Early age temperature control in mass concrete through incorporation of dispersed Phase Change Materials (PCMs)

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Abstract. There is a frequent need to take measures for temperature control in massive concrete structures, as to avoid thermal cracking risk at early ages (induced by temperature gradients inherent to hydration heat release). This research work has explored a procedure for temperature control through the incorporation of phase change materials (PCMs) in laboratory environment (mortar testing). Indeed, PCM's have the potential to store and release heat energy during phase change from solid to liquid, or vice versa. By choosing a PCM with a melting range between casting temperature and the expected peak temperature, it is possible to attenuate the temperature rise rate in concrete through heat storage (the melting process is endothermic). This paper presents and discusses an experimental work focused on the thermo-physical properties and thermal performance analysis of mortar with direct incorporation of pristine PCM (with a melting temperature of 34ºC and latent heat capacity of 240 J/g) in three volume fractions of 0, 10 and 20% in mixture compositions (volumetric percentage replacement with regard to sand particles), cast into partially insulated cubes with 320mm^3 size. The thermal performance tests revealed the impact of the PCM in the thermal behavior of the cast element, by reducing the maximum peak temperatures in comparison with the reference case (without PCM). Mechanical tests were also performed and revealed that, as expected, their compressive and flexural strength are reduced. Nonetheless, the observed reduction might still be compatible with structural applications in specific contexts, even for the case of high PCM content incorporation (20%).

Keywords: Phase change materials, mortar, heat of hydration, thermal and mechanical behavior.

1 Introduction

In concrete, the stress developed at early ages can lead to cracking due to the restrained volume changes, originally associated to the autogenous shrinkage and thermal expansion/contraction [1]. Early age cracking of concrete [2] may reduce performance levels for serviceability and durability [3]. Several measures can be taken to help to mitigate early age cracking, particularly at the definition of mix constituents and mix proportioning: using specific cement or admixtures [4], and phase change materials (PCMs) as additives [5-9].

The principle of operation of PCMs [10] through heat storage in the scope of concrete applications can be explained with the following example: in a concrete mixture containing PCM, when the increasing core temperature of concrete (induced by cement hydration heat) reaches the melting point of the PCM, it endures the corresponding phase change and absorbs heat (endothermic process). Therefore, while the heat storage capacity of PCM still exists, concrete temperature tends to remain in the vicinity of the PCM's melting point [5], therefore limiting the temperature rise, and inherently leading to smaller thermal cracking risk.

There are several methods of incorporation of PCM into mortar proposed in the literature, such as: suspension of phase change material (SPCM) in combination with water [6], self-compacting concrete cubes containing PCMs (dispersed form of incorporation) [11], concrete containing porous aggregates incorporated with PCMs (in the form of vacuum impregnation) [12], mortar and concrete mixes blended with microencapsulated PCM (by direct incorporation) [13], footing member and bridge pier containing PCM (added into the mixture) [14], and cement based materials incorporating microencapsulated PCM [15]. Regardless of the incorporation method, the selection of the PCM to incorporate is mostly related to two key characteristics [16]: the specific enthalpy and phase change temperature range.

The present work presents a study of thermal and mechanical behaviour of a mortar in which the volume of sand is partially replaced by PCM (directly incorporated into the mixture). Apart from the study of the reference case (no PCM added), two volumetric ratios of replacement of sand by PCM are considered: 10% and 20%.

2 Experimental program

2.1 Materials

The PCM selection was based on the typical temperature development for concrete structures. The initial selection of PCM melting temperature range was limited to 30 to 40ºC, which is a range that is usually attained during curing of most massive concrete structures, when the surrounding boundary temperature is ~20ºC [17]. The average hydration heat of cement is 300kJ/kg, therefore, the selected PCM needs to have high latent heat capacity [3]. For the experimental testing carried out, commercial PCM “RT35HC” (from Rubitherm paraffin RT series) [18] was selected for this study that has a latent heat capacity of 240kJ/kg and melting temperature ranges between 34 and 36ºC. Portland cement type I class 42.5R was used as a binder from the SECIL company, Portugal. River sand was used as aggregate. The fineness modulus of the sand was 3.2.

2.2 Development of mortars

The mix design of the investigated mortars together with their adopted designations are listed in Table 1. This study includes a reference mortar (REFM) and two mortar systems named as PCMM10 and PCMM20, which incorporate respectively 10% and 20% Vol. of PCM dispersed in the mixture (ratios defined in regard to the volume of sand). Fig. 1, shows a illustrative scheme on the comparison between the cross section structure of the PCM mortars dispersed PCM and reference mortar. This figure presents the effect of the PCM incorporation on the specific configuration of the mortar systems, i.e. possibly of homogeneity of dispersed form of the PCM particles.

