Comparison of Phase Change Slurries: Physicochemical and thermal properties

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Abstract

Phase change slurries (PCS) consist on a carrier fluid binary system, where water is mostly used as continuous phase and micro-encapsulated phase change materials (MPCM) are used as dispersed phase. PCS are used in Thermal Energy Storage (TES) for building applications, combining the latent heat capacity of the MPCM with the sensible heat capacity of the carrier fluid, and at the same time giving the PCM pumpable properties. In this study, two PCS samples are compared and characterized, the commercial Micronal® DS 5007 X from BASF and a laboratory made PCS28. Thereby, in this paper thermal stability is studied by using thermogravimetrical analysis (TGA) and the main components of the MPCM have been studied using Fourier transformed infrared spectroscopy (FT-IR). Moreover, TGA coupled with FT-IR is used to study deeply the thermal decompositions of the PCS microcapsules and products derived thereof. Finally, differential scanning calorimetry (DSC) is performed to study the melting enthalpy and the melting temperature range of the phase change material (PCM). This paper concludes that both types of PCS have good potential from thermal energy storage purposes such as solar space heating applications.

Keywords: Phase Change Materials; Microencapsulated Phase Change Material; Phase Change Slurry; Thermophysical properties; FT-IR; DSC.

1. Introduction

Phase Change Materials (PCM) absorb energy during the heating process and release the energy to the environment during the cooling process. One of the biggest drawbacks of the PCM incorporation for technical used is due to the liquid migration (leakage). Accordingly, microcapsules were thought to solve this problem. Microencapsulated Phase Change Materials (MPCM) are composed by a PCM as a core [1] and a polymer or an inorganic wall used as shell [2]. They are micro-containers with a hydrophobic core material and a hard shell which accept volume changes, maintaining the shape to conserve the form and avoid the PCM leakage when the phase change occurs. MPCM attribute thermoregulatory properties to materials [3] and they must be enclosed in thin and resistant polymer shells for changing solid to liquid phase change and back again within the shells.

Organic PCM have been proposed as one of the most significant thermal energy storage materials [4–6] because of their advantageous thermal and heat transfer characteristics [7]. Moreover, PMMA is also a widely used polymer as a shell [8–12]. Nevertheless, the PCM which is mainly used and studied is *n*-octadecane [13–16]. This PCM is commonly encapsulated with melamine-formaldehyde shell [7,17–19], although there are a lot of PCM possibilities as a core, and materials as a shell [20,21]. It is known that the physicochemical properties of all type of microcapsules after several thermal cycles change[22], and these changes are attributed to a partial degradation of the microcapsules by breakage [23].

Encapsulating PCM in a firm substance with small enough diameter to be suspended in a liquid (partially melting and solidifying slurries) can be done with high energy densities and heat transfer rates [24]. Phase change slurries (PCS) consist on a carried fluid binary system (water is mostly used as a continuous phase) and MPCM (as a dispersed phase) [25], and may be used in active or passive systems [26–28], having several applications in different fields [29] such as heating [30], air-conditioning [31] and ventilation [32], refrigeration [33] and heat exchangers, most of them in buildings [34–38].

In this study, an evaluation between two PCS able to be used for thermal energy storage (TES) in buildings was performed. The main objective is the characterization and the comparison of two different PCS, a commercial one and a laboratory made one. The most important reason to compare two PCS samples with different types of microcapsules is based on deciding the best candidate to be used in a TES field for active systems in building applications. Besides, as novelty, two different PCS sources (commercial and laboratory made) were compared to ensure that the obtained thermal properties for both samples are similar and good enough to choose both as possible samples to apply in an active system. This paper includes thermophysical and chemical properties of the two studied slurries, while other properties will be covered in a second paper [39]. It was evaluated different techniques to compare them physicochemically and thermally. Thermogravimetrical analysis (TGA) and differential scanning calorimetry (DSC) were used to measure the thermophysical properties and the thermal stability of the PCS under study. Furthermore, to study the composition of the polymer used as the shell of the microcapsules it was performed a Fourier transform infrared (FT-IR). Besides, the characterization of the chemical functionality for the volatile emissions during the decomposition of the PCS microcapsules was studied with TGA coupled with FT-IR.

