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# Addition of microencapsulated PCM into single layer mortar: Physical and thermal properties and fire resistance

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#### 1. Abstract

The energy consumption in buildings has increased in the last years due to the energy demand for thermal comfort. The improvement of thermal properties in building materials is one of the key points to achieve a reduction of the energy demand. Thermal energy storage (TES) is an alternative to save energy that has been investigated extensively in recent years. The addition of phase change materials (PCM) in a material increases its thermal inertia because PCM are able to store and release energy in sensible and latent heat. In this paper coatings used in the outer walls of buildings are studied, in particular single layer mortar. Microencapsulated paraffin PCM is added into the material to improve the thermal properties of the building envelope. The use of PCM combined with thermal insulation can reduce the energy consumption of buildings by absorbing heat gains and reducing the heat flow. Two types of single layer mortar Weber.Therm.Mineral commerzialized by Weber Saint Gobain incorporating different percentages of microencapsulated PCM type Micronal® DS5001 (20%, 10% and 0%) in their formulations are studied in this paper. The main objective of this work is to analyze the effect of PCM addition on the properties of two single layer mortars. Fresh mortar properties, as well as fire behavior, physical, mechanical and thermal properties of hardened mortar have been evaluated. The results show that physical and mechanical properties of single layer mortar are affected by the addition of microencapsulated PCM. Compressive strength decreases 60% with the addition of 20% of PCM in the formulation while the formulation with 10% of PCM has approximately the same compressive strength than a single layer mortar without PCM. The addition of PCM in the formulation decreases in 35% the adherence of the single layer mortar to a concrete surface. The presence of PCM in the mortar formulation worsens the fire behavior, due to the organic, and therefore flammable, composition of Micronal® DS5001. Flammability increases according to the percentage of PCM added. As a general conclusion, physical and mechanical properties of the single layer mortar become worse when the percentage of the added microencapsulated PCM is increased. Nevertheless, an improvement of thermal properties is expected and the quantification will be done that could justify the addition of PCM to some extent.

**Keywords:** single layer mortar, coating, building, thermal properties, fire resistance, thermal energy storage







#### 2. Introduction

The building sector accounted for 34% of total global final energy use in 2010 according to ETP 2012 [1]. The building industry is one of the largest end-use sectors worldwide. For this reason, the EU Member States faced new challenges with the approval of EPBD in 2010 (Directive 2010/31/EU) [2]. The achievement of nearly-zero energy buildings in 2020 (2018 in the case of Commercial buildings) will be one of the key points by the implementation of EPBD.

In order to improve the thermal response of the building envelope to reduce the energy demand several studies have been done in recent years. Aste et al. 2009 [3] conclude that the implementation of walls with high thermal inertia reduces around 10% of the heating demand and 20% of the cooling demand requirements. Increasing the thermal mass of the materials, which compose the building envelope, temperature fluctuations can be minimized [4[5]. Phase change materials are successfully used in some studies to increase the thermal mass of materials. In Alawadhi et al. 2008 [6] the PCM is added inside the cylindrical holes of building bricks and the results show a reduction of 17-55% of the heat flux through the indoor space if the cylindrical holes are placed at the centerline of the brick. The incorporation of microencapsulated PCM increases the heat transfer area, PCM reactivity towards the outside is reduced and volume changes are controlled when the phase change occurs. The most common studies are done at macro-scale, by numerical simulations or at laboratory or micro-scale in order to verify the improvement of microencapsulated PCM in the building envelope [7[12].

In most studies PCM is located inside the wall but, in [13] the author investigates the effect of the PCM placed outside the building envelope and up to 7.2% of energy savings on the cooling demand is achieved by the installation of 3 cm of PCM plaster on all exposures of the vertical building envelope. This technique has a substantial importance in refurbishment because it offers a non-invasive thermal improvement of old building envelopes.

The aim of this investigation is to study the effect of different percentages of microencapsulated PCM (0, 10 and 20%) in the properties of single layer mortars to be used as an external plaster. The materials are studied at micro and laboratory scale in order to evaluate thermal improvements. Furthermore, the modification of mechanical and physical properties and fire reaction are evaluated.

