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Development of ductile cementitious composites incorporating microencapsulated phase change materials

Branko Šavija¹  · Mladena Luković¹ · Geerte M. G. Kotte¹ · Stefan Chaves Figueredo¹ · Fernando França de Mendonça Filho¹ · Erik Schlangen¹

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Abstract In the past two decades, much research has been devoted to overcoming the inherent brittleness of cementitious materials. To that end, several solutions have been proposed, mainly utilizing fibres. One of the most promising classes of materials is strain hardening cementitious composite (SHCC). It utilizes PVA fibres, and it is relatively costly compared to regular concrete, so it is commonly used only in surface layers. In this paper, a multi-functional ductile cementitious composite based on SHCC has been developed. It uses microencapsulated phase change materials (PCMs), capable of reducing temperature fluctuations in the material due to their high heat of fusion. It is shown that, although addition of microencapsulated PCMs are detrimental to compressive strength, they have very little effect on the flexural strength and deflection capacity. In the future work, mixtures with higher PCM contents will be developed in order to exploit their heat storage capability better. This material has potential to reduce temperature effects on concrete surfaces, while at the same time being extremely ductile.

Keywords Ductile cementitious composite · Phase change materials · Temperature control

1 Introduction

Binders that utilise Portland cement are the most widely used building materials in the world today. Wide availability of raw materials that can be used for their

production, together with their relatively low price, is responsible for this. Furthermore, when extreme environmental and loading conditions are concerned, cement based materials have a clear advantage compared to other building materials, given their good durability. However, they are also prone to cracking [1]. Large cracks provide easy pathways for ingress of deleterious species such as water [2] and chloride [3]. This could lead to fast initiation of reinforcement corrosion [4] and end of service life [5]. Therefore, durability is the main reason why cracking in reinforced concrete should be limited. To overcome this major issue of reinforced concrete, in recent years a new type of cementitious composite called strain hardening cementitious composite (SHCC) has been proposed [6–8]. Unlike conventional mortar and concrete, which show strain softening behaviour and crack localisation when subjected to uniaxial tension, these advanced composites show strain hardening (i.e. metal like behaviour) due to their multiple cracking ability and a distribution of fine cracks. This is achieved by micromechanical design [9] and addition of a small volume of fine fibres (around 2% per volume), mostly PVA fibres [7, 10]. These composites can sustain large strains (up to 5%) which is achieved through formation of a large number of very fine cracks (typically 30–80 μm). In addition, small crack widths of SHCCs lead to improved durability in comparison to conventional cementitious materials [11–13].

Due to their excellent tensile properties, SHCCs have been mostly used in repair and retrofitting of existing structures [14–17]. Repair and retrofitting activities are good opportunities for adding functionalities to new (surface) layers of reinforced concrete structures. Consequently, much research in recent years has been devoted to developing multifunctional SHCCs which can, apart from their excellent mechanical performance, provide added

✉ Branko Šavija
b.savija@tudelft.nl

¹ Faculty of Civil Engineering and Geosciences, Delft University of Technology, Delft, The Netherlands

value to (existing) structures. For increasing long term durability of SHCC repairs, different methods of self-healing have been proposed, for example addition of bacteria [18] or super absorbent polymers [19]. Furthermore, use of piezoresistive SHCCs for self-sensing of mechanical damage has been recently proposed [20, 21].

Another interesting possibility would be to develop SHCCs which incorporate microencapsulated phase change materials (PCMs). PCMs are materials which have high heat of fusion which is able, by melting and solidifying at a certain temperature, to store and dissipate large amounts of energy in the form of heat [22]. Numerous research studies have been devoted to use of PCMs in cementitious materials to increase the energy efficiency of buildings [23, 24]. For use in cold regions, it has been proposed that suitable PCMs can increase freeze-thaw resistance of concrete [25, 26] and melt ice and snow on concrete pavements [27]. In moderate and warm climates, early age thermal cracking may be prevented by incorporating PCMs to the concrete mix [28, 29]. In both of these applications, a strain hardening cementitious composite containing microencapsulated PCMs would be of use.

