Benchmarking of useful phase change materials for a building application

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Abstract

Nowadays, there is an increasing interest in more efficient building materials and new technologies to accomplish the objectives defined by energy policies. The combination of energy efficient building designs and integration of renewable energies makes the use of thermal energy storage (TES) necessary. Within this context, the improvement of building envelopes by the use of phase change materials (PCM) has been widely studied. The PCM selection should fulfil the requirements of the specific application. In this study, a benchmarking of PCM available in the market was performed to undertake a material selection for a specific building application. The incorporation of PCM into a radiant wall technology present several requirements with especial interest on long-term thermal stability. A complete laboratory-scale characterization has been carried out considering the following properties: temperature of phase change, specific heat capacity, thermal conductivity, and stability.

Keywords: thermal energy storage (TES); phase change materials (PCM); buildings; material selection; benchmarking

1. Introduction

The building sector is responsible for one-third of the global energy consumption, with a significant importance on the energy consumed in residential buildings [1]. Energy intensity per square metre in buildings has improved in many regions, but not fast enough to offset the doubling of floor area since 1990 [2].

Within this context, the interest of using more efficient building materials, new technologies and new systems is growing to accomplish the objectives defined by energy policies. Building envelopes, design and orientation are some of the building aspects that directly affect the energy demand intensity. Net-zero energy buildings (NZEBs) represent the combination of energy efficient building designs and renewable energies to meet the whole building energy loads [3]. Due to the intermittency that characterizes renewable energies, thermal energy storage (TES) technology should be hand by hand when implementing these systems to ensure the energy supply available to the end user.

Therefore, TES technology could be implemented, basically for domestic hot water or space heating and cooling purposes. The use of phase change materials (PCM) allows taking advantage of the latent heat absorbed and release during the phase change from solid to liquid. With these materials a larger amount of thermal energy can be stored in a lower volume compared to sensible heat storage [4]. Moreover, the PCM works within a specific temperature range (phase change temperature), which provides the maximum amount of thermal energy in the desired temperature range of the application [5].

However, not all PCM are suitable for building applications. An ideal PCM candidate should fulfil a number of criteria such as high heat of melting and thermal conductivity, high specific heat capacity, small volume change, non-corrosiveness, non-toxicity, and exhibit little or no decomposition or subcooling [5,6]. Moreover, the temperature range where the phase transition occurs is a significant parameter in the selection of the material [7]. As an example, Saffari et al. [8] showed that the use of a PCM passive system in the building envelopes with optimized melting temperature for each climate conditions can lead to higher energy savings, Khana et al. [9] showed that the use of a PCM integrated in PV collectors with optimized melting temperature for each climate can lead to higher electricity production, or Young et al. [10] stated that the phase change temperature is the main selection criteria to choose the right PCM of concrete pavements.

Therefore, the selection of the PCM should be defined by the requirements of the application. In this case, the PCM is selected to be implemented in an active TES system, a radiant wall technology. This technology was designed in the framework of the project INPHASE (Spanish Retos Colaboración funding scheme) with the following requirements set: to use a PCM commercially available, the possibility of PCM inclusion in a building concrete matrix, the phase change temperature inside the comfort building range (between 21ºC and 26ºC), and long-term thermal stability. The design of the system was framed under the objective of the development a new precast concrete panel (enhanced using PCM) for new residential buildings and also of refurbishment of Spanish buildings built before 1979, since those are the buildings which will go to energy rehabilitation in the near future. Those existing building walls are usually made of an external wall, an air chamber and a thinner wall with the internal gypsum finishing. The design developed had the idea of including a concrete layer with the PCM incorporated in it and with water piping to actively charge and discharge the PCM. External thermal insulation was used in both cases (Figure 1).

