

# The direct incorporation of micro-encapsulated Phase Change Materials in the concrete mixing process – A feasibility study

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## Abstract

The present study refers to a set of tests using different amounts of micro-encapsulated PCM directly mixed into self-compacting concrete. This SCC is investigated regarding its fresh and hardened properties. It will be shown that increasing PCM amounts lead to lower thermal conductivity and increased heat capacity, which both significantly improve the thermal performance of concrete structures. On the other hand, a significant loss in strength and a micro-structural analysis both indicate that a large part of the capsules cannot withstand the mixing process, which is a result of insufficient mechanical and chemical resilience of the encapsulating material.

## Keywords:

PCM, self-compacting concrete, latent heat capacity, hydration heat

## 1 INTRODUCTION

Phase Change Materials have the ability to absorb and to release thermal energy at a specific temperature when their state changes. The heat capacity and high density of concrete combined with the use of the latent heat storage of PCM can provide new energy saving concepts, for example in combination with solar energy. In fresh concrete mixes PCM could be added to prevent high hydration temperature peaks, which can result in higher compressive strength and better durability.

The use of PCM in building materials and components has been researched for multiple years and different materials and components have been regarded to incorporate them (e.g. [1] and [2]).

Because the direct use of a micro-encapsulated mixture of paraffins in fresh concrete has not received much attention yet, a mixture of paraffins will be used in this research. It concerns a commercially available product for the building industry with a melting point of 23 °C named Micronal DS 5008 X. According to the product descriptions, it is described as a mixture of paraffin waxes in powder form, encapsulated in polymethyl methacrylate microcapsules. The technique of micro-encapsulation is used here in order to surround the liquid/solid paraffin phase with a hard shell. This way the liquid paraffin (< 23 °C) is transformed into a powder and prevented from entering the surrounding matrix. Due to the encapsulation, it should be theoretically possible to

disperse the paraffin in the fresh concrete mix and to melt it without any interaction between the PCM and the concrete constituents.

This research aims to contribute to existing knowledge on the use of micro-encapsulated PCM in self-compacting concrete (SCC) by conducting experiments regarding its behavior during mixing, hydration and after hardening. The properties of three recipes containing 1% PCM, 3% PCM and 5% PCM (by mass of concrete) were compared with one reference mix. These additions correspond to 2.5 – 12.4% of volume in the mix. The properties of fresh SCC were evaluated using the J-ring test and V-funnel test. During hydration the influence of PCM on the heat development was firstly modeled and secondly monitored in situ. Finally, the hardened concrete was subjected to compressive strength tests and tests to measure its thermal properties. Furthermore, densities were measured and visual observations were reported. Moreover, the micro-structure has been analyzed by using SEM.

## 2 CHOICE OF COMPONENTS – MIX DESIGN

A practical aim of this research is to prevent high hydration temperature peaks during the first day of hydration. Therefore, a deliberately high cement dosage of 450 kg/m<sup>3</sup> was selected. In order to increase the hydration heat development even more a mixture of cement with high fineness (micro-cement) as well as a R type cement with higher clinker content was selected.

In order to account for the varying PCM amounts in the mixes, a non reactive material with comparable particle size distribution is necessary to substitute the respective PCM volume. This way it is assured that all mixes are comparable from the granulometric point of view. Accordingly, all respective variations compared to the (PCM-free) reference mix can be assigned to the influence of the PCM. For this test a dolomitic marble powder is selected, which in former research was successfully applied in SCC production [3]. PCM and marble powder were substituted on volumetric base and are of similar particle size distribution. The 5% mix is an exception since the PCM volume in this case is higher than the available marble volume. Therefore, a slightly higher fines content is present in this mix.

Moreover, the concrete is designed with common aggregates such as fine sand 0-1, 0-4 sand, an intermediate gravel fraction 2-8 and a gravel fraction 4-16. All sand and gravel fractions are river aggregates and therefore show smooth and round shape. Finally, a third generation superplasticizer of the polycarboxylate ether (PCE) type was used to adapt the workability and to adjust the mixes to about the same slump-flow class.

