NANOPCM
New Advanced Insulation Phase Change Materials

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D6.3 Assessment of the performance of new advanced insulation phase change materials

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Summary

NanoPCM project aims to develop novel advanced insulation PU panels with improved thermal properties in terms of accumulated heat and isolation level in comparison to traditional materials.

For this, different phase change materials have been developed in WP2 and 3, resulting in innovative encapsulated, not encapsulated and chemically linked phase change materials.

Finally, eight prototypes were constructed within WP6, reported in the deliverable 6.1 “The prototypes of the developed materials designed and manufactured”. After that, decisions about the panels installation within the demo-parks was taken in WP7, told in the deliverable 7.4 “NanoPCM materials production and incorporation to insulation components”.

On the other hand, additional measurements were needed for the WP4, to include in the simulation models reported in the Deliverables 4.3 “Assessment of the thermal behavior of developed materials” and 4.4 “Assessment of the thermal behavior of sandwich panels”.

In this document, the lab experiences are reported to know the efficiency of the developed panels and materials. Within the tests, three different systems have been used: MDSC, HFM and one innovative machine designed by UCLM.

This way, the materials as been deeply defined and the properties were introduced in the micro and macro scale models.

Contents

Abbreviations

NANOPCM	New Advanced Insulation Phase Change Materials
PCM	Phase Change Materials

1.-Introduction

That report has the main objective of showing the behavior of panels tested by different partners present in the NanoPCm project.

2. - Prototypes construction

Following the results from WP2 and WP3, the best materials were selected to construct the prototypes.

Few of them are sandwich panels while others are layers of PU foam improved by adding different types of PCM.

For the construction of the PU foam samples (as the standard as the modified one) was constructed by one only step, mixing the components used in the standard case with or without PCMs.

For the construction of prototypes, it was used the lamination process to maintain adhered all the layers in an only panel.

Both processes are deeply told in the Deliverable 6.1 “The prototypes of the developed materials designed and manufactured”.

3. – Measurements

NanoPCM project has the purpose of getting a commercial insulation product which have a competitive price to be present in the market.

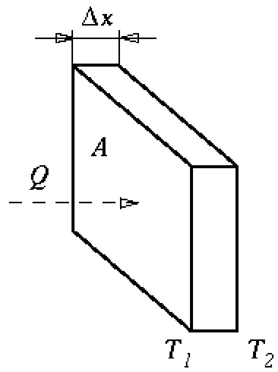
Because of this, the entire research, measurements and demonstration activities are focused in the thermal behavior of the product.

Thermal properties were measured to evaluate the influence of PCM in comparison to the standard PU foam and in order to compare between the novel products from the project.

3.1 Thermal conductivity

The thermal conductivity is a bulk property that describes the ability of a material to transfer heat. In the following equation, thermal conductivity is the proportionality factor k. The distance of heat transfer is defined as Δx, which is perpendicular to area A. The rate of heat transferred through the material is Q, from temperature T1 to temperature T2, when T1>T2.

$$k = \frac{Q\Delta x}{A(T_2 - T_1)}$$



The thermal conductivity was measured using few types of equipment.

On the one hand, Acciona measured the thermal conductivity using an HFM (Heat Flow meter) while UCLM used DSMC and an innovative equipment designed by themselves.

3.1.1 ACCIONA measurements

The equipment used is HFM 436/3 Lambda (NETZSCH)

For this, samples had to be cut with dimensions 30 cm x 30 cm. Before putting in the HFM, it is necessary to know the density and include that as parameter. About the thickness, it can be introduced manually or measured automatically by the machine.

- Operating

The Sample is placed between two heated plates, set at different temperatures. The heat flow (q) through the sample is measured by a (calibrated) heat flux transducer. After reaching a thermal equilibrium, the test is done. Only the sample center (100x100 mm for the HFM 436/3/x versions) is used for the analysis. The heat flux transducer output is calibrated with a standard.