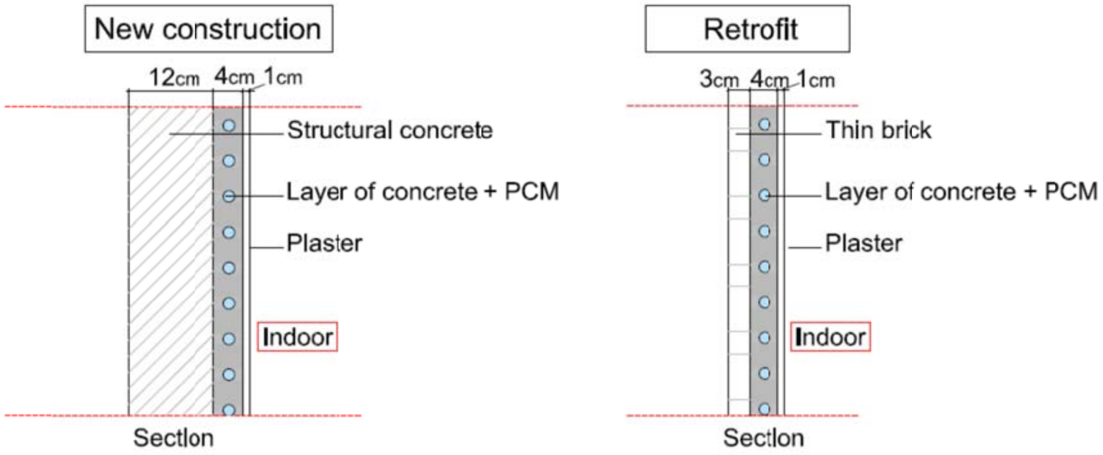

Figure 1. Scheme of the system considered.

The main objective of this study is to characterize the phase change materials currently available in the market that could be used fulfilling such requirements. With this purpose, products of a different nature and form were selected from different companies. The results obtained from this study will allow the PCM to be classified and selected to this specific application. The best materials will be those with high values of enthalpy, heat capacity and thermal conductivity, thermal cycling stability as well as a phase change temperature in the range of the application. This information will also allow comparing the results obtained with the data provided by the manufacturer.

2. Materials

The PCM analysed in this study are commercial available in the market classified as bulk PCM, micro-encapsulated PCM, and macro-encapsulated PCM. The phase change temperature and manufacturer of all the materials are listed in Table 1. The material used to microencapsulate or macro-encapsulate PCM are polymeric materials in order to avoid leakage which is one of the main drawbacks of PCM implementation in building systems which must be compatible with the PCM during all the life cycle. Therefore, the most used polymer to encapsulate PCM is polymethyl acrylates [11]. The PCM itself was always a paraffin.

Table 1. PCM under study

	PCM	**Phase change temperature Data sheet (ºC)**	**Manufacturer**
Bulk PCM	RT21 HC	21	Rubitherm
	RT22 HC	22	Rubitherm
	RT 25 HC	25	Rubitherm
	RT 27	27	Rubitherm
	PureTemp 23	23	Entropy Solutions
Micro-encapsulated PCM	Micronal DS5040X	23	BASF
	Micronal DS5008X	25	BASF
	MPCM24D	24	BASF
	Micronal DS5038X	25	Microteklabs
Macro-encapsulated PCM	MacroPCM28	28	Microteklabs
	MacroPCM24	24	Microteklabs

3. Methodology

PCM characterization is commonly based on their thermophysical properties evaluation before and after thermal cycling stability test, in order to ensure the proper performance over charging/discharging processes, and to guarantee chemical stability during all their service life. To achieve that, phase change temperature, phase change latent heat, as well as chemical structure composition must be studied after several thermal cycles. Moreover, specific heat capacity (*Cp*) and thermal conductivity are key properties for PCM selection as a proper candidate for a final application. Therefore, both properties were also included as part of the methodology of PCM characterization. Thermal conductivity and *Cp* change depending on the material state, hence liquid and solid phases were analysed.

3.1. Phase change thermal properties and thermal cycling stability

Thermophysical properties of PCM were measured with differential scanning calorimetry (Mettler Toledo DSC 822e). Depending on each PCM phase change temperature, several thermal dynamic methods were programmed.