The above described materials have been used to design self-compacting mixes with increasing amounts of PCM. Besides one reference mix without PCM three more mixes were designed containing 1%, 3% and 5% of PCM materials based on their total mass. The applied design method is similar to the SCC mix design of [3]. The method basically focuses on the optimization of the solid granular skeleton. Figure 16 presents the entire particle size distribution of the reference mix including the target grading and all individual materials, based on the design method elaborated in [4]. The micro-encapsulated PCM material is considered as a particle and therefore part of the optimization.

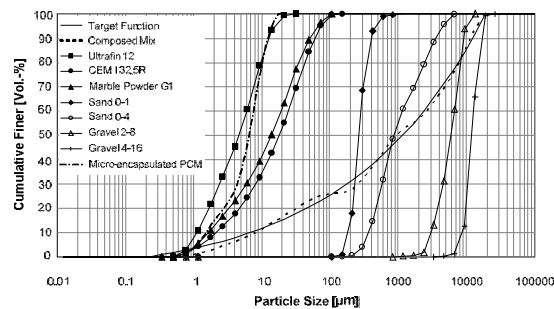


Figure 16: Plot of the total grading of the PCM reference mix, showing target grading compared to the achieved grading as well as all individual constituents. The PCM capsules are not constituent of the reference mix.

### 3 EXPERIMENTAL PROCEDURE

#### 3.1 Differential scanning calorimetry

For this test series a micro-encapsulated PCM was obtained which is a paraffin wax encased in polymethyl methacrylate microcapsules. A small sample of these microcapsules has been used for

the differential scanning calorimetry (DSC) experiment, deploying a Differential Thermal Analyzer (DTA) Perkin Elmer DSC7. For the given temperature range from -20 °C – 50 °C a cyclic heating/cooling/heating scan was conducted at a heating/cooling rate of 10 °C/min with 2 min isothermal holds at both minimum and maximum temperatures. Based on this, during solidification (cooling) an enthalpy  $\delta H_{exoth}$  of 102.8 J/g in a temperature range of 22.1 – 9.3 °C was measured. For the melting (heating) the enthalpy  $\delta H_{endoth}$  amounts to 99.7 J/g in a temperature range of 18.8 – 35.4 °C. The authors are aware of, that higher values of heating or cooling rate lead to broader melting ranges and vice versa [5]. Figure 17 provides the heating and cooling scans. The selected heating/cooling rate is responsible for the observed super-cooling effect shown in Figure 17. This behavior is normally not to be expected when a lower heating/cooling rate of 0.1 – 0.5 °C/min is applied. Nevertheless, the heating/cooling rate does not influence the total melting/freezing enthalpy.

#### 3.2 Concrete mixing

The mixing of the self-compacting PCM mixes took place in four steps. At first all solids except for the PCM are mixed for 30 seconds in order to homogenize the dry components. Thereafter, about 90% of the total water dosage is added and mixing is continued for further two minutes. In this range of time the superplasticizer is added directly after the water in order to assure sufficient mixing time for the plasticizer to be homogeneously dispersed and activated. At this latest possible moment the micro-encapsulated PCM is added to the mixing process in order to expose it as short as possible to the mixing process. After the PCM addition, part of the remaining SP and water is dosed to, again, obtain the desired self-compacting characteristics. This is for the time being controlled by visual inspection. A subsequent slump-flow and V-funnel test decides if an additional water or SP dosage is necessary to obtain equal workability in terms of relative viscosity and yield stress. All four mixes succeeded in achieving similar workability with only one additional dosage step. Table 2 refers to the final water and SP dosages used for the respective mixes.

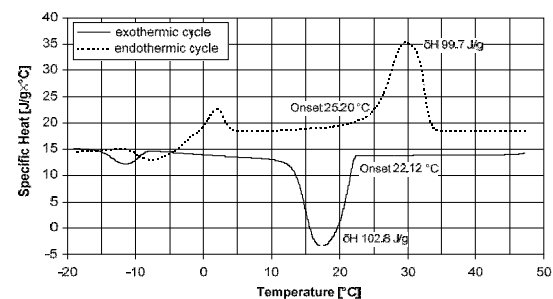


Figure 17: Plot of specific heat as a function of temperature for the heating and cooling cycles of the applied PCM.