The magnitude of the heat flow (q) depends on several factors:

• thermal conductivity of the sample (λ)
• thickness of the sample (Δx)
• temperature difference across the sample (ΔT)
• area through which the heat flows (A)

The Fourier heat flow equation (Equation 1) gives the relationship between these parameters when the test section reaches thermal equilibrium.

$$\dot{Q} = \lambda A \frac{\Delta T}{\Delta x}$$

One or two heat flow transducers measure the heat flow though the sample
. The signal of a heat flow transducer (in Volts (V)) is proportional to the
heat flow through the transducer. In the HFM 436 Heat Flow Meter instrument, the area of the heat flow transducer represents the area through which the heat flows and is the same for all samples; therefore:

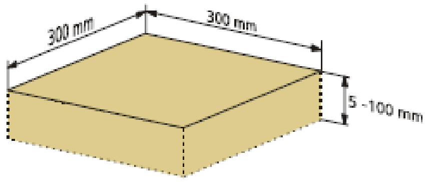
$$\dot{Q} = N \cdot V$$

where N is the calibration factor that relates the voltage signal of the heat flow transducer to the heat flux through the sample. Solving for λ we derive the thermal conductivity:

$$\lambda = \mathbf{k} = N\frac{V\Delta x}{\Delta T}$$

- How the equipment is working

Samples must have dimensions 30 cm x 30 cm and the thickness can change within a range, from 5 to 100 mm.



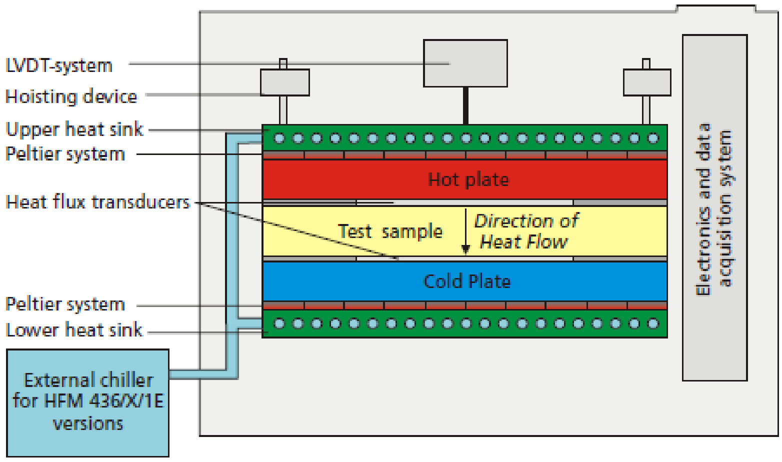
If the sample has smaller weidght or lenght, it can be placed within a frame constructed by the supplier.

In the case of NanoPCM project, the samples were constructed thinkin in it, so they are 30 cm x 30 cm.

To start measuring, the sample must be located in the equipment. After that, the uppwe plate is moved automatically by pressing the botton. This way, the sample is between both plates.

Before measuring, it was necessary to think carefully about the temperatures we want to use. On the one hand, there are three different temperatures: Upper temperature (temperature of the upper face), down temperature (temperature on the bottom of the sample) and intermediate temperature (temperature in the center of the sample). The last one is not measured but is calculated through the promedium of upper temperature and down temperature, as it is supossed a linear behaviour of the sample.

Before starting, it is necessary to think about the temperatures would be necessary to work with. This way, the expected measurements are done considering the two possible states of the PCM (melting and solid). After that, the intermediate temperature is fixed as well as a temperature increase (temperature increase between the upper and down plates). With a basic calculation, the expected upper temperature is the intermediate temperature summing the half of the increase. The expected down temperature is the intermediate temperature taking away the half of the increase.



After fixing the needed parameters, the system start working, heating the upper plate and cooling the down plate.

It is recomended to fix the temperatures, wait for a couple of hours and start measuring to avoid stabilization problems and consequently mistakes in the measurements.

- Standards considered

The heat flow meter method is a standardized test technique. The application (insulating materials) is strongly connected with standards:

• ISO 8301:

Standard test technique for measurements of insulating materials using the heat flow meter method.

• ASTM C518:

American standard for measurements of insulating materials using the heat flow meter method.

• DIN EN 12667/12939:

European standard for measurements of insulating materials using the heat flow meter method or the guarded hot plate technique.

• DIN EN 13163:

European standard for characterization of foam insulations for building applications using the heat flow meter method or the guarded hot plate technique.