2. Materials and methods

2.1. Materials

Micronal® DS 5007 X from BASF company is a microencapsulated slurry sample which is used in active systems and composed of an acrylic shell and *n*-octadecane as PCM with an average size of the microcapsules is around 7.5 μm. The other sample is a laboratory suspension of microcapsules prepared at the University of Ljubljana (Slovenia) named PCS28 based on analysis and experimental optimization of in situ polymerisation technology composed of melamine-formaldehyde (MF) as a shell and *n*-octadecane as PCM, and with an average size of 6.6 μm. Working temperatures for both samples are around 24 - 28 ºC, considered close to the indoor comfort temperature in buildings.

2.2. Methods

2.2.1. Fourier Transform Infrared Spectroscopy (FT-IR)

Chemical characterization of PCS shell composition was carried out using FT-IR spectroscopy. A Spectrum Two™ from Perkin Elmer supported by Dynascan™ interferometer and OpticsGuard™ technology was used to perform the analyses. This equipment can measure substances at liquid and solid state, and it was optimized by a wavelength range between 4000 cm^{-1} and 350 cm^{-1} and its standard spectral resolution is 0.5 cm^{-1}.

2.2.2. Thermogravimetrical Analysis (TGA)

The thermal stability of the samples under study was evaluated with a Simultaneous SDTQ600 from TA Instruments under nitrogen flow. The heating rate applied was 0.5 $K·min^{-1}$ between 25 and 30 ºC. Then, the temperature was increased applying 1 $K·min^{-1}$ heating rate from 30 to 100 ºC. The last heating ramp was using 5 $K·min^{-1}$ heating rate from 100 to 600 ºC. Sample mass used was around 38 mg for both samples.

2.2.3. TGA coupled with FT-IR

The TGA coupled with IR was performed in order to characterize the chemical functionality of volatile emissions during the decomposition of the microcapsules. The temperature range used to perform the evaluation of the microcapsules degradation was between 25 ºC and 250 ºC applying 10 $ºC·min^{-1}$ heating rate. All measurements were performed in alumina vessels (90 μl) under 50 mL min^{-1} flow rate of N_2.

2.2.4. Differential Scanning Calorimetry (DSC)

Differential scanning calorimetry (DSC) was used to assess the melting range, melting enthalpy, as well as thermal stability of compounds [40–42]. The analyses were carried out using a DS 822e by Mettler Toledo at 0.5 $K·min^{-1}$ heating/cooling rate between 10 - 45 ºC under constant 80 $ml·min^{-1}$ flow of N_2. Furthermore, crucibles used were 40 μL closed aluminium.

3. Results and discussion

3.1.Fourier Transformed Infrared Spectroscopy (FTIR)

3.1.1. Micronal® DS 5007 X

The spectrum of Micronal® DS 5007 X is shown in Figure 1. In this figure, it is shown the spectra of wet and dried sample, just to evaluate the suspension, comparing both spectra. Hence, the sample was dried at 35 ºC during 24 h in an oven to obtain the ratio microcapsules: water, being 1:2.4. The broad intense peak in Figure 1 at 3368.9 cm^{-1} corresponds to the aqueous suspension of the microcapsules. Then, the sharp intense peaks at 2921.0 cm^{-1} and 2852.0 cm^{-1} are due to the presence of the aliphatic C-H stretching vibration. The two peaks related to the presence of ester carbonyl group stretching vibration are in 1729.5 cm^{-1} for the dried sample and 1646.0 cm^{-1} for the slurry one. This wavenumber difference may be attributed to two possible reasons. The first one ascribed to the hydrogen bounding with the presence of the carbonyl group enfeebles the molecule producing a peak at less frequency. The second probable cause of this displacement may be assigned to the presence of monomers in the slurry, and when the air dried process is achieved, these monomers are evaporated causing a displacement of the carbonyl peak at higher frequencies. Besides, the peak at 1466.6 cm^{-1} was associated to the C-H bending vibration. The broad peak ranging at 1123.5 cm^{-1} is explained owing to the C-O (ester bond) stretching vibration. Moreover, the peak at 720.5 cm^{-1} corresponds to the bending of C-H. This study confirms that the suspension contains an acrylic polymer as microspheres wall shell.