	Reference mix	1% mix	3% mix	5% mix
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	Mass (kg)	Mass (kg)	Mass (kg)	Mass (kg)
Ultrafin 12	149.9	149.9	149.9	149.9
CEM I 32.5 R	299.7	299.7	299.7	299.7
Marble powder	170.2	98.0	0.0	0.0
PCM	0.0	23.3	70.0	113.7
Sand 0-1	139.6	139.6	139.6	139.6
Sand 0-4	655.3	655.3	655.3	655.3
Gravel 2-8	387.1	387.1	387.1	387.1
Gravel 4-16	319.6	319.6	319.6	319.6
SP - PCE Glenium 51	3.1	3.1	2.4	2.9
Water	203.2	207.4	211.5	248.4

Table 2: Mix composition.

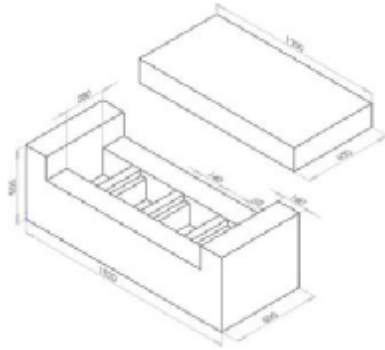


Figure 18: Depiction of the semi-adiabatic environment for four standard sized molds.

### 3.3 Semi-adiabatic curing

To measure the influence of PCM content on the peak temperature during hydration, an experimental setup has been constructed. A layer of cellular glass, 160 mm thick with a heat resistance of  $3.81 \text{ m}^2\text{K/W}$ , forms a semi-adiabatic environment for four standard cubic molds with an edge length of 150 mm according to EN 12390-1. The box-shaped environment measures  $1520 \times 600 \times 500 \text{ mm}^3$  ( $l \times w \times h$ ). The four molds were individually separated by three fixed septa of 50 mm cellular glass (see Figure 18). Around and within the molds thermocouples (type T) were placed to measure the temperature during the early stage of hydration. Data acquisition took place by using National Instruments USB-6215 and PICO TC-08 in combination with a personal computer to store the data. The basis of this setup is comparable to setups used before [6], [7].

### 3.4 Thermal conductivity measurements

A "CT-METRE" was used for the conductivity measurements. The device uses the transient hot wire method conforming to ISO 8894-1:1987, DIN 51046 and ASTM D2326. The operating principle is based on the association of a heating device with a temperature sensor (both connected inside the same probe) intended to measure the temperature increase undergone by the sensor during a

predetermined heating time. The method is not applicable when a phase change occurs in the material, as might be the case with the PCM containing mixes. To overcome the problem, the samples have been heated to  $30^\circ\text{C}$  and the measurements have been performed at this temperature, which is well above the melting range of the PCM.

### 3.5 Specific heat capacity/thermal efficiency measurements

Unlike thermal conductivity, specific heat capacity ( $c_p$ ) increases rapidly in the temperature range of phase change. This makes transient methods inappropriate for  $c_p$  measurements. The Differential Scanning Calorimetry method requires a representative sample of the material in the order of a few milligrams which is not possible with concrete samples consisting of particles up to 16 mm diameter. For the needs of inexpensive and reliable measurements an experimental setup (Figure 19) has been developed which allows measurements of heat capacity, thermal mass and thermal efficiency of building materials. The device applies variable thermal loads at the two sides of a flat-surface material sample, while measuring its thermal response. An extensive presentation of the concept and the corresponding operational principle can be found in [8].



Figure 19: Sample holder of the thermal mass measuring device (sample between two thermo-regulated plates).

## 4 RESULTS

### 4.1 Effects on the properties of fresh concrete

In order not to expose the PCM microcapsules to additional wear during pouring and compaction, a self-compacting mix was designed. These mixes start flowing at much lower yield stresses compared to plain concrete. Therefore, the concrete placing involves only a limited amount of shear stress for the microcapsules. Another reason for the choice of SCC is the faster heat development of SCC compared to standard plain concrete which is among others reported by [9].