- Results

Sample	Thickness (cm)	m (g)	ρ (kg/m³)	PCM Tmelt. (˚C)	‹T› (˚C)	ΔT (˚C)	k(T) (W/m·K)
Standard	3.50	22	72.00		24	15	0.0325
PU laminated	4.25	923	276,95	-	10	10	0.0346
PU laminated	4.25	923	276,95	-	30	10	0.0381
PU + 5% porous PCM	4.89	443	279,545		30	10	0.0437
PU + 5% porous PCM	4.89	443	279,545		10	10	0.0423
PU + masterbach laminated+ epoxy resin laminated	4.97	1402	386,93	13-33	35	10	0.0396
PU + masterbach laminated+ epoxy resin laminated	4.97	1402	386,93	13-33	10	10	0.0370
Standard	3.50	221	72	-	24	15	0.0325
Epoxy resin laminated	0.3	570	2111,48	-	10	10	0.1003
Epoxy resin laminated	0.3	570	2111,48	-	30	10	0.1066
Masterbach laminated	0.4	555	1542,5	13-33	35	10	0.1136
Masterbach laminated	0.4	555	1542,5	13-33	10	10	0.1030

Table 1 Thermal conductivity results of prototypes using HFM device (Note: Standard is a glass fiber panel from the supplier used for the equipment calibration)

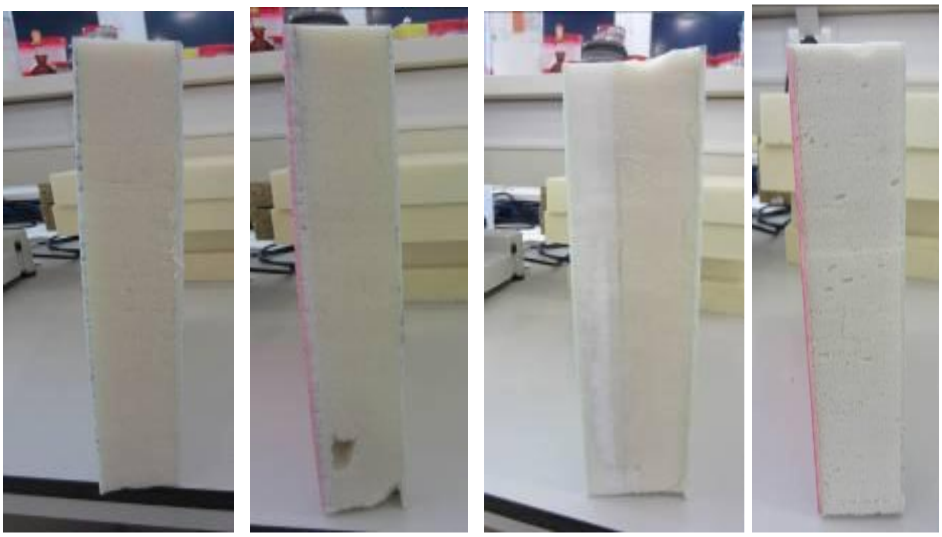


Figure 1 From the left-to-right: PU laminated; PU+masterbach laminated (laminated); PU+masterbach panel (laminated); PU+ 5% porous PCM (laminated);

Figure 2 From the left-to-right: epoxy resin laminated using glass fiber layers ; masterbach laminated using glass fiber layers

Other prototypes were tested although it was not possible to extract any result as the machine cannot work at a temperature out of the melting temperature range.

In the case of the prototypes were the masterbach was present, no conclusions can be extracted as it was not expected any answer from the system because of the melting temperature range.

PANEL (content 10%)	weight (Kg)	V(m3)	density (kg/m3)	k (W/mK)	Tmean	T delta	Melting point
fatty acids	0,1287	0,0036	35,75	0,02874	11	11	19
fatty acids	0,1287	0,0036	35,75	0,03144	30	10	19
RT27	0,162	0,0036	45	0,03709	15	10	27
RT27	0,162	0,0036	45	0,03943	35	10	27
octadecane	0,161	0,0036	44,72	0,02729	15	10	27,67
octadecane	0,161	0,0036	44,72	0,02986	35	10	27,67
Blank		0,0036	72	0,03169	24	20	-

Table 2 Thermal conductivity results of panels using HFM device

As it can be extracted from the table, the highest thermal conductivity is shown by the PU panel containing 10% of microencapsulated PCM in LDPE-EVA capsules. This behavior is extracted at low and high temperature. It means, for both cases were the PCM is melted or solid, that panel has a higher thermal conductivity than other cases.