One drawback of using microencapsulated PCMs in cementitious materials is that it may cause a drop in compressive strength [28, 30]. This study aims to develop a strain hardening cementitious composite that incorporates microencapsulated phase change materials as a partial substitute of one of the mixture components. The main aim of the study is to investigate whether this will affect the mechanical properties of the composite material. Specifically, the focus is on the tensile/flexural and compressive response, and whether the modified SHCC will retain its flexural strength, ductility, and multiple cracking ability.

2 Experimental program

2.1 Materials

An SHCC mixture developed by Zhou et al. [10], named M6, was used. Apart from ordinary Portland cement (OPC), this mixture contains blastfurnace slag and limestone powder as a filler. Furthermore, 2% of PVA fibres per volume are added to the mixture. The fibres had a length of 8 mm and a diameter of 40 μm . Tensile strength of the PVA fibres is 1600 MPa and their density is 1300 kg/m^3 . Note that the mixture does not contain sand or coarse aggregates. In mixtures where PCM was used, parts of the filler material (i.e. limestone) were replaced by microencapsulated PCMs. The reason was as follows: in previous studies (e.g. [28, 30]) it was noticed that, when microencapsulated PCMs were used as a substitute for binder material, a significant drop in strength resulted. If, however, it is used as a substitute for sand, this drop was significantly

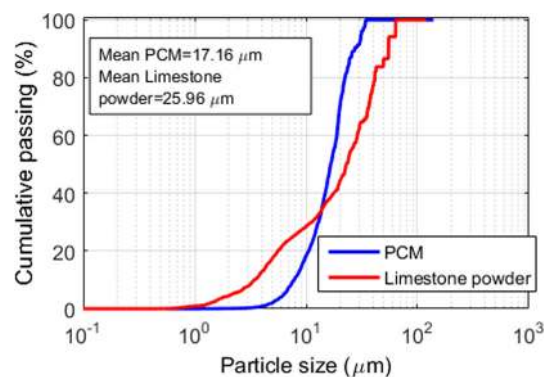


Fig. 1 Particle size distribution of limestone powder and microencapsulated PCM used

Table 1 Mixture designs used in the study

	Reference	10% PCM	20% PCM	50% PCM
Portland cement 42.5N (kg/m^3)	237	237	237	237
Blast furnace slag (kg/m^3)	553	553	553	553
Limestone powder (kg/m^3)	790	711	632	395
Microencapsulated PCM (kg/m^3)	0	27.8	55.6	139
PVA fibres (kg/m^3)	26	26	26	26
Water (kg/m^3)	411	411	411	411
Superplasticizer (kg/m^3)	6.3	6.3	6.3	6.3

lower [31]. Therefore, three different replacement levels were considered: 10, 20, and 50% of limestone per volume was replaced by microencapsulated PCMs. Given their somewhat different particle size distribution (Fig. 1), the workability of mixtures with different PCM percentages was altered. Material dosages for each of the mixtures are given in Table 1. Note that, since substitution of limestone powder by PCM was done per volume, the weight of added PCM in each mixture was significantly lower compared to the weight of limestone powder it substitutes due to a significant difference in density (2700 kg/m^3 for limestone and 950 kg/m^3 for microencapsulated PCM's).

Mixing of the ingredients was done in 1.5 l batches using a Hobart mixer. The mixing procedure was as follows: first, the dry ingredients (Portland cement, blast furnace slag, limestone powder, fibres, and PCMs where needed) were mixed for 2.5 min under low speed. Then, water (together with the dissolved superplasticizer) was added to the mix. The complete mixture was then mixed for 1 min under low speed. Finally, the complete mixture was then mixed for 4 min under high speed.

After mixing, specimens were cast. For uniaxial compression testing, 40 \times 40 \times 40 mm^3 specimens were

prepared. For bending tests, thin slabs (approximately $180 \times 190 \times 7 \text{ mm}^3$) were cast. These were then cut to size of the bending specimens prior to testing.

