. On days 1-2, we will set up the experiment and perform PXRD characterization of **POP-W-s1**, a sorbent synthesized in water (W) using polyamine-1 and triformylphloroglucinol as starting materials. On days 3-5, the study will be repeated on samples **POP-O-s1** and **POP-W-s2**. POP-O-s1: synthesized in organic (O) solvent, same starting compounds used for POP-W-s1; POP-W-s2: synthesized in water using triformylphloroglucinol and polyamine-2.

References:

1 K. S. Song et al., ***Chem. Soc. Rev.***, 2022,**51**, 9831-9852
2 D. Luo et al., ***Angew. Chem. Int. Ed.***, 2023, **62**, e202305225

Experiment Proposal

Experiment number GP2024134

Principal investigator	Mr Giuseppe Mastronardi, University of Turin, ITALY	
Co-investigator (*)	Professor Angiolina Comotti, Università degli studi di Milano Bicocca, ITALY	
Co-investigator	Dr Jacopo Perego, University of Milano-Bicocca, ITALY	
Co-investigator		
Co-investigator		
Co-investigator		
Co-investigator		
Co-investigator		
Co-investigator		
Experiment title	Powder X-ray Diffraction experiment on MOF	
MRF Instrument	**X-Ray Diffractometer**	**Days requested:** 5
Access Route	Direct Access	**Previous GP Number:** No
Science Areas	Chemistry, Engineering, Environment, Materials	**DOI:** -
Sponsored Grant	None	**Sponsor:** -
Grant Title	-	**Grant Number:** -
Start Date	-	**Finish Date:** -
Similar Submission?	-	
Industrial Links	-	

Non-Technical Abstract

The knowledge of the preferred adsorption sites of the MOF-303 after EDA diffusion is fundamental for application in CO 2 capture from the air. The proposed experiments will be focused on the study of:
1) crystal structures of the MOFs-303 and after distinct amounts of EDA insertion;
2) amino-functionalized MOF-303 after loading CO2 at 400 ppm and room temperature.
The final goal will be to identify the right amount of EDA for the most efficient CO 2 capture from
the air. The deep understanding of the active sites is essential for achieve the goal. For the future application of our materials, investigations using the ISIS@MACH ITALIA X-ray powder diffractometer, equipped with a chamber for performing in situ experiments in the presence of gases, is necessary.

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Professor Angiolina Comotti, Università degli studi di Milano Bicocca, ITALY
MRF Instrument **X-Ray Diffractometer** **Days Requested:** 5
Special requirements:

SAMPLE

Material	MOF-303 (without EDA), MOF-303 (with EDA)	MOF-303+EDA	-
Formula	AlC5N2O5H3	AlC5N2O5H3+(N2C2H6)	-
Forms	Friable powder	Friable powder	
Volume	5 cc	5 cc	
Weight	200 mg	200 mg	
Container or substrate	Simple Vials	Simple Vials	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	273 - 298 K	273 - 298 K	-
Pressure Range	0 - 1000 mbar	0 - 1000 mbar	-
Magnetic field range	0 - 0 T	0 - 0 T	-
Standard equipment	Gas Handdling	Gas Handdling	-
Special equipment	No	No	-

SAFETY

Prep lab needed	Yes	Yes	-
Sample Prep Hazards	No	No	-
Special equip. reqs	No	No	-
Sensitivity to air	No	No	-
Sensitivity to vapour	No	No	-
Experiment Hazards	No	No	-
Equipment Hazards	-	-	-
Biological hazards	No	No	-
Radioactive Hazards	No	No	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	Returned to user by instrument scientist (when inactive)	-

ANCHORING AMINE FUNCTIONALITIES TO MOFs FOR APPLICATIONS IN DIRECT CO_2 AIR CAPTURE

1. Background

Direct Air Capture (DAC) has been recognized as a fundamental way to reduce CO_2 in the atmosphere and mitigate greenhouse effect. The current atmospheric concentration of CO_2 is around 420 ppm: in these conditions, sorbents that capture CO_2 through chemisorption exhibit stronger and more selective interaction sites than physisorbent materials. Amine functionalities contribute to the chemical sorption of CO_2 by virtue of the reversible formation of carbamate moiety. The development of amino-functionalized solid sorbents (e.g., Metal-Organic Frameworks - MOFs) represents a benchmark strategy to create new possible DAC technologies.[1] In the present work, the alluminium-based MOF-303 has been chosen as main target[2]. This MOF could anchor amine moieties by acid-base interaction of its pyrazolic linker, this type of interaction seems to mimic the one of some classes of Ionic Liquids (IIs) constituted by azoles and amines.[3] In the current work, Ethylenediamine (EDA) has been chosen for the functionalization of MOF-303.

2. Proposed experiment

The knowledge of the preferred adsorption sites of the MOF-303 after EDA diffusion is fundamental for application in CO_2 capture from the air. The proposed experiments will be focused on the study of:

1) crystal structures of the MOFs-303 and after distinct amounts of EDA insertion;
2) amino-functionalized MOF-303 after loading CO2 at 400 ppm and room temperature.

The final goal will be to identify the right amount of EDA for the most efficient CO_2 capture from the air. The deep understanding of the active sites is essential for achieve the goal. For the future application of our materials, investigations using the ISIS@MACH ITALIA X-ray powder diffractometer, equipped with a chamber for performing in situ experiments in the presence of gases, is necessary.

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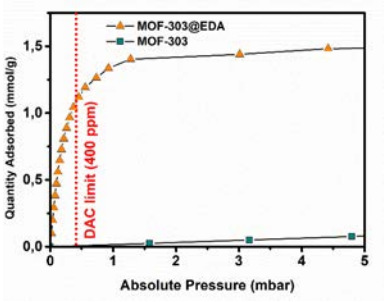
3. Summary of the previous experiments and characterization

The samples with and without amine molecules were characterized by several techniques including ^{1}H NMR, ^{13}C CP MAS NMR, IR spectroscopy, thermogravimetric analysis, powder X-ray diffraction. CO_2 adsorption isotherms of the amino-loaded MOF-303 collected at 273 K have shown encouraging results, thanks to the enhancement of the CO_2 adsorption capacity of the diamine-decorated MOF compared to the pristine MOF-303. Taking into account the absolute pressure of 0.4 mbar (target pressure for DAC technologies), the functionalized material shows an adsorption of about 1 mmol/g, a huge improvement of CO_2 capture capacity (in DAC conditions). Spectroscopic data confirm theoretical calculations, demonstrating high affinity between the MOF-303 structure and amines..