Figure 1.

3.1.2. PCS28

The study of the PCS28 by FT-IR elucidates precisely the functional groups of the shell. Several transcendental peaks such as the intense peak at 3367.9 cm^{-1} which reflected the overlapping stretching frequencies of ν_{OH} and ν_{NH} absorption are shown in Figure 2. Moreover, the peaks obtained at 2913.6 cm^{-1} and 2848.4 cm^{-1} provide information about the asymmetric and symmetric stretching vibration of the aliphatic methylene group C-H of the polymeric chains. Besides, the intense peaks at 1564.1 cm^{-1} and 1471.0 cm^{-1} are assigned to the C=N vibrations (triazine ring) and the weak ones at 1368.5 cm^{-1} represents the β_{CH} deformation of heterocyclic rings. Finally, the peak at 716.3 cm^{-1} was due to the bending C-H. Therefore, this spectrum is clearly recognizable a melamine-formaldehyde (MF) resins as wall material for the microcapsules.

Drying the sample at 35 ºC during 24 h in an oven showed the ratio microcapsules:water, which is 1:2, less fluid than Micronal® DS 5007 X. The dried PCS28 sample spectrum is also presented in Figure 2, and it shows that the carrier fluid is water. The rest of characteristics peaks are the same obtained for the sample PCS28 wet slurry.

Figure 2.

3.2.Thermogravimetrical Analysis (TGA)

3.2.1. Micronal® DS 5007 X

Micronal® DS 5007 X sample degrades in three steps as it is seen in Figure 3. The first weight loss is 72 % wt. due to water evaporation between 24 and 180 ºC. The second decomposition is 18 % wt. ending around 260 ºC, and it is attributed to the paraffinic wax decomposition. The third weight loss is around 6 % wt .and it corresponds to decomposition of the acrylic shell and it was complete at 385 ºC. A 5 % wt. solid residue is observed at 600 ºC and it is identified as a Si based compound [43].

Figure 3.

3.2.2. PCS28

Figure 4 shows the weight loss variation for this slurry over temperatures for PCS28. Also three steps of decomposition can be observed, from 25 to 125 ºC, from 125 to 325 ºC, and from 325 to 600 ºC, respectively. At the beginning of the first decomposition, water evaporation and the self-condensation of methylol groups represent a 59 % wt. The second weight loss is due to the PCM decomposition and partly degradation of MF shell and it is a 31 % wt. of the total amount of the sample, and occurs in the temperature range between 180 ºC and 330 ºC. Finally, the weight loss beyond 330 ºC is 8 % wt. attributed to the condensation reaction between melamine, methylol group, some emulsifying agent (in this case styrene-maleic acid anhydride copolymer), the elimination of formaldehyde from the ether bridge forming methylene bridge, the breakdown of the methylene bridges, and the thermal degradation of the triazine ring, and it is assumed that MF resin progressively transform to HCN. The final residue is around 2 % wt.

Figure 4.

3.3.FT-IR coupled to TGA

3.3.1. Micronal® DS 5007 X

The decomposition of PCS samples was deeply analyzed by coupling the FT-IR with TGA. In a three dimensional graph the Transmittance (%) in front of the Wavenumber (cm^{-1}) and *vs*. time (s) is represented (Figure 5). It is represented the most important spectra taking into account the degradation temperatures of the TGA results (Figure 3). It means six different spectra at 25 ºC (around 600 s), 75 ºC (around 1000 s), 200 ºC (around 1800 s), 300 ºC (around 2400 s), 350 ºC (around 2700 s), and 600 ºC (around 4200 s). As it can be observed in Figure 5, at the beginning of the experiment, no signals can be observed. Around 1800 s the degradation of the paraffin wax should be seen, although its spectrum is almost 100 % Transmittance. Then, the ones at 2400 s is represented, which is the most important spectrum, due to it is related to the shell degradation. This degradation can be observed until the end of the experiment.

Figure 5.

