Chapter 4
MPCM-based Porous Cementitious Composites for Enhanced Energy Efficiency of Smart Buildings

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Abstract This work reports a detailed experimental study that has the purpose of investigating the thermal energy storage (TES) performance of cement pastes enhanced with novel microencapsulated phase change materials (MPCMs). Three water-to-binder ratios and three MPCM volume fractions, leading to a total of nine different mixtures, were investigated at the *Institut für Werkstoffe im Bauwesen*– TU Darmstadt. Water-to-binder ratios of 0.33, 0.40, and 0.45 are considered, for the reference pastes, while a MPCM having a melting/solidification temperature range of 37 °C and a latent storage capacity of 190 kJ/kg has been mixed in the MPCM pastes. Thermal and mechanical tests, accompanied by SEM analyses, are performed to observe the effect MPCMs have on the resulting TES and strength results. The experimental data have been analyzed to evaluate the corresponding temperature-based material parameters like specific heat, conductivity, or more in general the energy storage capacity of these systems under transient heat transfer conditions.

Keywords Energy storage · Cement pastes · Bio-based PCM · Thermal energy analysis · Heat storage · Eco-friendly

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4.1 Introduction

The global energy consumption of buildings is one of the key issues that significantly contributes to the anthropogenic CO_2 emissions; thus, its reduction represents a major Sustainable Development Goals (SDGs) of tomorrow. According to the EU commission, the energy consumption of buildings' heating and cooling accounts for the biggest single end-use sector of all generated energy. Furthermore, 84% of this energy is still obtained from fossil resources [1]. Besides, building energy demand is expected to increase by 79% in residential buildings and 84% in commercial buildings until 2050 [2]. In this sense, EU has committed to at least double the annual energy renovation rate of residential and nonresidential buildings by 2030 and to foster deep energy renovations. To achieve this target, the focus on constructing the path toward the reduction of energy consumption of buildings is of key importance.

The effective management of the interacting thermal energy flowing inside a building (including sources and sinks of heat) can be used to (i) minimize its energy consumption, (ii) to level out daily temperature differences, and (iii) to enhance building's energy efficiency [3]. In this regard, a promising solution to improve the thermal energy storage (TES) performance of buildings through a smart use of sensible and/or latent TES is by incorporating phase change materials (PCM) into construction composites [4–7].

PCM can be used to store large quantities of heat, not only through their sensible capacity but also (often predominantly) via their latent TES [8]. These materials can store large amounts of latent heat, at quite constant temperature, and can contribute to the energy efficiency and thermal comfort of buildings [9]. Ordinary building materials, such as concrete, only can deal with their sensible heat storage capacity, which varies approximately between 0.75 and 1.00 kJ/ (kgK), whereas organic PCMs can possess latent heat storage capacity of approximately 180 kJ/kg [10]. Consequently, passive storage/release of latent heat through phase transitions from solid to liquid or vice versa allows to save considerable amount of primary energy [11–13]. Organic PCM are composed of a wide range of materials, including paraffin and non-paraffin (bio-based) components. The latter, which can be made (at least in part) from renewable resources, can be an alternative eco-friendly option to petroleum-based chemicals. In the building sector, PCM are commonly used in building materials with a phase change temperature in the range of human comfort, that is, between 18 °C and 24 °C (according to the World Health Organization [14]).

One of the major challenges of adding PCM in construction elements/materials is how to avoid liquid leakage during the phase change [15]. Various techniques have been proposed to integrate PCM directly into the building materials [16–18]. For example, microencapsulated PCM (viz., MPCM) is one of the most suitable techniques [19] to be used and integrate PCM directly into the composite material [20–22]. PCM can be encapsulated within microcapsules, and the result is a powderlike material, whose size ranges from less than 1 μm to 300 μm circa [19]. MPCM can be easily mixed together with the basic concrete components and has various advantages like an increased surface area for heat transfer, no leakage, reduced reactivity of PCM toward the external environment, and controlling the phase change process [23].

