INNOSTORAGE – USE OF INNOVATIVE THERMAL ENERGY STORAGE FOR MARKED ENERGY SAVINGS AND SIGNIFICANT LOWERING CO_2 EMISSIONS

Beneficiaries:

Partners:

D7.2 - Report on Staff Exchanges

	Name and Institution	Signature and date
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1 Objective

On October 2013, Prof Dr. M. Farid visited the University of Barcelona for one week. During his visit, the research program of the PhD student Jessica Giró to work at the University of Auckland was set within the frame of the IRSES project. Jessica Giró accordingly has started her work at University of Auckland from 10th February and ended the 7th May 2014.

The initial working plan was very ambitious and there was not enough time to carry out the proposed synthesis of neither phase change materials (PCM) nor microencapsulated phase change materials (MPCM). Nevertheless, the rest of planned activities about characterization of MPCM were fully developed and will be published. The main objective of this secondment was to determine the physico-chemical and mechanical characterization of some MPCM. The studied samples were commercial ones and also samples manufactured in the Department of Chemical and Materials Engineering, University of Auckland, under the responsibility of Prof. Dr. M. Farid. Description of work

An exhaustive characterization of morphological aspects as well as physico-chemical and mechanical properties was performed by using different techniques.

2 Introduction

Microencapsulated phase change materials (MPCM) are widely known in advanced technologies for their usage in passive and active systems (as Phase Change Slurries, PCS). These materials have the ability to absorb and release the latent heat involved in a phase change process. MPCM consist on little polymeric containers and as PCM, paraffin wax in the inner part of the MPCM. The MPCM usage has many advantages due to microcapsules can handle phase change materials as core permitting the slurries preparation. Nevertheless, there are few drawbacks about the thermal cycling reliability of these MCPM and slurries because of the possible rupture of the MPCM shell during charging/discharging process and the subsequent loss of effectiveness. This fact encourages the study of the mechanical properties for these type of materials, as well as the physico-chemical characterization to better understand their behavior.

3 Materials and Methodology

The samples used to perform the physico-chemical and mechanical characterizations were:

- Sample A: BASF

- Sample B: 18D, Microtek

- Sample C: 24D, Microtek

- Sample D: 28D, Microtek

- Sample E: M-2, University of Auckland

- Sample F: M-2 3 fridge cycles, University of Auckland

- Sample G: M-2-RT-58, University of Auckland

- Sample H: Sanelle_sample 25, University of Auckland

Besides, the characterization was executed through the usage of some techniques, as:

Scanning Electron Microscopy (SEM): By using SEM, the size and shape of the samples were characterized. The device used was a FEI Quanta 200 F (FEG = Field Emission Gun), with an EDS Detector SiLi (Lithium drifted) with a Super Ultra-Thin Window. The sputter coater used for standard SEM samples is a Quorum Q150RS sputter coater. It was designed to give a very thin, minimal metal coating suitable for SEM viewing. The normal target used is Pt or Au (current 20mA for Pt). The coating thickness and its uniformity depend on the distance between sample and target, the topography of sample, the affinity of material for metal coating, the surface area of target (increases with use), the applied voltage, the original vacuum, the cleanliness of the chamber, the partial pressure of argon gas, and any oily material or solvent present (affects vacuum at surface).

Fourier-Transformed Infrared Spectroscopy (FT-IR): This technique was used to chemically characterize the shell of the MPCM. A Spectrum 100 FT-IR Spectrometer from Perkin Elmer was used to study the polymeric shell of the different MPCM samples was used. Also, it was needed the OMNIC Specta software, from Thermo Scientific, to analyze the results interpretation.

Nanoindentation technique: This technique was used to characterize the mechanical properties of the samples. This methodology is recognized as a suitable way to test the strength of the shell for individual microcapsules. The MTS Nano Indenter XP was the used device (Figure 2a). Aluminum stubs of 20 mm height and 30 mm diameter were needed to stick the samples at the top (Figure 2b). A red color nail polished was used as a glue to stick the samples (Figure 2c). The samples were put at the same time in the holder, and the instrument parameters were the same to avoid different variables.