2.2 Experimental methods

2.2.1 Differential scanning calorimetry

Thermal properties of microencapsulated PCMs (onset and peak temperatures, enthalpy) were determined by means of a differential scanning calorimeter. The thermal program was as follows: the sample was heated from -20 to 100°C and then cooled back to -20°C in a nitrogen environment. Rate of heating and cooling was set to $5^\circ\text{C}/\text{min}$.

2.2.2 Scanning electron microscopy

The microstructure of microencapsulated PCMs and imaging of fracture surfaces was performed using a Philips XL30 Environmental Scanning Electron Microscope. The microcapsules were sprinkled on top of glass which was coated with superglue to ensure bonding, and were subsequently imaged in the secondary electron (SE) mode. Microstructures of (fracture surfaces) of SHCC incorporating the PCM microcapsules were imaged using the BSE mode.

2.2.3 Mechanical testing of the composite

Mechanical properties of different mixtures were measured at 1, 3, 7, and 28 days. After casting, specimens were sealed with plastic sheats until reaching testing age. Compressive strength was determined on $40 \times 40 \times 40 \text{ mm}^3$ cube specimens using a loading rate of 1 kN/s .

Flexural strength and deflection hardening capability of designed mixtures was determined using four-point bending tests as shown in Fig. 2. For flexural testing, coupon specimens of $180 \times 190 \times 7 \text{ mm}^3$ were cast. Prior to testing, they were cut in four, resulting in beam specimens of $120 \times 30 \times 7 \text{ mm}^3$ that were tested in four-point bending.

Loading of the sample was controlled by the displacement of the loading rig using a constant rate of 0.01 mm/s . In stress-displacement curves, therefore, displacement of the loading rig is given. Specimens were loaded until the load started

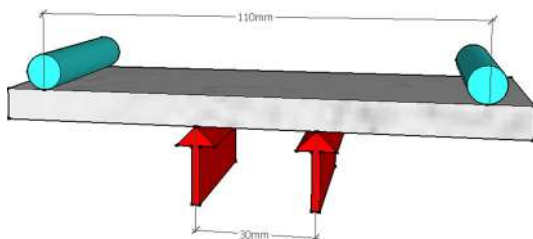


Fig. 2 Four-point bending test setup

decreasing. For each specimen, flexural strength and flexural deflection capacity were determined as shown in Fig. 3.

2.3 Functional testing

In order to assess the influence of microencapsulated PCM, temperature evolution in the material was measured over time. For each mixture, a specimen of $40 \times 40 \times 160 \text{ mm}^3$ was cast in styrofoam molds, then wrapped in bitumen/aluminium foil and stored in laboratory conditions for more than 6 months. In addition, a thermocouple was cast inside each of the specimens in order to monitor temperature. It was positioned exactly in the middle of the specimen in all three directions. Mature specimens were subjected to a heating cycle (from 10 to 30°C) inside of a freeze-thaw chamber to observe the effect of PCM addition on the internal temperature during heating.

3 Results and discussion

3.1 Characterization of PCM microcapsules

Differential scanning calorimetry (DSC) curves of the PCM microcapsules are shown in Fig. 4 for both the heating and the cooling regime. The heat of fusion during

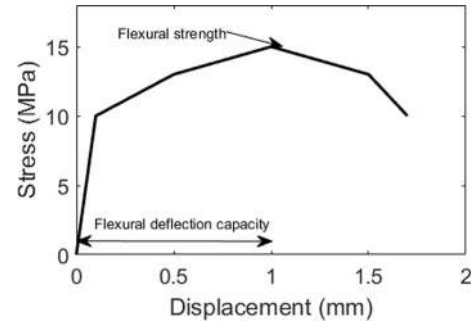


Fig. 3 Definition of flexural strength and flexural deflection capacity as determined by four-point bending tests