Incorporating MPCMs in cementitious materials is a very efficient way of reducing energy demand in buildings due to their enhanced ability to absorb and release large quantities of heat energy at an almost constant temperature [24]. The great advantage of incorporating MPCMs in cementitious materials is represented by their vast area they offer for storage and heat transfer. However, for a successful TES implementation, research is needed to overcome major drawbacks such as crack formation and unsatisfying thermal conductivity [25]. Therefore, to produce stable MPCM cementitious composites, it is crucial to study the mixture design concerning different MPCM dosages and different water-to-binder (w/b) ratios. Integrating MPCM into cementitious matrix can potentially mitigate thermal cracking, caused by early-age temperature rise due to cement hydration and freeze-thaw damage [26]. On the other hand, the incorporation of MPCM into cementitious matrix possesses a wide range of issues due to the nonuniform nature of cement paste and the chemical reactions associated with the hydration. Although the implementation of MPCM into cementitious composites has been regarded as a good technique to overcome the material leakage during phase transition, undesired lower thermal conductivity has been reported and needs to be improved [27, 28].

In this regard, there is a need to investigate the thermal and mechanical behavior of cement paste by changing a number of factors, such as type and amount of MPCM, fillers and supplementary cementitious materials, and the mixing procedure. The selection of MPCM used for the application in cementitious matrix depends on its thermophysical properties [29]. A large number of studies have already been addressed, which, from an experimental point of view, emphasized the hydro-thermo-chemo-mechanical properties of cement-based materials containing MPCM at several scales of applications [15, 30, 31]. There are some common techniques like differential scanning calorimeter (DSC) [32], Fourier transform infrared spectroscopy (FTIR), thermal gravimetric analyzer (TGA), and scanning electron microscopy (SEM) [33] by which thermophysical properties of MPCM-based cementitious composite have been investigated.

Besides the use of traditional experimental methods, computer-aided simulations present themselves as an efficient method to further understand the effects of MPCM incorporations into cementitious composites. It has been seen that most studies focus more experimentally on the influence of PCM (or MPCM) in cementitious matrices. However, it is much less common to see the relationship between the experimentally obtained results and predicted data based on numerical simulations.

As an overview, the major objectives of this study are the following:

1. To evaluate the TES behavior of MPCM incorporated into porous cementitious composites.
2. To better understand the microstructure and mechanical behavior changes of cementitious systems upon the incorporation of MPCM.

3. To prepare a wide experimental database for investigating the influence of the morphological effect on the thermal properties of hydrating cement paste systems.

The latter will be linked to numerically generated cement pastes, created with the HYMOSTRUC model [34] for pastes combined with MPCM.

Finally, the goal of this study is to find a clear understanding of available studies focused on MPCM in cementitious composites and their applications tailored for the building market. Another very important issue is to present proper testing procedures, boundaries, and heating loads that should be taken under consideration, in order to obtain the correct performance potential of the investigated MPCM composites.

4.2 Materials and Methods

The following section deals with the materials employed in this study as well as the methods to characterize the thermal and mechanical properties of the selected MPCM, plain, and MPCM cement paste systems.

4.2.1 Materials

For the cement paste design, a commercially available ordinary Portland cement (CEM I 52.5 R) was used. In addition to the cement, metakaolin (Centrilit NC II) was added as a binder (33% in volumetric fraction substitution of the cement powder). Binder's main properties are shown in Table 4.1. Regarding the MPCM, this study focused on the use of paraffin-based Nextek37D by the Microtek Laboratories Inc. (see Table 4.2). The chosen MPCMs in powder form were applied into the cement paste matrices, in different volume fractions, as cement paste replacement.