The results on the fresh SCC properties are extensively discussed in [10]. Here, it can be summarized that all four mixtures resulted in good and partly excellent self-compacting properties. The increasing PCM dosage did not seem to influence any of these measures in a verifiable range. Only the visual inspection during the V-funnel and in particular the slump-flow experiments resulted in

some uncommon observations. Here a white liquid was accumulated on top of the mixture in areas of slow flow or on resting concrete areas. This observation became especially obvious at the flow area behind the rods of the J-ring as well as along the circumferential line of the spreading concrete during slump-flow tests (cp. Figure 20). This behavior was only observed for the three mixes containing PCM and also became more prominent with increasing PCM content.



Figure 20: SCC after J-ring test with obvious flow marks behind the rods and around the spreading concrete.

#### 4.2 Effects on the hydration temperature peak

The setup offers the space to store four molds in an environment of  $20 \pm 2$  °C. After casting the temperature development of the four kernels was monitored for about 80 hours, taking measurements every minute. These results are shown in Figure 21.

The reference mix shows a peak temperature of 41 °C. This temperature is significantly lower than the results of [6] have shown for other Portland cement mixtures during hydration. The observed cooling refers to a leakage of heat. Otherwise, under totally adiabatic conditions, a temperature rise of about 76 °C to an absolute temperature of 98 °C would be expected, considering complete hydration of the cement. For very massive constructions, where adiabatic conditions can be assumed for the core volume, such a temperature rise would imply the boiling of the free water and cause fundamental durability problems.

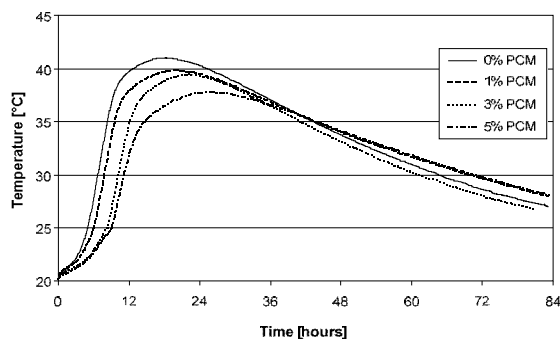


Figure 21: Temperature development of 4 self-compacting mixes in the kernel of the molds in a semi-adiabatic environment during the first 3.5 days after production.

Figure 21 shows that an increasing amount of PCMs results in a lower peak temperature. Because

of the comparable availability of internal or chemical energy in all four mixes, more time is needed to come to the ambient temperature when the amount of PCMs is higher. The 3% and 5% PCM mixes show a small bow around 25 °C, being -according to the DSC experiment - the onset for the endothermic cycle.

Surprisingly, the 3% mix had a larger temperature rise than the 1% mix. In this case it could be that 1) the adiabatic surrounding did not suffice, that 2) more PCM particles were destroyed during mixing in the 3% mix than in the 1% mix, or that 3) the thermocouples were not able to register the temperature correctly. In future experiments these three aspects will be taken in consideration.

Regarding option number two, the analysis of the porosity using SEM showed that many PCM capsules were in fact destroyed. Nevertheless, the temperature registration showed that to a large extent, especially in the 5% mix, the peak temperature could be lowered. This could imply that, despite the partial destruction of the PCM capsules, a large part of the wax is still present in the concrete and could be potentially functioning.

#### 4.3 Effects on mechanical properties

In order to evaluate the effect of PCMs on the mechanical properties of concrete, compressive strength measurements were executed. Therefore, five standard cubes per mixture with an edge length of 150 mm according to EN 12390-1 (2000) have been tested at the 28th day after production. The test procedure for compressive strength is conforming to EN 12390-3 (2001).

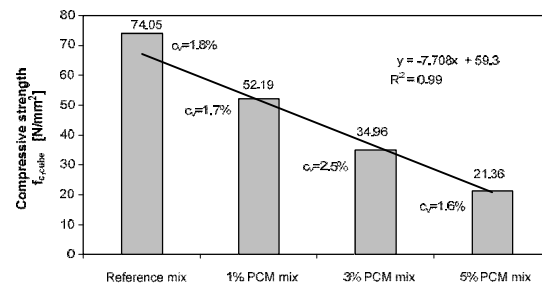


Figure 22: Compressive strength of the PCM mixes after 28 days [N/mm<sup>2</sup>] including the coefficient of variation ( $c_v$ ).