#### 3. Materials and formulation

The single layer mortar (SLM) used in this experimentation Weber. Therm. Mineral is commercialized by Weber of Saint Gobain. In order to study the variability of the properties, two types of SLM are studied, without coloring agents (white) and with coloring agents (yellow). SLM are composed by cement, lime, high dispersion glass fibers, aggregate with compensated granulometry, organic additives and, hydrofuged resins (and mineral pigmentations in case of yellow SLM).

A proper selection of the PCM is mandatory, so the main parameters to take into account are: the melting temperature (around the range of operation), the chemical stability, the toxic elements, and the cost. In this study the selected PCM was based on paraffin waxes due to its low cost, the absence of subcooling and the chemical stability without phase segregation. On the contrary, its low thermal conductivity limits their applications [4]. The selected PCM is







Micronal DS5001 microencapsulated paraffin waxes. It is commercialized by Basf and has a melting point around 26° and a heat absorption capacity of 145 kJ/kg.

The amount of PCM used in this experimentation is 0%, 10% and 20% in weight in relation to the mixing water and the powder mortar. In order to guarantee a satisfactory workability of the cement mixtures a plastic consistency was fixed for all mortar samples according with the standard UNE-EN 1015-2 [14]. The mortars with PCM addition required higher amounts of water to achieve similar consistency values. Determination of bulk density and flow value of the mortars following (UNE-EN 1015-3 [15]) is necessary to calculate the consistency.

#### 4. Methodology

# 4.1 Non-destructive tests. Determination of dynamic modulus of elasticity $(MOE_D)$ by fundamental resonance

Dynamic modulus of elasticity (longitudinal and transversal) can be calculated by the characteristic fundamental resonance frequency, which is obtained by the application of a mechanical impulse or by a hit. This impulse generates sound waves through the material, and a microphone collects them. The received signal is synthetized by specific software and it indicates the resonance frequency of the material (Figure 1). This method follows the standard UNE- EN ISO 12680-1 [16] with samples of 40x40x160mm.



Figure 1. Equipment used in fundamental resonance frequency test.

### 4.2 Destructive tests. Determination of compressive strength and adherence

The compressive strength of nine samples (40x40x160mm) with different PCM percentages (0%, 10% and 20%) and different ages (7, 28 and 90 days) has been tested following the standard UNE-EN 1015-11 [17]. The equipment used was WYKEHAM FARRANCE (Figure 2), with a compressive strength device, a low load adapter 2T and a load reader; the load velocity used is 200N/s according to [17].











Figure 2. WIKEHAM FARRANCE Sample tested (left). Compressive strength adapter (right)

The adherence is tested in KN-30 of NEURTEK (Figure 3) following the standard UNE-EN 1015-12 [18]. This test consists on the application of a perpendicular traction force in a SLM sample fixed to a holder by dual-component resins using samples with 40 mm of diameter and 18 mm of thickness (Figure 3).





Figure 3. KN-30 of NEURTEK Tester (left). Samples fixed in the holder by dual-component resins (right)

### 4.3 Thermal analysis. Thermal conductivity, thermal diffusivity and specific heat

The equipment Quickline-30 manufactured by Anter Corporation (Figure 4) is a multifunctional equipment to test the thermal conductivity ( $\kappa$  [W/m·K]), thermal diffusivity ( $\alpha$  [m²/s]) and heat capacity by density (Cp [J/m³]) of materials at room temperature using a sensor with a range of 0.04 - 0.3 W/m·K. The measures are based on periodical registers of temperature depending on the time response of the sample thermal excitation by a directional heat flux. Heat flux is generated heating an electric resistance inside the sensor. This sensor is placed in contact with the analysed material.



Figure 4. Conductivity meter Quickline-30 Anter Corporation







#### 4.4 Fire reaction tests

Paraffin are highly inflammable so the fire reaction must be analyzed when paraffin are used in buildings. A dripping test is done in order to study the fire response of the SLM with microencapsulated PCM.

A dripping test was performed on the samples to determine the time to ignition, the number of combustions and the average extent of the combustions. In this analysis, samples of 70x70x18mm are tested in a combustion chamber (¿Error! No se encuentra el origen de la referencia.) .The samples are placed on a metallic grid below a heat source of 500 W, which is taken away and put back after each ignition and extinction during 5 minutes. The parameters determined are the time to ignition, the number of ignitions and the average time of combustion extent. This test follows the standard UNE 23725 [19].

