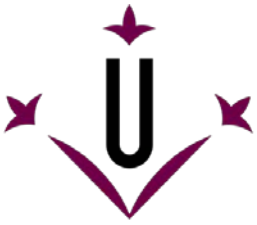

Comparison of three different devices available in Spain to test thermal properties of building materials including phase change materials

Camila Barreneche[1,2], Alvaro de Gracia[1], Susana Serrano[1], M. Elena Navarro[2], Ana María Borreguero[3], A. Inés Fernández[2], Manuel Carmona[3], Juan Francisco Rodriguez[3], Luisa F. Cabeza[1]

[1]GREA Innovació Concurrent, Universitat de Lleida, Edifici CREA, Pere de Cabrera s/n, 25001, Lleida, Spain. Tel: +34.973.00.35.77. Email: lcabeza@diei.udl.cat

[2]Department of Materials Science & Metallurgical Engineering, Universitat de Barcelona, Martí i Franqués 1-11, 08028, Barcelona, Spain. Tel: +34.93.402.12.98. Email: ana_inesfernandez@ub.edu

[3]Institute of Chemical and Environmental Technology, Department of Chemical Engineering, University of Castilla – La Mancha, Av. Camilo José Cela s/n, 13071 Ciudad Real, Spain. Tel: +34.902204100. Email: manuel.cfranco@uclm.es

Abstract

Thermal properties of materials used in building envelopes must be analysed in order to evaluate the thermal response of the constructive system. This thermal characterization is a key point during the design phase of a building. However, thermal characterization of constructive systems at laboratory scale is difficult to be carried out under real environment conditions. In this paper, three devices developed by three different research groups in Spain were used to compare in an inter-laboratory test the performance, capabilities and thermal properties of construction systems at lab scale. Tested materials were gypsum blocks containing phase change materials (PCM) and made by three different ways: using microencapsulated materials Micronal® DS5001, a suspension water/PCM and impregnation with RT21. The effective thermal conductivity, the total amount of heat accumulated, and the specific heat were measured using these homemade devices. *k* results followed same trend but there was a drift between them due to the samples porosity and thickness. Moreover, the *k* decreased when adding PCM but this behaviour was not followed by impregnated samples; due to the PCM filling gypsum pores instead of air. The Cp results followed same trend $Cp_{Blank} < Cp_{Suspension} < Cp_{Microencapsulated} < Cp_{Impregnated}$ but a gap between results was observed due to different amount of incorporated PCM.

Keywords: Phase Change Materials (PCM), Thermophysical characterization, Thermal Energy Storage (TES), building materials, building envelope.

1. Introduction

A substantial increase in global energy consumption has been recorded in recent years. There is a high concern for the exhaustion of fossil fuels energy resources and how their use can impact the environment. In developed countries, the buildings contribution (private and offices consumption) to total energy consumption is between 20% and 40% [1].

Thermal properties of materials used in building envelopes must be analysed during the design phase to evaluate the thermal response of the building. Several researchers have studied the thermal behaviour of building insulation materials in situ [2-4]. However, the thermal properties of these constructive systems are poorly characterized at laboratory scale with samples sizing several cm. Therefore, it is important to test the materials that will be used in the construction of building envelopes before implementing them. This type of characterization is difficult to reach due to the difficulty in measuring samples at the macro scale under real environmental conditions. The scientific community is doing a great effort to develop experimental equipment to carry out thermal measurements using macroscopic samples at lab scale which reproduces real constructive systems [5,6].

This problem increases when phase change materials (PCM) are added in the construction systems, because steady-state characterization is not enough, since it does not take into consideration the thermal inertia increase due to the PCM.

These limitations guided different research groups to develop their own homemade equipment to test different thermal properties of the materials used in building envelopes. First, the University of Lleida researchers developed a device [7] which can measure the thermal transmittance in steady-state (U-value) and the heat storage capacity of the sample. Second, the University of Barcelona researchers developed the Conductimeter F14. This equipment can analyse the effective thermal conductivity of the materials and the effect of the PCM is visualized graphically. And finally, the University of Castilla-La Mancha developed a device which can measure the thermal energy storage capacity of samples and their effective thermal conductivity [8-9].

The samples analysed in this paper have in their formulation phase change materials (PCM) and gypsum. PCM provide high thermal inertia to the building envelope when its latent heat is used

(that is when thermal conditions allows phase change). The use of PCM combined with thermal insulation smoothes the daily temperature fluctuations and may reduce the energy consumption of buildings by absorbing heat gains and reducing the heat flow [10-12].

The main objective of this paper is to compare in an inter-laboratory test the performance, capabilities and results of the three devices developed by three different research groups in Spain.

2. Materials and methodology

2.1 Materials

Samples used in this inter-laboratory experience were made at the University of Lleida at same time. Then, they were sent to different laboratories to be tested by other universities, independently.

Gypsum (E-35) was the building material used for this inter-laboratory experience. The thermal conductivity (κ) and the *Cp* of the gypsum found in the literature are 0.3 – 0.4 W/m·K and 1090 J/kg·ºC, respectively [13].

The gypsum blocks made for testing had PCM in their formulations, introduced using three different methods:

- Microencapsulated PCM: Micronal® DS5001 from BASF was mixed with the gypsum in dry state and then the gypsum block was manufactured.
- PCM suspension: A suspension with the needed water and PCM (RT 21 from Rubitherm) in liquid state was produced, and it was used as liquid suspension for the manufacture of the gypsum block.
- Impregnation: Dry gypsum blocks were impregnated with liquid RT-21 from Rubitherm.

The samples were made using a mixer with a rotor speed of 25 rpm during 5 minutes. After mixing, a manual vibration process was carried out to evacuate trapped air and to reduce the sample porosity.

