ISIS@MACH ITALIA

Science and Technology Facilities Council
ISIS Neutron and Muon Source

# Experiment Proposal

Experiment number GP2024013

| | |
|---|---|
| **Principal investigator (*)** | Dr Francesco Saliu, Universita&039; Milano Bicocca, ITALY |
| **Co-investigator** | Dr Massimiliano Clemenza, INFN, ITALY |
| **Co-investigator** | Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY |
| **Co-investigator** | |
| **Co-investigator** | |
| **Co-investigator** | |
| **Co-investigator** | |
| **Co-investigator** | |
| **Co-investigator** | |
| **Experiment title** | Continuation of Nanofibers from textiles: determining photo-degradation induced physicochemical modification of natural and synthetic fibers surface by AFM-RAMAN |
| **MRF Instrument** | **AFM Raman** |
| **Access Route** | Direct Access |
| **Science Areas** | Environment |
| **Sponsored Grant** | None |
| **Grant Title** | - |
| **Start Date** | - |
| **Similar Submission?** | - |
| **Industrial Links** | - |

**Days requested:** 3
**Previous GP Number:** GP2023074
**DOI:** -
**Sponsor:** -
**Grant Number:** -
**Finish Date:** -

**Non-Technical Abstract**

Nowaday Microfibers (MFs) pollution is widespread. Textile washing has been identified as the major source. However, the identification of the factors that influence the MFs release from textiles is underway. Moreover, MFs negative effects on living organisms has been less studied than those associated with spherical particles and very few research has focused on submicrometric fibers (NFs).This may accounted as a significant knowledge gap. The research aim to define the chemo-physical process causing microfiber and nanofiber release, identify the main modification occuring on the fiber surface and suggest technical solution to limit their environmental impacts. The present proposal represent the second stage of experiments of a previous proposal. The MRF instrumentation will be used for the identification of the key chemo-physical modfiication induced by photo-degradation leading to NFs release

**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** | |

# Sample record sheet

**Principal contact** Dr Francesco Saliu, Universita&039; Milano Bicocca, ITALY
**MRF Instrument** **AFM Raman** **Days Requested:** 3
**Special requirements:**

## SAMPLE

| | | | |
|---|---|---|---|
| **Material** | polymeric microfibers (cellulose, polyethilene, polyesther subjected to artificial weathering) deposided on a glass slide (or we can provide colloidal solution to be despersed on the stub) | cellulose nanofibrillated | Photoaged PET fibers |
| **Formula** | CxHxOx ( polypropylene with unknow degree of surficial oxidation) | CnHnOn nanofibrillated cellulose with unknow degree of photo-degradation | CnHnOn PET with unknow degree of surface oxidation |
| **Forms** | Solid | Solid | Solid |
| **Volume** | cc | cc | cc |
| **Weight** | 50 mg | 100 mg | 100 mg |
| **Container or substrate** | samples can be provided despersed as colloidal solution in a glass vial or deposited onto a glass slide or onto the required stab | - | - |
| **Storage Requirements** | - | - | - |

## SAMPLE ENVIROMENT

| | | | |
|---|---|---|---|
| **Temperature Range** | 290 - 310 K | 290 - 320 K | - K |
| **Pressure Range** | - mbar | - mbar | - mbar |
| **Magnetic field range** | - T | - T | - T |
| **Standard equipment** | Sample Changer | - | - |
| **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 | No | No |
| **Experiment Hazards** | no | - | - |
| **Equipment Hazards** | - | - | - |
| **Biological hazards** | no | - | - |
| **Radioactive Hazards** | no | - | - |
| **Additional Hazards** | - | - | - |
| **Additional Details** | - | - | - |
| **Sample will be** | Removed By User | Disposed by IS | Disposed by IS |

**Continuation of Nanofibers from textiles: determining photo-degradation induced physicochemical modification of natural and synthetic fibers surface by AFM-RAMAN**

***Background and Context***

Recent environmental research highlighted how microfibers (MFs) are widespread in the environment, from subsurface oceanic seawater to the deep sea, from the atmosphere to the living organism (Suaria et al 2020). Textile washing has been identified as the major source of MFs. However, the identification of the factors that influence the MFs release from textiles is underway (Saliu et al. 2021). Moreover, the negative effects of MFs on living organisms have been less studied than those associated with spherical particles and very little research has focused on submicrometric fibers (NFs). This may be accounted as a significant knowledge gap in the current literature. The research carried out by the Environmental Chemistry research group at the University of Milano Bicocca aims to define under laboratory conditions the chemo-physical process causing microfiber and nanofiber release and identify the main modification occurring on the fiber surface caused by weathering. Moreover, a model to understand fragment aggregation in water is required. The information collected through the experiment may help in understanding MFs environmental fate and impacts. The material submitted to the MRF facility were previously submitted to artificial weathering under laboratory-controlled conditions to mimic different environmental stresses and were characterized by employing a wide range of analytical techniques but AFM-Raman. The research is currently financed by University of Milano-Bicocca funds (FAR and MUSA) and involves several international collaborations (University of Stockholm, University of South Carolina, CNR-ISMAR, University of Foggia)

***Proposed Experiment***

The AFM-RAMAN MRF instrumentation will be used to add new information regarding the photodegradation mechanism of fibers in textiles through the identification of the key surface chemo-physical modification, directly on the nanofiber surface Specifically, it is requested the chemical characterization of the surface of polypropylene nanofiber obtained after photodegradation under Xenon lamp with a focus on the recognition of the degree of oxidation and the identification of the main functional groups originated by the chemical photo-oxidation, concerning the nanofiber morphology (e.g., cracks and pitches). AFM and Raman spectroscopy have been used to characterize plastic particles with sizes < 100 nm (Fang et al., 2020, Stawikowska and Livingston, 2013). Compared with SEM, AFM can characterize particles more comprehensively (Fu et al., 2020). Coupling Raman spectroscopy affords the nanoscale chemical description of the sample surfaces (Fu and Zhang, 2017). To date, only a few studies applied hybrid AFM techniques (AFM/IR or AFM/Raman) to detect and characterize nano plastics, and no NFs.