The purpose of this study is to assess nine paste mixtures, made with three different w/b ratios of 0.33, 0.40, and 0.45, respectively. For each w/b ratio, MPCM is added at minimum and maximum dosages to ensure powder volume fractions of 20% and 40%, by volume substitution of cement paste. All mixtures were

Table 4.1 Outlines of the cement and metakaolin properties

Binder	Type	Strength after two days	Strength after 28 days	Apparent density	Density
		[MPa]	[MPa]	[kg/m^3]	[kg/m^3]
Cement	CEM I 52.5 R	≥30	≥52.5	900–1200	3100
Metakaolin	*See Centrilit NC II datasheet https://www.mc-bauchemie.de/*				

Table 4.2 Outlines of the main properties of the considered PCM and MPCM

PCM core	Encapsulation	T_m	Heat storage capacity sensible+latent	Apparent density	Density
		[°C]	[kJ/kg]	[kg/m^3]	[kg/m^3]
Mix of paraffin	Nextek 37D	$37^{+/-2}$	Under the datasheet of https://www.microteklabs.com/nextek-37d		

Table 4.3 Mixture proportions, overview of the nine cementitious pastes

Labels	S-45-ref [kg/m^3]	S-45-20 [kg/m^3]	S-45-40 [kg/m^3]	S-40-ref [kg/m^3]	S-40-20 [kg/m^3]	S-40-40 [kg/m^3]	S-33-ref [kg/m^3]	S-33-20 [kg/m^3]	S-33-40 [kg/m^3]
w/b ratio	**0.45**			**0.40**			**0.33**		
Cement	983.4	786.7	590.0	1043.1	834.5	625.8	1140.0	912.0	684.0
Metakaolin	161.8	129.4	97.1	171.6	137.3	103.0	187.6	150.1	112.5
Water	515.3	412.3	309.2	485.9	388.7	291.5	438.1	350.5	262.8
Superplasticizer	5.7	4.6	3.4	6.0	4.8	3.6	6.6	5.3	3.9
Stabilizer	33.1	26.5	19.8	35.1	28.0	21.0	38.3	30.7	23.0
MPMC [V.-%]	–	20	40	–	20	40	–	20	40
Hardening accelerator	22.9	18.3	13.7	24.3	19.4	14.5	26.5	21.2	15.9

experimentally prepared based on the German Standard EN 196-1 and according to the mixture designs highlighted in Table 4.3.

By considering different mixing procedures, based on the EN 196–1 standards, the amount of additives, the addition order of the components, and also the mixing time for the most optimized pastes were adapted. The chosen mixing procedures were the following: (i) first, the cement and metakaolin (Centrilit NC II) are mixed for 60 s.; (ii) the water with the superplasticizer is added and mixed for another 60 s. by mechanical stirring at low speed, followed by extra 60 s. at high speed; and (iii) the stabilizer is added to the mixture and mixed for 30 s. at high speed, followed by (iv) the MPCM mixing the paste for 30–60 more s. at low speed. Finally, (v) the hardening accelerator is mixed with the paste for 30 s. at low speed. At this point, the paste was molded. For each mixture, nine prisms were prepared and stored in a climatized room (20 °C), to be used later for mechanical tests, mercury porosimetry, and SEM characterization. One day after casting, the prismatic samples were removed from the molds, wrapped in plastic foil, and stored back in the climatized room, until the desired testing age (i.e., 3, 7, and 28 days). Subsequently, after 28 days, the samples were dried at 50 ° C. The temperature during drying was increased gradually: $30\ ^{\circ}C \rightarrow 40\ ^{\circ}C \rightarrow 50\ ^{\circ}$C. During drying, the mass of the samples was checked every 1–2 days until the weight is stabilized. Then, DSC and thermal conductivity measurements were performed, as well as final mechanical tests.

The batch was big enough to prepare also one cylindrical specimen of 2 cm high (*h*) and 4 cm of diameter (*d*) for Hot Disc (HD) measurements. Furthermore, one specimen ($h = 2$ cm, $d = 1.2$ cm) for laser flash technique (LFT) for thermal diffusion characterization was also prepared as well as three crucibles for differential scanning calorimetry (DSC) and discs of $h = 0.5$ cm and $d = 1.5$ cm size for electrical conductivity measurements. However, for brevity, this work will show the results of mechanical, DSC, and SEM tests only.