Each PCM sample was heated up to 50 ºC and cooled down to 10 ºC applying 10 K/min heating/cooling rate in order to homogenize, in a first stage, all the materials under study and placing the material in a perfect contact with the bottom of the crucible. Afterwards, the thermophysical properties were measured by applying 0.5 K/min heating rate from 10 ºC to 50 ºC. Analyses were performed using 40 mL aluminium crucibles under 50 mL/min N_2 flow. The equipment accuracy is +/-0.1 ºC for temperature measurements and +/-10% regarding enthalpy measurements [12].

The thermal cycling stability is one of the key attributes of PCM because allow suitable and realistic implementation at the final application taking into account the thermal degradation during their charging/discharging life cycle. Thereby, the phase change temperature and the latent heat absorbed/released during the phase change should remain constant during the thermal cycling test. Moreover, the chemical composition and structure of PCM must remain safe during this test.

The PCM studied in this paper were brought up to 10000 melting/solidification thermal cycles simulating 30 working service years. To carry out the thermal test, a Bioer Gene Q T-18 thermal cycler was used, which allow cycling 12 samples each time. The materials were thermally cycled between 10 ºC and 40 ºC, as the Figure 2 shows, in order to assure the complete charging/discharging processes.

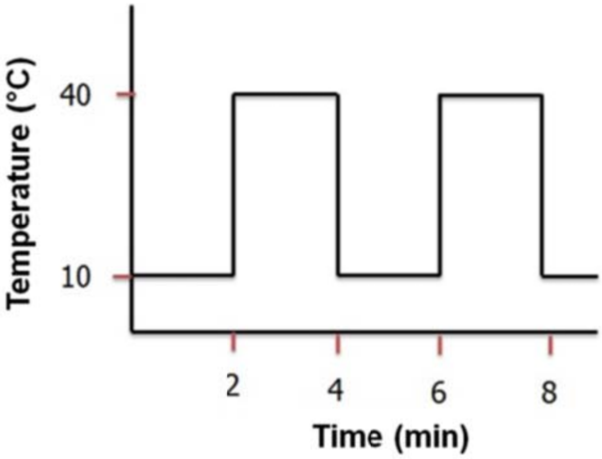

Figure 2. Thermal cycles applied to the samples under study

Samples were analysed after 10, 100, 1000, and 10000 thermal cycles with DSC applying the same conditions described before. Moreover, the chemical structure evaluation to follow a possible chemical decomposition was followed by FT-IR with Spectrum Two™ de Perkin Elmer coupled with an ATR (Attenuated Total Reflectance).

3.2. Specific heat capacity

A Mettler Toledo DSC 822e was used to measure the *Cp* at atmospheric pressure applying the areas method described by Ferrer et al. [13] (Figure 3) and using sapphire as pattern material. The *Cp* was measured at 10 ºC (solid state), 40 ºC, and 50 ºC (liquid state). Analyses were performed using 40 mL aluminium crucibles under 50 mL/min N_2 flow. The equipment accuracy is 3% for *Cp* measurements [13].

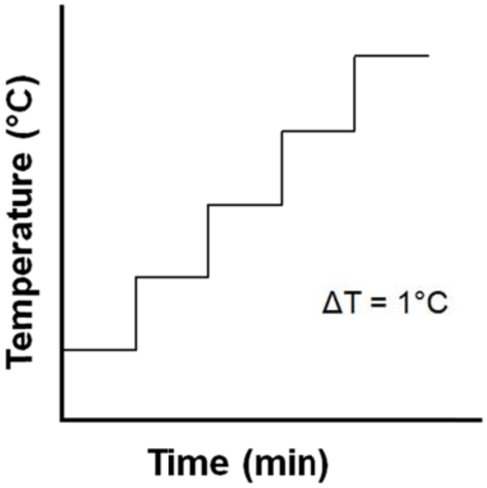

Figure 3. Areas method used with DSC for *Cp* analyses

3.3. Thermal conductivity

Hot wire K2D Pro de Decagon Device was used to analyse the thermal conductivity of the samples under study. The thermal conductivity was measured at 10 ºC, 35 ºC, and 50 ºC in order to obtain results at liquid and solid states. In order to minimize errors due to the environmental conditions, two different series of ten measurements were performed by waiting 15 minutes between each measurement. The equipment accuracy is +/- 5%.