4. Justification of experimental time requested

The application of our materials as sorbents for CO_2 from the air needs a thorough characterization using the ISIS@MACH ITALIA diffractometer, which is equipped with a chamber for performing experiments at various temperatures (200–300 K) and in the presence of target gases such as CO_2. The experiment will be conducted over 5 days, focusing on 4 samples.
The samples will be characterized at 273 K and 298 K and in the presence of CO_2.
On days 1-2, the PXRD characterization of MOF-303 with and without amino moieties.
On days 3-5, the study will be performed MOF-303 with distinct amount of amino moieities after loading CO_2 at distinct temperatures, especially 273 and 298 K.

References:

[1] S. Bose, D. Sengupta, T. M. Rayder, X. Wang, K. O. Kirlikovali, A. K. Sekizkardes, T. Islamoglu, O. K. Farha, Adv Funct Mater, 2023.
[2] Z. Zheng, H. L. Nguyen, N. Hanikel, K. K. Y. Li, Z. Zhou, T. Ma, O. M. Yaghi, Nat Protoc, 2023, 18, 136–156.
[3] J. Wu, B. Lv, X. Wu, Z. Zhou, G. Jing, ACS Sustain Chem Eng, 2019, 7, 7312–7323.
[4] R. Dovesi, A. Erba, R. Orlando, C. M. Zicovich-Wilson, B. Civalleri, L. Maschio, M. Rérat, S. Casassa, J. Baima, S. Salustro, B. Kirtman, Comput Mol Sci. 2018;8:e1360.

XRD TOMOGRAPHY

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Experiment Proposal

Experiment number GP2024049

Principal investigator	Dr Pier Francesco Fabbri, Museo e Istituto Fiorentino di Preistoria, ITALY
Co-investigator (*)	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Carla Andreani, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Alessandro Cianchi, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Ancient DNA analysis of a Neolithic human tooth from eastern Sicily XRD TOMOGRAPHY: insights and implications

MRF Instrument	**XRD TOMOGRAPHY**	**Days requested:** 1	
Access Route	Direct Access	**Previous GP Number:** No	
Science Areas	Biology and Bio-materials, Cultural Heritage	**DOI:** No	
Sponsored Grant	None	**Sponsor:** -	
Grant Title	-	**Grant Number:** -	
Start Date	-	**Finish Date:** -	
Similar Submission?	-		
Industrial Links	-		

Non-Technical Abstract

This is the first of three proposals which aim to analyse with multilevel techniques a tooth belonging to the first Neolithic individual buried at Rocchicella Paliké (CT) in eastern Sicily. The burial is dated back between 5210- 4840 BC (calibrated) (LTL12788A). In this proposal we want to perform a non-destructive 3D characterisation of the human tooth using XRD TOMOGRAPHY (at the CNR-IPCB Unit). In distinct proposals, further characterisations with Small/Wide Angle X-ray Scattering (SAXS GISAXS instrument, CSGI-Unit), neutron diffraction and neutron tomography at INES and at IMAT beamlines at ISIS Facility (UK) will be requested. Finally, we will focus on the genetic composition, migration patterns, and interaction of this individual with local Mesolithic communities. Thus, we will request the use of the DNA sequencing NGS instrument Univ Tor Vergata Unit, for a destructive analysis, i.e. extracting ancient DNA (aDNA) from the tooth and to follow by sequencing the human genome.

Publications

Lonoce et al. 2023. Journal of Archaeological Science 155, 105790
Viva et al. 2023. Archaeological and Anthropological Sciences 15:193
Vincenti et al. 2023. American Journal of Biological Anthropology 183: e24911.

Instruments	**INES**	**Days Requested:** 1	
Access Route	Direct Access	**Previous RB Number:** No	
Science Areas		**DOI:** No	
Sponsored Grant	None	**Sponsor:**	
Grant Title	-	**Grant Number:**	
Start Date	-	**Finish Date:**	
Similar Submission?			
Industrial Links			

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Science and Technology Facilities Council
ISIS Neutron and Muon Source

Sample record sheet

Principal contact	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY		
MRF Instrument	**XRD TOMOGRAPHY**	**Days Requested:** 1	
Special requirements:			

SAMPLE

Material	intact tooth (enamel, dentine and cementum) about 2x3x1 cm^3	-	-
Formula	Not known	-	-
Forms	Solid		
Volume	6-8 cc		
Weight	200-500 mg		
Container or substrate	standard INES holder	-	-
Storage Requirements	plastic box	-	-

SAMPLE ENVIROMENT

Temperature Range	273 - 320 K	-	-
Pressure Range	1000 - 1010 mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	No	-	-
Special equip. reqs	No	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	No	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	-	-

Ancient DNA analysis of a Neolithic human tooth from eastern Sicily XRD TOMOGRAPHY: insights and implications

1. Background and Context

The Neolithic period, marking the transition from hunter-gatherer societies to agricultural communities, represents one of the most transformative periods in human history (Barker et al. 2015). This shift, often referred to as the Neolithic Revolution, began around 10,000 years ago in the Near East and gradually spread across Europe and the Mediterranean. During the Neolithic transition, farming communities originated in the Middle East and then started to expand into new territories (Hofmanová et al., 2016). These groups spread through Anatolia and the Balkans (Lazaridis et al., 2014; Mathieson et al., 2018), progressively admixing with local hunter-gatherers (Lipson et al., 2017). There are profound differences in the spatiotemporal patterns of Neolithization across Europe, with significant shifts in genetic ancestry varying by region (Allentoft et al., 2024). This genetic transition was particularly extensive in southern Europe, especially in Italy. In this framework east Sicily, strategically located in the central Mediterranean, offers a unique vantage point for studying the diffusion of Neolithic culture and technology. It holds the potential to shed light on the genetic diversity of early Neolithic farmers, their origins, and their genetic legacy. The peopling of Sicily from the Upper Palaeolithic to the Mesolithic has already shown a peculiar pattern involving migrations and partial replacements of local populations (Mathieson et al., 2018; Catalano et al., 2020; Yu et al., 2022; Scorrano et al., 2022).

This project aims to delve deeper into understanding whether the arrival of Neolithic communities in this area involved complex interactions between incoming agriculturalists and indigenous Mesolithic hunter-gatherers by analysing the ancient DNA (aDNA) of the first Neolithic individual from the site Rocchicella Paliké (CT) in eastern Sicily, dated back to the years 5210-4840 BC (calibrated) (LTL12788A) in East Sicily (Figure 1). These interactions likely facilitated the exchange of ideas, technologies, and genetic material, leading to the establishment of early farming villages.