Since a new mix design, as described above, has been applied to this concrete, elevated strength compared to standard composition with equal cement content was expected. Therefore, a modification of Férét's equation was applied to get an indication for the possible compressive strength at 28 days [11]. This modification includes the contribution of other cementitious materials and reads as:

$$f_c = \frac{K_g R}{\left(1 + 3.1 \frac{W + A}{C(1 + K_1 + K_2) + BFS}\right)} \quad (1)$$

where  $K_g$  is an aggregate coefficient (typical values are 5.4 for crushed aggregates and 4.8 for rounded aggregate),  $R_c$  is the standard cement strength (cp. Table 3),  $W$  is the total effective water content,  $A$  is the volume of entrapped air and  $C$  is the weight of cement (in  $\text{kg}/\text{m}^3$ ). The other variables,  $K_1$ ,  $K_2$  and  $BFS$ , refer to pozzolanic and latent hydraulic effects. Since none of these materials were used, these latter variables are zero. Including the relevant data in Eq. (3), the expected strength of the reference mix can be calculated to 46.2  $\text{N}/\text{mm}^2$ . In fact a compressive strength of 74.1  $\text{N}/\text{mm}^2$  was determined. The significantly higher strength could for a large extent be attributed to the improved packing of the new mix design.

Cement type	Heat release after 1 day $Q_{1day}$ [J/g]	Standard strength $R_c$ [ $\text{N}/\text{mm}^2$ ]
CEM I 32.5 R	139.3	57,3
Ultrafin 12	250.0	70.0

Table 3: Thermodynamic and strength properties of the deployed materials.

Results of the compressive strength measurement are presented in Figure 22. From the given data it can be clearly observed that increasing PCM dosages lead to significantly lower compressive strengths. From Figure 22 it can furthermore be concluded that the compressive strength of this specific mixture decreases by 13% for each additional percentage of PCM. This linear relation holds for the range of PCM contents considered.

#### 4.4 Microstructural analysis

An analysis by means of scanning electron microscopy shows a porous microstructure and many, spherical voids which presumably contained PCM capsules before. As a mixture of paraffin probably also slightly varies in its melting temperature, softer paraffin melts earlier than the harder paraffin components. This causes a slight segregation during the melting process. Due to the immiscibility of the leaked paraffin with other concrete ingredients, solidification appears in the cavity of the matrix and at pore walls. An example is given with the SEM micrograph in Figure 23 (a). Here flaked structures of solidified wax cover the inner walls of the pore. Close to the pore walls it forms a type of cell wall which appears to be smooth from the outside (cp. left boundary). The surrounding structure is densely occupied with PCM capsules of the predominant size of about  $6\mu\text{m}$  (cp. Figure 23 right). These capsules appear to a large extent deformed and broken. These observations are very similar to those by Gschwander et al. [12], who found broken micro-capsules of the same type after pumping PCM slurries.

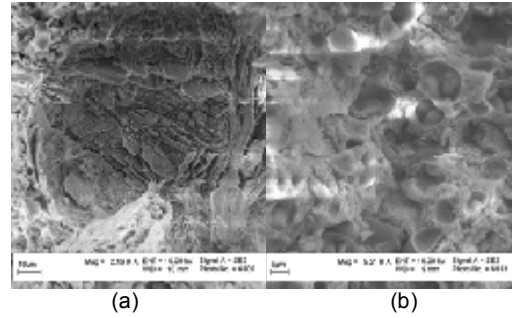


Figure 23: SEM micrographs of (a) an open pore covered with solidified wax, and (b) a higher magnified (5.5k) part of the matrix with deformed and broken capsules, partly pure leaked wax is visible.

#### 4.5 Effects on the thermal properties of hardened concrete

The study of the effects of PCM on the thermal properties of concrete includes measurements of thermal conductivity and specific heat capacity.

##### Thermal conductivity

In order to evaluate the effect of PCM on the thermal conductivity of concrete three mixes containing 1% PCM, 3% PCM and 5% PCM per weight, and a reference material were measured.

According to the standards, two samples of  $100 \times 100 \times 50 \text{ mm}^3$  of every mixture were prepared for the measurements. Thermal conductivity measurements are presented in Figure 24. It is clearly indicated that the addition of PCM particles into the mass of the concrete results in a reduction of thermal conductivity. This can be explained by the enhanced air content and by the lower thermal conductivity of paraffin.