4. Results and discussion

The results of the experimental characterization of all the PCM under study is shown in this section along with a general discussion. The final selection is based on the specific requirements given by the application, detailed in the introduction, which are the use of a PCM commercially available, the possibility of PCM inclusion in a building material matrix, the phase change temperature inside the comfort building range, and long-term thermal stability.

4.1. Phase change thermal properties and thermal cycling stability

Phase change enthalpy and temperature (melting and solidification) for all the eleven PCM under study was experimentally determined before and after 10, 100, 1000 and 10000 melting/freezing cycles (Table 2).

The results show that, in general, bulk PCM present higher phase change enthalpy than the microencapsulated and macro-encapsulated. These results were expected since micro and macro-encapsulated PCM present less PCM concentration due to their morphology and composition of their capsules, usually made of polymeric materials. However, it is noteworthy to point out that macro-encapsulated PCM MacroPCM28 presents a phase change enthalpy as high as some bulk PCM, such as RT21 HC and RT22 HC, of around 130 kJ/kg. The micro-encapsulated PCM as well as the other macro-encapsulated, MacroPCM 24, show enthalpies between 86 kJ/kg and 110 kJ/kg. All the standard deviations of the results are between ± 0.01 and ± 0.09.

Table 2. Phase change enthalpy and temperature of the PCM before and after 10, 100, 1000 and 10000 cycles