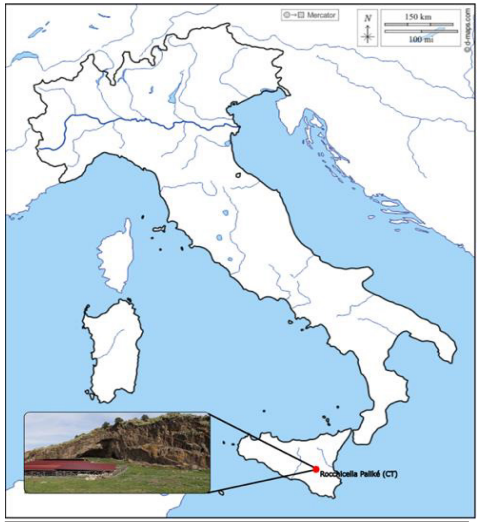

Figure 1: localization of the site.

To characterise the human tooth, we will firstly perform non-destructive X-ray computed tomography (XCT) by XRD TOMOGRAPHY (at the CNR-IPCB Unit), Small/Wide angle X-ray Scattering (SAXS GISAXS instrument, CSGI-Unit), and neutron tomography and diffraction [at the IMAT and INES beamlines, ISIS (UK)]. The elemental composition of the sample will be quantified through Rietveld refinement of X-rays data acquired with XRD TOMOGRAPHY and SAXS GISAXS, and neutron diffraction data, using INES, and neutron tomography, using IMAT, will be cross compared to verify consistency. XCT scan will be complemented with neutron tomography data, which is more sensitive to detect organic-like material. After this analysis a destructive shotgun sequencing of aDNA will be performed on the tooth, using the DNA Sequencing NGS of Rome Tor Vergata Unit.

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2. Proposed experiment

In this experiment we aim to perform a non-destructive 3D X-ray tomography on the human tooth belonging to the first Neolithic individual buried at Rocchicella Paliké (CT) in eastern Sicily, using the XRD TOMOGRAPHY instrument operating at the IM@IT Unit-CNR-IPCB. The elemental composition of the sample will be quantified by the 3D rendering and analysis of the reconstructed XCT scan will be compared and complemented by neutron computed tomography reconstructed data that will be measured at the IMAT beamline operating at ISIS Facility. In a second distinct IM@IT proposal the elemental characterisation of the human tooth will be done by Rietveld refinement of X-ray data acquired with the Small/Wide angle X-ray Scattering data (SAXS GISAXS instrument, CSGI-Unit) and by means of neutron diffraction performed at the INES@ISIS beamline. Finally, in a third IM@IT proposal, destructive shotgun sequencing of aDNA of the sample will be assessed by the DNA Sequencing NGS operating at the IM@IT Unit-University of Rome Tor Vergata.

4. Justification of experimental time requested

The selected intact human tooth sample with dimension of 20 mm x 30 mm x 10 mm. We aim to measure the sample using a field of view of 20 mm x 40 mm, pixel size of 10 μm, and about 3150 projections to fulfil the Niquist-Shannon sampling theorem. With an exposure time per projection of 5 s, the tomography will last about 4.5 hours. Hence, after discussion with the instrument scientist, we request **1 days** of instrument time including set-up and calibration time.

References

Allentoft et al. Nature 625, 301–311 (2024).
Barker, G. & Goucher, C. (Cambridge Univ. Press, 2015).
Catalano et al. Quaternary International 537, 24-32 (2020).
Hofmanová, Z. et al. Proc. Natl Acad. Sci. 113, 6886–6891 (2016).
Lazaridis, I. et al. Nature 513, 409–413 (2014).
Lipson, M. et al. Nature 551, 368–372 (2017).
Mathieson, I. et al. Nature 555, 197 (2018).
Scorrano et al. Communications Biology 5, 1262 (2022)
Yu et al. iScience 25, 104244 (2022)

Experiment Proposal

Experiment number GP2024081

Principal investigator (*)	Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY
Co-investigator	Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Training on XRD TOMOGRAPHY (CNR-IPCB)
Training MRF	**XRD TOMOGRAPHY**
Days requested:	1
Access Route	Direct Access
Previous GP Number:	No
Science Areas	Materials
DOI:	-
Sponsored Grant	None
Sponsor:	-
Grant Title	-
Grant Number:	-
Start Date	-
Finish Date:	-
Similar Submission?	-
Industrial Links	RGF SRL

Non-Technical Abstract

Consorzio Physis SRL S.B. is a benefit company dedicated to the production of high-quality metallic components and surface treatments for fashion and luxury. We aim to enhance product quality and longevity by non-destructive structural analysis: we are interested in the XRD TOMOGRAPHY instrument at the CNR-IPCB Unit, which can provide valuable insights into crack detection in brass components manufactured through hot and cold stamping and voids distribution and shrinkage during processes like Metal Injection Molding (MIM) and 3D printing of steel. We seek training on the RIGAKU Nano3DX instrument, focusing on XRD TOMOGRAPHY principles and practical applications. The knowledge obtained from this training would be valuable for the submission of future experimental proposals.

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Science and Technology Facilities Council
ISIS Neutron and Muon Source

Sample record sheet

Principal contact	Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY
Training Instrument	**XRD TOMOGRAPHY** **Days Requested:** 1
Special requirements:	

SAMPLE

Material	-	-	-
Formula	-	-	-
Forms			
Volume			
Weight			
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	-	-	-
Pressure Range	-	-	-
Magnetic field range	-	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	-	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	-	-	-
Sensitivity to vapour	-	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	-	-	-

Training proposal on XRD TOMOGRAPHY (CNR-IPCB)

1. Background and context

Consorzio Physis SRL S.B. is actively engaged in the production of fashion accessories, since it collects the facilities producing metallic fashion accessories and surface treatments as well as chemicals providers for the surface treatments. As part of our ongoing efforts to enhance our technical capabilities and address critical challenges related to quality and longevity of the items produced, we are seeking to deepen our understanding of internal structural defects in manufactured articles and pores and cavities that are residual to some kind of production processes. We are particularly interested in the RIGAKU Nano3DX, a submicron resolution computed tomography scanner useful for structural characterization, including 3D reconstruction. This technique is especially beneficial for enabling targeted interventions in the production manufacturing processes to improve the final products performances. To fully leverage this technology, we seek training on the instrument, focusing on the technique principles and practical applications since Consorzio Physis SRL S.B. is a benefit company collecting societies dedicated to the production of high-quality fashion metallic components for fashion and luxury. To enhance the quality and longevity of the products, we aim to deepen our understanding on this technique.

