Experimental analysis of the thermal performance of plastering mortars comprising hybrid phase change materials for increased energy efficiency in buildings

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Abstract: Several research studies have forwarded the possibility of incorporating microencapsulated phase change materials (PCM) in plastering mortars for building façades, in pursuit of increased energy efficiency associated to the heat storage capacity of PCM. Nonetheless, most of these studies are centred in the use of a single type of PCM, which is bound to be more adequate for a given season of the year (e.g. winter or summer) than for other seasons, particularly in the case of South-European countries. The study proposed in this work regards the evaluation of the possibility of using more than one kind of PCM (with distinct melting ranges) in plastering mortars, in order to achieve adequately advantageous performance in both heating and cooling seasons. In order to characterize the PCM there are two parameters (enthalpy and phase change temperature) that should be adequately measured. The main purpose of this study was to show feasibility and to evaluate the behaviour of mortars that contain hybrid PCM's with Differential Scanning Calorimeter testing (DSC). The knowledge obtained from the experimental testing shall establish bases for a framework of numerical simulation of real scale applications, which can be used to ascertain the feasibility of the hybrid PCM concept for increased energy efficiency in buildings.

1. Introduction

The energy consumption of any building is critically dependent on indoor air temperature that the building must maintain. Heat is gained/lost from the building through its surfaces or by ventilation, with strong dependence on the temperature difference in regard to the outside environment [1]. In order to move towards increased sustainability in buildings it is important to investigate methods that can reduce the cooling/heating energy demand that is necessary to assure proper thermal comfort levels. One such method that can help in the reduction of peak cooling loads and stabilization of indoor temperature is the use of Phase Change Materials (PCM) in a building construction, taking advantage of the latent heat storage that is intrinsic to these materials. One of the simplest ways of using PCM in buildings consists in incorporating them into porous materials such as plaster mortar and concrete. For such purpose, the PCM is delivered to the mortar/concrete mix within micro-scale polymer capsules in the form of powder (microencapsulation). A detailed review on this kind of use for PCM's can be found in [2].

A broad knowledge of the thermo-physical properties of PCM is necessary for the correct design/optimization approaches for their use in building constructions. In this way, one of the most important properties to determine is the temperature-enthalpy relationship.

Generally, the standard measurement method for the analysis of many thermo-physical properties is the Differential Scanning Calorimeter (DSC) [3]. From the literature survey [3-5] there are different possibilities of operation methods to analyse PCM through DSC testing. The two most common methods are: the dynamic method which corresponds to a constant heating rate and the step method which consists in applying variable heating rates to the sample [4]. Barreneche *et al.* [5] state that dynamic method and step method are suitable for the organic materials such as paraffin. The drawbacks of the step method are related to its programming complexity, the fact that it is time consuming and the difficulties in data treatment/interpretation [6]. In this work, we present measurement data using the dynamic method.

The main objective of this paper is the evaluation of the enthalpy performance of the PCM when incorporated into plastering mortar. The study is related to microencapsulated PCM's alone and the study of the mortars that contain such PCM's (both single PCM and hybrid PCM).

2. Experimental setup

2.1. Materials and sample preparation

The studied materials for this paper were microencapsulated PCM's and mortars containing microencapsulated PCM's. Regarding the microencapsulated PCM's, three types of organic PCM paraffin were considered (Devan Mikrathermic D series): MC18 (melting temperature of 18˚C), MC24 (melting temperature of 24˚C) and MC28 (melting temperature of 28˚C), with mean particle size of approximately 18 micron (μm). From those three types of PCM's, four samples prepared for DSC testing of PCM samples: three samples that solely contain one type of PCM (MC18, MC24 and MC28 respectively) and one sample that contained a 50%-50% mass proportion mix of MC18 and MC28, here termed as MC18_28. The tested samples had average weight of ~4mg.

The experiments also involved the study of two mortars containing PCM's. The first mortar, here termed as SPCMM24, is a single PCM mortar and that contains MC24. The second mortar, here termed as HPCMM18_28, involves the combined use of MC18 and MC28 in the same total quantity as the first mortar contained only MC24. Detailed information on the mix proportions and adopted materials are shown in Table I. The formulation of the mortars was fixed in respect to criteria of the European Norm EN 998-1 [7]. Plastic cylindrical moulds with height of 1 cm, diagonal of 10 cm and thickness of 0.3 cm considered for the mortar samples. The few milligrams of mortars samples necessary for DSC testing were gathered by drilling of the hardened mortar.

Table I Mix proportions of formulations SPCMM24 and HPCMM1828

Materials	Formulations (percentage of the total weight of mortar)	
	SPCMM24	HPCMM18_28
Cement type I class 42.5R	31.32	31.32
Sand	30.59	30.59
Water	18.79	18.79
Super Plasticizer (water reducer)	0.94	0.94
MC18	-	9.175
MC24	18.34	-
MC28	-	9.175

2.2. Methodology and rationale

The DSC calorimeter submits the sample to controlled temperatures and records the corresponding heat fluxes, thus providing information about temperatures and enthalpies associated to phase changes. From the heat flux, the specific heat as a function of temperature can be obtained, and the enthalpy is determined by integration procedures [3]. In this study, the methodology of enthalpy calculation follows the strategy adopted in ref. [5].