4.2.2 Methods

This section reports the methods used to investigate the TES and mechanical properties of components and composites presented in Sect. 4.2.1.

Heat Capacity Tests – Differential Scanning Calorimetry (DSC) German Standards DIN 51005 and DIN 51007 have been considered as a reference to perform the DSC tests (Fig. 4.1a), while the IEA standard procedure [10] is followed to determine the effective heat storage capacity of the samples during the phase transition of the MPCM. The samples were prepared in aluminum DSC crucibles (Fig. 4.1b), cured until 28 days, and completely dried. A small hole was made in the lid of the crucible, as seen in Fig. 4.1c, in order to enable an isobaric measurement.

Mechanical Tests – Flexural/Compressive Strength To characterize the mechanical response of the produced energy storage cementitious systems, small-sized prismatic samples (Fig. 4.2a) were tested under both bending (Fig. 4.2b) and compression (Fig. 4.2c) according to the procedures described in EN 196-1. For each mixture, nine prisms (40 mm × 40 mm × 160 mm) were prepared and tested under a three-point bending scheme after 3, 7, and 28 days. Three identical samples per each time and mixture were considered. For the bending test, the distance between supports was 100 mm (± 0.5 mm), and the vertical load was applied with a loading rate of 50 N/s until failure. Subsequently, compression tests were performed, under a load cell of 200 kN with each of the remaining halves of the broken beams by bending tests.

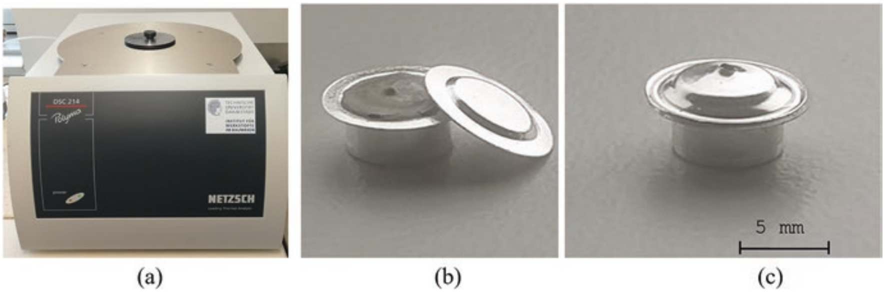


Fig. 4.1 (**a**) Differential scanning calorimetry (DSC) 214 Polyma from Netzsch (Selb, Germany); (**b**) aluminum sample holders (maximum volume capacity of 40 µL); (**c**) Prepared sample inside crucible with a small hole on the top

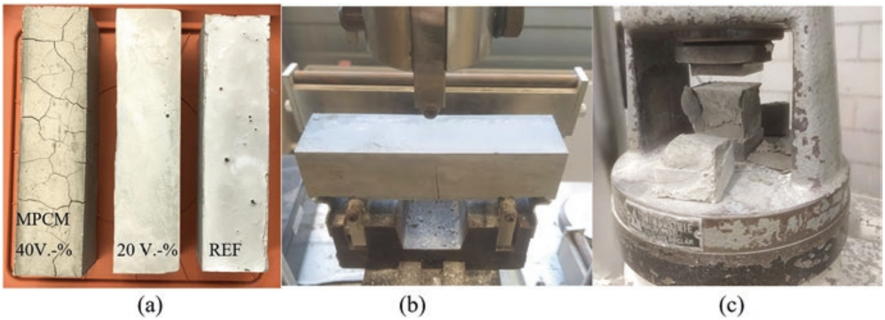


Fig. 4.2 (**a**) Prisms (w/b = 0.40) after 28 days curing and drying in an oven at 50 °C: (**a**) from left to right, 40%, 20%, and 0% volumetric MPCM; (**b**) prismatic sample setup under three-point bending test; (**c**) one-half of the initial sample after compression strength test

Microstructural Analysis, Scanning Electron Microscope (SEM), MPCM Pastes

The microstructure of the cementitious samples was observed through scanning electron microscopy analysis (by using a Zeiss EVO LS25 – atmospheric scanning electron microscope available at the WiB TU Darmstadt).