2. Proposed training

This training proposal aims to provide comprehensive instruction on the utilization of X-ray tomography, a crucial tool for non-destructive testing and detailed internal analysis. Emphasizing the strategic importance of this training, it will enable consortium companies to submit more informed and sophisticated experimental proposals in the future. By acquiring advanced knowledge in X-ray tomography, participants will enhance their ability to innovate and optimize manufacturing processes, particularly in the metal accessories sector for fashion and luxury markets.

X-ray tomography offers in fact significant applications in the analysis of metal accessories, such as detecting internal and non-visible cracks in products manufactured through the conventional production techniques for brass, such as hot and cold stamping, that could lead to corrosion upon time. Additionally, analysis performed with this technique could also supports the industrialization of production processes of steel articles, such as Metal Injection Molding (MIM), providing critical insights into critical production aspects, such as void distribution and structural modifications during sintering. Furthermore, it could aid in the comparative analysis of pre- and post-sintering voids for 3D printing of steel as well, helping in the industrialization of the production process, ensuring higher quality and reliability of the final products.

In summary, this training not only enhances the current capabilities of participants but also lays a solid foundation for future experimental proposals, driving forward innovation and excellence within the consortium.

Therefore, we request a training on the instrument to gain knowledge in the technique and on the analytical capabilities of the instrument.

The training would be carried out at CNR-IPCB, with whom we are already in contact and agreed on the proposed training.

3. Summary of previous training proposals

No previous training proposal has been presented. However, this training will empower us to perform more accurate and detailed analyses, ultimately leading to improved quality and durability of the fashion accessories since XRD TOMOGRAPHY stands out as a robust analytical method with a wide array of benefits

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for non-destructive structural analysis. It provides invaluable data concerning It can detect cracks in components manufactured through hot and cold stamping of brass, identify inclusions and structural defects, and analyse void distribution and shrinkage during processes like Metal Injection Molding (MIM) and 3D printing of steel [1-3]. Such in-depth knowledge would allow for targeted and efficient actions to improve the production processes in order to enhance the performance and longevity of metallic accessories designed for the fashion and luxury segment, including footwear, leather goods, and jewellery.

4. Justification of experimental proposals request

We request a training on the instrument to gain knowledge in the technique and on the analytical capabilities of the instrument; specifically, our objectives for the training course would include:

1. Learning the general principles of XRD TOMOGRAPHY and specific potential of Rigaku 3D Nano.
2. Instrumental session to apply the technique on some samples.

Therefore we request 1 day of training, as discussed and agreed with the instrument scientists, in order to provide strong know-how on the principles of the technique during the first part of the day, with valuable examples on data analysis, while the second part of the day will be dedicated to an experimental session on selected samples in order to explore the analytical capabilities of the instrument.

[1] Quantitative X-ray tomography E. Maire & P. J. Withers (2014) Quantitative X-ray tomography, International Materials Reviews, 59:1, 1-43.

[2] Exploring Micro-focus X-ray computed tomography for metal injection moulded green parts, M Seerane 2018 IOP Conf. Ser.: Mater. Sci. Eng. 430 012032.

[3] X-ray computed microtomography studies of MIM and DPR parts by N.S. Muchavi, L. Bam, F.C. De Beer, S. Chikosha and R. Machaka, Journal of the Southern African Institute of Mining and Metallurgy · October 2016.

Experiment Proposal

Experiment number GP2024082

Principal investigator (*)	Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY
Co-investigator	Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Wire bending and adhesion investigation with XRD TOMOGRAPHY (CNR-IPCB)
MRF Instrument	**XRD TOMOGRAPHY**
Days requested:	4
Access Route	Direct Access
Previous GP Number:	NO
Science Areas	Materials
DOI:	-
Sponsored Grant	None
Sponsor:	-
Grant Title	-
Grant Number:	-
Start Date	-
Finish Date:	-
Similar Submission?	-
Industrial Links	RGF SRL

Non-Technical Abstract

In the fashion industry, rings of various sizes and finishes are widely used for structural and decorative purposes, such as connecting multiple decorative elements, like charms on leather bags. Given the size requirements for these charms, the rings are produced by bending brass wires to the desired dimensions. These rings are then subjected to surface finishing treatments that are both protective (to prevent brass corrosion) and decorative (to meet the aesthetic demands of design offices). To achieve diverse aesthetic needs, painting is often employed, allowing for a wide range of colours. However, painting can mask structural defects like cracks caused by bending. Such cracks pose a risk as they can lead to the fracturing of the outer paint layer over time, causing product oxidation and impacting the final product (the bag). This study aims to analyse internal cracks and potential fissures/voids/bubbles at the brass-paint interface to optimize production processes and improve paint

Publications -

ISIS neutron and muon source

E-platform: No

Instruments	
Access Route	
Science Areas	
Sponsored Grant	
Grant Title	
Start Date	
Similar Submission?	
Industrial Links	
Days Requested:	
Previous RB Number:	
DOI:	
Sponsor:	
Grant Number:	
Finish Date:	

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Sample record sheet

Principal contact	Dr ESTER FALLETTA, CONSORZIO PHYSIS SRL SB, ITALY		
MRF Instrument	**XRD TOMOGRAPHY**	**Days Requested:**	4
Special requirements:			

SAMPLE

Material	Brass	-	-
Formula	Cu, Zn	-	-
Forms	Solid		
Volume	4 cc		
Weight	10 g		
Container or substrate	No special need	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	RT - K	-	-
Pressure Range	Atmospheric pressure - mbar	-	-
Magnetic field range	No - T	-	-
Standard equipment	-	-	-
Special equipment	No special equipment needed	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	No	-	-
Special equip. reqs	No	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	No	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Wire bending and adhesion investigation with XRD TOMOGRAPHY (CNR-IPCB)

1. Background and context

In the fashion industry, rings of various sizes and finishes are widely used for structural purposes, such as connecting multiple decorative elements, like charms on leather bags, as well as decorative purposes, as aesthetical connection elements on leathergoods. For the production of these rings for charms, the required dimensions are achieved through wire bending of brass wires. These rings are then subjected to surface finishing treatments that serve both protective and decorative purposes. The protective coatings prevent brass corrosion, while the decorative finishes meet the aesthetic demands of design offices. To cater to diverse aesthetic needs, painting is often employed, offering a wide range of colours. However, painting can conceal structural defects such as cracks caused by the bending process. These cracks pose a risk as they can lead to the fracturing of the outer paint layer over time, resulting in oxidation of the product, which impacts the final quality of the item, such as a bag.