The melting and freezing behaviours of the PCM were analysed by a DSC model Mettler Toledo DSC 822e. The DSC was calibrated with Indium standard [8], using a single point-calibration method. The DSC has an accuracy of ±0.2˚C for temperature measurements. All the samples were tested within aluminium crucibles with volume of 40 μL under nitrogen (N2) atmosphere flow of 80mL.min-1. The samples were weighted by an analytical balance with accuracy of ±0.01mg.

A constant heating/cooling rate of 1˚C.min-1 was applied for all the samples, as recommended by [5, 9]. The applied program steps for the test procedure of samples were the following: (i) initial isothermal period at -5˚C for 5 minutes; (ii) dynamic heating up to 50˚C at a rate of 1˚C.min-1; (iii) stabilization at 50ºC for 5 minutes; (iv) dynamic cooling to –5ºC at a rate of -1˚C.min-1. Samples of each material were analysed and each sample was cycled once (heating and cooling). The main purpose of this experimental process was to determine whether the enthalpy data calculated from DSC test can be used to predict the energy performance of PCM materials when incorporated into the plastering mortars. Another question was whether the entire volume of hybrid PCM sample will work with similar effectiveness in the composite experiment when compared with single PCM composite as it was made during DSC test with a few milligrams of samples. It is important to note that the DSC provides the heat flow (mW) at each temperature for a specific sample mass, and that these values are converted to energy (kJ/kg) properly in order to make comparison between the archived enthalpies.

3. Results and discussion

Table II shows the DSC results for different samples of tested PCM. The melting temperature (peak point) of PCM samples occurred almost according to the supplier's information for MC18 and MC24. Sample of MC28 presented a peak of freezing temperature shifted by -3.74˚C in regard to 28˚C. Also, in the sample of MC18_28 the phase change melting temperatures were coherent with those of the components of the mix (MC18 and MC28). This shows that, the mixing of two PCM's has a similar behaviour to the independent PCM's and therefore, it can be said that they have not interacted with each other.

In the sample of MC18_28, enthalpy values of solid-liquid phase transition of 134.17 kJ/kg and 100.35 kJ/kg obtained. It can thus be observed that, the enthalpy value of MC18_28 at ~18ºC is almost half of its enthalpy of

independent MC18 sample at the same transition temperature, which is coherent with the fact that MC18_28 has half the quantity of 18ºC PCM. A similar tendency is observed for the 28ºC transition (however with slightly higher deviation).
The difference between the melting peak temperature and the freezing peak temperature was of 3.29˚C, 4.5 ˚C and 2.42˚C for MC18, MC24 and MC28 respectively. This is coherent with reports of hysteretic behaviour of the phase change by Castellon et al [3]. For the sample of MC18_28, the difference between the melting peak temperatures and the freezing peak temperatures was of 2.7˚C and 1.86˚C for the phase changes corresponding to MC18 and MC28, respectively.

Table II - Pure PCM DSC enthalpy, onset and end temperatures for different PCM samples

Designation	Weight (mg)	Melting				Freezing			
		Onset temp. (C)	End Temp. (C)	Enthalpy (kJ/kg)	Peak temp. (C)	Onset temp. (C)	End temp. (C)	Enthalpy (kJ/kg)	Peak temp. (C)
MC18	3.5	15.67	18.97	270.63	17.75	15.4	12.44	275.08	14.32
MC24	4.4	18.21	23.86	162.4	22.74	22.36	15.67	136.53	17.94
MC28	3.8	24.19	28.79	253.89	24.26	25.96	13.42	256.89	27.08
MC18_28	4.3	15.8 , 24.72	18.29 , 27.8	134.17, 100.5	17.61, 27.08	15.31, 25.96	13.51, 22.62	139.59, 82.23	14.7, 24.78

The results for mortars incorporating the PCM microcapsules are shown in Table III. From the observation this table, it can be observed that the mortar sample SPCMM24 (powder) has values of onset temperature and end temperature that are consistent with those of the MC24 sample (see Table II) with small differences (less than 1˚C).

The mortar sample with formulation of HPCMM18_28, also has a similar behaviour to the corresponding MC18 and MC28 for the onset temperatures and end temperatures of the solid-liquid phase transition. Moreover, the peak melting temperature of SPCMM24 and HPCMM18_28 occurred almost in the same positions (with less than 1˚C differences) in comparison to the independent PCM samples.