RT-21 has a melting point of 21 ºC and a fusion enthalpy of 100 kJ/kg. Micronal® DS5001 has a melting point of 26 ºC and its fusion enthalpy is 110 kJ/kg. Samples analysed in this inter-laboratory test are listed in Table 1.

2.2 Physical characterization

Density and porosity of studied materials were measured since they affect their thermal properties [14,15]. The bulk density was measured through a Helium pycnometer and the geometrical density was measured weighting the sample. The open porosity was calculated following Eq. 1 were P_O (%) is the open porosity, ρ_{He} is the density obtained by Helium pycnometer, and ρ_{bulk} is the geometrical density [16]:

$$P_O(\%) = \frac{\rho_{He} - \rho_{bulk}}{\rho_{He}} \cdot 100 \qquad \text{Eq. 1}$$

2.3 Experimental set-up and methodology

In the thermal characterization tests performed during this paper, three different devices were employed. In addition to the thermal transmittance in steady-state and the effective thermal conductivity measurements, transient tests are also carried out in order to analyse the increase of thermal inertia of the material due to the PCM addition.

2.3.1 Equipment developed at the University of Lleida [7]

This equipment is divided in two cavities as Figure 1 shows. The sample is placed in the middle of the device, being the boundary between the two environments. Each cavity had a copper cooling coil connected to a programmable water bath; those water baths simulated different thermal conditions, changing the temperature in the cavity and applying different heating and cooling ramps.

The placement of the used sensors is shown in Figure 2. The temperatures in each cavity were measured using calibrated Pt-100. The temperatures of both surfaces and the sample centre were measured using thermocouples type T. In addition, two heat flux sensors were fixed at the sample surfaces to measure input and output heat flux (Hukseflux HFP01). For this equipment, the tested samples had the dimensions of 19×19×4 cm.

The equipment developed at the University of Lleida was used to perform two different experiments. During the first experiment, a thermal gradient between sample surfaces was created and the thermal transmittance in steady-state of the sample was calculated. In the second experiment the sample was homogeneously heated from an initial temperature of 20 ºC to 40 ºC

(steady-state was achieved at initial and final conditions). Therefore, an average heat storage capacity of the sample was calculated.

The thermal transmittance of the sample (U_{sample}) was calculated with Eq.2, where T_{down} and T_{up} are the surface temperatures and q_{sample}/A is the measured heat flux across the sample per m^2, this value was calculated as an average between the heat flux measured in the top and bottom surfaces. Although the equipment is insulated, there are differences between the heat fluxes due to the heat losses. The measurement always started once the experiment achieved steady-state conditions.

$$U_{sample} = \frac{q_{sample}}{A} \cdot \frac{1}{T_{down} - T_{up}} \quad \text{Eq. 2}$$

The average heat capacity of the sample (Cp_{sample}) was calculated with Eq. 3, where q_{acc} is the amount of accumulated heat in the sample during the experiment, m_{sample} is the mass of the sample, and T_f and T_i are the temperature at the final and initial conditions:

$$Cp_{sample} = \frac{q_{acc}}{m_{sample} \cdot (T_f - T_i)} \quad \text{Eq. 3}$$

Furthermore, the University of Lleida equipment can also performed another experiment in order to measure the dynamic thermal response. The experiment is to create daily oscillations of temperature in the upper cavity of the facility between 15 ºC and 42 ºC. Then, the delay between the temperature peaks is measured and the dampening of the temperature wave (thermal stability coefficient [17]) is evaluated. This last experiment was not performed in this inter-laboratory experience because the other devices used in this study are not capable to perform it.

2.3.2 Conductimeter F14

Conductimeter F14 available at the University of Barcelona is a reliable, repeatable and comparable equipment to determine the effective thermal conductivity of samples at macroscale [18]. In Figure 3 a schematic cross section view of it can be seen. A surrounding insulation made of refractory brick gives to the equipment a total dimension of 825 x 825 mm to achieve a z-axis thermal gradient.

This equipment was developed following the standard UNE-EN 12664 for measuring thermal resistance of building materials using the guarded hot plate method. In this case, the sample was placed between a hot plate and a cold plate and both plates are insulated with polyurethane foam to minimize the ambient temperature effect. The thermal conditions were stabilized in steady-state using a thermostatic system in one side of the sample. In the opposite side, the temperature was increased by an electrical resistance. When both surfaces of the system were at steady-state conditions again, the effective thermal conductivity was measured. The guarded plate area surrounding the measurement area assures a unidirectional heat flow and this is controlled by differential thermocouples.

Two type-T thermocouples (according to the European standard UNE – EN 12664) were placed in each sample surface and another one was placed outside measuring the room temperature. The dimension of the samples analysed by Conductimeter F14 was 30×30×3 cm. Samples which incorporated impregnated PCM in their formulations were not analysed with this equipment since it was not possible to prepare samples big enough for this equipment. Moreover, the Conductimeter device, by adjusting the hot plate and cold plate, is able to characterize different samples incorporating different temperature of fusion, from nearly 100 ºC to subzero temperatures and this device is able to measure the thermal properties of materials with low to medium resistance with accuracy close to ± 2% [19].

Effective thermal conductivity (κ_{eff}) depends on the thermal gradient achieved. This parameter (κ_{eff}) was calculated at the mean temperature of the measurement following Eq. 4, where ϕ is the heat flux expressed in watts (W), Δx is the thickness of the sample, A is the surface of the sample and ΔT is the gradient of temperature between both sample surfaces:

$$k_{eff} = \frac{\Phi \cdot e}{A \cdot \Delta T} \qquad \text{Eq. 4}$$

Using Conductimeter F14, the effective thermal conductivity was measured once the system reached steady-state conditions. Moreover, the thermal profile of the heating and cooling processes was acquired in order to visualize the thermal inertia of the sample. The test procedure used was as follow: from 15 ºC achieved using the cold plate connected to a water bath until the maximum temperature was achieved at steady state conditions for each sample under 10 V of power supplied with the hot plate.