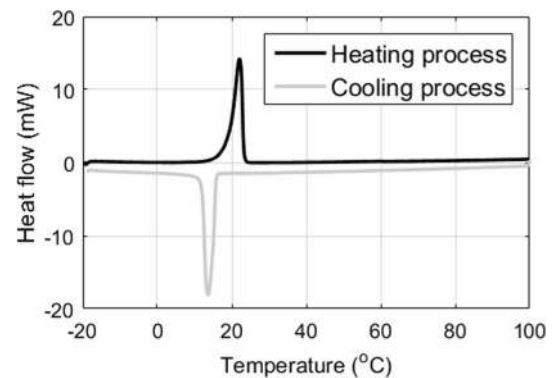


Fig. 4 DSC thermograph of the PCM microcapsules

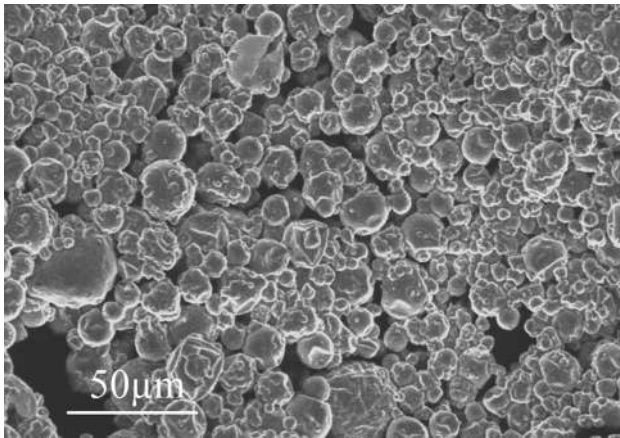
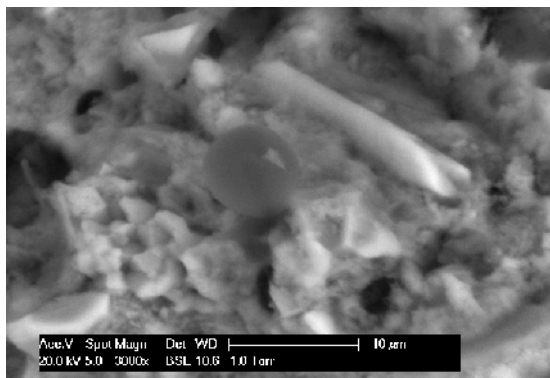


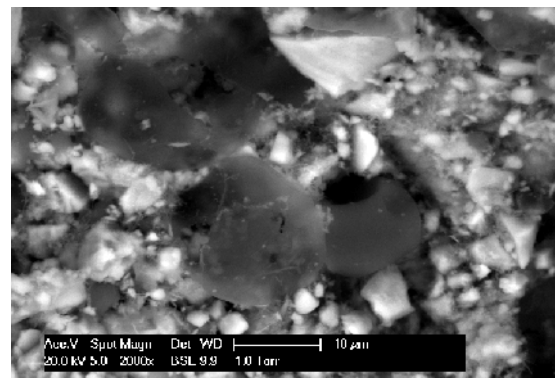
Fig. 5 A micrograph of dispersed PCM microcapsules

the phase change was determined as the area under the heat flow curve during the phase transition. Measured heat of fusion was 146.7 J/g, which corresponds well with the value provided by the manufacturer (143.5 J/g). The onset of phase change corresponding to melting is measured at 19.07 °C, and the endothermic peak at 22.07 °C.