The samples after 28 days of curing were cut into approximately $2 \times 2 \times 2\ \text{cm}^3$ and placed in cylindrical molds as shown in Fig. 4.3. For the sample preparation, a vacuum impregnation device (CitoVac from Struers) was used to imbed the samples in an epoxy mixture (25 g of epoxy to 3 g of hardening accelerator). Afterward, the surface was polished with a proper polishing device (LaboForce 100 from Struers).

4.3 Thermal Experimental Results

In this section, the impacts of MPCM on the cement paste TES properties are reported. The thermal behavior analysis has been carried out via DSC tests for all cement paste systems produced with and without the substitution of MPCM with three different water/binder ratios. For all the samples, three specimens were investigated by considering three cycles and a heating or cooling rate of 10 K/min for the reference samples and 0.5 K/min for the MPCM paste samples.

From the thermal point of view, it was found that the inclusion of the MPCM in the cement paste increases its thermal inertia and also the specific heat capacity of all the samples. The response of the three reference paste systems is characterized by almost the same kind of sensible behavior, as analyzed in the temperature range between 20 and 40 °C. Their sensible specific heat storage capacity is 1.03, 0.98, and 0.95 J/g K, for the w/b ratios of 0.45, 0.40, and 0.33. A slightly higher specific heat capacity (sensible) turned out for those cement pastes with higher water-to-binder ratio, for example, the w/b = 0.45 results when compared with the 0.40 and 0.33 ones. The results also showed an almost temperature independency behavior of the $\rho \times C_p$ response.

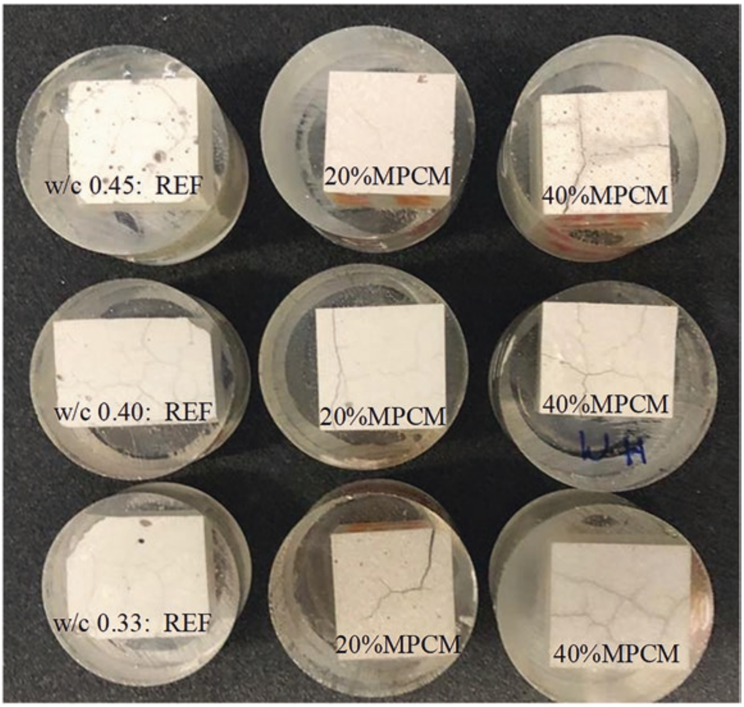


Fig. 4.3 (**a**) Paste/MPCM paste samples after epoxy treatment for SEM analyses

In the DSC thermograms of Figs. 4.4 and 4.5, there are plotted ρ × Cp vs. T for heating and cooling of the systems with 20% and 40% MPCM volume fractions, respectively. The curves show an evident latent peak in the region close to the temperature where the phase change occurs (under solidification/melting).