#### 5. Results

Results of MOE<sub>D</sub> by fundamental resonance test for compositions with 0, 10 and 20% and ages of 7, 28 and 90 days are presented in Figure 5. The results show an increase of the flexural dynamic modulus of elasticity with the age. Nevertheless, the velocity of hardening varies depending on the amount of PCM added. The composition without PCM achieves the 81.6% of its total flexibility during the first 7 days, and the 87.81% at 28 days of age, reaching 4702 N/mm² in 90 days. The composition with 10% of PCM has a slower velocity of hardening, achieving the 66.9% of its total flexibility during the first 7 days and 88.29% at 28 days, reaching 2871 N/mm² in 90 days. However, composition with 20% of PCM achieves 36.75% of its flexibility during the first 7 days and 39.24% at 28 days of age, reaching 2620 N/mm² in 90 days.

The final elasticity decreases with the addition of PCM. The SLM with 10% of PCM exhibits a reduction of 38.94% with regard to the SLM without PCM. This decrease reaches the 44.8% for the formulation with 20% of PCM.

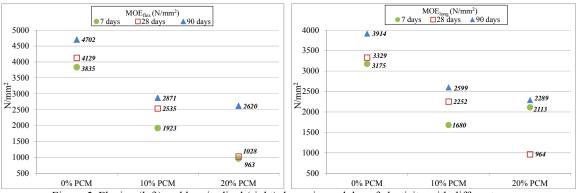


Figure 5. Flexion (left) and longitudinal (right) dynamic modulus of elasticity with different ages

The behaviour of the compressive strength follows the same trend tan MOE<sub>D</sub>, compressive strength decreases according to the amount of PCM added and it increases with the age.

Compressive strength of SLM (Figure 6) with 10% of PCM shows a decrease of 14.45% compared to the sample without PCM at the age of 7 days, 1.90% at the age of 28 days, and 4.69% at the age of 90 days. The compressive strength of SLM with 20% of PCM decreases drastically if results are compared with samples without PCM, 38.94% at the age of 7 days, 43.81% at the age of 28 days and 59.76% at the age of 90 days. Considering the results of the SLM with 10% of PCM it seems that the presence of PCM hinders the hydration of the mortar







and therefore, the achievement of mechanical strengths at early stages. However, with 10% PCM addition the compression strength results at 28 days and from this point forward are comparable with the conventional SLM. The addition of higher percentages of microencapsulated PCM penalises the compressive strength of the SLM probably due to both the lack of mineral aggregate, and the excess of water in the mixture.

The results of the SLM adherence (Figure 6) show better results in white composition than yellow composition, regardless of the amount of the PCM added. Furthermore, the adherence is also reduced by the addition of PCM.

White SLM without PCM has an adherence of 3.37N/mm<sup>2</sup>; if 10% of microencapsulated PCM is added into the material the adherence will be reduced in 0.36 times, and the addition of 20% of PCM reduces the adherence 0.63 times. Yellow SLM without PCM has lower adherence (1.85N/mm<sup>2</sup>) than white SLM; this adherence is reduced 0.25 times by the addition of 10% of PCM, and 0.43 times by the addition of 20% of PCM. In both SLM, results show a linear relationship between the addition of PCM and the adherence.

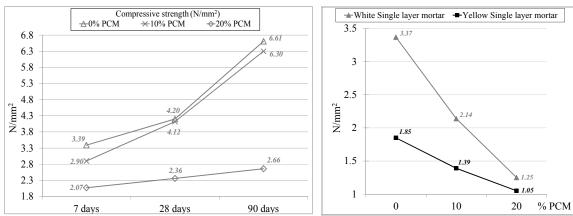


Figure 6. Results of compressive strength test (left) and values of single layer mortar adherence (right)

The obtained samples (Figure 7) show a decrease of the thermal conductivity by the addition of microencapsulated PCM. Microencapsulated PCM has a lower thermal conductivity (0.18W/m·K) than SLM, therefore the addition of PCM decreases the final thermal conductivity of the material. White SLM without PCM has a thermal conductivity of 0.51 W/m·K. The addition of 10% of PCM diminishes thermal conductivity in 3.92%, and in 17.65% in the case of samples with 20% of PCM. Thermal conductivity of yellow SLM decreases a little bit more with the addition of PCM reaching 0.54 W/m·K without PCM, 7.41% less with the addition of 10% of PCM, and 11.11% with the addition of 20% of PCM. In all cases, white SLM has raised lower thermal conductivities than yellow for all the compositions.