3.3.2. PCS28

FT-IR device coupled with TGA give the decomposition spectra of the PCS over time in a three dimensional figure (Transmittance in % *vs*. Wavenumber in cm^{-1} *vs*. time in s) (Figure 6). Five different spectra related to TGA temperatures in Figure 4 are represented: 25 ºC (around 0 s), 60 ºC (around 360 s), 250 ºC (around 660 s), 400 ºC (around 2400 s), and 600 ºC (around 3600 s). The three first spectra have very high transmittance, therefore it is very difficult to attribute and characterize peaks, although as it is exposed in 3.2.2 section, these are related to the water and PCM decomposition. The spectrum related to the MF shell is the one at 2400 s, and its degradation is prolonged until the end on the experiment.

Figure 6.

3.4.Differential Scanning Calorimetry (DSC)

DSC curves results of DSC samples characterization under study are summarized in Table 1 and presented in Figure 7.

Table 1.

Results show that the latent heat observed is almost equal between melting and solidification processes for both samples. Besides, the phase change temperature remains roughly constant for Micronal® DS 5007 X, but accounts 7 ºC in the case of PCS28 due to the solidification will present subcooling as was stated by Günther *et al.* [44].

Figure 7.

The main property in this article to highlight that the two reported PCS are proper candidates to be used in TES building heating and cooling applications is DSC, as it provides highly enough values for melting and solidification latent heat to confirm their capability of developing their role in thermal energy storage field, concluding a good potential from TES functions for both PCS.

4. Conclusions

Two different PCS samples which contain different materials for the shell and the core were evaluated. FT-IR results reported that for Micronal® DS 5007 X the shell is an acrylate polymer. The FT-IR spectra corresponding to PCS28 sample concluded that the external polymeric shell is melamine-formaldehyde. Thermogravimetrical investigation (TGA) showed that Micronal® DS 5007 X slurry decomposed in three steps and in two steps for the dried sample. Moreover, PCS28 decomposed in three steps (the carrier fluid, the PCM and the wall shell) and shows better thermal stability than Micronal® DS 5007 X. In addition, the temperature and latent heat of melting and solidification of the PCS samples were determined using DSC analysis method. For the Micronal® DS 5007 X is shown a phase change between 21 ºC and 23 ºC and an enthalpy around 90 $kJ\cdot kg^{-1}$. Otherwise, PCS28 slurry has a melting temperature between 22 ºC and 27 ºC with an enthalpy of 50 $kJ\cdot kg^{-1}$. Overall, the studied PCS are totally capable of developing their role in the thermal energy storage field, and they can be considered as PCM candidates for TES in active building applications.

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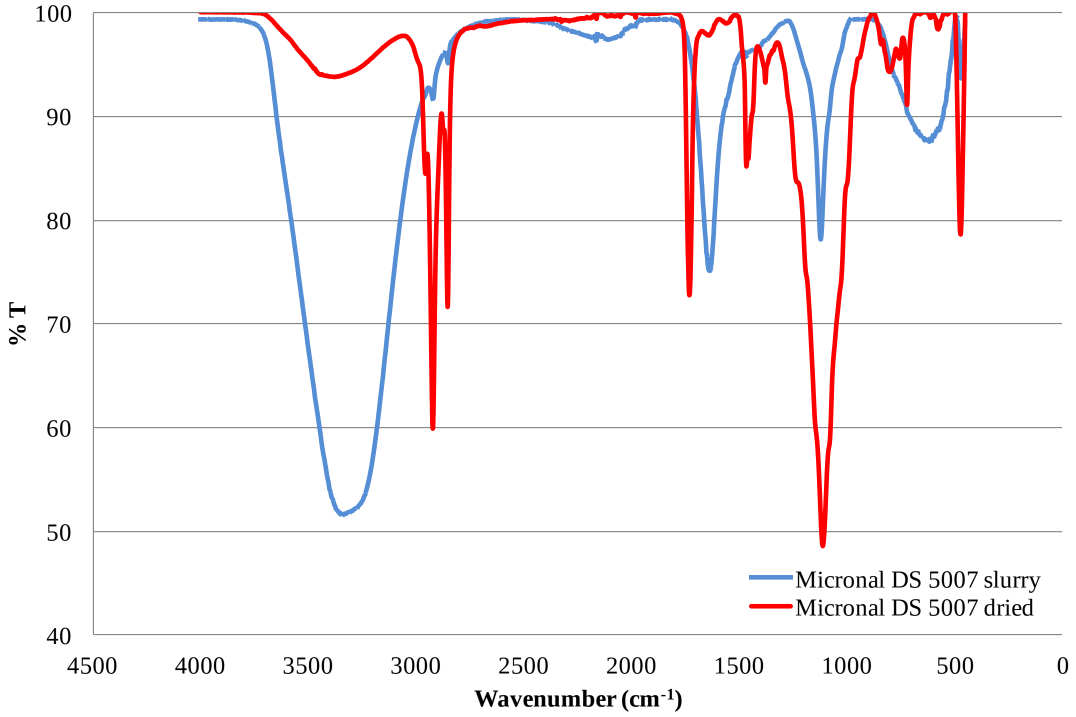