4.4 Mechanical Experimental Results

In this section, the impacts of MPCM have on the mechanical properties of the tested cement pastes are reported and discussed. Figure 4.6 shows the impact of the various percentages of MPCM have on the compressive strength of all mixtures by considering the same w/b ratio. It shows that the compressive strengths mainly reduce between 20% and 40% for those mixtures having 20% of MPCM volume fraction added, while significantly larger loss (more than 50%) occurred when 40% of MPCM were substituting the cement paste. The reduced mechanical properties of MPCM pastes are mainly due to either the low strength of the MPCM capsules, especially under failure controlled by compression stress states, or also to the weak interface between MPCM hydrophobic capsules and the surrounding cement matrix.

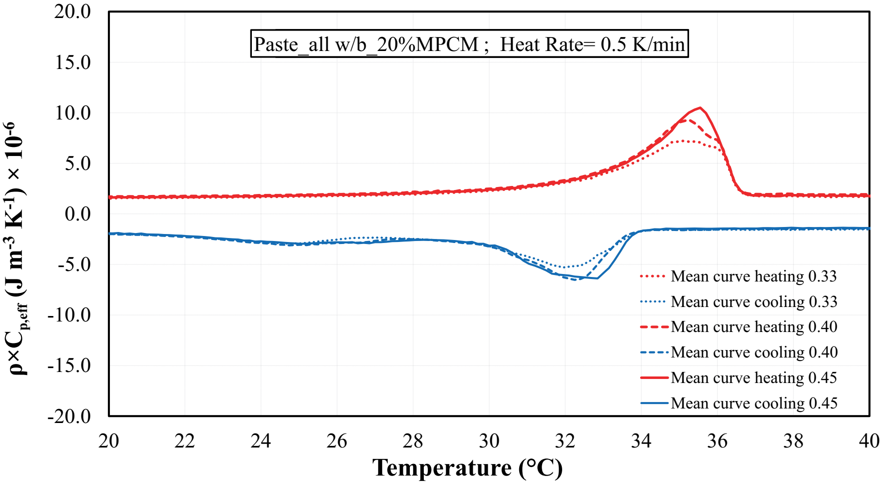


Fig. 4.4 Results of DSC measurements in terms of ρ × C_p of cement pastes with 20% MPCM volume fraction obtained with a heating/cooling rate of 0.5 K/min

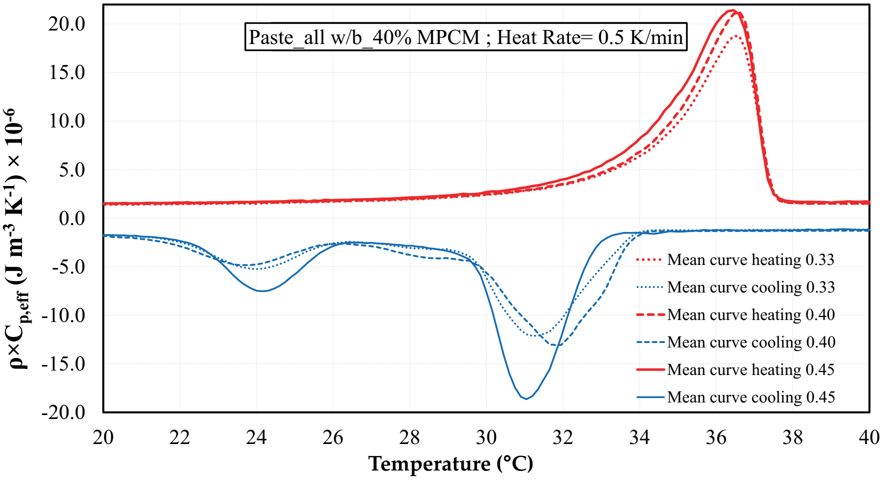
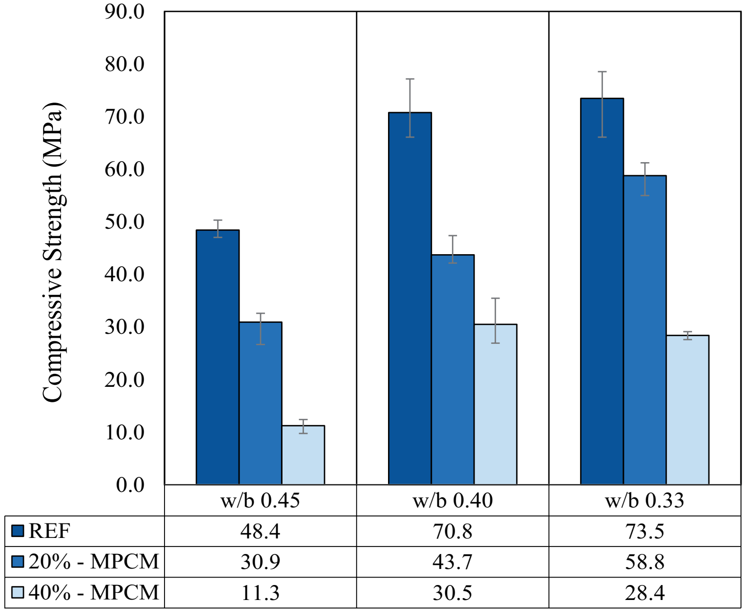