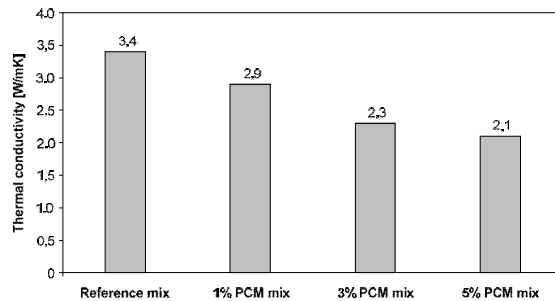


Figure 24: Thermal conductivity of the PCM mixes.

##### Specific heat capacity/thermal mass

For the specific heat capacity measurements four samples of the four different mixes were prepared at the appropriate dimensions,  $200 \times 200 \times 30 \text{ mm}^3$  ( $l \times w \times h$ ). The samples were introduced in the sample holder of the thermal analysis device at a temperature of 19 °C and were heated up to 28 °C. The temperature of the device during the heating process was maintained constant at 32 °C. The temperature of the samples and the heat flux from the device to the samples were recorded. Temperature and heat flux measurements allow the

calculation of the heat capacity and thermal mass of the samples (Figure 25 and 11) as:

$$c_p = \frac{A\dot{q}}{m\left(\frac{dT}{dt}\right)} \quad (2)$$

$$M_{th} = mc_p \quad (3)$$

where  $c_p$  is the heat capacity of the sample,  $M_{th}$  the thermal mass,  $A$  the heat exchange area of the sample,  $q$  the heat flux per square meter,  $m$  the mass of the sample,  $T$  the temperature of the sample and  $t$  the time.

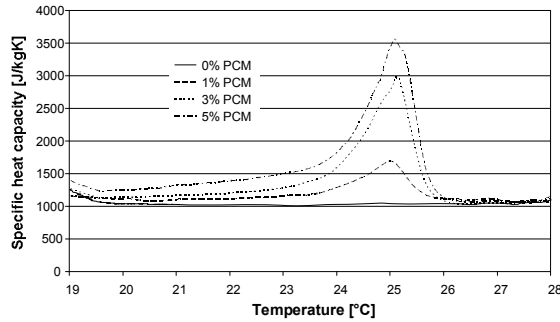


Figure 25: Specific heat capacity of the PCM mixes versus temperature.

Figure 25 and 11 present the measured specific heat capacity and thermal mass for the four samples versus temperature. In both Figures, the effect of increasing the percentage of PCM in the mixture is apparent in the melting temperature range of the PCM (23 °C – 26 °C). Comparison of Figure 25 and 13 indicates that, as expected, increasing the amount of PCM in the mixture increases significantly its specific heat capacity (up to 3.5 times for the 5% PCM content). However, there seems to be an upper limit to the increase of the thermal mass. The 5% PCM mixture has slightly more thermal mass than the 3% mixture inside the melting range of the PCM but less outside of this region. This can be the consequence of decreasing concrete density with increasing PCM content. As a result, a percentage more than ca. 5% (or 4%) of PCM in the mixture does not increase the thermal mass of the material.

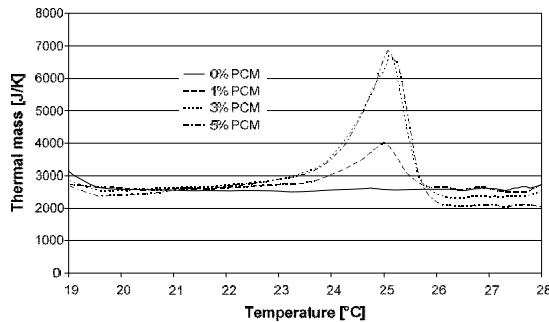


Figure 26: Thermal mass of PCM mixes vs. temperature.

