Experiment Proposal

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Experiment title	SEM FEI morphological analysis of polyninylalcohol/polyacrylic acid/2D filler nanocomposites		
MRF Instrument	**SEM FEI**	**Days requested:** 3	
Access Route	Direct Access	**Previous GP Number:** -	
Science Areas	Engineering	**DOI:** -	
Sponsored Grant	None	**Sponsor:** -	
Grant Title	-	**Grant Number:** -	
Start Date	-	**Finish Date:** -	
Similar Submission?	-		
Industrial Links	-		

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 [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.

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. To further improve their performances, a detailed morphological analysis by SEM FEI available at IPCB CNR to evaluate the filler spatial distribution in the polymer matrix is needed.

Publications   -

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

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
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Similar Submission?	
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Sample record sheet

Principal contact   Dr Gennaro Gentile, IPCB CNR, ITALY
MRF Instrument   **SEM FEI**   **Days Requested:** 3
Special requirements:

SAMPLE

Material	HAVOH/PAA/graphene derivatives (5 samples)	HAVOH/PAA/ZrP (3 samples)	HAVOH/PAA/Hectorite (1 sample)
Formula	HAVOH/PAA/graphene derivatives (5 samples)	HAVOH/PAA/ZrP (3 samples)	HAVOH/PAA/Hectorite (1 sample)
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	- K	- K	- K
Pressure Range	- mbar	- mbar	- mbar
Magnetic field range	- T	- T	- T
Standard equipment	None	None	None
Special equipment	n/a	-	no

SAFETY

Prep lab needed	Yes	Yes	Yes
Sample Prep Hazards	NO	no	no
Special equip. reqs	-	-	-
Sensitivity to air	Yes	Yes	Yes
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	Disposed by IS	Disposed by IS	Disposed by IS

SEM FEI morphological 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) at variable molecular weight. 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 Ti_3C_2, prepared by etching the aluminium from the MAX phase Ti_3AlC_2. 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 further 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 morphological analysis by SEM FEI available at IPCB CNR to evaluate the filler spatial distribution in the blend polymer matrix 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:

- Morphological analysis by scanning electron microscope (SEM FEI) (Unit IPCB CNR): to obtain info about the effect of the blend composition and the additivation of 2D fillers on the

morphology of the samples. It is proposed to measure n. 9 samples. Cryo-fractured surfaces of the samples will be analyzed. SEM analysis should be performed at suitable acceleration voltage using secondary electron detectors.

In distinct proposals the same samples will be analyzed by small and wide angle X ray diffraction (SAXS WAXD), 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 request the SEM FEI equipment available at the IPCB CNR Unit to evaluate the effect of the composition on the morphology of the composites.
We request 3 days of SEM FEI beam time, necessary for the morphological characterization of the 9 above described materials, after discussion with the instrument scientist. The foreseen beam time accounts for 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.