It can therefore be inferred that the several constituents of the mortar mixes seem to have none or negligible effect on the transitions of the microencapsulated PCM's (both on the temperatures and on the enthalpies). The observed differences can probably be justified by potential inaccuracies caused by the algorithm used to estimate onset and end temperatures, or by possible inhomogeneity within the samples.

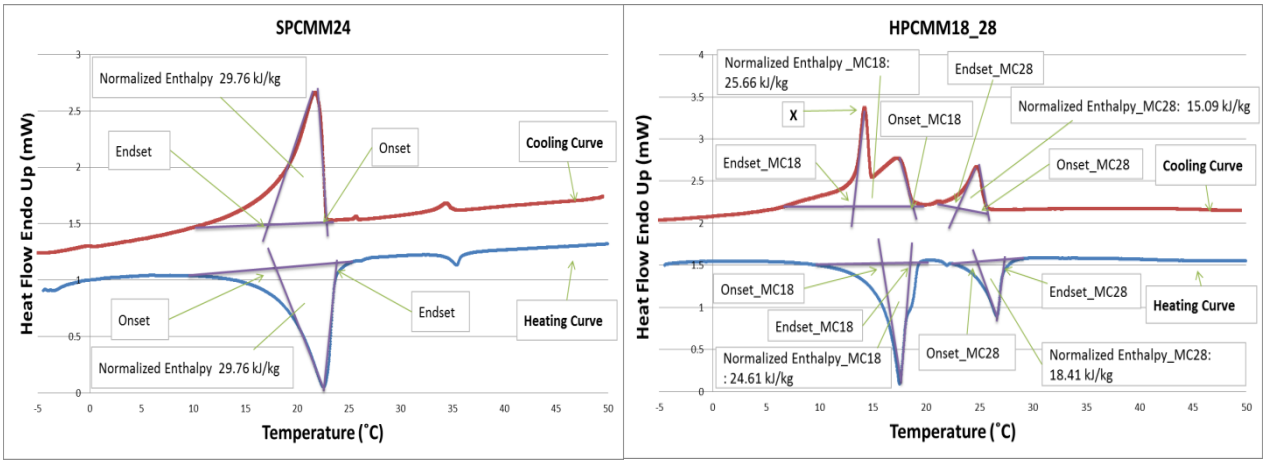
Table III - PCM incorporated into mortar DSC enthalpy, onset and end temperatures for different plaster mortars

Designation	Weight (mg)	Melting				Freezing			
		Onset temp. (C)	End Temp. (C)	Enthalpy (kJ/kg)	Peak T (C)	Onset temp.(s) (C)	End temp. (C)	Enthalpy (kJ/kg)	Peak T (C)
SPCMM24	10.0	17.77	23.61	29.76	22.56	22.79	16.79	25.08	21.77
HPCMM18_28	10.3	15.36, 24.32	18.5, 27.22	24.61, 18.41	17.49, 26.63	25.52, 18.73	22.55 , 13.16	25.66, 15.09	14.29, 24.8

For the sample of SPCMM24, a latent heat value of 29.76 kJ/kg was obtained for the melting phase transition of the PCM which is, approximately, 18.35% of the corresponding values obtained for the independent MC24. This relation of 18.35% is consistent with the mass ratio of MC24 in the mortar SPCMM24, which is of 18.34% according to Table I, thus pointing to the possibility of predicting enthalpy of mortars that incorporate PCM. This feasibility for enthalpy prediction has also been observed by Vaz sa et al. [10]. The enthalpy values for the HPCMM18_28, which contains 18.34% of PCM in the mix, correspond to 18.35% of enthalpy values obtained for MC18_28 (Table II), thus corroborating the previously mentioned proportionality.

Fig. 1 represents the DSC curves of the SPCMM24 and HPCMM18_28 for heating and cooling processes. In specific regard to HPCMM18_28, two main peak points are identifiable in the cooling process curve (Fig. 1b) of the DSC. The first peak point corresponds to phase change transition of MC18 (labelled as X in Fig. 1bThe second peak

corresponds to the MC28 phase change transition (peak_MC28 labelled in Fig. 1). The smaller peak points were omitted for the calculation of the enthalpy values for both heating and cooling processes for all samples.



(a) (b)

Figure 1 (a) DSC result of sample SPCMM24; (b) DSC results of HPCMM18_28 sample for heating/cooling processes.

4. Conclusion

Based on the research made with the different DSC tests the following main conclusions can be drawn: (1) the mixing of two PCM's has a similar behaviour to the superposition of effects of independent PCM's and therefore, it can be inferred that they have not interacted with each other; (2) DSC testing of different samples shows that both plain microencapsulated paraffin samples (PCM) and plaster mortars containing microencapsulated PCM (SPCMM24 and HPCMM18_28) exhibit hysteretic behaviour of the phase change temperature in regard to heating/cooling cycles; (3) enthalpy is linearly proportional to the mass fraction of PCM in the mortar sample as compared to the behaviour of plain PCM samples.

5. References

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