#### 4.6 Effect of PCM quantity on thermal performance

Evaluation of thermal performance of building materials used for building envelopes is generally performed by measuring the decrement factor and time lag. In the case of building materials containing PCM, the above two measures may not be representative since they do not take into account the large amount of heat contributions/subtractions in the temperature range that the phase change occurs [13]. An appropriate method of evaluating the effect of PCM on thermal efficiency of structural elements is the comparison of the heat flux at the indoor surface of a wall with different amounts of PCM [14].

In the current work the above described thermal analysis device is used to simulate indoor and outdoor temperatures and the corresponding temperature profiles are imposed at the two sides of the sample. The outdoor temperature is assumed to have a sinusoidal variation from 18.5 °C – 28.5 °C for 48 hours (for instance resembling temperature variations in a South European country), while the indoor temperature is set stable at a level of 23.5 °C. Temperatures and heat fluxes on both surfaces of the same samples as the ones used for the specific heat capacity measurements are recorded.

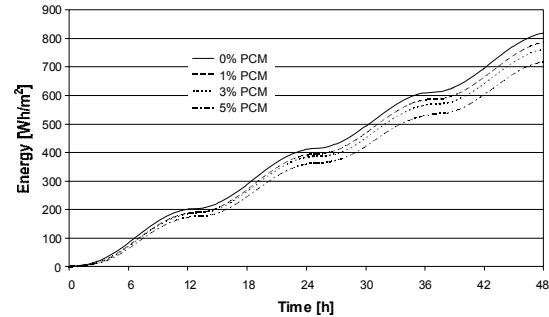


Figure 27: Energy required for maintaining indoor temperature stable at 23.5°C.

Integration of the measured heat flux on the inner side of the sample provides a measure of the total heat losses towards the indoor environment (Figure 27). The heat flux measurements demonstrate an up to 11% variation in the measured maximum and minimum peak values for the sample with 5% PCM content.

## 5 CONCLUSION

The mix design method presented in [3] for SCC containing marble powder was successfully adopted and applied to the three mixes containing micro-encapsulated PCM. Based on the J-ring and V-tunnel test, all four mixtures featured good self-compacting properties. Using the developed recipes the increasing PCM dosage did not seem to influence the properties of the fresh concrete.

The modeling and experiments involving hydration showed that the temperature peak of hydration could be reduced up to 28.1% by increasing the PCM content to 5%. However, the heating rate cannot be changed by the PCM, only the absolute

temperature peak is lowered by the amount of energy temporarily stored in the PCM. The emission of heat from the sample will therefore continue for a longer time when the PCM content is higher.

Regarding the thermal properties of hardened self-compacting concrete with PCM, the experiments showed a reduction of thermal conductivity with increasing amounts of PCM.

The measurements have indicated that the overall behavior of each sample is the combined result of conductivity and specific heat variations. Moreover, with increasing PCM content the thermal mass of the sample increases significantly. Although the specific heat capacity increased with increasing amount of PCM in the considered temperature range (24 – 26 °C), the thermal mass seems to be bound by a maximum of approximately 6800 J/K at 4% to 5% PCM.

The increase in thermal mass could improve significantly the thermal performance of concrete in terms of energy saving. For example, savings up to 12% could be expected as a result of the inclusion of 5% PCM in the mix.

More experiments with samples incorporating dedicated amounts of PCM above chosen percentages could provide better insight into the exact value of this maximum. In practice, both the lower thermal conductivity and the increased heat capacity significantly improve the thermal performance of concrete and could lead to energy savings in building applications.

Although the loss of compressive strength is significant, concrete with PCM content up to 3% and an accompanying compressive strength of 35 N/mm<sup>2</sup> is still for most constructional purposes well acceptable. The loss of compressive strength can be assigned to destructed shells of the PCM capsules for reasons mentioned in paragraph 4.4. Observations made during a side experiment show that the respective micro-encapsulated PCM material is not suitable for the requirements of a concrete application [10]. The released wax from the microcapsules interferes into the surrounding concrete matrix and hinders a sufficient strength development in multiple ways. Here, the possible inhibition of the water transport and hence an interruption of the hydration, and the possible development of phase interfaces by the wax are to be mentioned. Therefore, the main recommendation to be given involves the development of stronger shells for microencapsulated PCM. These stronger shells have to withstand the highly alkaline conditions, which in addition to the mechanical impact have a negative influence on the strength of the shells.

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