ISIS@MACH ITALIA

Science and Technology Facilities Council
ISIS Neutron and Muon Source

Experiment Proposal

Experiment number GP2024031

Principal investigator	Professor Anita Grozdanov, Skopje University, MACEDONIA
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 structural analysis of polyninylalcohol/polyacrylic acid/2D filler nanocomposites
MRF Instrument	**SAXS WAXD**
Access Route	Direct Access
Science Areas	Engineering
Sponsored Grant	None
Grant Title	-
Start Date	-
Similar Submission?	-
Industrial Links	-

Days requested: 4
Previous GP Number: -
DOI: -
Sponsor: -
Grant Number: -
Finish Date: -

Non-Technical Abstract

With the objective of preparing new nanocomposites with high gas barrier properties, in this activity new polymer blends filled with different 2D nanofillers have been realized at variable composition. As a polymer matrix, an easy water soluble polvinylalcohol, high amorphous polyvinylalcohol (HAVOH) has been used, blended with polyacrylic acid (PAA). Indeed, after thermal treatments, HAVOH/PAA blends are prone to give light crosslinking, with improvement of their stability to high relative humidity environments. Activities are on-going to evaluate the effect of 2D fillers (graphene oxide - GO, reduced graphene oxide - rGO, lamellar zirconium phosphates - ZrP, hectorite-Hect) on gas barrier properties of HAVOH/PAA blends. These systems have already shown very interesting barrier properties to oxygen. In order to further improve their performances, a detailed structural analysis by SAXS WAXD available at IPCB CNR is needed.

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:

Sample record sheet

Principal contact Dr Gennaro Gentile, IPCB CNR, ITALY
MRF Instrument **SAXS WAXD** **Days Requested:** 4
Special requirements:

SAMPLE

Material	HAVOH/PAA/graphene derivatives (5 samples)	HAVOH/PAA/ZrP (3 samples)	HAVOH/PAA/Hectorite
Formula	HAVOH/PAA/graphene derivatives (5 samples)	HAVOH/PAA/ZrP (3 samples)	HAVOH/PAA/Hectorite
Forms	Solid	Solid	
Volume	1 cc	1 cc	1 cc
Weight	1 g	1 g	g
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
Special equipment	N/A	N/A	N/A

SAFETY

Prep lab needed	Yes	Yes	Yes
Sample Prep Hazards	No	No	No
Special equip. reqs	-	-	-
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 structural analysis of polyninylalcohol/polyacrylic acid/2D filler nanocomposites

1. Background and Context

Polymer composites with nanoparticles as fillers are a growing group of materials with interesting properties for variety of application. Although numerous composites with nanofillers have been prepared and studied in last decade, mainly with carbon based fillers as carbon nanotubes or graphene, there are still challenges when new type of nanoparticles are discovered or synthetized.
Different types of 2D nanofillers are very useful to impart gas barrier properties to composite materials.
With the objective of preparing new nanocomposites with high gas barrier properties, in this activity new polymer blends filled with different 2D nanofillers have been realized at variable composition. As a polymer matrix, an easy water soluble polvinylalcohol, high amorphous polyvinylalcohol (HAVOH) has been used [2], blended with polyacrylic acid (PAA). Indeed, after thermal treatments, HAVOH/PAA blends are prone to give light crosslinking, with improvement of their stability to high relative humidity environments. In an on-ongoing activity, HAVOH/PAA blends have been additivated with MXenes, in particular Ti3C2, prepared by etching the aluminium from the MAX phase Ti3AlC2. These nanocomposite systems have been characterized by morphological and structural analysis in previous experiments carried out at ISIS@MACH Italy.
Further activities are on-going to evaluate the effect of other 2D fillers (graphene oxide - GO, reduced graphene oxide - rGO, lamellar zirconium phosphates - ZrP, hectorite-Hect) on gas barrier properties of HAVOH/PAA blends. These systems have already shown very interesting barrier properties to oxygen. In order to further improve their performances, a detailed structural analysis by SAXS WAXD available at IPCB CNR is needed.

2. Proposed experiment

The HAVOH/PAA nanocomposites additivated with graphene oxide (GO), reduced graphene oxide (rGO), zirconium phosphates intercalated with different ions, and hectorite, have been realized by Skopje University - Faculty of Technology and Metallurgy, in cooperation with IPCB-CNR. In particular, HAVOH/PAA blends in water solutions have been prepared and additivated with the 2D fillers. Then films (about 50 micrometer thick) have been prepared by water casting. On the obtained films thermal treatments have been performed in oven to promote crosslinking between the HAVOH and the PAA phase. The following samples have been prepared for their characterization by SEM FEI: 1) HAVOH/PAA; 2) HAVOH/PAA + 3 phr GO; 3) HAVOH/PAA + 3phr rGO; 4) HAVOH/PAA + 3phr ZrP1; 5) HAVOH/PAA + 3phr ZrP2; 6) HAVOH/PAA + 3phr ZrP3; 7) HAVOH/PAA + 3phr Hec; 8) HAVOH/PAA + 5 phr GO; 9) HAVOH/PAA + 5phr rGO.
The following characterization will be performed on these samples to evaluate the effect of the composition on the morphology of the composites:

- Small and Wide-Angle X-ray Diffractometer (SAXS/WAXD) to obtain info about orientation of 2D fillers and crystallinity degree of polymeric phase. It is proposed to measure n. 9 samples by modulating the acquisition time to optimize the spectra and highlight the presence of the

fillers, by scanning the accessible q range from 0.06nm-1 to 40.7 nm-1. Hence, we request 4 days of beamtime which account also for setup time, and eventual beam loss time.

In distinct proposals the same samples will be analyzed by Scanning Electron Microscopy (SEM FEI), available at the IPCB CNR Unit.

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 to evaluate the effect of the composition on the structure of the composites.

We request 4 days of SAXS WAXD beam time, necessary for the structural characterization of the 9 above-described materials, after discussion with the instrument scientist. The foreseen beam time accounts set up and for the data collection on the samples.

References

[1] M. Naguib, M. Kurtoglu, V. Presser, J. Lu, J. Niu, M. Heon, L. Hultman, Y. Gogotsi, and M. W. Barsoum. Two-dimensional nanocrystals produced by exfoliation of Ti 3AlC 2. Adv. Mater. 23 (2011), p. 4248–4253.

[2] C. Santillo, A.P. God, R.K.Donato, R.J.Espanhol Andrade, G.G. Buonocore, H. Xia, M. Lavorgna, A. Sorrentino. Tuning the structural and functional properties of HAVOH-based composites via ionic liquid tailoring of MWCNTs distribution. Composites Science and Technology, 207, 2021, 108742.