Figure 1.- FT-IR spectrum of Micronal® DS 5007 X in PCS form and dried form.

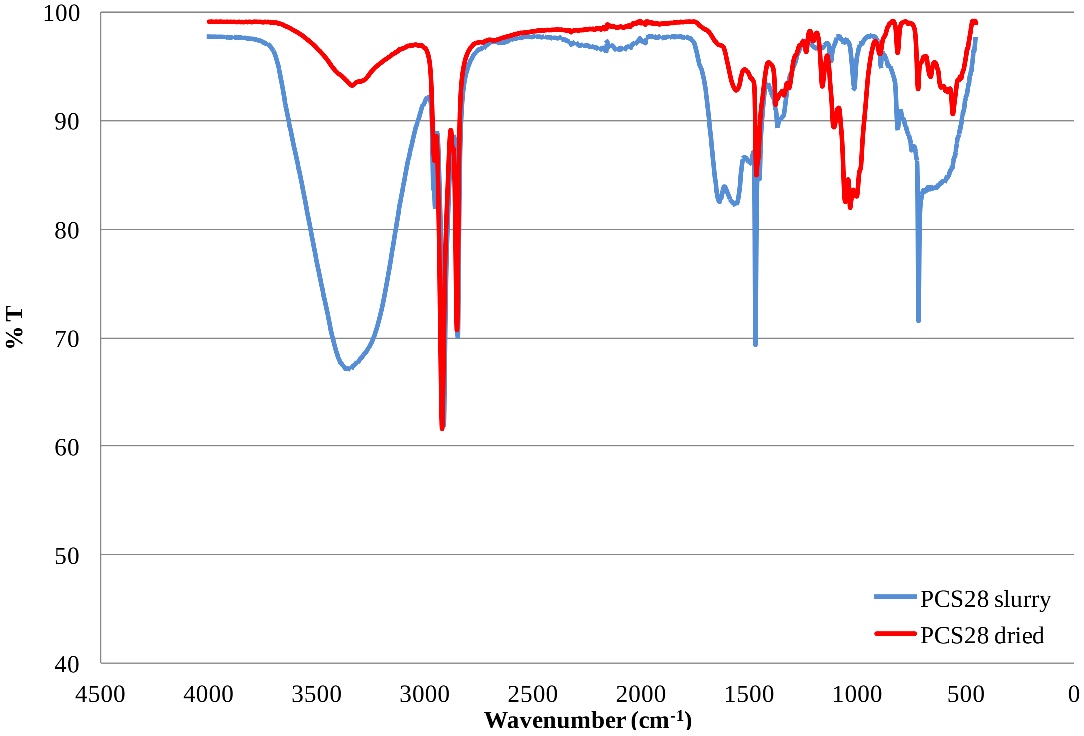

Figure 2.- FT-IR spectra of the PCS28.