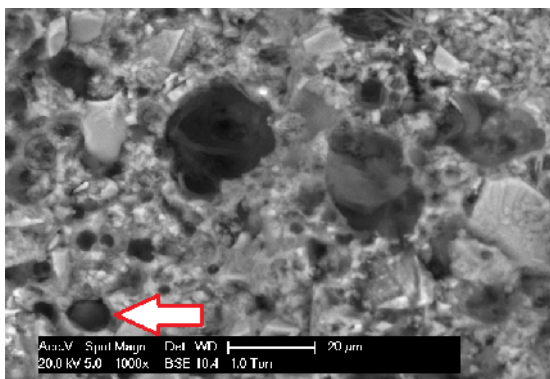
In order to observe individual PCM microcapsules, they were sprinkled on a superglue layer on top of a glass substrate and placed inside the SEM chamber. Imaging was performed using the secondary electron mode, acceleration voltage of 7 kV and 200× magnification. A micrograph of microencapsulated PCMs is shown in Fig. 5. It can be seen that microcapsules are spherical in shape with a range of different diameters, which is beneficial for proper dispersion inside the cementitious matrix [31].



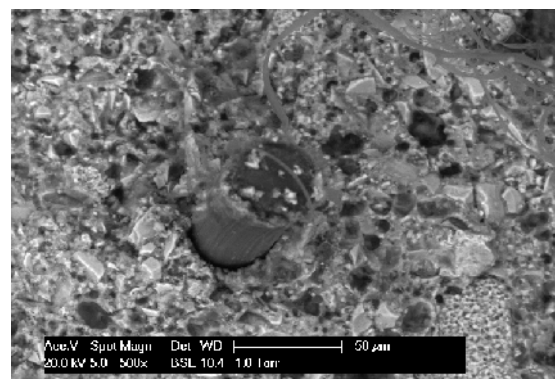
(a) Intact microcapsule in a 28-day 50%PCM specimen



(b) Dispersed microcapsules in a 3-day 50%PCM specimen



(c) Microcapsules in a 28-day 50%PCM specimen (intact PCM microcapsule is marked)



(d) Broken PVA fibre in a 28-day 50%PCM specimen

Fig. 6 Micrographs of fracture surfaces of SHCC specimens incorporating PCM microcapsules. **a** Intact microcapsule in a 28-days 50% PCM specimen. **b** Dispersed microcapsules in a 3-days 50% PCM specimen. **c** Microcapsules in a 28-days 50% PCM specimen

(intact PCM microcapsule is marked). **d** Broken PVA fibre in a 28-days 50% PCM specimen

It is also of interest to observe the PCM microcapsules inside the microstructure of the SHCCs. For this purpose, selected specimens that were subjected to mechanical testing (as described in Sect. 3.2) were split along their largest crack and the fracture surface was observed in the ESEM using the BSE mode. Selected micrographs are shown in Fig. 6. In Fig. 6a, an intact microcapsule can be seen. In Fig. 6b, multiple PCM microcapsules are visible, and some of them appear damaged by the fracture (either during the mechanical testing or specimen preparation). Figure 6c shows broken microcapsules but also an intact microcapsule (marked with an arrow), which seems to have deflected the crack. Figure 6d shows a fibre that was broken, possibly when the manual splitting was performed. From the micrographs it can be seen that a large proportion of the microcapsules that are at the fracture surface break during the mechanical loading. This is probably due to the low strength and stiffness of the microcapsules compared to the matrix, which determines the crack path during fracture propagation [32]. Breakage of the microcapsules will cause the release of the encapsulated phase change material. As this material may leak through the crack (when the temperature is above the phase change temperature), it has to be noted that the total heat storage capacity

of this type of SHCC may be somewhat diminished compared to the uncracked condition. However, this was not quantified in the current study.

3.2 Mechanical properties

Development of compressive strength in time as a function of different levels of microencapsulated PCM addition is shown in Fig. 7. It can be clearly observed that limestone powder replacement by microencapsulated PCMs does lead to a reduction in compressive strength. It is clear that higher levels of replacement lead to higher strength loss for all tested ages: this is expected when a hard filler (i.e. limestone powder) [33] is substituted by softer inclusions (microencapsulated PCM) [28, 30]. The same trend was reported previously for mortars where microencapsulated PCM was used to partially replace fine aggregate (sand) [31]. For the highest replacement level (50%), the (average) compressive strength drops, compared to the