2.3.3. C.R. equipment [8]

The equipment developed at the University of Castilla-La Mancha can provide controlled changes in the external temperature of the samples using a thermostatic bath [8]. The test was carried out applying a thermostatic bath set-point step change from 18 to 42 ± 0.1 ºC while the different external block temperatures and inlet-outlet heat fluxes were registered. The equipment is shown in Figure 4. The sensors distribution and the scheme of the C.R. device are also shown in this figure. The samples size analysed in these experiments performed with the C.R. device was 6×10×2 cm.

As shown in Figure 4, the sample was placed on the upper surface of the aluminium cell and further insulated with foam boards of 3.9 cm thickness. Seven thermocouples type-K were used to measure temperatures: two were put in the external sample surface (T_{up}), other two were placed at the cell (T_{down}), two more in the middle of the sample (T_{centre}), and the last one was placed in the upper foam surface.. Four heat flux sensors were also used to measure heat fluxes: two were installed up and down the sample, one more at the front and the last one in a lateral of the wallboard. All these signals were registered continuously using the NOKEVAL program and recorded with a computer. Using these signals it is possible to quantify the Cp_{sample} and k of the analysed samples [9]. Although k value could be calculated at the steady-state using different temperature gradients, with the aim to minimize the heat losses effect, the selected gradient was between the down and centre temperatures.

Therefore, the maximum thermal conductivity can be obtained applying the heat conduction equation in one dimension (Eq. 5), where Q_{inlet} is the inlet heat flux to the gypsum sample (W/m^2) Δx is the blocks thickness and ΔT is the difference between temperatures T_{down} and T_{centre}.

$$\kappa_{eff} = \frac{Q_{inlet} \cdot \Delta x}{\Delta T} \qquad \text{Eq. 5}$$

3. Results

3.1. Physical characterization

The open porosity results are presented in Figure 5. The results show that the open porosity changes depending on the sample size. The sample setting is different although they were made at same time because the gypsum mixture was located in the bigger mold (University of

Barcelona) at the beginning and in the smaller mold at the end (University of Ciudad Real). The porosity was measured and the results showed that a bigger sample size has lower porosity. In addition, impregnation samples have the PCM filling the porous; hence, the impregnation sample porosity is lower than the other samples.

3.2. University of Lleida device

Figure 6 shows the temperature profile of all samples analysed with the University of Lleida device. All samples with PCM have lower temperature than the Blank, and the samples with PCM show the phase change of the PCM at the expected temperature range.

As an example of the experiment carried out at the University of Lleida, Figure 7 shows the thermal profiles of the temperature evolution over time of the sample with microencapsulated PCM compared to the Blank. In this experiment, the temperature of three points of the sample is tested. Figure 7 shows that in all points, the addition of PCM decreases the temperature of the sample. Also, at the centre point, the phase change of the PCM can be seen in the change of slope of the temperature.

The accumulated heat power over time of the samples with microencapsulated PCM and the Blank tested during Experiment 2 is presented in Figure 8 as an example of the profiles obtained with the University of Lleida equipment. These results were obtained by subtracting the output to the input heat flux. The area under the curve is the accumulated heat power by the samples. The difference of areas between both curves is due to the PCM effect.

3.3. Conductimeter F14 (University of Barcelona)

The thermal profiles of the samples analysed in this paper to measure the effective thermal conductivity with the Conductimeter F14 are visualized in Figure 9. The temperature of the samples with PCM is higher than the Blank, showing the effect of the inclusion of the PCM. Both PCM samples show very similar behaviour.

3.4. C.R. equipment (University of Castilla-La Mancha) [8]

T_{up} profiles of the analysed samples and thermostatic bath temperature are shown in Figure 10. Thermostatic bath temperature profile confirms the step change from 18 to 42 ºC. Comparing the slope of T_{up} profiles in the PCM melting range (20-27 ºC), it can be seen that the impregnated sample is able to absorb the largest amount of energy because it is able to hold the

temperature practically constant around 23 ºC, indicating that the incorporated PCM is melting. This figure also indicates that the three studied samples do not have the same thermal energy storage (TES) capacity. TES capacity includes sensible and latent heat that depend on the total amount of PCM incorporated and the PCM type. According to this figure the TES capacity order seems to be: Blank<Suspension<Microencapsulated<Impregnated. The time required to achieve the steady state also confirm the TES capacity order. Taking into account the melting range of the PCM and the initial and final temperatures of all the samples, it can be concluded that the PCM has completely melted in all these samples.

Values of κ agree with those reported in the literature by Feldman and Chen and by Borreguero for gypsum sample containing PCM [20,21]. It is important to point out that the lower thermal conductivity of the composite materials respect to the pure gypsum is due to the lower conductivity of the PCM.

As an example of the variation of the temperature on the middle of the blocks, Figure 11 shows the T_{centre} and T_{up} for the Blank and Microencapsulated samples. As expected, the temperature profiles in the middle of the samples also showed a change in the slope when the temperature reaches the melting temperature range of the PCM, but less significant than when considering the whole sample. T_{centre} follows the above commented TES capacity order indicating that PCMs are distributed in the whole block.

Figure 12 shows the obtained accumulated heat power for each gypsum sample analysed at the University of Castilla-La Mancha as function of time. The area under each curve corresponds to the accumulated heat by the sample, confirming that the impregnated sample is able to absorb the highest amount of heat.