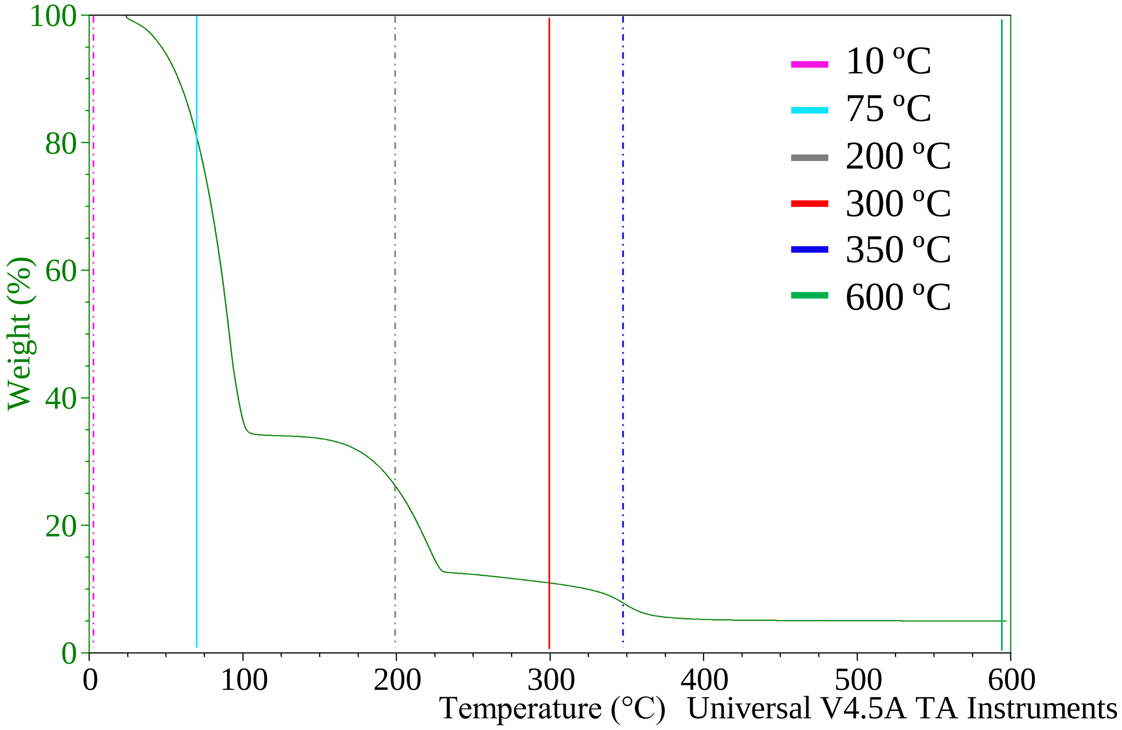

Figure 3.- Thermogravimetrical Analysis of PCS Micronal® DS 5007 X.