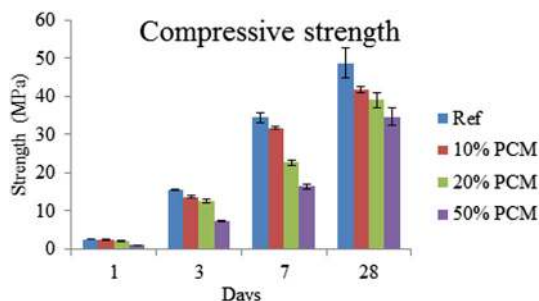


Fig. 7 Compressive strength as a function of time for SHCC mixtures containing different amounts of microencapsulated PCM (error bars indicate standard deviation)

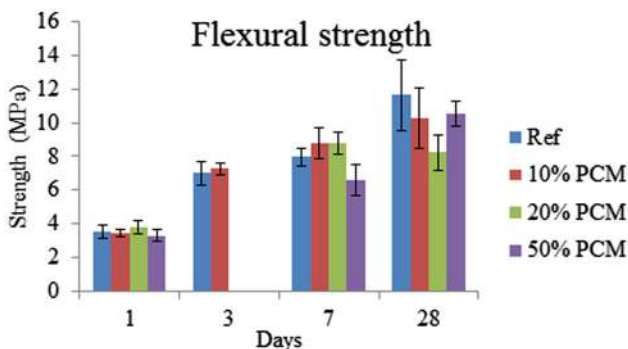


Fig. 8 Flexural strength as a function of time for SHCC mixtures containing different amounts of microencapsulated PCM (error bars indicate standard deviation)

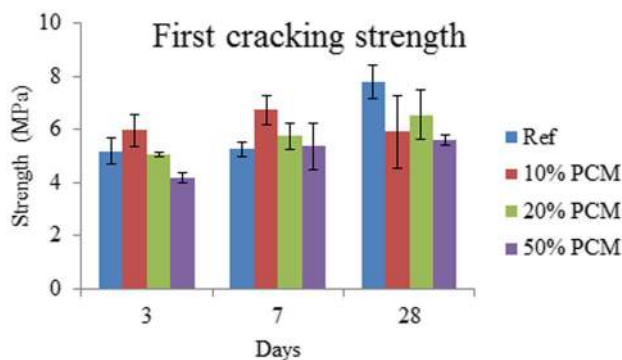


Fig. 9 First cracking (flexural) strength as a function of time for SHCC mixtures containing different amounts of microencapsulated PCM (error bars indicate standard deviation). Note that 1 day is not included because first cracking strength cannot be unambiguously determined

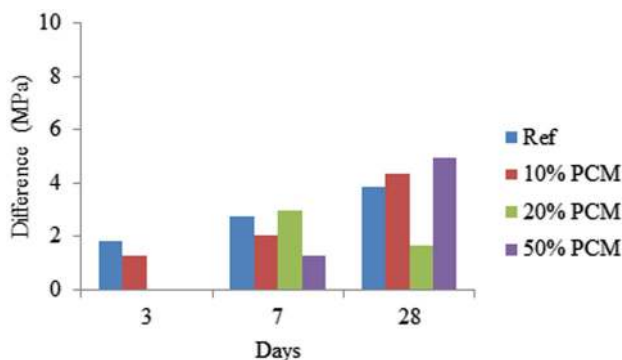


Fig. 10 Difference between average flexural strength and first cracking (flexural) strength as a function of time for SHCC mixtures containing different amounts of microencapsulated PCM (error bars indicate standard deviation). Note that 1 day is not included because the first cracking strength is not clear

reference, by 62.5, 52.4, 52.4, and 28.9% for 1, 3, 7, and 28 days respectively. Therefore, it seems that the decrease in compressive strength is reduced with increasing curing time. This is probably related with improvements of the bond between microencapsulated PCMs and the matrix which results from prolonged hydration and latent hydraulic reaction of blastfurnace slag.