4. Discussion

The thermal transmittance (U-value) was analysed with the University of Lleida equipment, and the results are presented in Table 2 (called Experiment 1). The effective thermal conductivity was calculated with Eq. 6, where Δx is the sample thickness (0.04 m):

$$\kappa_{eff} = U_{value} \cdot \Delta x \qquad \text{Eq.6}$$

The effective thermal conductivity decreases when PCM is added in the formulation. However, the sample impregnated with PCM has a higher effective thermal conductivity than the other

two samples with PCM and also higher than the blank (sample without PCM). Moreover, the effective thermal conductivity of the samples where PCM was added microencapsulated or by suspension have very similar effective thermal conductivity.

The following trend could be established in the effective thermal conductivity obtained by Lleida and Barcelona devices: $k_{Blank} > k_{Microencapsulated} \geq k_{Suspension}$. The higher porosity observed for the Microencapsulated sample tested by the C.R device could explain the lower conductivity obtained for this Microencapsulated sample, the porosity was 80 % compared to 60 or 53 % for the Lleida and Barcelona Microencapsulated samples. Rahmanian and Wang [14] stated that the higher the gypsum porosity, the lower the thermal conductivity.

The impregnated sample was not analysed with the Conductimeter F14 but the results obtained with the University of Lleida device and the one developed at the University of Castilla-La Mancha demonstrate that this sample had higher thermal conductivity than other samples with PCM. This higher effective thermal conductivity is due to the fact that the PCM fills up the gypsum porous, substituting air and, therefore, showing higher thermal conductivity.

The effective thermal conductivity of the Blanks obtained with the University of Lleida and University of Castilla-La Mancha devices are close to the thermal conductivity the range found in the literature for gypsum [13]. In addition, there is a drift between results from the different compared devices that could be explained by their different porosity and thickness or size.

Table 3 shows the results of the total heat accumulated and average heat capacity measured by the University of Lleida equipment (called Experiment 2) and the C.R. device. The gypsum Cp found in the literature is 1090 J/kg·ºC [13], close to that obtained with the University of Lleida device (1059 J/kg·ºC); on the other hand, with the C.R. device the value obtained was 1396 J/kg·ºC. All other samples always gave a higher Cp value when analysed with the C.R. device than with the University of Lleida equipment. Results that could be related with the porosity and the higher amount of PCM incorporated. As expected the Cp tendency was $Cp_{Blank} < Cp_{Suspension} < Cp_{Microencapsulated} < Cp_{Impregnated}$.

The q_{acc} is obtained as contribution of sensible and latent heat (Eq. 7). Assuming that all the composites have same sensible heat and similar to that obtained for the pure gypsum, the PCM mass fraction (*x)* into the sample could be obtained. PCM mass fraction for each samples are shown in Table 4.

$$q_{acc} = (1 - x) \cdot q_{Sensible} + \cdot\, x\Delta H_f \qquad \text{Eq. 7}$$

These results confirm the water evaporation from the composite gypsums (Microencapsulated and Suspension) that increases the PCM percentage respect to the 10 wt% in the formulation. In the case of C.R samples, these PCM mass percentages are higher than those from the Lleida, differences that could be explained by the higher water evaporation confirmed by the porosity analyses. Furthermore, the impregnation step was performed around 80 ºC and two are taking place at the same time: PCM impregnation (mass gain) and water evaporation (mass loss). Thereby, the difference of the PCM final amount in the impregnation samples is also caused by this fact.

Impregnated samples present the maximum amount of PCM which agrees with the lower slopes of the temperature profiles. As expected from the porosity results, the Impregnated sample tested in the C.R. device has a higher amount of PCM incorporated.

5. Conclusions

The improvement in the thermal behaviour of gypsum board samples due to the addition of PCM was analysed using three different devices developed by three Spanish Universities: University of Lleida equipment, Conductimeter F.14 and C.R. device. The comparison of the obtained results was used in order to verify the suitability of the different home-made devices for the composite materials thermal characterization.

The obtained effective thermal conductivities of the pure gypsum and the gypsum samples containing PCM were in the values range reported in the literature for this type of materials. The higher the amount of PCM, the lower the effective thermal conductivity measured except for Impregnated samples. The increase of the impregnated sample conductivity was due to the air substitution by the liquid PCM, decreasing the gypsum porosity. However, there is a drift between them that is attributed to the different porosity and thickness of the studied samples.

The average *Cp* was estimated by the University of Lleida equipment and C.R. device, finding same trend $Cp_{Blank} < Cp_{Suspension} < Cp_{Microencapsulated} < Cp_{Impregnated}$ but higher values were observed for samples analysed with the C.R. device. The higher values were in agreement with the samples porosity and the higher amount of PCM incorporated. In addition, the gypsum *Cp* values found in this work 1059 and 1396 J/kg·ºC for Lleida and C.R, devices, respectively, were in concordance with the value reported in literature 1090 J/kg·ºC [13] for the Blank sample.

Finally, it can be concluded that the thermal properties of real materials used for building applications can properly be measured by the different home-made devices described in this paper since the obtained results were consistent and in the range of those values found in literature.

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Table 1: Formulation of studied samples

Sample	**Gypsum**	**H_2O**	**PCM**	**PCM type**
Blank	55.5%	44.5%	0%	---
Microencapsulated	44%	46%	10%	Micronal® DS5001
Suspension	50%	40%	10%	RT-21
Impregnated	50%	40%	10%	RT-21

Table 2. U-value and effective thermal conductivity (κ_{eff}) of the samples analysed

Parameter	**Units**	**Device**	**Blank**	**Microencapsulated**	**Suspension**	**Impregnation**
U-value	$W/m^2 \cdot K$	Univ. of Lleida	8.48	7.31	6.14	12.47
κeff	W/m·K	Univ. of Lleida	0.34	0.28	0.25	0.50
		Conductimeter F14	0.50	0.36	0.35	---
		C.R. device	0.31	0.22	0.25	0.29

Table 3. Total accumulated heat and average heat capacity

Parameter	**Units**	**Device**	**Blank**	**Microencapsulated**	**Suspension**	**Impregnated**
q_{acc}	[J/kg]	Univ. of Lleida	28,399	42,041	38,823	44,380
		C.R. device	23,450	48,320	42,926	73,472
Cp_{sample}	[J/kg·ºC]	Univ. of Lleida	1,059	2,150	1,659	2,159
		C.R. device	1,396	2,988	2,643	4,579

Table 4. PCM mass fraction into the samples.