On the other hand, the panel with a lowest thermal conductivity value is the PU panel containing microcapsules of SiO_2 containing octadecane.

3.1.2 UCLM measurements

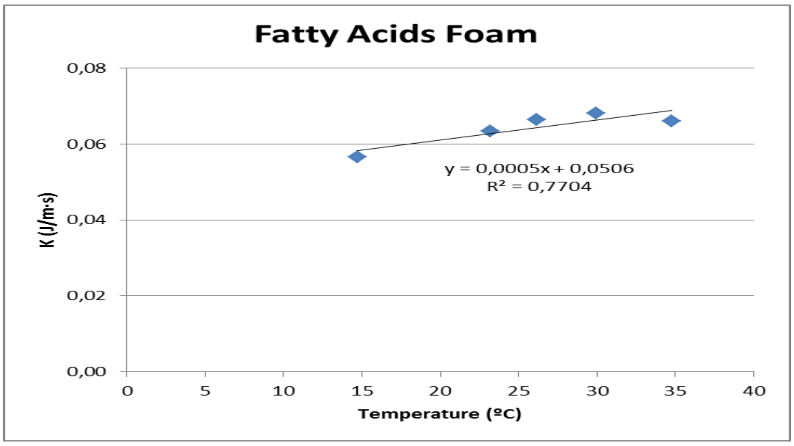


Figure 3 Thermal conductivity of PU foam containing 10% of SiO2 microcapsules confining fatty acids mixture

From the graph, although the thermal conductiviuty of the PU sample containing microencapsulated fatty acids increases with temperature increases, the grown is not so high as other cases.

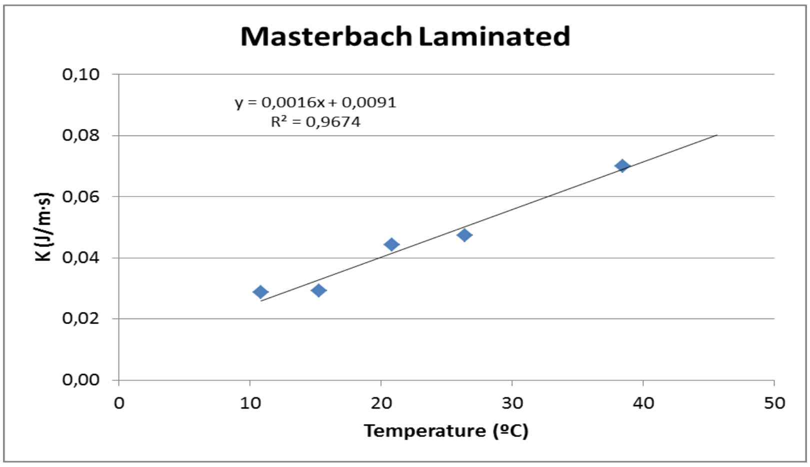


Figure 4 Thermal conductivity of laminated masterbach

In the case of the masterbach laminated, the thermal conductivity is increased as proportional function of the temperature.

This way, the insulator behavior of that material is not conserved along the temperature increase.

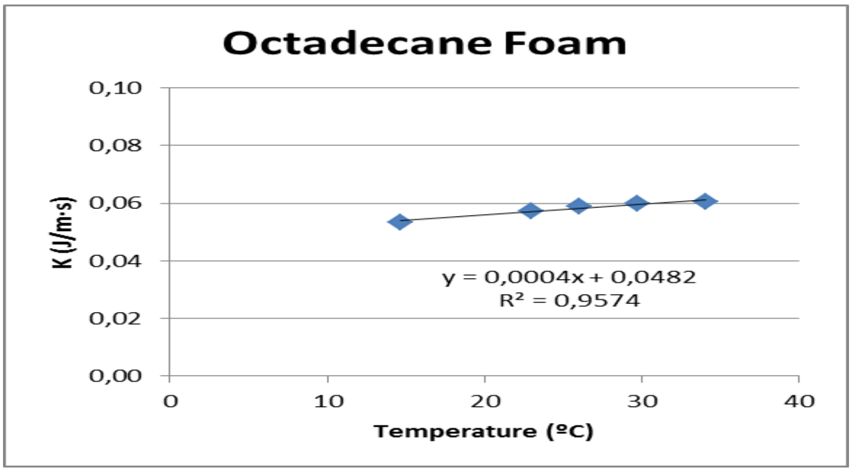


Figure 5 Thermal conductivity of PU foam containing 10% of SiO2 microcapsules containing octadecane

As it can be observed from the graph, the sample containing octadecane show a stabilizated thermal conductivity. It means that the K value will not be increased along the temperature.