Table 1. Mix design of reference mortar and mortars with grated PCM.

Components (kg/m3)	**Reference mortar**	**PCM mortar**	
	REFM	PCMM10	PCMM20
Cement Type I - 42.5R	500	500	500
Water	245	265	285
Sand	1579	1435.5	1315.8
PCM	0	41.7	76.5

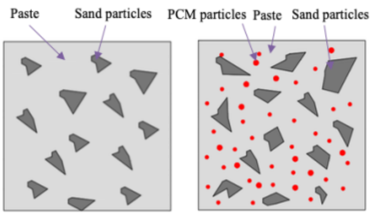

Fig. 1. Illustrative scheme of the internal structure of reference (left), dispersed PCM (right) mortars.

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3 Strategies and test procedures

3.1 Characterization of the mortars

3.1.1 Flow table test

The flow table test was used to maintain the mortar workable according to the flow value based on diameter of standard frustum. The workability tests were conducted through flow table test based on the European standard EN 1015-3 [19]. The mortar was considered workable only when the value of 140±5mm was monitored/recorded for proposed mortar.

3.1.2 Density

For the density measurement, three specimens were considered for each type of mortar. The test was made following recommendations of EN1015:10 [20]. Firstly, the specimens were casted into prism moulds (with dimensions of 160mm×40mm×40mm). Then, the specimens were kept sealed with a plastic wrap at room temperature (20 ± 1ºC) for 24hr. Then, the specimens were submerged at 20 ± 1ºC for 7 days. Finally, the specimens were dried at 70ºC until recording constant weight for each specimen. The density of the specimens was calculated directly. The average density of different formulations is reported.

3.1.3 Flexural strength

Flexural strength testing was performed at age of 28 days following European standard EN1015-11 [21]. Three prism specimens with dimensions of 40mm×40mm×160mm were prepared and sealed by plastic wrap at room temperature (20ºC) until testing. A three-point loading method was deployed using apparatus model SHIMADZU-AG-IC with the capacity of 100kN. Average of the measured valued was used on the analysis. For the flexural strength measurement, three specimens were considered for each type of mortar and average values are reported.

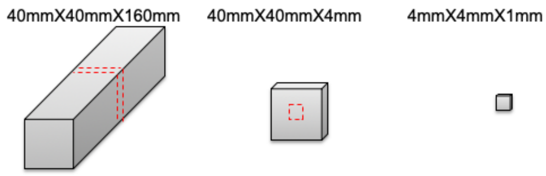
3.1.4 Compressive strength

The compressive strength tests were performed to evaluate the effect of the PCM on the mechanical strength of the mortars taken from ruptured specimens in flexural strength testing. For each mortar mixture, three specimens were tested at the age of 28 days. The testing recommendations of EN1015-11 [21] were followed. All the specimens were kept sealed with a plastic wrap in laboratory environment until testing (28 days). Then, the compressive strength tests were carried out using apparatus model SHIMADZU-AG-IC with 100 kN capacity. The average of the measured values was used for the analysis.

3.2 Field thermal analysis of the mortars

3.2.1 Thermal energy storage

A total number of 3 specimens were prepared for differential scanning calorimetry (DSC) testing. This includes a representative specimen of each mortar containing PCM (PCMM10 and PCMM20), one specimen for reference mortar (REFM) and one specimen was also taken from bulk PCM (RT35HC). The specimen preparation for DSC testing is shown in fig. 2. All test samples were obtained from mortar cast prism molds with dimensions of 40mm×40mm×160mm, which were cured for 28days before preparation for DSC testing (fig.2a). Central part of the prism was cut out, resulting in a slice with size of 40mm×40mm×4mm (fig. 2b). A small sub sample with dimensions about 4mm×4mm×1mm was extracted and submitted to DSC testing (fig. 2c). The detained classification of the type of specimen as well as weight of the samples for DSC testing is presented in Table 2.



(a) **(b)** **(c)**

Fig. 2. Schematic of specimen preparation for DSC test: (a) fabricated sample in the shape of prism cured for 28days; (b) the thin layer of 4 mm width were cut from the middle of the prism in the form of slice; (c) finalized sample for DSC testing, extracted from the center of the slice.

Table 2. Identification of the samples taken and weight for DSC testing.

