ISIS@MACH ITALIA

Science and Technology Facilities Council
ISIS Neutron and Muon Source

Experiment Proposal

Experiment number GP2023011

Principal investigator	Miss Giulia Pallottini, Fondazione Museo Antichità Egizie, ITALY
Co-investigator	Professor Carla Andreani, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator (*)	Mrs Valentina Turina, Fondazione Museo Antichità Egizie, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	WAXS/SAXS characterisation of ancient Egyptian linen textiles from Museo Egizio
MRF Instrument	**SAXS Xenocs Xeuss**
Access Route	Direct Access
Science Areas	Cultural Heritage
Sponsored Grant	None
Grant Title	-
Start Date	-
Similar Submission?	-
Industrial Links	Fondazione Museo Egizio

Days requested: 2
Previous GP Number: -
DOI: -
Sponsor: -
Grant Number: -
Finish Date: -

Non-Technical Abstract

We propose the characterisation of ancient Egyptian linen textiles intended for daily use using Small and Wide-Angle X-ray Scattering (SAXS and WAXS). SAXS and WAXS data will be used to access the decrease of the degree of polymerization of cellulose in naturally aged linen fabrics as a function of the aging time. Additional measurements using Fourier-transform infrared spectroscopy (FT-IR), and Raman spectroscopy are also planned. All the instruments required for this study are operating at the MRF1 of ISIS@MACH ITALIA Research Infrastructure in Italy. FT-IR and Raman spectroscopy data will provide the opto-chemical and neutron analyses of the linen fabrics. Additional measurements using inelastic neutron scattering and epithermal neutrons will complement the pieces of information gathered using the MRF1 instruments.

Publications

G. Festa, T. Christiansen, V. Turina, M. Borla, J. Kelleher, L. Arcidiacono, L. Cartechini, R.C. Ponterio, C. Scatigno, R. Senesi and C. Andreani, Sci. Rep., 9, 7310 (2019)

ISIS neutron and muon source

IM@IT E-platform: Yes - ISIS Facility

Instruments	**MAPS**
Access Route	Direct Access
Science Areas	
Sponsored Grant	None
Grant Title	-
Start Date	-
Similar Submission?	
Industrial Links	Fondazione Museo Egizio

Days Requested: 3
Previous RB Number:
DOI:
Sponsor:
Grant Number:
Finish Date:

Sample record sheet

Principal contact Mrs Valentina Turina, Fondazione Museo Antichità Egizie, ITALY
MRF Instrument **SAXS Xenocs Xeuss** **Days Requested:** 2
Special requirements:

SAMPLE

Material	linen	-	-
Formula	CnHm	-	-
Forms	Solid		
Volume	1 cc		
Weight	1 mg		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	300 - 300 K	-	-
Pressure Range	1000 - 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	None	-	-
Radioactive Hazards	None	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed of by instrument scientist	-	-

Scientific Background

Textiles contain important archaeological information about everyday life, religion, art, technical culture, and history of ancient peoples. Unfortunately, ancient textiles are rare in archaeological sites compared to ceramic, metal, and wood artifacts because of their structural fragility under the action of humidity, temperature, fungi, microbes, and insects. Nevertheless, especially in Egypt or the Near East, there are many cases in which well-preserved textiles have been discovered in crypts and ground caves [1]. This is the case of the textile collection preserved in the *Museo Egizio* (Turin, Italy), an example of which is shown in Fig. 1 and Fig. 2, and that represents a unicum for quality, quantity, and variety covering a vast chronological period of over 4500 years of history. Among the mentioned collection of textiles, a number of items of fabrics found in the Tomb of Kha [2] and already studied [3] with different techniques will be investigated further in this new set of measurements.

Figura 2. Inv. N° S. 08543 - Perfectly preserved tunic

Figura 2. Inv. N° ST 27/4 - Example of a fragmented and fragile shroud (it is possible to see the flax powder from the textile)

Archeological textiles can comprise braided and woven structures from animal or vegetable fibers. Flax is a vegetable textile frequently used by populations of the Mediterranean geographical area. One reason is that linen fibers are the most resistant vegetable fibers to environmental aging agents (oxygen, humidity, light, etc.). Deterioration of vegetable fibers is related to cellulose degradation, the main component of their structure. Cellulose in linen fabrics is a polymer, which contains an hydroglucose units bonded by β-glycosidic bonds. Aging of textiles can have thermal, hydrolytic, photolytic, photochemical, and enzymatic origins. All these physical and chemical agents degrade cellulose, provoking the scission of the polymeric chains by breaking the β-glycosidic bonds [4-6]. Within any glucose unit, three hydroxyl groups are available for oxidation, leading to a great variety of structural modifications and combinations of the cellulose through oxidative processes. Depending on the particular stress imposed on cellulose, the individual hydroxyl groups in both the polymeric chain and the hydroglucose units are involved to a varying extent in giving rise to carbonyl and carboxyl groups. These groups are usually introduced in the structure of cellulose through material processing steps, such as bleaching. Furthermore, high energy radiation can cause a considerable increase in carbonyl groups in addition to chain scission, mainly through the action of the generated radical species. For example, the number of carbonyls introduced by irradiation is directly correlated to the applied dosage [7].