Parameter	**Device**	**Microencapsulated**	**Suspension**	**Impregnated**
x	Univ. of Lleida	0.167	0.146	0.223
	C.R. device	0.287	0.225	0.578

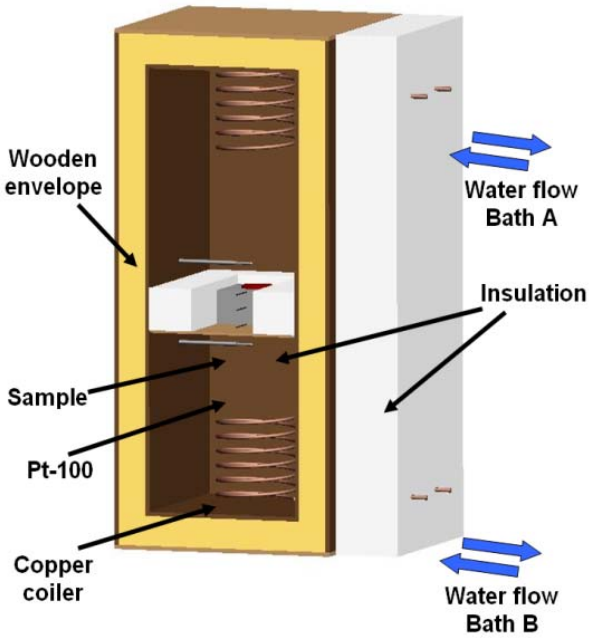

Figure 1. Sketch of the University of Lleida device

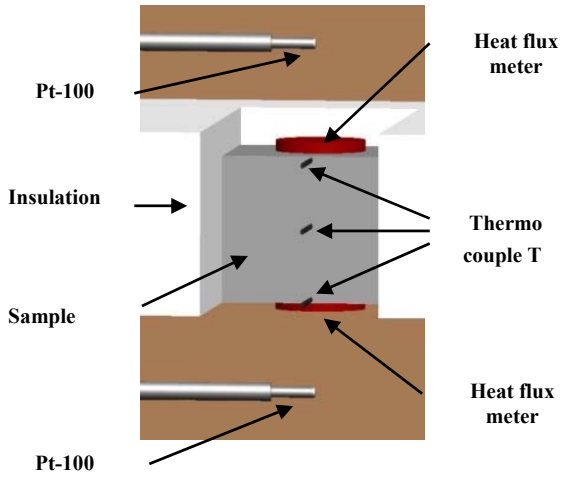

Figure 2. Sensor distribution in the sample

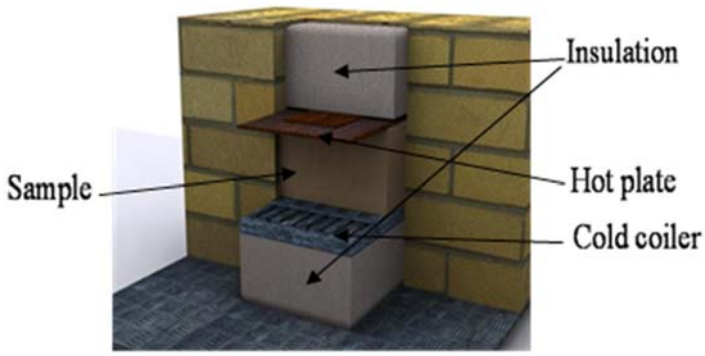

Figure 3. Sketch of the Conductimeter F14

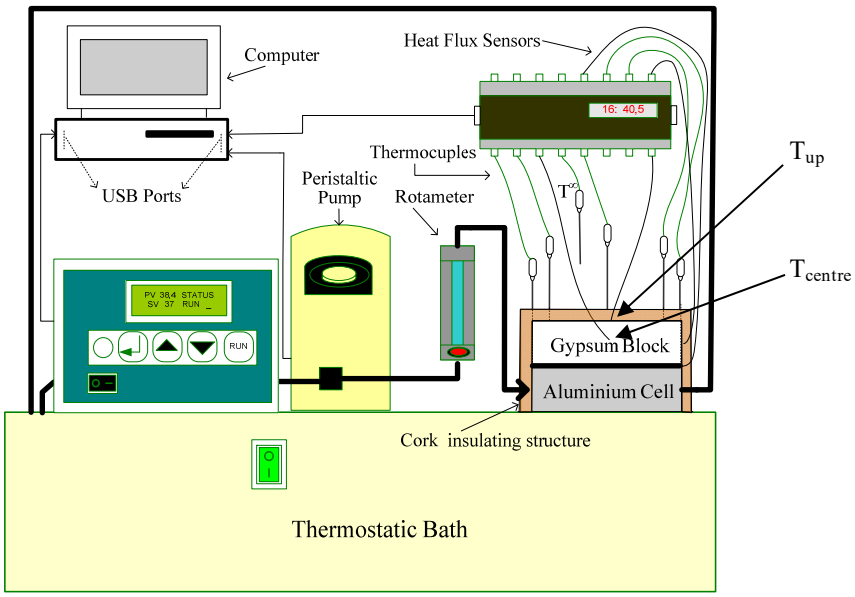