Material		H_m (kJ/kg)	Property loss (%)	H_s (kJ/kg)	Property loss (%)	T_m (ºC)	Property loss (%)	T_s (ºC)	Property loss (%)
Bulk PCM									
RT21 HC	**Not-cycled**	137.02	0.00	126.03	0.00	22.21	0.00	21.47	0.00
	Cycled (10)	127.94	6.63	114.12	9.45	22.69	-2.15	21.87	-1.86
	Cycled (100)	124.88	8.86	113.77	9.73	22.76	-2.46	21.91	-2.08
	Cycled (1000)	141.22	-3.07	139.37	-10.58	22.11	0.47	21.15	1.46
	Cycled (10000)	109.47	21.74	110.71	12.16	21.32	4.03	20.73	3.45
RT22 HC	**Not-cycled**	141.17	0.00	144.74	0.00	23.06	0.00	22.27	0.00
	Cycled (10)	137.01	3.04	127.91	13.16	22.94	0.55	22.21	0.30
	Cycled (100)	130.49	8.18	130.47	10.94	22.07	4.52	21.18	5.16
	Cycled (1000)	130.58	8.11	125.97	14.90	23.16	-0.43	22.41	-0.62
	Cycled (10000)	128.20	10.11	124.28	16.46	21.96	5.01	21.19	5.11
RT25 HC	**Not-cycled**	177.29	0.00	186.03	0.00	25.27	0.00	n.a.	n.a.
	Cycled (10)	128.86	27.32	129.09	30.61	26.66	-0.75	25.40	n.a.
	Cycled (100)	125.34	29.30	121.99	34.42	24.54	0.39	23.77	n.a.
	Cycled (1000)	126.88	28.43	130.7	29.74	26.56	-0.69	25.85	n.a.
	Cycled (10000)	129.10	27.18	130.79	29.69	26.58	-0.70	25.94	n.a.
RT27 HC	**Not-cycled**	164.43	0.00	151.54	0.00	26.46	0.00	26.45	0.00
	Cycled (10)	120.93	26.45	122.88	18.91	26.84	-1.44	25.49	3.65
	Cycled (100)	112.02	31.88	100.54	33.66	25.14	4.96	24.61	6.97
	Cycled (1000)	111.315	32.30	110.33	27.20	27.31	-3.23	26.76	-1.16
	Cycled (10000)	69.28	57.87	69.16	54.37	27.07	-2.30	26.49	-0.12
Pure Temp 23	**Not-cycled**	215.02	0.00	215.79	0.00	24.17	0.00	20.66	0.00
	Cycled (10)	172.75	19.66	181.06	16.10	22.97	4.96	17.69	14.36
	Cycled (100)	183.88	14.48	193.77	10.21	23.39	3.23	19.29	6.63
	Cycled (1000)	166.30	22.66	175.58	18.64	22.84	5.52	17.77	13.99
	Cycled (10000)	170.71	20.61	170.23	21.11	22.23	8.04	18.51	10.42
Macro-encapsulated PCM									
MacroPC M24	**Not-cycled**	86.81	0.00	86.25	0.00	23.61	0.00	19.28	0.00
	Cycled (10)	82.94	4.46	99.63	-15.51	23.71	-0.42	18.87	2.13
	Cycled (100)	82.65	4.79	86.74	-0.56	23.49	0.49	18.94	1.75
	Cycled (1000)	83.75	3.52	99.18	-14.99	23.56	0.18	18.58	3.63
	Cycled (10000)	59.81	31.10	59.06	31.53	23.91	-1.28	18.94	1.75
MacroPC M28	**Not-cycled**	128.59	0.00	138.65	0.00	28.82	0.00	21.82	0.00
	Cycled (10)	130.68	-1.63	139.07	-0.30	28.99	-0.58	21.64	0.81
	Cycled (100)	130.06	-1.15	134.58	2.93	29.19	-1.28	21.58	1.08
	Cycled (1000)	129.97	-1.07	134.79	2.78	28.70	0.43	22.07	-1.18
	Cycled (10000)	132.37	-2.94	136.39	1.63	28.96	-0.49	21.40	1.93
Micro-encapsulated PCM									

MC DS5040X	**Not-cycled**	111.99	0.00	100.15	0.00	22.97	0.00	20.87	0.00
	Cycled (10)	99.23	11.39	69.18	30.92	22.63	1.48	20.59	1.34
	Cycled (100)	73.98	33.94	68.62	31.49	22.61	1.55	20.59	1.37
	Cycled (1000)	73.56	34.32	61.64	38.45	22.64	1.44	20.60	1.31
	Cycled (10000)	73.30	34.55	50.57	49.51	22.67	1.31	20.49	1.82
MC DS5008X	**Not-cycled**	109.04	0.00	102.27	0.00	24.63	0.00	23.19	0.00
	Cycled (10)	88.78	18.58	94.31	7.78	24.62	0.05	23.18	0.03
	Cycled (100)	88.24	19.08	90.03	11.97	24.64	-0.03	23.18	0.04
	Cycled (1000)	85.67	21.43	92.19	9.85	24.65	-0.07	23.17	0.09
	Cycled (10000)	83.50	23.42	92.69	9.37	24.60	0.12	23.17	0.09
MC DS5038X	**Not-cycled**	84.28	0.00	78.64	0.00	25.07	-1.80	21.54	7.12
	Cycled (10)	83.22	0.97	77.30	1.31	25.03	-1.64	21.68	6.50
	Cycled (100)	82.81	1.35	77.38	1.23	25.02	-1.60	21.67	6.55
	Cycled (1000)	83.20	0.99	77.55	1.06	25.02	-1.60	21.71	6.40
	Cycled (10000)	80.22	3.72	75.44	3.13	25.10	-1.89	21.74	6.27
MPCPM24D	**Not-cycled**	99.69	0.00	88.65	0.00	23.14	0.00	20.43	0.00
	Cycled (10)	88.20	11.53	83.13	6.23	22.66	2.10	20.23	1.01
	Cycled (100)	89.02	10.70	84.08	5.16	22.71	1.88	19.36	5.27
	Cycled (1000)	84.09	15.64	79.36	10.48	22.81	1.44	20.21	1.08
	Cycled (10000)	82.94	16.80	73.04	17.61	22.66	2.09	20.25	0.90