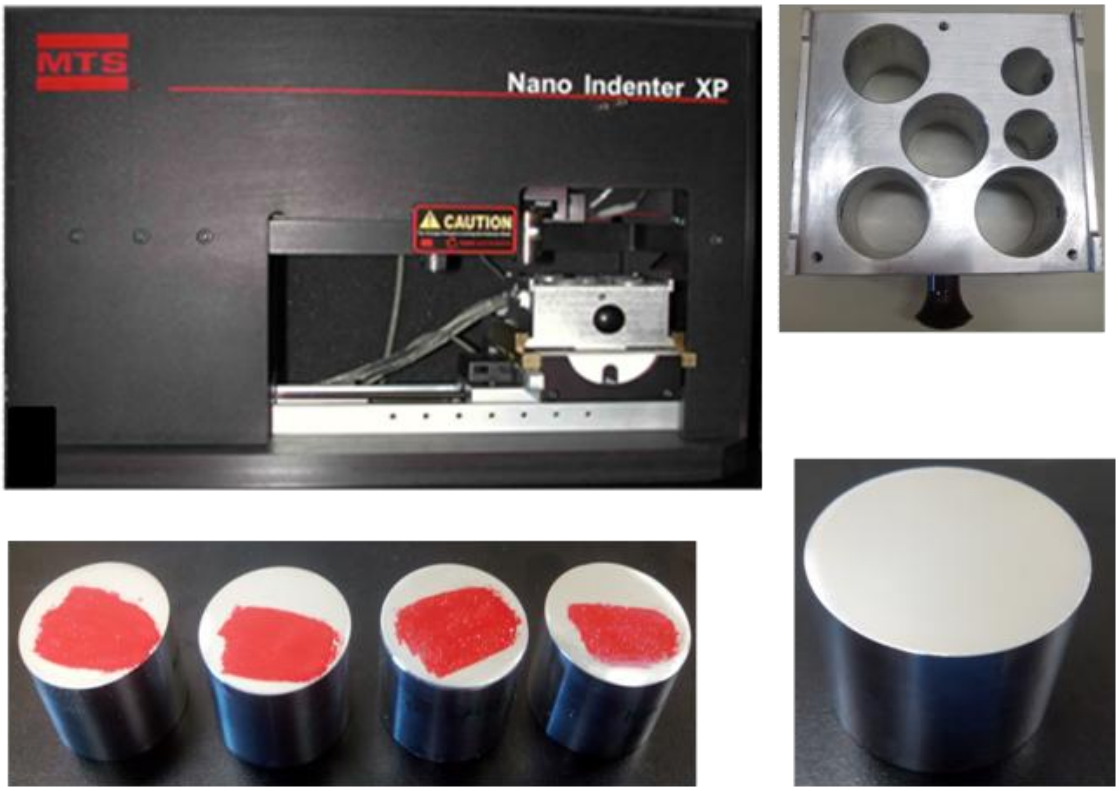

Figure 1. Instrument, stub, holder

Gas Chromatography / Mass Spectroscopy (GC/MS): The objective of using this equipment was to analyze the organic volatiles of the MPCM when heated. A GC-17A Gas chromatograph Shimadzu instrument was used coupled to a GCMS-QP5000 Gas chromatograph Mass Spectrometer Shimadzu device. A calibration of the instrument was conducted with pure compounds (paraffin). A vial of 0.5 ml was used of each pure component from $C_{12}H_{24}$ to $C_{17}H_{36}$, and 0.5 g of each component from $C_{18}H_{38}$ to $C_{24}H_{50}$. Figure 2 shows some images of the SPME holder, the water bath and the heating ramp.

Figure 2. SPME holder inside the vial, septum, needle, water bath, diagram of heating ramp.

4 Results

Scanning Electron Microscopy (SEM): Figure 3 shows the size and shape of two MPCM images of the studied samples at different magnifications.