Previous studies using similar microencapsulated PCM materials [28, 31] have suggested that the drop in flexural strength due to PCM addition is less pronounced compared to the drop in compressive strength. Development of flexural strength as a function of time is shown in Figure 8.

Unlike the compressive strength development, where a clear drop can be observed for increased PCM replacement levels, flexural strength seems to be only mildly affected. For example, the highest PCM replacement level (50% PCM) results in a drop of (average) flexural strength compared to the reference mixture by 6.5, 1.2, 16.9, and 9.7% for 1, 3, 7, and 28 days respectively. These numbers lie within the statistical variability of the material itself, as

indicated by the error bars in Fig. 8. Furthermore, the trend of decrease in flexural strength with increasing PCM levels is not consistent. Therefore, it cannot be said that PCM addition causes a clear decrease in flexural strength. This can probably be explained by the nature of the SHCC material itself. Unlike conventional cementitious materials, such as mortar and concrete, which exhibit strain softening after crack localization [1], SHCCs exhibit a pseudo-plastic response [6]. Tensile (or flexural) response of SHCC will not depend on the strength of the matrix *only*, but also on the fibre-matrix bond. Therefore, it is possible that the first cracking strength (Fig. 9) will be influenced to a certain extent by a weaker matrix caused by the addition of microencapsulated PCMs. This is consistent with an observed correlation between flaw size and first cracking strength in SHCC [34]. After the crack formation, it will be bridged and arrested by PVA fibres, which will prevent (macro)crack localization and cause formation of a new crack elsewhere in the sample. This process will repeat multiple times until the specimen is saturated with cracks



Fig. 11 Flexural load-displacement curves of different mixtures tested after 1 day

and ultimate failure occurs. Therefore, the flexural strength will not depend only on the matrix strength, but also on the fibre/matrix interface and the ability of the material to sustain multiple cracking. In Fig. 10, the difference between the maximum stress and the first cracking stress is shown. This difference seems to be increasing with the increase in PCM addition, suggesting that the strength contribution of fibres and multiple cracking may be higher in mixes containing more PCM microcapsules. Similar trend was observed by Li [35], who used polystyrene beads to create artificial flaws in the SHCC matrix to induce multiple microcracking.

One of the main features of SHCC is its quasi-plastic behaviour in tension and flexure. All stress/displacement curves are shown Figs. 11, 12, 13, and 14 for 1, 3, 7, and 28 days, respectively.

At 1 day (Fig. 11), all specimens are quite soft and show little deflection hardening. They do exhibit multiple cracking, with very fine cracks forming and no clear drops can be observed in stress-displacement curves.

After 3 days, all mixtures show clear deflection hardening behaviour (Fig. 12). Drops in the load displacement curves, which signify occurrence of cracking, can be clearly observed. Specimens incorporating microencapsulated PCM show behaviour similar to reference specimens irrespective of the PCM dosage. Due to the settings of the loading rig, however, the 20% and the 50% PCM specimens reached the maximum displacement before the stress started decreasing.

Similar behaviour is observed for specimens tested after 7 days (Fig. 13). All specimens show deflection hardening behaviour, and it is not influenced by substitution of limestone powder by PCM microcapsules. Multiple successive microcracking is observed in all cases.

Specimens tested after 28 days also show deflection hardening behaviour typical of SHCC materials (Fig. 14). As cementitious materials are commonly designed to reach certain properties after 28 days, it is very important that the mixtures incorporating microencapsulated PCMs remain ductile and show multiple cracking also at this age.