Note: m – melting; s – solidification; H_m – melting enthalpy; H_s – solidification enthalpy; T_m – melting temperature; T_s – solidification temperature

The PCM with higher phase change enthalpy are PureTemp 23, RT27 HC, and RT25 HC. Nonetheless, the last two bulks PCM decrease their storage capacity remarkably after cycling, the differences compared to the initial enthalpy are between 21% and 56%. This is also the case for MC DS5040, and to a less extent of Macro PCM24 which shows a decrease of around 31 % after 10000 cycles. On the contrary, Macro PCM28, RT21 HC, RT22 HC, and MC DS5038X are thermally stable ensuring their storage capacity and phase change temperature after 10000 cycles. In addition, PureTemp 23 and MC DS5008X enthalpy loss trend when cycling is up to 20% compared to the initial value. Solé et al. [14] claim that considering that up to 20% in enthalpy difference is acceptable when cycling, but this will depend on the design and the expected thermal performance of the application itself.

When looking at the phase change temperature, no significant changes are suffered because of the cycling process in any PCM.

The IR spectrograms (transmittance vs. wave number) show that some of the characteristic peaks are shifted. This is the case of RT22 HC, Pure Temp23, all the macro-encapsulated and micro-encapsulated. However, no new peaks are observed when comparing the spectrograms of each material before and after 10000 cycles which clearly indicates that none of the PCM presents chemical degradation over cycling.

4.2. Specific heat capacity

The results of the specific heat capacity of the eleven commercial PCM under study are gathered in Table 3. The specific heat capacity of the commercial PCM available for building comfort ranges between 1.26 kJ/kg·ºC and 2.61 kJ/kg·ºC. In general, bulk PCM, all the paraffin (RT21 HC, RT22 HC, RT25HC, and RT 27) and the ester (PureTemp 23) present higher specific heat than the microencapsulated ones (MC DS5040X, MC DS5008X, MC DS5038X). The PCM with the highest specific heat is RT25 HC. The crucibles of the DSC technique do not allow accurate measurements of the specific heat of the macro-encapsulated PCM (MacroPCM28 and Macro PCM24) due to the morphology of these PCM (dimensions of the capsules) and therefore no results for these PCM are available.

The temperature range where the specific heat has been evaluated corresponds to passive building applications, being quite narrow from 10 ºC to 50 ºC. No general pattern of the specific heat with the temperature increase neither with the state of the matter (solid or liquid) can be deduced. These results along with the standard deviation are valuable to quantify the storage capacity of the final product in terms of sensible heat as well as an input property when modelling any application implementing one of these eleven PCM. Depending on the application, more experimental points might be needed to evaluate the complete application temperature range.

Table 3. Specific heat capacity of the PCM under study at three different temperatures

PCM	Cp (kJ/(kg·ºC))		
	10 ºC (solid)	40 ºC (liquid)	50 ºC (liquid)
RT21 HC	1.76 ± n.a.	1.78 ± 0.03	1.81 ± 0.03
RT 22 HC	1.73 ± 0.05	1.89 ± 0.06	1.93 ± 0.06
RT 25 HC	2.61 ± 0.43	2.06 ± 0.03	2.16 ± 0.00
RT 27	2.10 ± 0.03	1.96 ± 0.03	1.98 ± 0.01
PureTemp 23	1.56 ± 0.01	2.06 ± 0.03	2.06 ± 0.05
MacroPCM28	n.a.	n.a.	n.a.
MacroPCM24	n.a.	n.a.	n.a.
MC DS5040X	1.48 ± 0.02	1.30 ± 0.09	1.45 ± 0.03
MC DS5008X	1.58 ± 0.16	1.26 ± 0.00	1.33 ± 0.01
MC DS5038X	1.65 ± 0.08	1.26 ± 0.09	1.30 ± 0.11
MPCM24D	1.56 ± 0.04	1.31 ± 0.03	1.40 ± 0.02