Figure 4. Sketch of the C.R. device

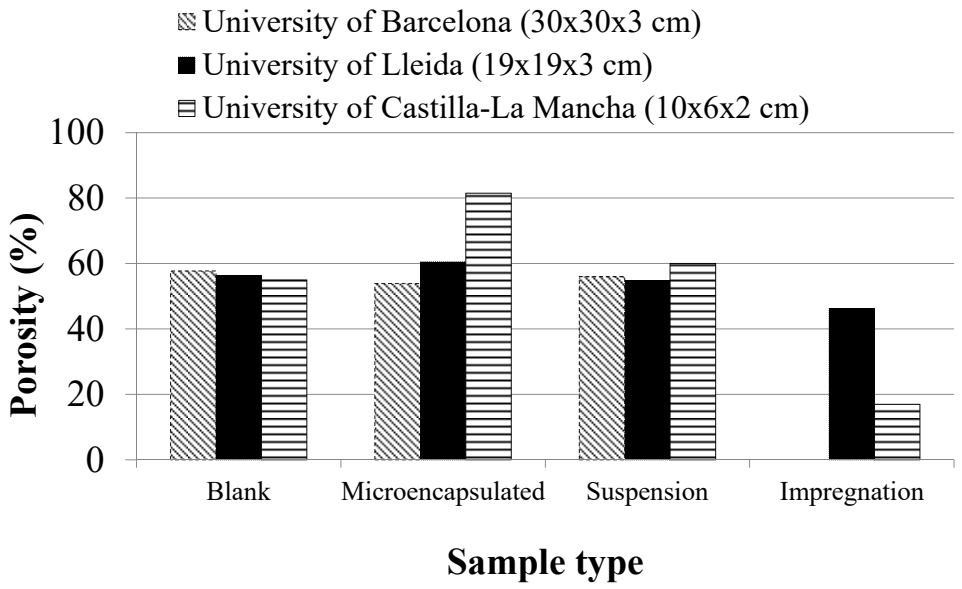

Figure 5. Open porosity results of the samples under study

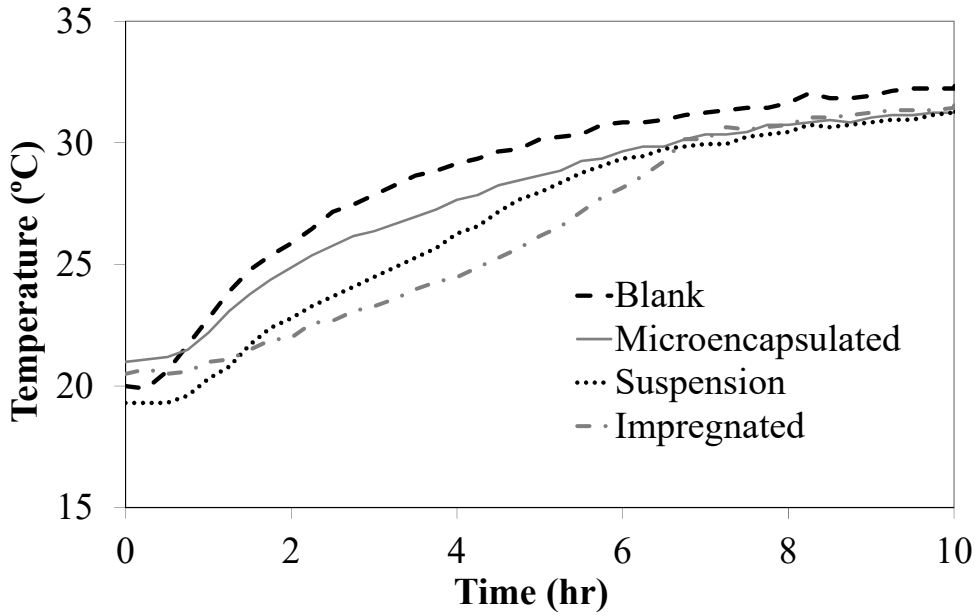

Figure 6. Temperature profile of the samples analysed at the University of Lleida.

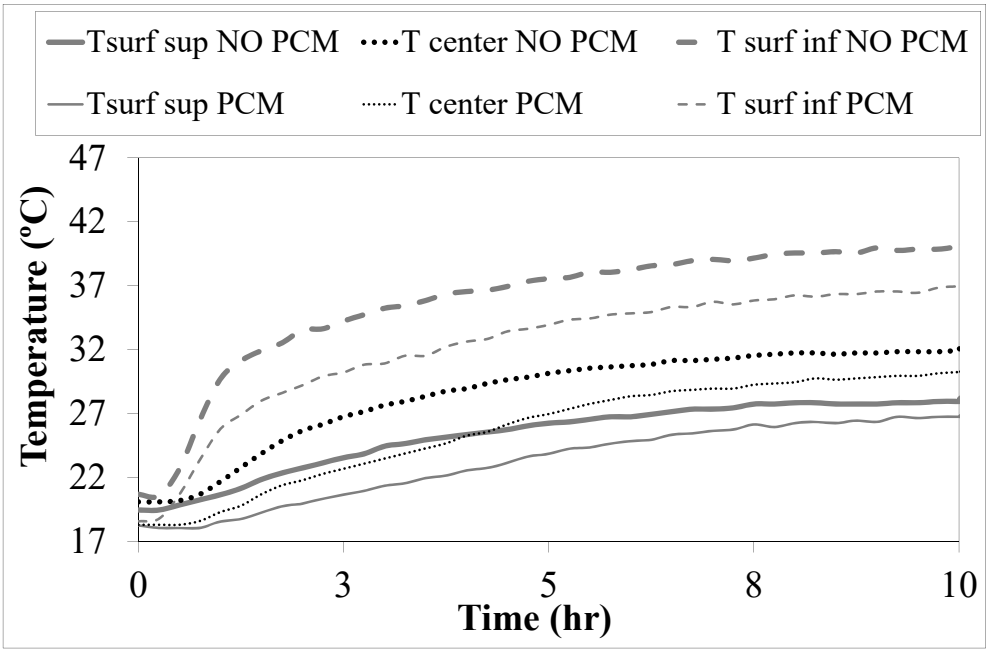

Figure 7. Temperature profiles of the sample with microencapsulated PCM compared to the blank during Experiment 1 (University of Lleida)