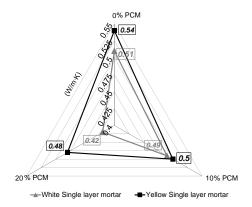


Figure 7. Thermal conductivity of single layer mortar







Results in Table 1 show that yellow SLM has a thermal diffusivity slightly higher than white SLM and values decrease with the addition of PCM.

Table 1. Thermal diffusivity of the samples studied

	0% PCM	10% PCM	20% PCM
White SLM	$3.47 \cdot 10^{-7}$	$3.25 \cdot 10^{-7}$	$3.22 \cdot 10^{-7}$
Yellow SLM	4.01·10 <sup>-7</sup>	$3.31 \cdot 10^{-7}$	3.04 · 10 <sup>-7</sup>

The following results of fire reaction test show the time to ignition, the number of ignitions and the average duration of the flame. As general observations in the results obtained in fire reaction tests, the temperature of SLM samples without PCM increases with the application of the hot focus but are not flammable. White SLM do not change its colour with the application of the hot focus but, yellow SLM suffers a variation of its colour becoming orange (Figure 8) probably due to the calcination of the pigment.





Figure 8. Yellow SLM. Before fire reaction test (left). After fire reaction test (right)

SLM samples have differences on the roughness of the surfaces and this fact can cause differences in the fire behaviour due to higher flame persistence in rough surfaces. Therefore samples with 20% of PCM have been tested on both surfaces: Rough and Smooth. Table 2 summarizes the results obtained for the different formulations in the dripping test. As it can be observed the first ignition (TTI) occurs at higher times for the samples with 10% of PCM than for samples with 20% of PCM. Moreover, a higher number of ignitions are observed for 10% of PCM samples together with short average combustion extent. Usually this combination is a sign of auto-extinguish capability of the material, which is important to reduce the risks of fire spread in case of fire. In both SLM, yellow and white, samples with 20% of PCM exhibit a worse fire reaction, and the flame persistence is higher in the case of rough surfaces. However, it should be noticed the remarkably high combustion extent values for the yellow SLM with 20% of PCM. This fact could be due to the inhomogeneous distribution of the PCM through the sample with higher concentrations on the surfaces of the material.

Table 2. Results obtained from the dripping test

	TTI (s)	Nr of ignitions	Avg. combustion extent (s)
W 0% PCM	-	-	-
W 10% PCM	27	35	4
WR 20% PCM	13	9	31
WL 20% PCM	14	13	20
Y 0% PCM	-	-	-
Y 10% PCM	23	43	4
YR 20% PCM	11	2	253
YS 20% PCM	10	2	202







#### 6. Conclusions

Addition of microencapsulated PCM to single layer mortars requires higher amounts of water to maintain the same workability. Both single layer mortars with and without pigments show the same trends regarding the effect of PCM in their formulation. Hydration of mortar samples is affected by the presence of PCM, which is reflected on lower values of mechanical properties at early stages. Dynamic module of elasticity, adherence and compressive strength strongly decreases with the addition of PCM, specially in the case of 20% PCM samples. The lack of adhesion between the Portland cement and the plastic microcapsule of the PCM together with the increase of water during mixing and the small size of the PCM that modifies the particle size distribution of the mortar could be some of the factors affecting mechanical properties. Taking into account that single layer mortars are not used in structural applications mortar with 10% PCM which compressive strength is only reduced in 2 and 5% at 28 and 90 days will probably fulfil the requirements for plaster applications.

Thermal conductivity and thermal diffusivity decrease with the amount of PCM, probably due to the lower thermal conductivity of the microcapsules compared with the mortar. As expected the presence of organic PCM worsens the fire behaviour of the mortar. Samples containing 10% PCM produce flame, but exhibit auto-extinguish ability once the heat source is removed. In several samples with 20% PCM the flame persists during larger times which could contribute to flame spread in case of fire.

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