n.a. – not available

4.3. Thermal conductivity

The thermal conductivity of the PCM was studied at 10 ºC, 35 ºC and 50 ºC (Figure 4). Bulk PCM, all the paraffin and the ester show higher thermal conductivity than microencapsulated and macro-encapsulated PCM. The commercial ester, Pure Temp 23, presents the highest thermal conductivity among all the tested PCM, being 0.232 W/m·K at 10 ºC and 0.156 W/m·K at 50 ºC.

On one hand, RT27, RT21 HC and RT22 HC show a slight increase of thermal conductivity with temperature while the tendency of Pure Temp23 and RT25 HC is the opposite. At 50 ºC, the thermal conductivity of the five bulk PCM ranges between 0.136 W/m·K and 0.156 W/m·K. On the other hand, thermal conductivity of macro-encapsulated and micro-encapsulated PCM is negatively affected by the polymeric material of the capsules which leads to values between 0.058 W/m·K and 0.108 W/m·K. The slightly decrease in thermal conductivity at 50 ºC compared to 10 ºC values of the encapsulated PCM is not observed for MC DS5038X.

It should be taken into account that the dimensions of the capsules of the macro-encapsulated PCM (around 2 mm to 4 mm) favours the air content in between capsules giving a value which does not corresponds to the thermal conductivity of the PCM itself but an effective thermal conductivity depending on the air content.

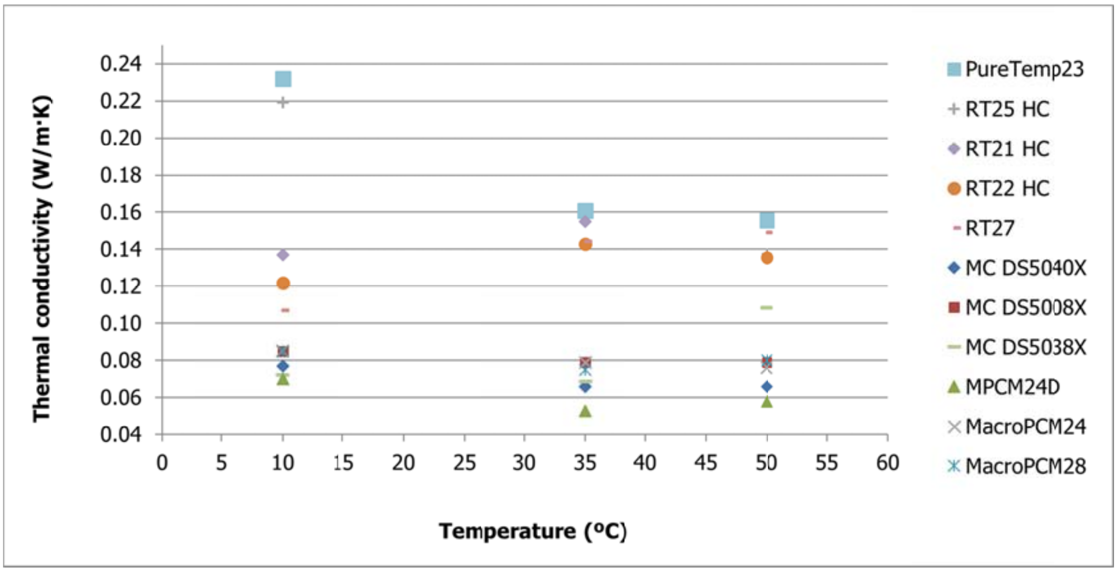

Figure 4. Thermal conductivity of the eleven PCM under study at 10 ºC, 35 ºC and 50 ºC