Samples	Names	Weight of specimen for DSC testing (mg)
Mortar with 10% PCM	PCMM10	16.202
Mortar with 20% PCM	PCMM20	15.860
Conventional mortar	REFM	16.401
Pristine PCM	RT35HC	7.564

In performed DSC analyses (Perkin Elmer model AD-4), the system measures the difference in the amount of heat required to increase the temperature of a material sample (sample pan) and an empty sample (reference pan) as a function of temperature. The DSC equipment has an accuracy of ±0.3ºC. The specific heat and the specific enthalpy was determined following the methodology detailed in [16]. The phase change and temperature were obtained from the DSC heat fluxes signal response by integration. The volumetric heat capacity of the material is calculated by multiplying the density and specific heat capacity according to Eq.(1):

$$C_{pv}=C_p \times Rho \quad (1)$$

Such value ascribes the ability of the material in terms of energy storage in a certain volume while undergoing a given temperature change. The theoretical latent heat capacity of these PCM mortars was also determined as Eq.(2):

$$H(T)=H_{pcm} \times (W_{pcm}\%/100) \quad (2)$$

Where $H(T)$ is the calculated latent heat of the mortar (J/g), H_{pcm} is the latent heat of the PCM, W_{pcm} is the weight percentage of the PCM in the matrix.

3.2.2 Design of the specimen prototypes

In order to assess the effect of different quantities of dispersed PCM into the mortar, for measuring early age temperature, three cubic specimens were casted. The cubes for the testing REFM, PCMM10 and PCMM20 mixtures, have dimensions of 200×200×200 mm3, as presented in fig. 3. Lateral faces, base and top of the element were insulated with 40mm thick extruded polystyrene (XPS) and 20mm thick plywood formworks. The casting process and the experiments themselves took place in controlled conditions, inside a climatic chamber to guarantee a constant temperature of 20ºC and relative humidity of 50%. Data acquisition started at the end of casting operations. Regarding the experimental monitoring equipment, a temperature sensor type K with a precision of ±1.1ºC was placed at the geometrical centre of the mortar. A total of three experiments were conducted by submitting the specimens to the climatic chamber room with constant temperature of 20ºC with during 4 days. The temperature sensor was connected to the data logger (AGILENT 34970A) with a acquisition period of 15min. The physical arrangement of this setup can be observed in fig. 4.

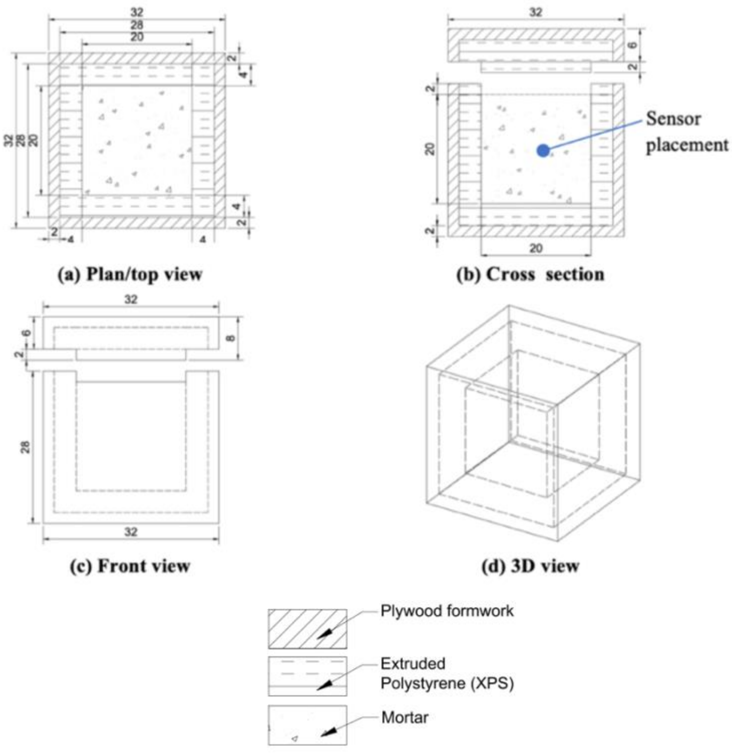
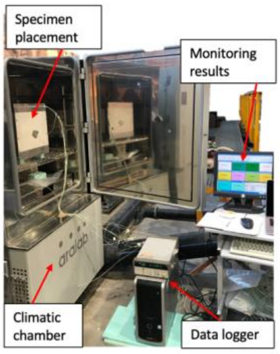
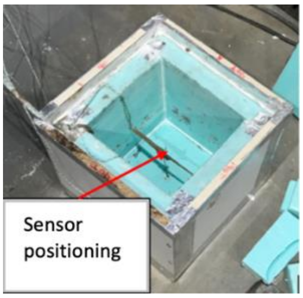


Fig. 3. Schematic representation of the experimental set up for reference mortar (REFM) and distributed form of grated pristine PCM mortars (PCMM10 and PCMM20): mixtures and sensor location. Units [cm].