2. Proposed experiment

This proposal is part of a dual campaign, integrating findings from Scanning Electron Microscopy (SEM) with XRD Tomography, to investigate the microstructural defects in bended brass wire rings and their interaction with varnish coating, applied as surface treatment.
The objective of this study is to conduct a non-destructive analysis using X-ray tomography to detect internal cracks in the material (the defect should be in the range of several microns), which are not visible to the naked eye, as well as any potential delamination of the paint layer that could create bubbles or voids at the interface between the brass base and the paint applied on the rings. This analysis will help optimize production processes and potentially improve the formulations of the paints used. Additionally, the study aims to compare the formation of cracks between raw brass rings (without surface painting) and painted rings. By conducting a comparative analysis between two sets of samples—rings subjected to repeated bending in both raw brass and painted brass—we aim to better understand the impact of opening and closing during assembly on structural integrity.
This proposal aims to provide comprehensive instruction on the utilization of X-ray tomography, a crucial tool for non-destructive testing and detailed internal analysis.

We therefore request experimental time on the X-ray tomography machine RIGAKU Nano3DX to conduct non-destructive measurements on brass rings manufactured through wire bending. These rings undergo a surface painting treatment and are then subject to further deformation during the assembly process on finished products, such as attaching charms to bags.

During this assembly process, the rings are opened to insert the charms and then closed again. This repeated bending may cause internal cracks within the brass and issues with the adhesion between the brass base and the paint layer. Currently, these assembled pieces are inspected visually; however, over time, they may exhibit corrosion phenomena due to the aforementioned defects.

Therefore, it is crucial to conduct this non-destructive analysis using X-ray tomography to detect any internal cracks in the material, which are not visible to the naked eye, as well as any potential delamination of the paint layer, which could result in bubbles or voids at the interface between the brass base and the paint. The tomography is crucial and essential for this purpose because it allows to conduct the investigation on the samples without the need of mechanical deformations such as in cross-section analysis, allowing to investigate the sample as is.

Additionally, it is important to compare the formation of cracks between raw brass rings (without surface painting) and painted rings. We aim to conduct a comparative analysis between two sets of samples: rings subjected to repeated bending in both raw brass and painted brass.

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The experiment would be carried out at CNR-IPCB, with whom we are already in contact and agreed on the proposed experiment.

3. Summary of previous experimental proposals

The link between the wire bending of brass, varnish application and cracking formation upon opening and closing of the rings during assembly of the final product has traditionally relied on a trial-and-error approach, supplemented by empirical knowledge. This method, while offering some insights, lacks the precision and predictability afforded by scientific analysis. Small and medium-sized enterprises (SMEs) in the fashion sector, in particular, encounter obstacles due to limited access to advanced analytical methods like XRD Tomography and the necessary expertise to interpret data effectively. Scientific literature provides findings that XRD Tomography is an essential tool for the investigation of cracks formation in brass and degradation mechanisms with surface coatings [1-2] but a detailed study on brass articles for fashion and luxury accessories has not been performed so far. This proposal seeks to fill that knowledge gap through focused research, in order to investigate the effects of widely used production processes such as wire bending that would be essential for production technological improvement.

4. Justification of experimental proposals request

As detailed in section 2, XRD Tomography is a crucial tool for this experiment due to its unique capabilities. The first day will be dedicated to the analysis of the brass base material rings, in order to have reference measurements on the behaviour of the wire upon bending; the second day will focus on the varnished sample and to data analysis and comparison.

In detail, we request 4 days of experimental time to analyse 8 samples: 4 samples with no varnish finishing, in order to have a reference measurement of the internal structure by itself, without surface varnish, to compare the cracks formations before opening and closing and after the opening and closing procedure; the other 4 samples will be produced with the same wire bending procedure and will also have the final surface varnish coating, allowing to perform the same set of measures and to compare the results: on the rings as produced and after the “opening and closing” procedure for the final assembly of the finished product. This number ensures comparison between the behaviour upon mechanical stress on the base material itself and with the varnish coating, heling in detecting cracks formation, delamination phenomena between brass and varnish and detection of voids at the interface between base material and finishing layer. This structured approach allows for thorough examination while maintaining a strict timeline.

[1] E. Maire & P. J. Withers (2014) Quantitative X-ray tomography, International Materials Reviews, 59:1, 1-43.

[2] Katsunori Shimizu, Tomohiro Miyata, Tomohiko Nagao, Akemi Kumagai, Hiroshi Jinnai, Visualization of the tensile fracture behaviors at adhesive interfaces between brass and sulfur-containing rubber studied by transmission electron microscopy, Polymer, Volume 181, 2019, 121789.

Experiment Proposal

Experiment number GP2024097

Principal investigator	Dr Chimenti Stefano, CROMOGENIA UNIT, SPAIN
Co-investigator	Dr Gennaro Gentile, IPCB CNR, ITALY
Co-investigator (*)	Dr Marino Lavorgna, CNR, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Analysis of the distribution and morphology of hydrophobic/oleophobic coatings onto textiles by XRD tomography
MRF Instrument	**XRD TOMOGRAPHY**
Days requested:	5
Access Route	Direct Access
Previous GP Number:	no
Science Areas	Chemistry, Engineering, Materials
DOI:	-
Sponsored Grant	None
Sponsor:	-
Grant Title	-
Grant Number:	-
Start Date	-
Finish Date:	-
Similar Submission?	-
Industrial Links	Cromogenia

Non-Technical Abstract

Water-dispersed polymer nanoparticles, obtained by radical emulsion polymerization, are key ingredients in many technological and industrial products, such as coatings, paints, adhesives, sealants, paper and textile additives, drug delivery systems and cosmetics. Cromogenia UNITS, in this context, is actively working in the research and development of fluorine-free polymer based nanoparticles capable of providing the hydrophobic/oleophobic effect to natural fabrics, such as cotton. For the tailoring of the developed products, a deep morphological/structural characterization by XRD tomography of coatings obtained by applying nanoparticles dispersions onto cotton fabrics is needed. In separate experiment proposals, nanoparticles and coatings applied onto fabrics will be characterized by TEM, SEM&C-AFM and SAXS/WAXD.