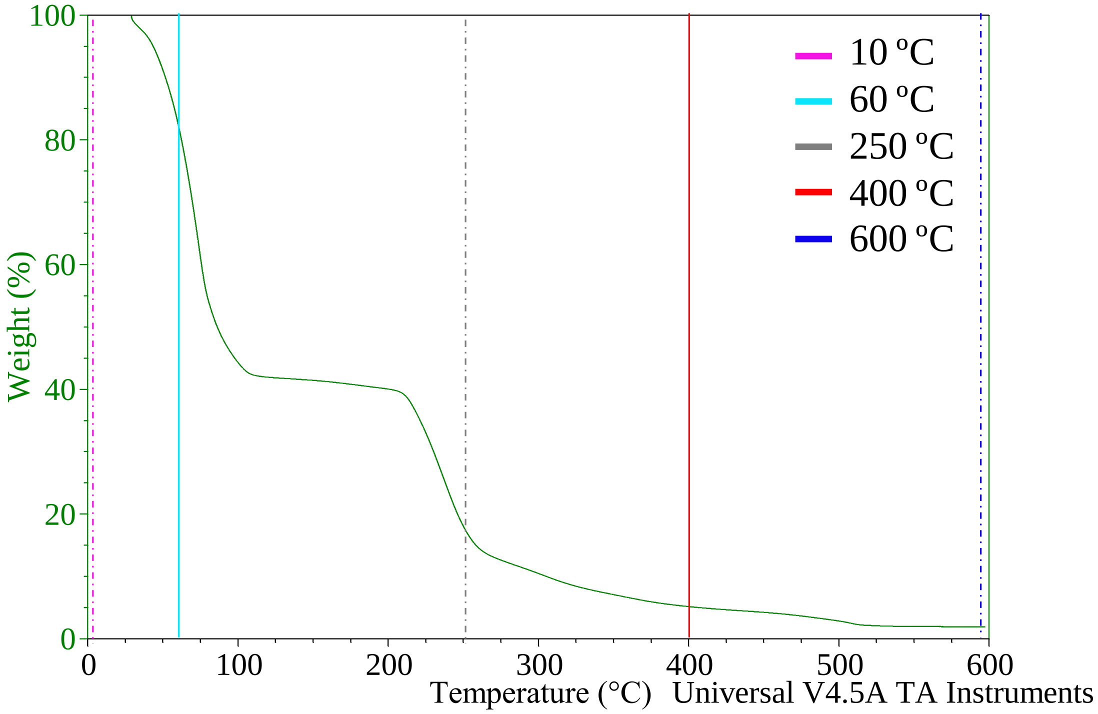

Figure 4.- Thermogravimetrical Analysis of PCS28.

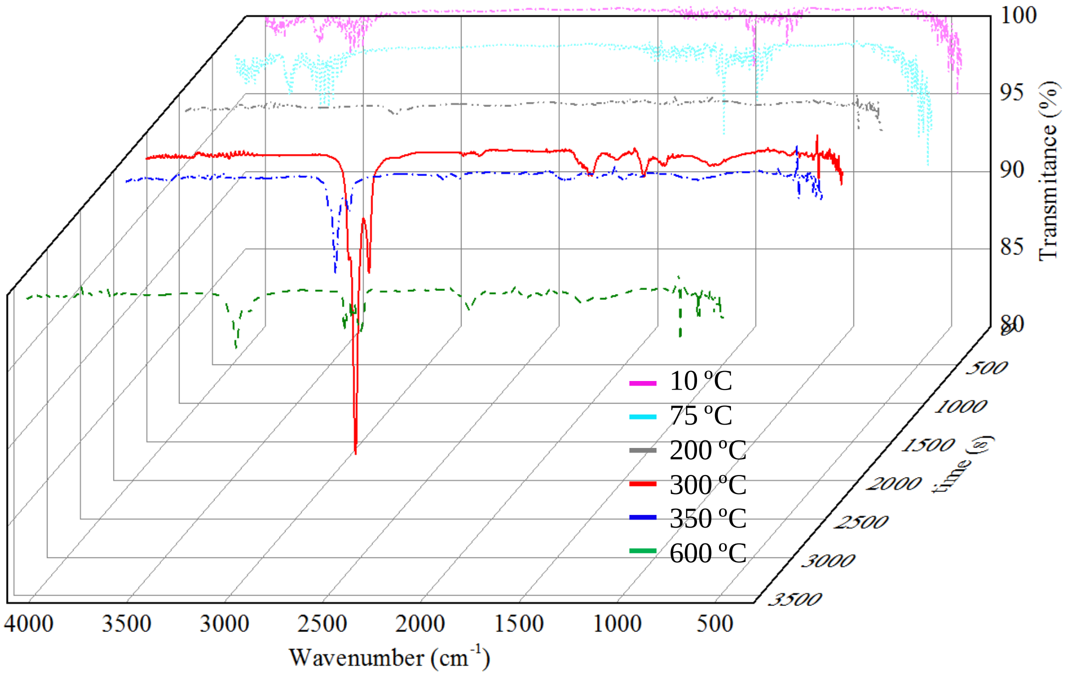

Figure 5.- FT-IR results after TG analysis of PCS Micronal® DS 5007 X.