In this case, the purpose of NanoPCM project was approximately reached, as the insulator behavior is not affected.

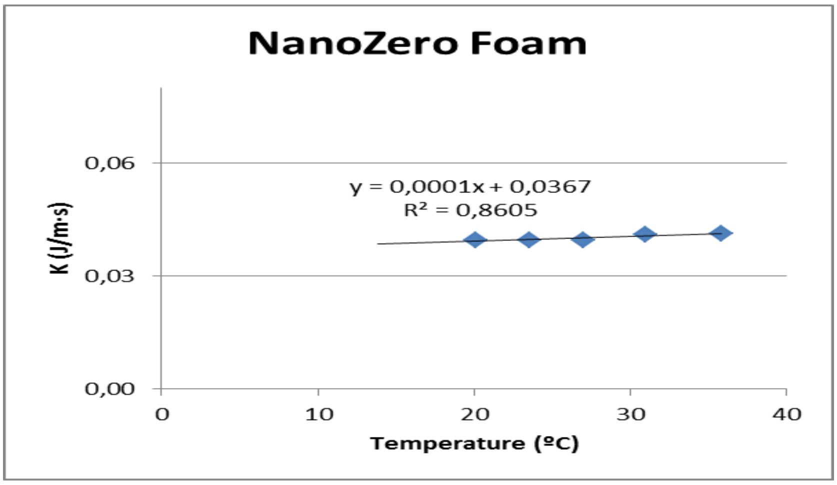


Figure 6 Thermal conductivity of standard PU foam

As it can be extracted from the graphs, the highest dependance of the thermal conductivity on the temperature is shown by the masterbach laminated. In that case, the lamination was done using glass fiber layers. Then, that behaviour is considering not only the masterbach material.

In that case, nanoparticles of aluminium oxide were used as additive. It can be the reason of the thermal conductivity grown.

3.2 Cp (Heat capacity)

The heat capacity Cp of a substance is the amount of heat required to change its temperature by one degree, and has units of energy per degree. The heat capacity is therefore an extensive variable since a large quantity of matter will have a proportionally large heat capacity.

In the case of NanoPCM project, the Cp value is directly related to the capacity of store heat. It is expected that the PCM will be the main responsible of storing heat because of its thermal properties: when the PCM melts, it absorbs some energy (related to the type of PCM) which can be dropt when it becomes a solid again.

Because of that, the purpose of NanoPCM project is getting a product with a high capacity of storing energy. It means, NanoPCM looks for a final product with a high Cp.

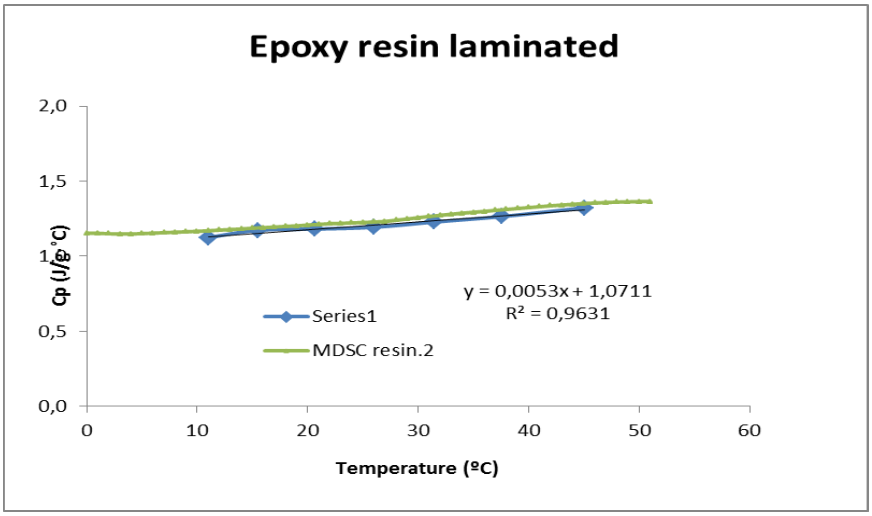


Figure 7 Heat capacity of epoxy resin laminated using glass fiber layers. The measurements were carried out using MDSC machine and a novel devide designed by UCLM

As it can be seen in the graph, the epoxy resin has a low value of energy stored. It means that it has almost no capacity of storing heat.