(a)



(b)

Fig. 4. (a) Photo of the prototype within climatic chamber before starting the test; (b) position of the temperature sensor for REFM, PCMM10 and PCMM20 cases.

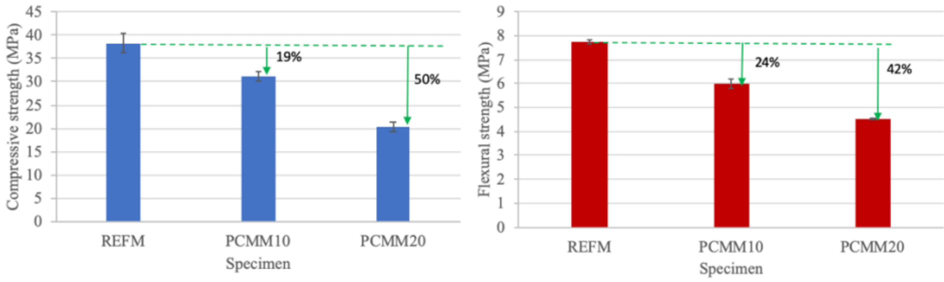
4 Results and discussion

4.1 Physical and mechanical performance

The workability test proved that the incorporation of the PCM leads to an increase in water content by 8% and 16% for the PCMM10 and PCMM20, respectively. This is maybe due to the particle size distribution of the PCMs in the mixture compared to the sand particles.

The density values for the REFM, PCMM10 and PCMM20 were 2188kg/m3, 1904 kg/m3, 1870 kg/m3, respectively. The density of the mortars with PCM are 13% and 17% lower than that of reference mortar (REFM) for the MPCMM10 and MPCMM20, respectively. This is attributed to the density of the PCM, which is lower than the sand particles.

Fig. 5a shows the compressive strength values for different specimens at 28 days of age. PCM mortars have lower compressive strength than the reference mortar, REFM. The strength of the PCMM10 and PCMM20 was reduced about 19% and 50%, respectively, when compared with REFM. A similar trend was observed for the flexural strength of the mortars with PCM (shown in fig. 5b). The flexural strength reduces for the cases of MPCMM10 and MPCMM20 by 24% and 42%, respectively, when compared with REFM.



(a) **(b)**

Fig. 5. Strength performance of different mortars and average with standard deviation of the measured values: (a) compressive strength; (b) flexural strength.

4.2 Thermal performance

4.2.1 DSC analysis

The results of DSC testing on the pristine PCM (RT35HC), REFM, PCMM10 and PCMM20 specimens are shown in Fig. 6. The absence of phase change can be confirmed for the case of REFM. The melting peak temperatures were obtained by 38.2ºC, 36.8ºC and 37.8ºC for pristine PCM (RT35HC), REFM, PCMM10 and PCMM20, respectively. The peak temperatures of PCM and PCMM20 are in a similar fashion. While, the tendency is though less clear because the PCMM10 involved smaller quantity of the PCM. The results suggested that the PCM peak temperature shifts in the direction of the imposed flux, thus, showing higher peaks for mortars containing more PCM incorporation. The DSC thermograms have been zoomed to clearly visualize the peak values in the case of mortars with PCM.

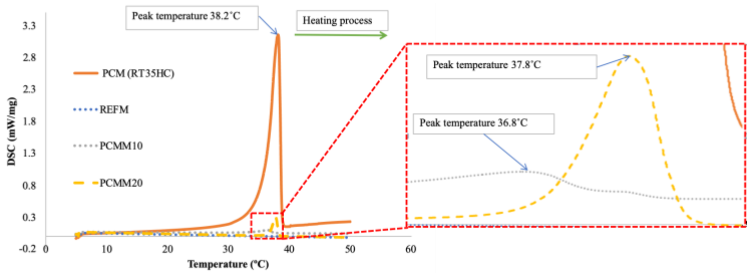


Fig. 6. DSC thermograms for mortars with and without PCM plus pristine PCM at the rate of 2ºC/min.