4.4. General discussion

PureTemp 23 shows the best thermal performance in the sense of higher phase change enthalpy and thermal conductivity compared to the other commercial PCM available in this temperature range. Also, the specific heat of PureTemp 23 is in the upper range if all the studied PCM are compared. Nonetheless, the requirement of the application to incorporate the PCM in a building material matrix, like concrete, makes the micro-encapsulated PCM the most appropriate candidates for this application [15]. Among the commercially available microencapsulated PCM for this temperature range, the four candidates present similar thermal conductivity, specific heat and phase change enthalpy. However, regarding thermal cycling stability, it is clearly observed that the microencapsulated PCM DS5038X shows the best results with a variation of less than 4% in its phase change enthalpy and temperature after 10000 melting/freezing cycles. When PCM are implemented in building comfort applications, usually one cycle is expected per day, therefore the life span of the PCM DS5038X is ensured for, at least, 30 years. Finally, MC DS5038X is selected, among the other micro-encapsulated materials, since it was demonstrated thermally stable after 10000 cycles. Also, the phase change temperature in MC DS5038X was higher than MC DS5040X, which is more suitable for this application.

5. Conclusions

Several PCM available in the market were studied in this paper to be used in a specific building application. In the framework of a Spanish project there was a need of PCM selection to be incorporated in a new technology of radiant wall. This application had specific requirements that the PCM selected should fulfil, to use a PCM commercially available, the possibility of PCM inclusion in a building concrete matrix, the phase change temperature inside the comfort building range, and long-term thermal stability. Therefore, the materials under study were characterized by their thermophysical properties, and their thermal cycling stability.

The long-term stability involves that the thermophysical properties should remain constant during the cycling tests, and also that there is no chemical degradation in the material itself to allow suitable and realistic implementation at the final application.

The specific heat capacity, thermal conductivity, and phase change temperature (melting and solidification) were analysed in all the PCM at different cycling points up to 10000 melting/solidification thermal cycles, which simulates 30 years of working service.

Results showed that the materials in bulk have higher thermal conductivity and specific heat values than micro-encapsulated and macro-encapsulated materials. In particular, PureTemp 23 has the higher values in the thermal properties measured and also, more stable conditions along the cycles compared to the other two bulk materials.

In the encapsulated materials, initially the results of specific heat capacity and thermal conductivity do not show significant differences between them. However, a decrease on these properties after 10000 cycles up to 31% was observed in most of the encapsulated materials, except MC DS5038X and Macro PCM28.

Although the good properties of the bulk materials, authors considered that the methods of incorporation (direct incorporation, impregnation) into a concrete matrix of these materials would be complex. For this reason, the micro-encapsulation method was selected in this case. MC DS5038X is a good option due to its long term stability properties, which is one of the most important requirements for the application, since it should last the whole building lifetime.

Acknowledgements

Financiado por el proyecto RTC-2015-3583-5 (INPHASE) del Ministerio de Economía y Competitividad, dentro del Programa Estatal de Investigación, Desarrollo e Innovación Orientada a los Retos de la Sociedad, en el marco del Plan Estatal de Investigación Científica y Técnica y de Innovación 2013-2016, y ha sido cofinanciado con FONDOS FEDER, con el objetivo de promover el desarrollo tecnológico, la innovación y una investigación de calidad. The work is partially funded by the Spanish government (ENE2015-64117-C5-1-R (MINECO/FEDER) and ENE2015-64117-C5-2-R (MINECO/FEDER)). The authors would like to thank the Catalan Government for the quality accreditation given to their research group (2017 SGR 1537 and 2017 SGR 188). GREiA is certified agent TECNIO in the category of technology developers from the Government of Catalonia. Camila Barreneche would like to thank ACCIO for her Grant TecnioSpring Plus TECSPR17-1-0071. Aran Solé would like to thank Ministerio de Economia y Competitividad de España for Grant Juan de la Cierva FJCI-2015-25741.

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