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Marino Lavorgna, CNR, ITALY
MRF Instrument	**XRD TOMOGRAPHY** **Days Requested:** 5
Special requirements:	

SAMPLE

Material	6 water dispersed polymer nanoparticles, differing for their composition (polymer and inorganic nanoadditives), applied as coatings onto cotton fabrics	-	-
Formula	polymer coatings onto cotton fabrics	-	-
Forms	Solid		
Volume	2 cc		
Weight	2 g		
Container or substrate	vial	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	298 - K	-	-
Pressure Range	1000 - mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	N/A	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	no	-	-
Special equip. reqs	no	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	no	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	no	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Analysis of the distribution and morphology of hydrophobic/oleophobic coatings onto textiles by XRD tomography

Background and Context

Water-dispersed polymer nanoparticles, obtained by radical emulsion polymerization, are key ingredients in many technological and industrial products, such as coatings, paints, adhesives, sealants, paper and textile additives, drug delivery systems and cosmetics.

In the field of the textile industry, for example, some technical properties of fibres, such as repellency to water or other substances, do not meet the needs required of garments during their useful life. To give the repellent effect, a finishing treatment must be applied to the fabric. Currently, fluorinated polymers in aqueous dispersion make it possible to obtain a water and oil repellent finish in the various substrates on which they are applied. Hence their wide use in many sectors and most companies that are dedicated to the marketing of products for textile finishing, constantly find themselves developing new products with new properties and substantial improvements that they give to fabrics in general.

However, in recent years ECHA has placed traditional fluorinated resins under scrutiny as they often contain long-chain perfluorocarbons (PFCs), which are known to persist in the environment and potentially have adverse effects on human health and wildlife. Consequently, research has been oriented towards the development of alternatives with similar hydrophobic and oleophobic properties but with a lower environmental impact given the absence of fluorinated monomers.

Cromogenia UNITS, in this context, is actively working in the research and development of polymers in aqueous dispersion as alternatives to fluorinated polymers which are capable of providing the same repellency to water and good repellency to oils.

Although homogeneous particles consisting of one or single components can be used in many textile applications, multiphase polymer particles (comprising several monomers and/or nanofillers) are preferred, since they synergistically combine various physical and chemical properties of different materials. As a result, a wider range of properties can be achieved, e.g. fast drying hardness and/or high hydrophobicity in water-based systems.

The final performance of multiphase particles depends critically on their morphology and chemical composition. Both morphology and chemical composition are the result of complex kinetic and thermodynamic processes that occur during polymerization.

For this reason, the distribution of the coatings obtained by applying water dispersed polymer nanoparticles on cotton fabric, performed by XRD tomography, is of fundamental importance to tailor the properties of the synthesized materials and their performances.

2. Proposed experiment

The characterization of the coatings obtained by applying the nanoparticles onto cotton substrates will be performed by using the XRD tomography facility available at IPCB-CNR.

In separate experiment proposals, the nanoparticles will be characterized by the TEM-FEI available at IPCB-CNR Unit. Moreover, the nanoparticles and the coated cotton substrates will be characterized by SEM&C-AFM and optical profiler, available at Tor Vergata Unit, and the coatings will be analysed by SAXS-WAXD tavailable at the IPCB CNR Unit, to investigate the the coating structure on fabrics.

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3. Summary of previous experimental proposals or characterisation

No previous experiments have been carried out on these samples

4. Justification of experimental time requested

We have requested the XRD tomography equipment available at the IPCB CNR Unit to evaluate the distribution of 6 types of coatings obtained by applying water dispersed polymer based nanoparticles, differing for their composition (polymer matrix, inorganic additives) onto cotton substrates.

Therefore, a total of 6 samples will be analysed. After discussion with the instrument scientist, we request 5 days of XRD tomography access, for a fully and thorough characterization of the materials. The foreseen beam time accounts set up and for the data collection on the samples.

References

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(2016) ACS Nano, 10 (3), pp. 3042 - 3068.
DOI: 10.1021/acsnano.5b08176

Kausar A.
Nanomaterials for design and fabrication of superhydrophobic polymer coating
(2019) Superhydrophobic Polymer Coatings: Fundamentals, Design, Fabrication, and Applications, pp. 77 - 90
DOI: 10.1016/B978-0-12-816671-0.00005-9

Attia N.F., Moussa M., Sheta A.M.F., Taha R., Gamal H.
Effect of different nanoparticles based coating on the performance of textile properties
(2017) Progress in Organic Coatings, 104, pp. 72 - 80
DOI: 10.1016/j.porgcoat.2016.12.007

Experiment Proposal

Experiment number GP2024107

Principal investigator	Professor Gabriele Gentile, University of Rome Tor Vergata, ITALY
Co-investigator (*)	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Carla Andreani, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Gabriele Scorrano, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Multi-instrumental characterization of Oniscidean isopods to establish the nature and function of their cuticle and tubercles: XCT measurements
MRF Instrument	**XRD TOMOGRAPHY**
Days requested:	5
Access Route	Direct Access
Previous GP Number:	No
Science Areas	Biology and Bio-materials, Physics
DOI:	-
Sponsored Grant	None
Sponsor:	-
Grant Title	-
Grant Number:	-
Start Date	-
Finish Date:	-
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

Andronicus is a genus of oniscidean isopods that includes less than a dozen species, most of which are confined to caves in the Italian Pre-Alps. Andronicus offers the unique opportunity to investigate the nature of the cuticle in cave (A. brentanus) and non-cave-dwelling species (A. dentiger), within the same evolutionary lineage, characterizing mineralization in the different species. The proposed investigation will also extend to including tubercles on the surface of the body, the nature of which, currently unknown, could be of a sensorial nature. Preliminary observations would suggest the presence of a possible innervation at the base of these tubercles, which if confirmed, would prove their nature and function. For the characterization on the mineralization of the cuticle and tubercles we envisage to use EDX, FT-IR, Raman, and X-ray diffraction, whereas the morphology characterization will be done by SEM, TEM and nano-XCT. Here, this proposal is focussed on the XCT analysis.

Publications

Vittori, M. et al., Arthropod Struct Dev. 46 (2016), pp. 96-107.
Gentile, G. and Allegrucci, G., International Journal of Speleology 26 (1997), pp. 47-61.
Neues, F. et al., Cryst. Eng. Comm. 9 (2007), pp. 1245-1251.