Fig. 4.5 Results of DSC measurements in terms of ρ × C_p of cement pastes with 40% MPCM volume fraction obtained with a heating/cooling rate of 0.5 K/min

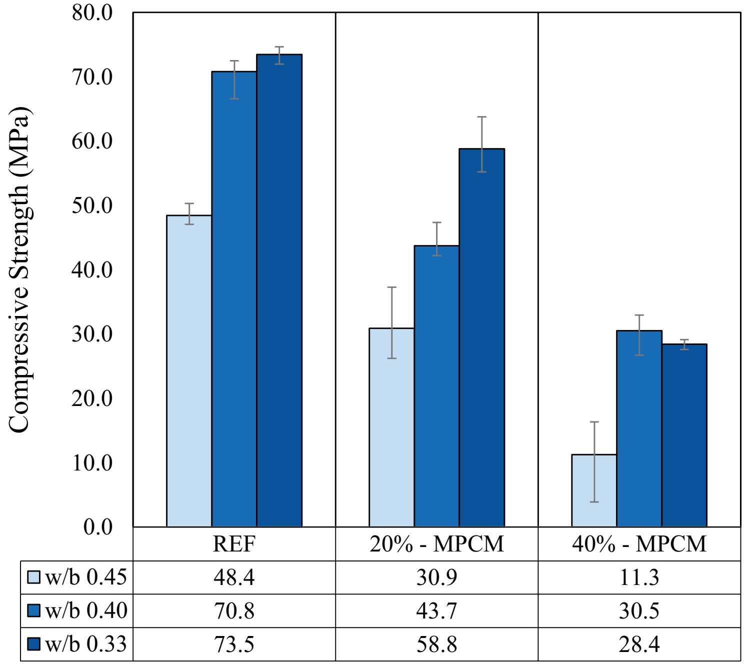
As expected, a higher percentage of MPCM added leads to a huge impact on the reduction of the mechanical properties.

Figure 4.7 plots the same results as Fig. 4.6 but this time emphasizing the impact of various w/b ratios have for fixed MPCM contents. The results show that the compressive strengths of all mixtures were influenced by the w/b ratio. Higher w/b values are characterized by comparatively lower compressive strengths. These trends were observed for all three cases REF, MPCM 20%, and MPCM 40%. Only the



	w/b 0.45	w/b 0.40	w/b 0.33
■ REF	48.4	70.8	73.5
■ 20% - MPCM	30.9	43.7	58.8
□ 40% - MPCM	11.3	30.5	28.4