***Summary of previous instrument time or characterization***

Materials to be submitted to the AFM-Raman analysis were already preliminarily characterized by FTIR and Raman, photo-aged, characterized by SEM, DLS, and Mass Spectrometry, and toxicological assayed with different biological models. Moreover on the first round of the proposal the MRR- facilities has already provided interesting results regarding the NPs characterization and the proposal is renewed for additionat 3 days of analysis and completation of the survey on different NPs materials

***Justification of instrument time request***

The request is for 3 days of the AFM-RAMAN instrument time needed for the collection of a statistically significant number of Raman signals taken from different collection points. The aim is to characterize the main chemical modification of the material induced by photo-degradation. We assume, after the first round of experiments, about 1 hour for set-up of the experimental parameters for the new materials, and, up-to 1.5 hours per sample

measurement, and an overall number of 10 samples (from 3 different polymeric materials). These measurements should be divided between the 3 days requested.

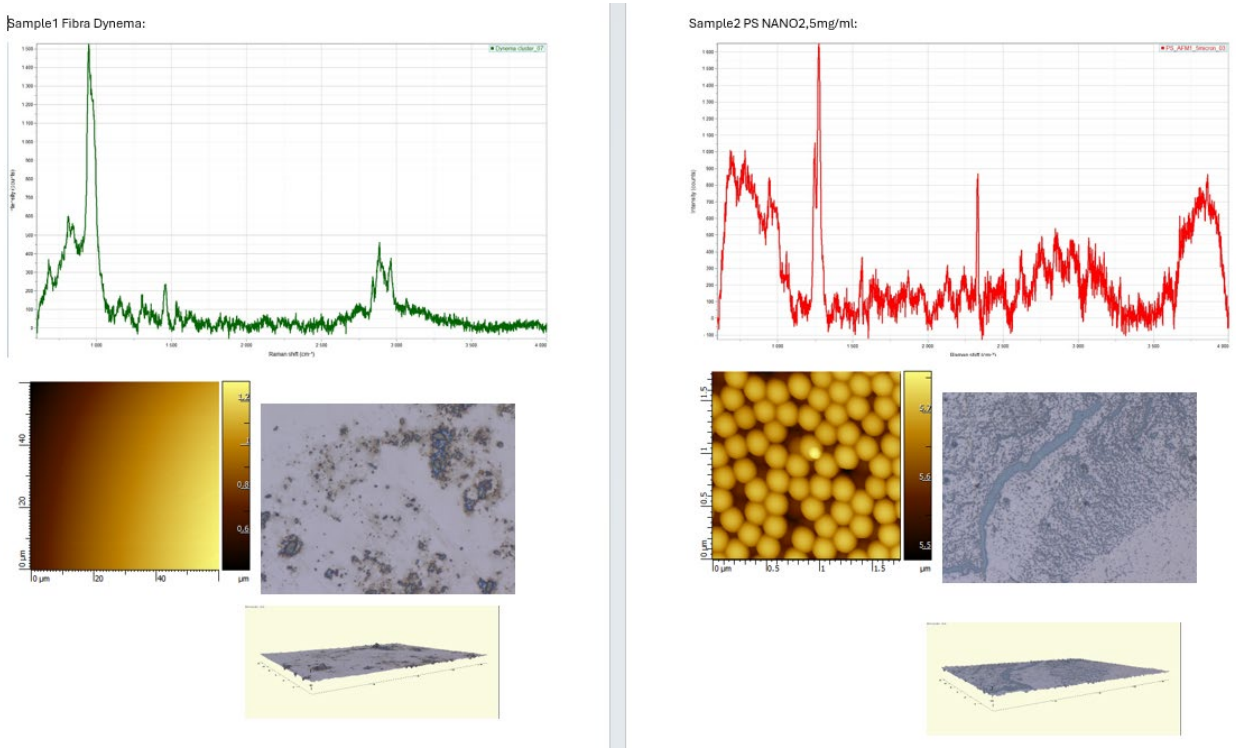


**Figure 1.** Representative characterization of NFs an NPs obtained from the previous access to the AFM-Raman MRF related to the proposed experiment.

***References***

G. Suaria et al. Microfibers in oceanic surface waters: A global characterization. Sci Adv. 23, (2020):

F. Saliu et al . The release process of microfibers: from surgical face masks into the marine environment. Environ. Adv.4 ( 2021)

C. Fang, et al. Identification and visualisation of microplastics/ nanoplastics by Raman imaging :smaller than the diffraction limit of laser? Water Res. (2020), p. 183

W. Fu, W. Zhang Hybrid AFM for nanoscale physicochemical characterization: recent development and emerging applications Small, 13 (2017)

J. Stawikowska, A.G. Livingston Assessment of atomic force microscopy for characterisation of nanofiltration membranes J. Membr. Sci., 425 (2013), pp. 58-70