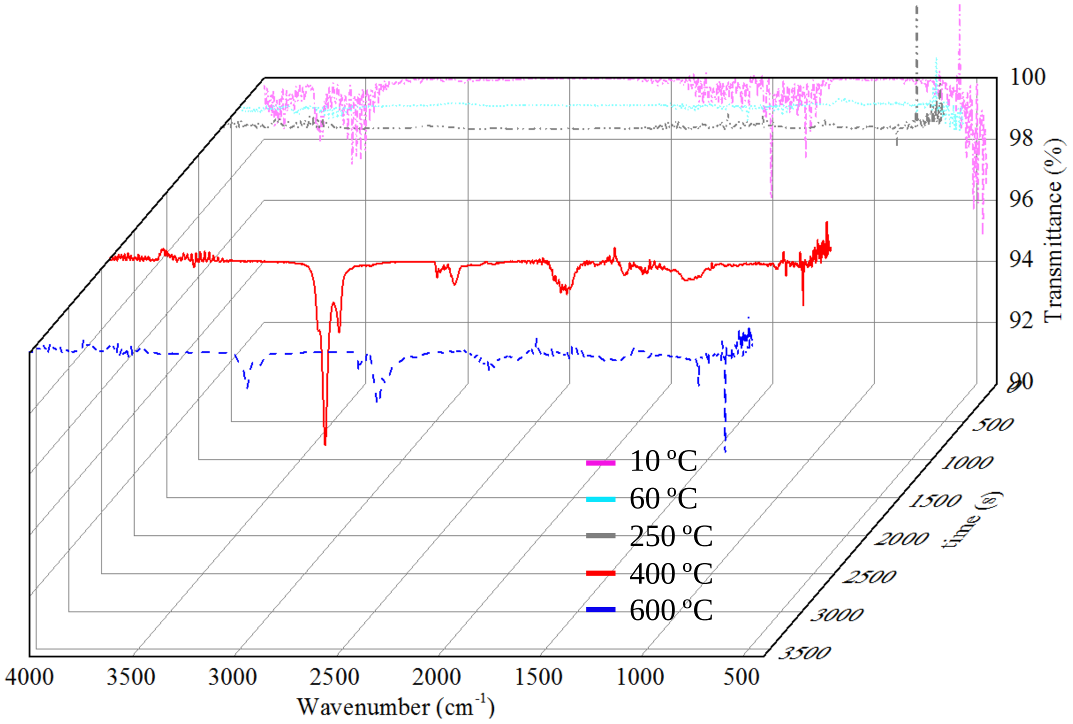

Figure 6.- FT-IR results after TG analysis of PCS28.

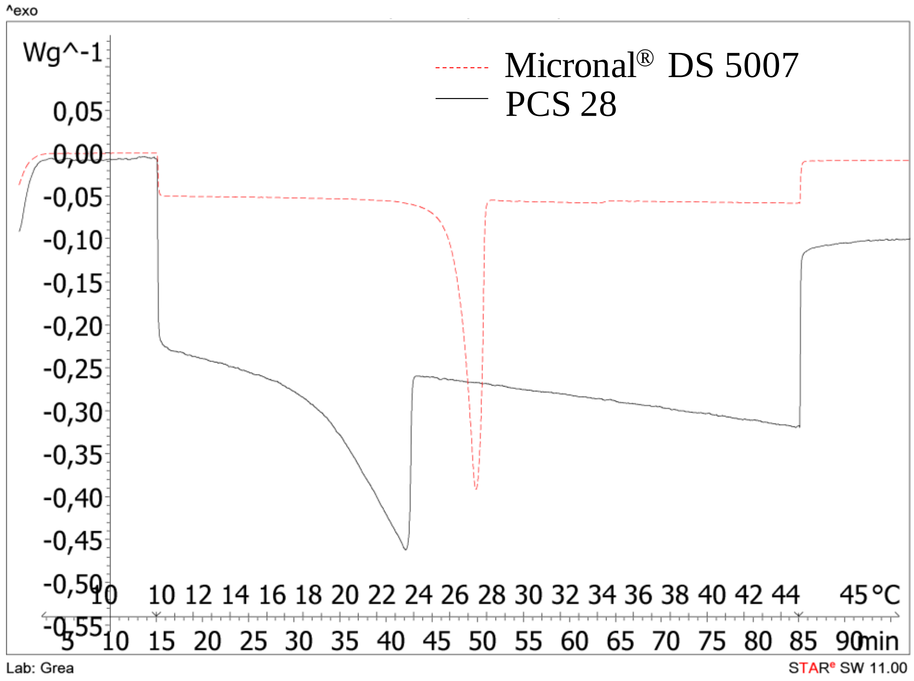

Figure 7.- Differential Scanning Calorimetry curves of the two samples using Dynamic method.

Table 1.- Differential Scanning Calorimetry results for Micronal® DS 5007 X and PCS28.

	Micronal® DS 5007 X		**PCS28**	
	Melting	solidification	melting	solidification
Enthalpy ($kJ \cdot kg^{-1}$)	87.48	89.73	50.36	50.78
T (ºC)	23.54	21.07	27.68	20.34