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E-platform: No

Instruments	
Days Requested:	
Access Route	
Previous RB Number:	
Science Areas	
DOI:	
Sponsored Grant	
Sponsor:	
Grant Title	
Grant Number:	
Start Date	
Finish Date:	
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY		
MRF Instrument	**XRD TOMOGRAPHY**	**Days Requested:**	5
Special requirements:			

SAMPLE

Material	Oniscidean isopod	-	-
Formula	Organic material, Calcite	-	-
Forms	Solid		
Volume	0.03 cc		
Weight	1-2 g		
Container or substrate	-	-	-
Storage Requirements	Freezer (-20C)	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	-	-

Multi-instrumental characterization of Oniscidean isopods to establish the nature and function of their cuticle and tubercles: XCT measurements

1. Background and Context

Among Crustaceans, oniscidean isopods are uniquely adapted to terrestrial life, exhibiting strongly mineralized cuticles. Oniscideans include several species adapted to the caves. Among the most important evolutionary adaptations found in troglobitic oniscideans (i.e. bound to cave environments, from which they cannot escape due to strict ecological and physiological constraints) are the thinning of the cuticle with a reduce layer of calcite, although calcium carbonate is present in the exocuticle and the endocuticle [1]. Additionally, other adaptations include the lengthening of the appendages, the loss of the eyes, the development of sensory systems alternative to sight such as hygrosensors and chemosensors, usually located in different areas of the body. *Androniscus* (Fig. 1) is a genus of oniscidean isopods that includes less than a dozen species, most of which are confined to caves in the Italian Pre-Alps. Among these, *Androniscus dentiger* is the one that shows the least constraints, being present even in the most superficial layers of the soil in non-cave environments and showing a wide geographical distribution [2]. Indeed, by combining atomic absorption spectroscopy, thermogravimetry and X-ray diffraction, the composition of cuticles in several isopods has been analyzed [3-4]. The use of high-resolution Raman microscopy enabled the determination of the distribution of different mineral phases in the tergal cuticles of some rollers, clingers, and runners [5,6].

Fig. 1 *Androniscus dentiger* (a) and *Androniscus brentanus* (b). Contrary to the second, the first is not troglobite, is pigmented, has thick cuticle, and shows a prominent single-ommatidium eye (arrow).

Androniscus offers the unique opportunity to investigate the nature of the cuticle in cave (*A. brentanus* and more) and non-cave-dwelling species (*A. dentiger*), within the same evolutionary lineage, characterizing mineralization in the different species. The proposed investigation will also extend to including tubercles (tricorns) on the surface of the body, the nature of which, currently unknown, could be of a sensorial nature. Some preliminary observations would suggest the presence of a possible innervation at the base of these tubercles, which if confirmed, would prove their nature and function. For the characterization on the mineralization of the cuticle and tubercles in the two different species (*A. brentanus and A. dentiger*) we plan to use Energy Dispersive X-ray Analysis (SEM-EDX), Fourier-transform infrared spectroscopy (FT-IR), Raman spectroscopy and X-ray diffraction, whereas the morphology characterization will be done by means of electron microscopy techniques (SEM, TEM) and X-ray nano tomography. The benefit of using a multi-instrumental approach would allow us not only to cross compared results to verify their consistency, but also to investigate the degree of resorption/failure to develop the eye in these isopods species, allowing us to observe the presence of vestigial or residual structures, such as for example the presence of an optic nerve, in the absence of the ommatidium (eyeball).

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2. Proposed experiment

In this specific proposal we aim to use the XRD TOMOGRAPHY instrument available at the CNR IPCB Unit for assessing the morphology of the cuticle and tubercles on a n. 6 *Androniscus brentanus and n. 6 Androniscus dentiger* isopods samples. Results of this experiment will be cross compared to verify consistency with data obtained by separate proposals where we request FT-IR, Raman spectroscopy, XRD, SEM-EDX, and TEM measurements on the same set of samples.

3. Justification of experimental time requested

Each of the n. 12 samples of the two Oniscidean isopod species (n. 6 Androniscus brentanus and n.6 Androniscus dentiger) will be washed for 1–2 s in double distilled water to remove tissue saline at the surface and then for 2–5 s in 100% methanol to remove water. Specimens will be air dried and stored at −20 °C until its use on the instrument. For the XCT scanner sample will be fixed to prevent any sample movement on the tomography rotation stage with a field of view of 10.64 mm x 10.64 mm, pixel size of 5.2 μm, and about 1570 projections to fulfil the Niquist-Shannon sampling theorem. With an exposure time per projection of 5 s, each tomography will last about 2 hours. Eventual XCT scans with a reduced field of view for increasing spatial resolution will be evaluated if necessary. Hence, after discussion with the instrument scientist, we request 5 days of instrument time including sample preparation, set-up and calibration time.

References

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[2] Gentile, G. & Allegrucci, G. (1997) Geographic variation and genetic relationships in populations of the *Androniscus dentiger* complex from Central Italy (Isopoda, Oniscidea, Trichoniscidae). International Journal of Speleology, 26: 47-61.
[3] Neues, F., Ziegler, A. & Epple, M. (2007) The composition of the mineralized cuticle in marine and terrestrial isopods: a comparative study Cryst. Eng. Comm. 9: 1245-1251.
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[6] Štrus, J., Žnidaršič, N., Hild, S. & Ziegler, A. (2008) Microscopic anatomy and mineral composition of cuticle in amphibious isopods Ligia italica and Titanethes albus (Crustacea: Isopoda) A. Aretz, B. Hermanns-Sachweh, J. Mayer (Eds.), EMC 2008: Life Sciences, Springer Verlag, Berlin:185-186.
[7] Hornung, E. (2011). Evolutionary adaptation of oniscidean isopods to terrestrial life: Structural-physiological-behavioural aspects. Terrestrial Arthropod Reviews. 4: 95-130.

Experiment Proposal

Experiment number GP2024153

Principal investigator	Dr Monica Carosi, Università Roma Tre, ITALY
Co-investigator	Dr Federica Spani, Università Campus Bio-Medico di Roma, ITALY
Co-investigator (*)	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Multi-instrumental characterization of primate baculum and baubellum bone tissues to support functional hypotheses: nanometric QCT measurement
MRF Instrument	**XRD TOMOGRAPHY** **Days requested:** 5
Access Route	Direct Access **Previous GP Number:** No
Science Areas	Biology and Bio-materials, Physics **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	To better understand the function of primate bacula and baubella bone tissues, we aim to study the micro-architecture of the bone focussed for the first time on characteristics related to either observed shapes and physical-chemical features of this tissue. Key aspects include trabecular density and orientation, bone mineral composition, and the distribution of minerals along the bones. The proposed study will involve and multi-instrumental characterization of different bones which will allow us to fine reconstruct a digital twin of these tissues, and for open exploring image-based finite element analysis to assess the mechanical forces involved during copulation. Here, this proposal is focussed on the nanometric QCT analysis.
Publications	Spani F, Morigi MP, Bettuzzi M, Scalici M, Carosi M, PLoS ONE 15(1): e0228131. Spani, F., Morigi, M., Bettuzzi, M. et al., Sci Rep 11 (2021), 11245. Reznikov, N., Bilton, M., Lari, L., Stevens, M. M. & Kröger,Science 360 (2018), eaao2189.