Carbonyl and carboxyl group formations in cellulose are also caused by natural aging, which happens at room temperature and humidity. Indeed, the presence of oxygen, often in the presence of light, induces many auto-oxidative reactions [8-14]. Radical reactions come into play leading to formation of carbonyl or carboxyl groups, to chain cleavage, and thus also to a loss in the degree of polymerization and in the fiber strength.

For this reason, the aim of this proposal is to study by means of SAXS and WAXS the decrease of the degree of polymerization of cellulose in naturally aged linen fabrics as a function of the aging time, mainly due to thermal and hydrolytic actions which have occurred where they have been preserved for centuries. This could in principle enable a non-invasive method for dating ancient linen threads by inspecting their structural degradation, and results could be compared with standard

radiocarbon dating data available for the set of samples under investigation. Furthermore, we aim to complement these measurements, with FT-IR and Raman spectroscopy data to get the opto-chemical analyses of the linen fabrics. A complementary investigation using Inelastic Neutron Scattering is planned using the MAPS beamline and with epithermal neutrons on the VESUVIO spectrometer, both at ISIS Facility to collect a neutron analysis of the fabrics.

Proposed Experiments and requested beam time on MRF1.

We aim to measure n. 20 linen samples on the Xenocs XEUSS 3.0 system equipped with a copper anode microsource (λ = 0.15405 nm) in the range of scattering vector from 1.5 to around 31 nm^{-1}. We propose to measure WAXS data for each linen sample directly mounted on the sample holder with an acquisition time of 1800 s. Hence, we request two days of beamtime on the Xenocs XEUSS 3.0 system accounting for the set-up and change of each specimen. Moreover, we aim also to collect Raman spectroscopy data on the same linen samples by means the Raman Confocal Microscopy instrument (inViaTM QontorTM model) and using the FT-IR spectroscopy and microscopy instrument (Nicolet Nexus 870 model). All the mentioned pieces of equipment are part of the IM@IT MRF1 and access is being requested through separate proposals.

References

[1] Cybulska, M.; Maik, J., "Archaelogical Textiles—A need for new methods of analysis and reconstruction", Fibers Text. East. Eur. 2007, 15, 64-65.
[2] Schiaparelli, E. Relazione sui lavori Della Missione Archeologica Italiana in Egitto (anni 1903-1920), volume secondo: La Tomba Intatta dell'Architetto Cha Nella Necropoli di Tebe, Torino 1927. The intact tomb of the architect Kha in the necropolis of Thebes (reprint with english translation). (AdArte, 2008).
[3] G. Festa, T. Christiansen, V. Turina, M. Borla, J. Kelleher, L. Arcidiacono, L. Cartechini, R.C. Ponterio, C. Scatigno, R. Senesi and C. Andreani, *Scientific Reports*, **9,** 7310 (2019).
[4] Potthast, A.; Rosenau, T.; Kosma, P., "Analysis of Oxidized Functionalities in Cellulose", Adv. Polym. Sci. 2006, 205, 1-48.
[5] Ding, H.Z.; Wang, Z.D., "On the degradation evolution equation of cellulose", Cellulose 2008, 15, 205-224.
[6] Stephens, C.H.; Whitmore, P.M., "Comparison of the degradation behaviour of cotton, linen, and kozo papers", Cellulose 2013, 20, 1099-1108.
[7] Takacs, E.;Wojnarovits, L.; Földvary, C.; Borsa, J.; Sajo, I., "Effect of combined gamma-irradiation and alkali treatment on cotton–cellulose", Radiat. Phys. Chem. 2000, 57, 399–403.
[8] Feller, R.L.; Lee, S.B.; Bogaard, J., "The kinetics of cellulose deterioration", Adv. Chem. Ser. 1986, 212, 329–347.
[9] Bouchard, J.; Overend, R.P.; Chornet, E.; Van Calsteren, M.-R.J., "Mechanism of dilute acid hydrolysis of cellulose accounting for its degradation in the solid state", Wood Chem. Technol. 1992, 12, 335–354.
[10] Calvini, P., "The Influence of Levelling-off Degree of Polymerisation on the Kinetics of Cellulose Degradation", Cellulose 2005, 12, 445–447.
[11] Whitmore, P.; Bogaard, J., "Determination of the Cellulose Scission Route in the Hydrolytic and Oxidative
Degradation of Paper", Restaurator 1994, 15, 26–45.
[12] Whitmore, P.; Bogaard, J., "The Effect of Oxidation on the Subsequent Oven Aging of Filter Paper", Restaurator 1995, 16, 10–30.
[13] Kolar, J., "Mechanism of Autoxidative Degradation of Cellulose", Restaurator 1997, 18, 163–176.
[14] Margutti, G.; Conio, G.; Calvini, P.; Pedemont, E., "Hydrolytic and Oxidative Degradation of Paper", Restaurator 2001, 22, 67–83.