Moreover, it shows a linear behavior what means that there is not any phase change and it does not change their properties along the temperature range.

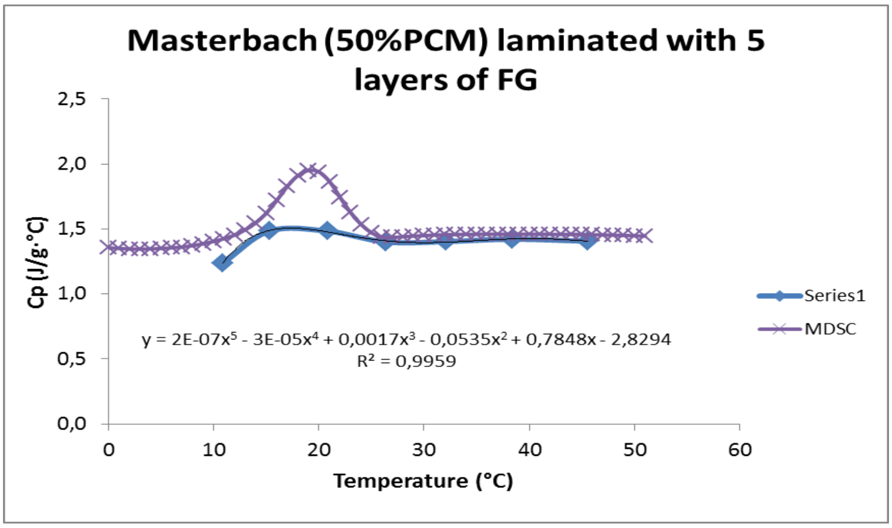


Figure 8 Heat capacity of masterbach laminated using glass fibeer, measured using MDSC and novel equipment

In the case of the masterbach laminated, the blue line shows the results using the novel equipment designed by UCLM. In this case, the results can be more reliable using the MDSC device, as it can be seen the phase change.

The results show an increase in the heat storage capacity when the PCM melts. Moreover, in that case, where the melting temperature range is from 13 to 33ºC, the range has been even shorter than the expected one. The curve is observed from 12 to 26ºC, which shows a high presence of some eutectic components and low of others.

Using the novel machine, it cannot be seen any change when there is.

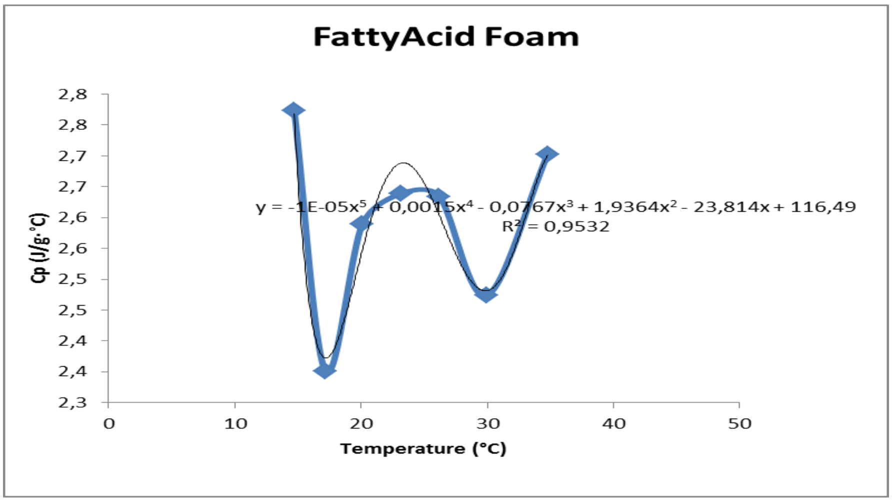


Figure 9 Heat capacity of PU foam containing 10% of SiO_2 microcapsules containing a fatty acids mix

In the case of PU foam containing encapsulated fatty acids mix, the results are weird and it was concluded that they are not good enough, as using the MSCD as novel equipment.