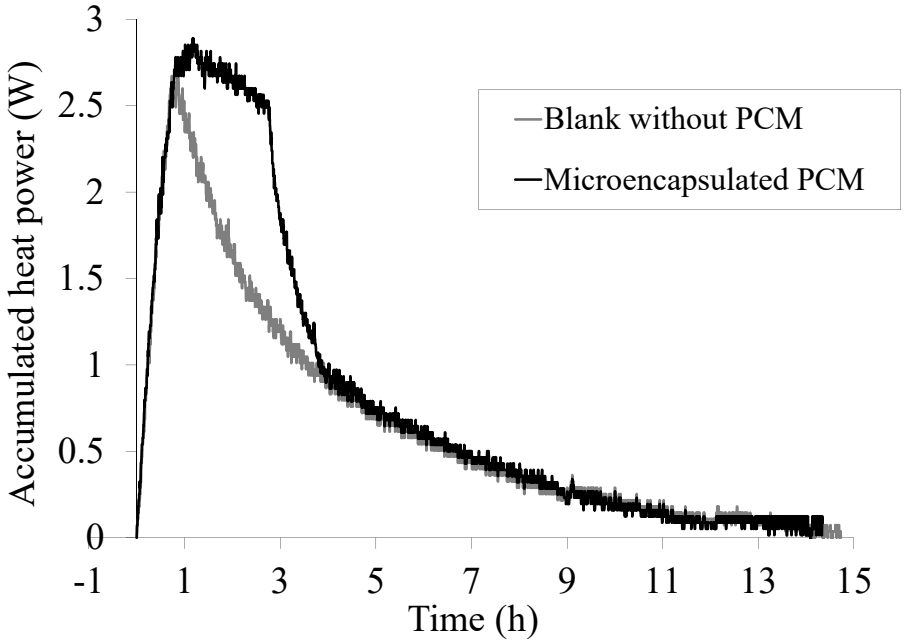

Figure 8. Rate of heat accumulation of the sample with microencapsulated PCM and the Blank, during the Experiment 2 (University of Lleida)

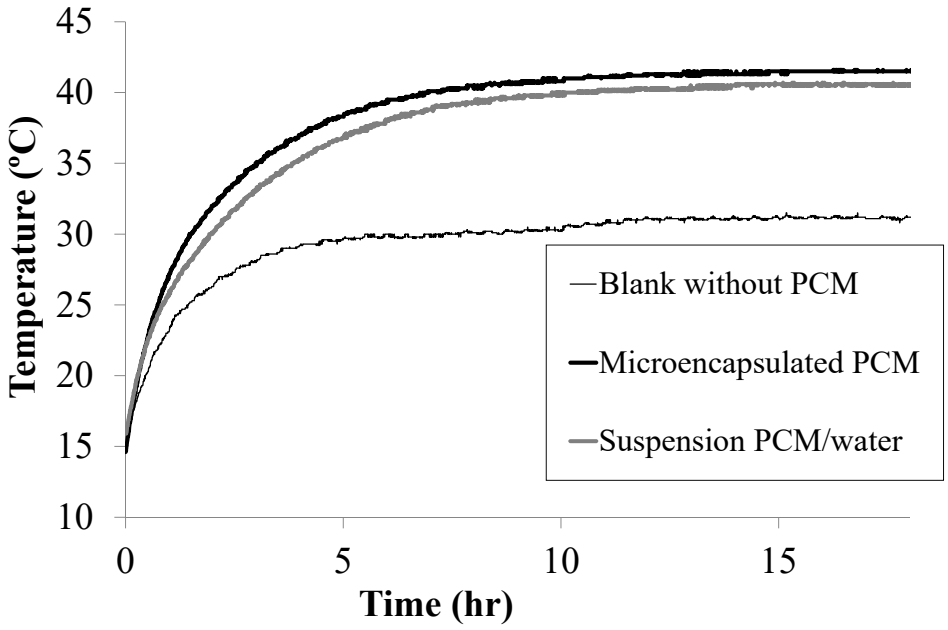

Figure 9. Temperature profiles of the samples analysed by Conductimeter F14 (University of Barcelona)