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
MRF Instrument **XRD TOMOGRAPHY** **Days Requested:** 5
Special requirements:

SAMPLE

Material	Primate bone tissue	-	-
Formula	Ca10(PO4)6(OH)2	-	-
Forms	Solid		
Volume	0.3 cc		
Weight	0.5 g		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	-	-

Multi-instrumental characterization of primate baculum and baubellum bone tissues to support functional hypotheses: nanometric QCT measurement

1. Background and Context

Inside the external genitals of some placental mammals, including Primates, genital bones are present in one or both sexes: the *baculum* (penile bone; pl. *bacula*) and the *baubellum* (clitoral bone; pl. *baubella*). *Bacula* are common in most primate species, whereas *baubella* are rare. Both bones occur in some species of Hominoidea (the human evolutionary lineage), but not in humans. Although homologous, *baubellum* is only present in species where males have a *baculum,* whereas species with *bacula* may lack *baubella*. Various functions have been proposed for the *baculum* (none for the *baubellum*), however only one is supported by correlational data: baculum supports erection and prevents urethral collapse, aiding sperm transport in species with prolonged copulations and high levels of sexual competition. *In fact, baculum* length positively correlates with copulation duration. Recent studies published the most comprehensive dataset on primate *bacula* and *baubella* occurrence, collecting data from primary literature and samples from fresh cadavers and museum specimens (Natural History Museum La Specola in Italy, the American Museum of Natural History in New York, and the National Museum of Natural History in Washington, DC). Using 3D high-resolution, non-invasive micro-Computed Tomography and a new landmark-free shape analysis (the *alpha*-shape technique), these studies identified three distinct internal and external morphologies in primate *bacula* for the first time.

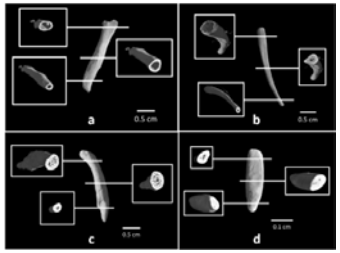

Fig. 1 *3D virtual volumes of 4 different types of primate genital bones, three bacula and one baubellum. For each type, internal structures and cross sections of epiphyses and diaphysis are shown. A: totally hollow structure (Chlorocebus aethiops). B: hollow epiphyses and solid diaphysis with few channels (Otolemur crassicaudatus). C: totally solid structure in both epiphyses and diaphysis with a network of Haversian channels (Papio cynocephalus). D: totally solid structure of baubellum (Sapajus apella) with some Haversian channels*

To better understand the function of primate bacula, the micro-architecture of baculum bone tissue should be investigated focusing for the first time on characteristics related to either observed shapes and mechanical forces exerted on bacula during copulation. Key aspects include trabecular density and orientation, bone mineral composition, and the distribution of minerals along the bones. Potential sex-based differences in these characteristics will aid in interpreting the results. Hence, the proposed study will involve and multi-instrumental approach as follows:

- trabecular density will be assessed by means of quantitative computed tomography (QCT) at different length scale and photon energy which will enable us reconstructing the 3-D bone geometry and volumetric bone mineral density (vBMD);
- trabecular orientation of the bone tissue will be studied in the bulk of the sample by small-angle X-ray scattering (SAXS) to measure crystal shape, their average crystal thickness and their crystal orientation;

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- the structure of bone mineral will be assessed by means of X-ray diffraction (XRD) which is considered as the gold standard for this type of measurement;
- for the compositional characterization of the bone tissue we aim to use nuclear magnetic resonance (NMR) which is uses the responses of isotopes to an external magnetic field to generate compositional information about the sample being scanned, and results will be verified for consistency checks with Raman spectroscopy, Fourier transform infrared spectroscopy (FTIR), X-ray Fluorescence (XRF) spectroscopy, and Energy Dispersive X-ray Analysis (SEM-EDX) data that are used here to determine the chemical and molecular signature of the sample.

2. Proposed experiment

In this specific proposal we aim to use the XRD TOMOGRAPHY instrument available at the CNR IPCB Unit to perform quantitative computed tomography (QCT) measurement that will allow us reconstructing the 3-D bone geometry and volumetric bone mineral density (vBMD) at sub-micrometric length scale on a n. 3 baculum bone tissue and n. 2 baubella bone tissue. Standard sample solutions based on calcium hydroxyapatite (CHA) on distilled water at different concentration will be measured as well for calibration purpose (required for vBMD). Results of this experiment will be cross compared to verify consistency with a coarser spatial resolution (micrometric) QCT data measured on the same set of samples with the RETINA instrument available at the POLIMI Unit, and requested by means of a separate proposal.

3. Justification of experimental time requested

Each of the n. 5 samples will be fixed to prevent any sample movement on the tomography rotation stage with a field of view of 10.64 mm x 10.64 mm, pixel size of 5.2 μm, and about 1570 projections to fulfil the Niquist-Shannon sampling theorem. With an exposure time per projection of 5 s, each tomography will last about 2 hours. Eventual XCT scans with a reduced field of view for increasing spatial resolution will be evaluated if necessary. Hence, after discussion with the instrument scientist, we request 5 days of instrument time including sample preparation, set-up and calibration time.

References

[1] Reznikov, N., Bilton, M., Lari, L., Stevens, M. M. & Kröger, R. Fractal-like hierarchical organization of bone begins at the nanoscale. Science 360, eaao2189 (2018).
[2] Glimcher, M. J. Bone: Nature of the Calcium Phosphate Crystals and Cellular, Structural, and Physical Chemical Mechanisms in Their Formation.*Rev. Mineral. Geochem.*64, 223–282 (2006).
[3] Delmas, P. D., Tracy, R. P., Riggs, B. L. & Mann, K. G. Identification of the noncollagenous proteins of bovine bone by two-dimensional gel electrophoresis. *Calcif. Tissue Int.***36**, 308–316 (1984).
[4] Schultz NG, Lough-Stevens M, Abreu E, Orr T, Dean MD. The Baculum was Gained and Lost Multiple Times during Mammalian Evolution. Integr Comp Biol. 2016 Oct;56(4):644-56. doi: 10.1093/icb/icw034. Epub 2016 Jun 1. PMID: 27252214; PMCID: PMC6080509.
[5] Spani, F., Morigi, M., Bettuzzi, M. et al. The ultimate database to (re)set the evolutionary history of primate genital bones. Sci Rep 11, 11245 (2021). https://doi.org/10.1038/s41598-021-90787-2