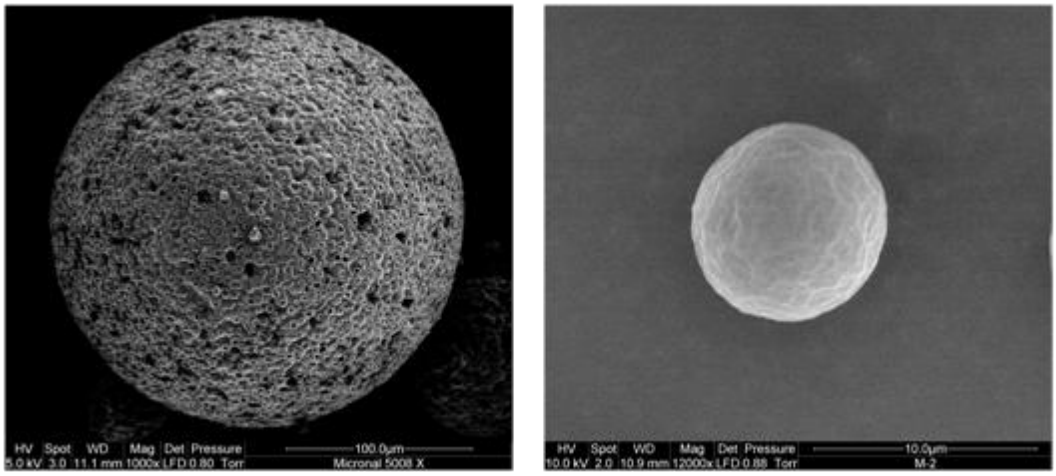

Figure 3. Sample A and sample E

Fourier-Transformed Infrared Spectroscopy (FT-IR): Figure 4 shows the spectra for one of the studied samples. The main result is that samples A, E, F, G and H show the same spectra, because they have the same polymeric shell. Otherwise, spectra B, C, and D are also the same polymer, but different from the other one.

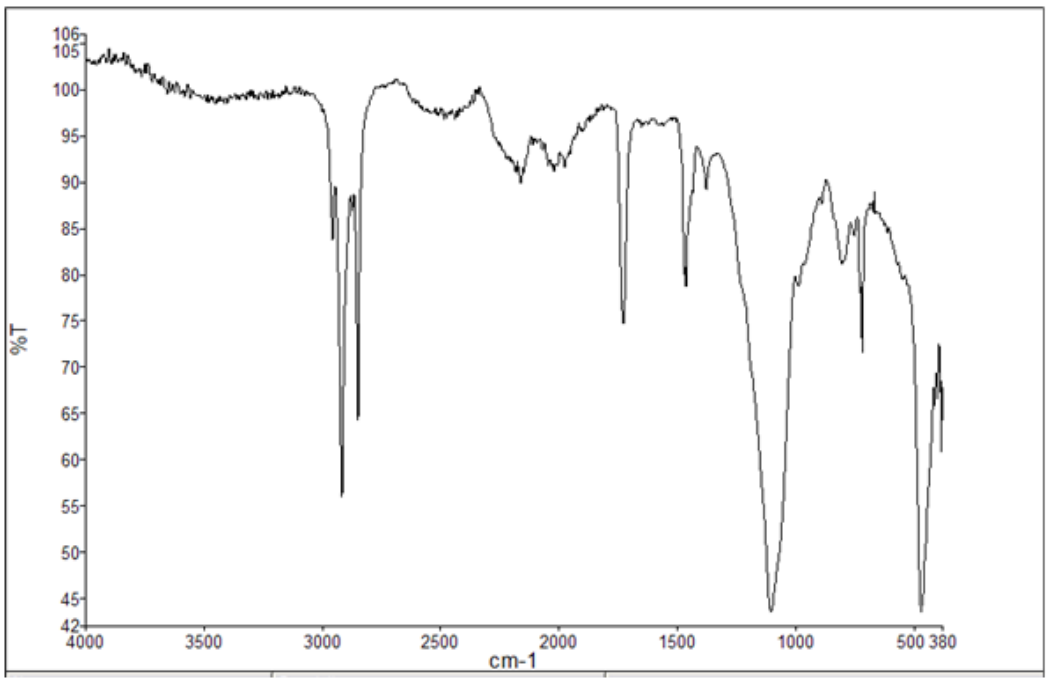

Figure 4. FT-IR of sample A.