Fig. 7 shows the volumetric heat capacity calculation for the studied mortars. Volumetric specific heat capacity indicates the ability of the material in terms of energy storage in a certain volume while experiencing a given temperature change.

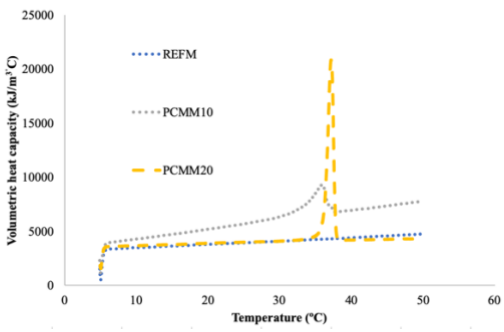
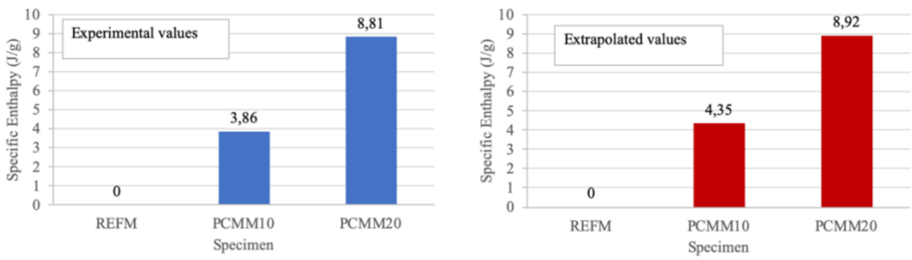


Fig. 7. Results for volumetric specific heat capacities of mortars.

The high thermal capacity indicates a material is characterized by high thermal mass and high thermal conductivity. In general, it can be stated that, the PCM mortars have higher thermal inertia than that of the reference mortar, thus, higher potential for attenuating the effect of hydration heat of cement based material.

Fig.8a shows the values of the specific enthalpy of mortars. It can be observed that the specific enthalpy values increased with incorporation of more PCM quantity. It is also interesting to calculate the specific enthalpy values through extrapolation in order to predict the energy performance of PCM mortar. Fig.8b shows the estimated specific enthalpy for mortars (based on the characteristics of the PCM and its volume within each mortar) in which the values are in the same magnitude as those already obtained experimentally (less than 10% difference). These results confirm the expectable proportionality between specific enthalpy of PCM and mortar containing PCM, based on the mass fraction of the PCM.



(a) **(b)**

Fig. 8. (a) measured specific enthalpy values of the mortars with PCM; (b) extrapolated specific enthalpy values.

4.2.2 Transient behavior of cube specimen

Temperature values measured in the geometrical center of the specimens are presented in Fig. 9. It can be noted that the temperature rises after casting of the mortar continues up to about 1 day and then, reduces until the end of testing. It is interesting to note that the peak temperature recorded for the cases with PCM is lower than that of REFM. as expected, the specimen with more PCM shows a lower peak temperature. It is also noticeable that, the phase change temperature of the PCM ranges between 33-36ºC, flatting the temperature evolution in the cooling phase. The peak temperatures reduction was recorded by 5ºC and 9ºC for the PCMM10 and PCMM20, respectively, when compared with REFM.

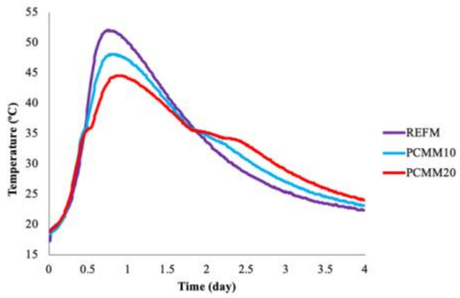


Fig. 9. Interior temperature of the cubic specimens: comparison between REFM, PCMM10 and PCMM20.

5 Conclusions

In this study, mortars incorporated with PCM have been developed and experimentally characterized. Based on the experimental campaign results, the following main conclusions can be drawn:

- The addition of the dispersed form of PCM into the mortar has led to 13% and 17% reduction of the density, when compared with the reference mortar.
- Addition of PCM leads to reduction of mechanical properties. However, it may still be compatible in structural applications.
- The DSC test results showed that the specific enthalpy is proportional to the mass fraction of PCM in the mortar sample as compared to the behavior of the pristine PCM sample. The numerical estimation of enthalpy values for PCM mortars matched the measured values quite closely, indicating the applicability of the estimation procedure.
- Thermal performance of the transient test has demonstrated the reduction of temperature rise, enhancing thermal capacity of the mortars with PCM.

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