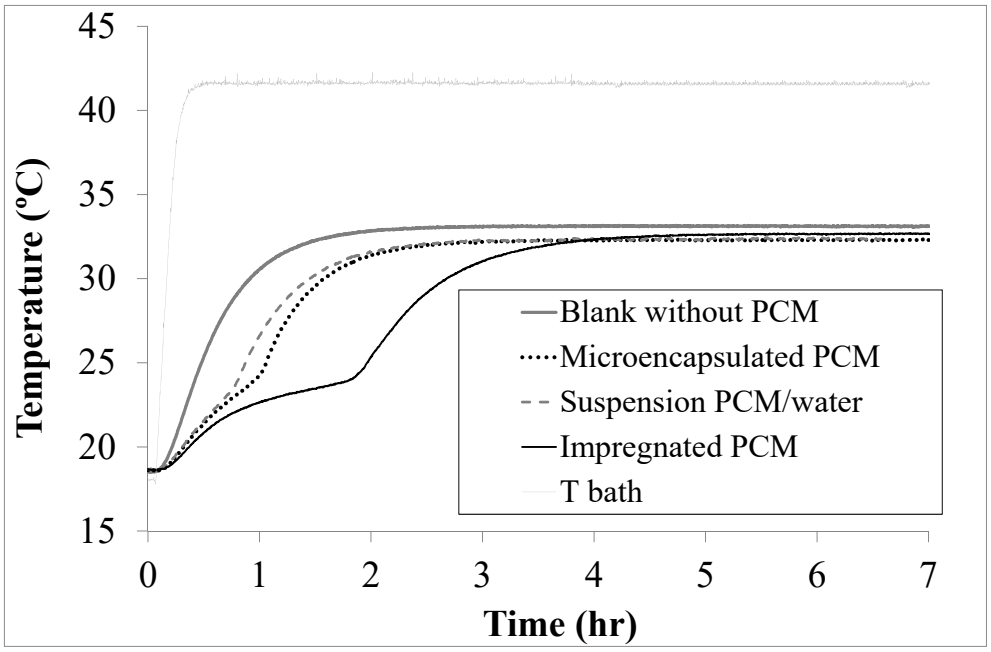

Figure 10. Temperature profiles of thermostatic bath and external surface for studied gypsum samples (University of Castilla-La Mancha)

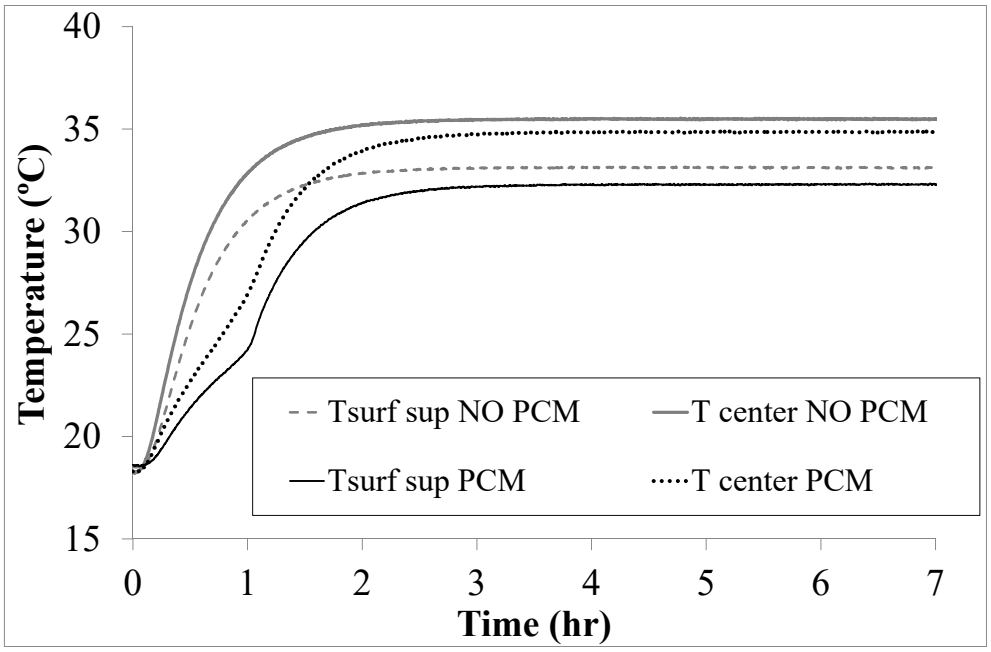

Figure 11. Temperature profiles of the external surface and in the middle of the blank and the sample with microencapsulated PCM (University of Castilla-La Mancha)

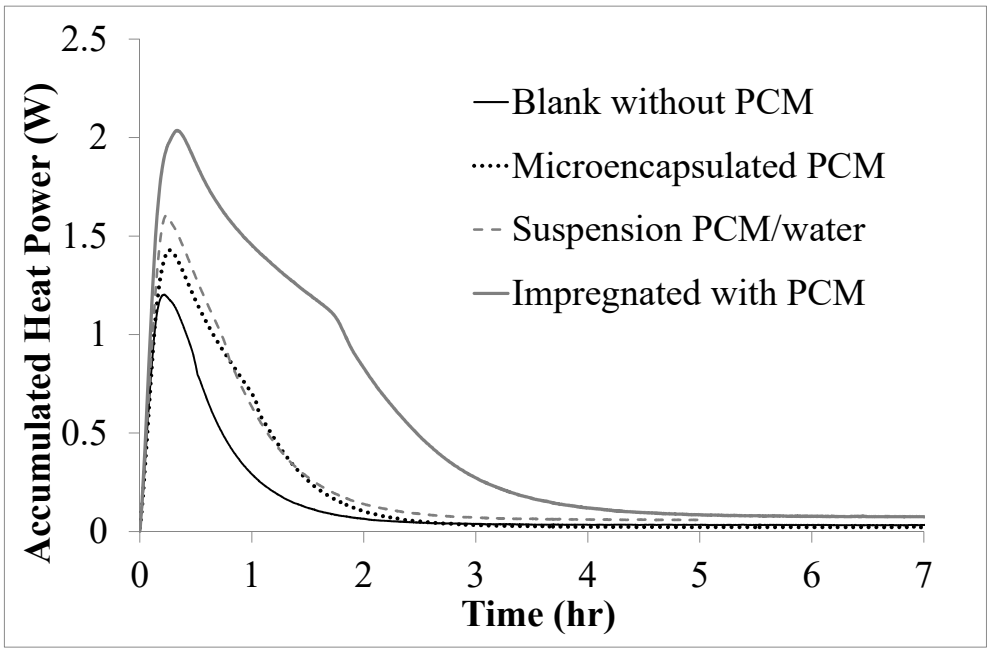

Figure 12. Accumulated heat flow for analysed samples (University of Castilla-La Mancha)