Fig. 4.6 Compressive strengths (28 days) of MPCM cement pastes. Comparisons between REF – 20% MPCM and 40% MPCM. The vertical segments represent the min./max. Data scatters



	REF	20% - MPCM	40% - MPCM
□ w/b 0.45	48.4	30.9	11.3
■ w/b 0.40	70.8	43.7	30.5
■ w/b 0.33	73.5	58.8	28.4

Fig. 4.7 Compressive strengths (28 days) of MPCM cement pastes. Comparisons between w/b = 0.45, w/b = 0.40, and w/b = 0.33. The vertical segments represent the min./max. Data scatters

case w/b = 0.40 and MPCM 40% is characterized by surprisingly high strength probably due to experimental scatter/variability.

Same results and conclusions have been obtained by analyzing both tensile strengths and the compressive results at different hydration stages (3 days, 7 days, and 28 days). However, the full experimental data are omitted for brevity in this work and will be published in future works.

4.5 SEM Analysis

This section is showing some relevant analyses of MPCM paste microstructure analyzed via SEM. The analysis of these images helped to evaluate the paste morphology, dispersion homogeneity of MPCM, interfacial regions, and average size of MPCM capsules of about 25 µm. It was revealed that MPCM particles – with dimensions ranging between 15 and 30 µm (see Fig. 4.8) – have in fact spherical shapes.

The results also indicate that the hydrophobic outer shell is unfavorable to the interface between the MPCM particles and the surrounding cement paste matrix. There is almost no binding evident between the essentially hydrophobic and insoluble outer shell and the cement matrix. It can be seen that the hydrophobic nature of the capsules (made of melamine formaldehyde) leads to increase the appearance of microcracks through the paste. The internal structure of the paste with MPCM is less cohesive in comparison with the reference paste that exhibits few microcracks. However, MPCM shows a quite good distribution, without signs of ruptures and/or damages. The latter demonstrate that the MPCM can resist the process of the paste preparation, including mixing, application, and curing.

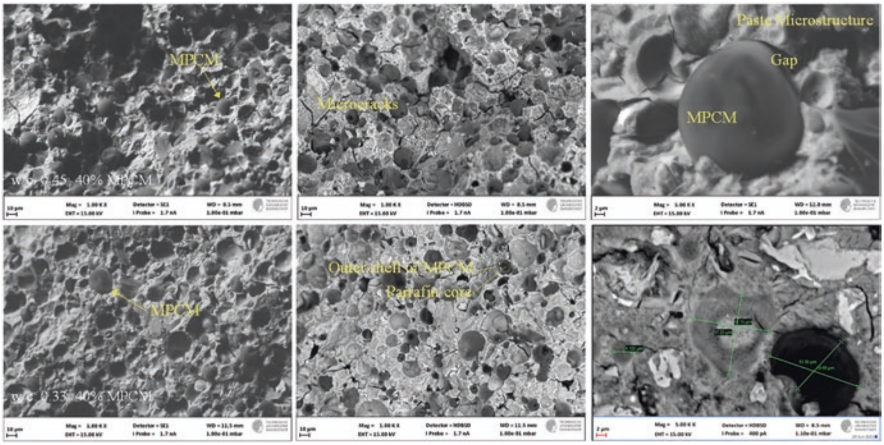


Fig. 4.8 SEM images of pastes highlighting microcracks, MPCM, and MPCM sizes/distributions

4.6 Conclusions

This work presented the results of an experimental study which investigated the thermal energy storage (TES) and mechanical performance of cement pastes made with MPCM. Several water-to-binder ratios (0.33, 0.40, and 0.45) and Nextek-37D MPCM volume fractions (0, 20, and 40%) were tested and analyzed. The results confirmed that the thermomechanical properties of cement paste can be (either positively or negatively) affected by the increasing dosages of MPCM. They positively enhance both the sensible and latent heat storage capacities of the pastes. However, MPCM can negatively affect the thermal conductivity/diffusivity of the composites and their compressive/flexural strengths.

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