The reason is that observing a quite high Cp value from the beginning is not logical.

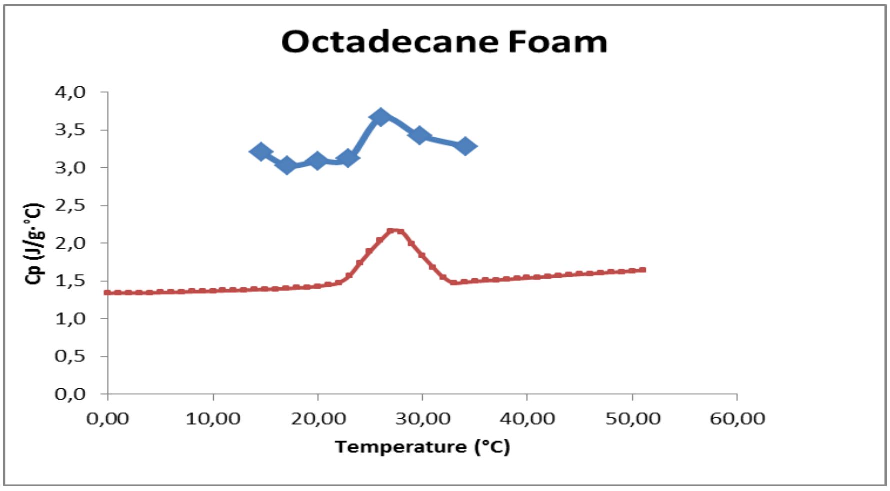


Figure 10 Heat capacity of PU foam containing SiO_2 microcapsules containing octadecane

For the PU sample containing SiO2 microcapsules confining octadecane, two results were extracted from two different equipment. In that case, the differences between both results are highly noted as it can be extracted from the last graph.

If the blue line is considered, the panel containing 10% of octadecane confined is the one which shows the best result in therms of accumulated heat. If not, the panel containing masterbach (50% PCM) can be the best option related to the absorbed heat.

On the other hand, the samples were not as dimensionally similar as it should. Then, the comparison between panels is not possible.

3.3 Physical properties

The panels were received for the installation in the Polish and Spanish demo-park. Before installing them, it was observed some differences:

- Panels containing microencapsulated fatty acids are slightly yellowish than others.
- Panels containing SiO2 microcapsules, as containing octadecane as fatty acids mix, show poor mechanical properties. In this case, the mechanical parameters were not tested but touching them, it was easily observed some release of particles.
- Panels containing RT27 microcapsules show better results in terms of relead particles although almost every panel was broken. It means that the manufacturing process was not optimized.

4. - Conclusions

In that report, the thermal properties of samples from the NanoPCM project were tested.

On the one hand, a standard PU foam sample was tested in terms of thermal conductivity and heat capacity measurements. On the other hand, masterbach laminated by glass fiber layers was tested following the same procedure in order to compare thermal behavior of all the samples extracted from the Work Packages 2 and 3.

Additionally, some experiences extracted by touching the samples are exposed.

For the thermal conductivity measurements, two partners involved were working in it. In the case of Acciona, it was used a HFM with which the best results was shown by the PU foam containing 10% of fatty acids mixture encapsulated by SiO_2. The highest K value was shown by the PU panel containing 10% of RT27 in LDPE-EVA microcapsules. On the other hand, the prototype of PU foam containing 5% of porous PCM (SiO2 impregnated with paraffin) showed even higher results than others.

In the case of UCLM measurements, the thermal conductivity values can be extracted in a dynamic way. It means, that the results are not values related to a specific temperature, so additional conclusions can be taken. In that case, it has been compared the stabilization along the temperature, which was shown by the PU panel containing 10% of octadecane confined in SiO_2 microcapsules. The highest value was shown by the epoxy resin without PCM.

In terms of Cp measurements, the best results are shown by the masterbach panel (50% PCM). It means that it is the material with high heat storage capacity and it could maintain the comfort temperature better than other materials.

The results showed in that report will be used in the deliverables 4.3 “Assessment of the thermal behavior of developed materials” and 4.4 “Assessment of the thermal behavior of sandwich panels” (month 28).

Acknowledgements

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