Nanoindentation technique: These results can't be published because it is needed to check the obtained results.

Gas Chromatography / Mass Spectroscopy (GC/MS): After the calibration, each sample was situated inside a crystal vial HS 50 ml capacity. A clamp was used to hold the vial in a tempered water bath at 25 ºC for 30 min. After that, a fiber solid-phase microextraction (SPME) holder is punctured on the top of the silicone cap. The lot number of the SPME holder was P268618D 57330-U. This SPME holder was provided by Supelco. Therefore, a 10 min desorption was done. The ramp inside the device was 60 ºC during 2 min. After that, a ramp of 15 ºC min-1 was programmed. Finally, it was heated during 10 min to 230 ºC. This experimental procedure was done at 25 ºC, 35 ºC, 45 ºC, 55 ºC, and 65 ºC, to evaluate the differences.

The column was HP-5MS, with a serial number 1553434H, having a thickness of 0.5 μm, 30 m of length, and 0.32 cm of diameter. Besides, in the Gas Chromatographer part of the whole device, the injection and interface temperature were 200 and 280 ºC, respectively. There were more parameters to be taken into account, such as: the split mode, the inlet pressure: 1 kPa, the flow 1.1 ml min^{-1}, the lineal velocity 38.7 cm sec^{-1}, split ratio between peaks of 20, and finally, the total flow for the he gas is 23.1 ml min^{-1}. On the other hand, in the Mass Spectrometer part of the device was important to notice the start and end of the m/z: 35-350. Moreover, the acquisition mode was scan, so the scan speed was 4000 and the solvent cut time 0.5 min. Each sample was studied at 25 ºC, 35 ºC, 45 ºC, 55 ºC, 65 ºC

The results of the retention time, tr, (1min = 100 sec) for each component were:

- 94 % of probability to be $C_{12}H_{24}$ with a retention time of tr= 7.115min

- 91 % of probability to be $C_{14}H_{30}$ with a retention time of tr= 9.255 min

- 91 % of probability being $C_{15}H_{32}$ the compound with tr= 10.180 min

- 91 % of probability, this compound was $C_{16}H_{34}$ with tr=11.060 min

- 90 % of probability was $C_{17}H_{36}$ with tr=11.89 min

- 90 % of probability to be $C_{18}H_{38}$ with a retention time of tr= 12.675 min

- 88 % of probability, this compound is $C_{20}H_{42}$ with tr= 14.225 min

- $C_{22}H_{46}$ signal non observed

- $C_{24}H_{50}$ signal non observed

5 Outcomes or future work

Giro-Paloma, J.; Al-shannaq, R.; Fernández, A.I.; Farid, M. Quality testing of microencapsulated Phase Change Material for use in thermal energy storage in building. *In preparation. To be submitted to Solar Energy Materials and Solar Cells Journal, 2014.*

6 References

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Jessica Giro-Paloma, Camila Barreneche, Mónica Delgado, Mònica Martínez, A. Inés Fernández, Luisa F. Cabeza Physicochemical and thermal study of a MPCM of PMMA shell and paraffin wax as a core. Energy Procedia 48 (2014) 347-354.

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7 Assessment

I am very glad to have had the opportunity on doing this secondment in Auckland (New Zealand) with my supervisor Prof. M. Farid. It was a great pleasure to work with him and learn from him due to his extended knowledge in this field. Besides, all the University staff, as well as the rest of students were very collaborative with this this project and their help during the secondment were constant.