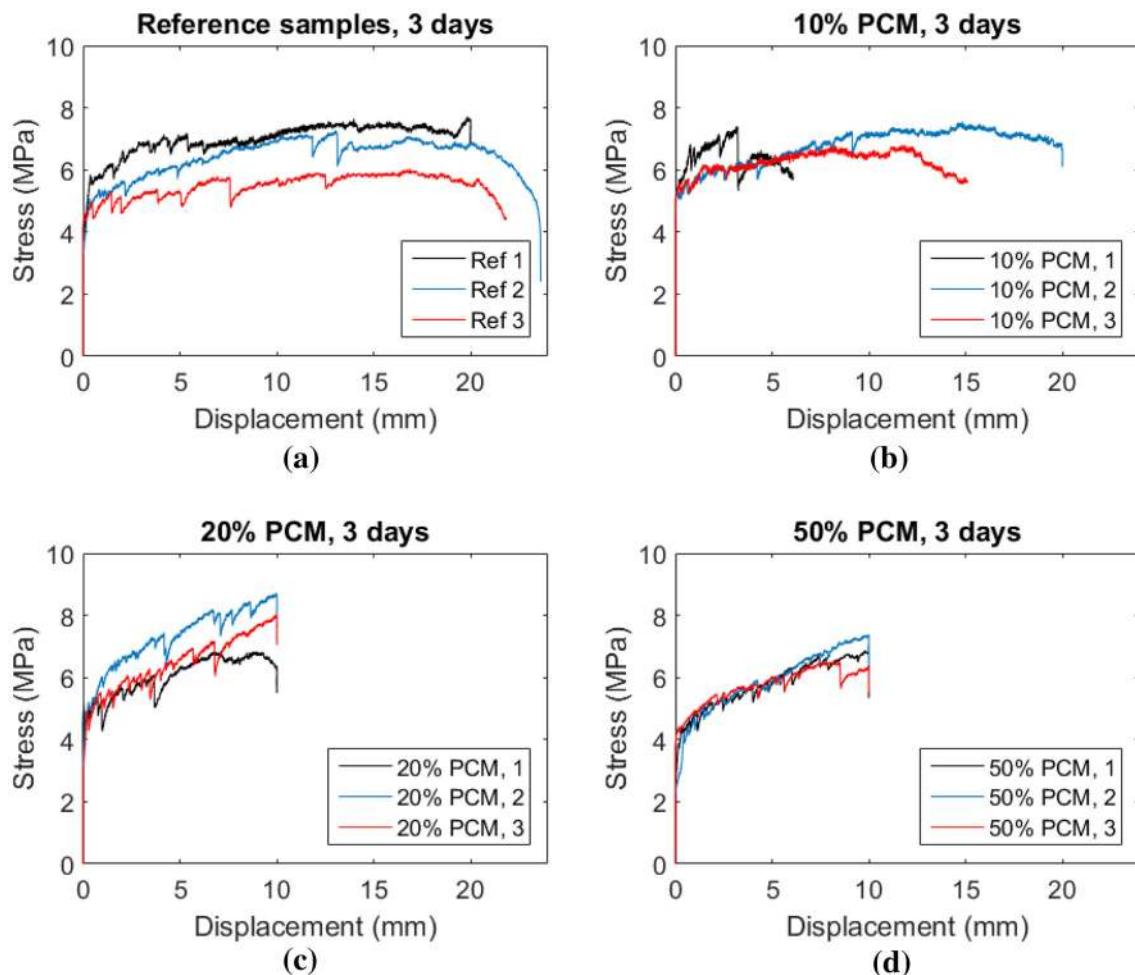


Fig. 12 Flexural load-displacement curves of different mixtures tested after 3 days (Note that 20 and 50% PCM specimens reached the maximum displacement of the machine for the settings used)

After 28 days, all mixtures exhibit multiple cracking when subjected to four-point bending. This can be seen in Fig. 15. No significant difference can be observed between the reference mixture and mixtures with PCM addition.

Another important parameter for characterization of SHCCs is their deflection (or, in uniaxial tension, strain) capacity (Fig. 3). The deflection capacity of SHCC is, in general, very high, and can be of great importance when certain loading types such as e.g. impact loading [36] or cyclic loadings [37] such as earthquakes are concerned. It is therefore crucial that any modifications of the material do not compromise this capability. Deflection capacity for all tested ages and mixtures is shown in Fig. 16. It can be seen that, apart from the reference mixture tested at 3 days which seems to be an outlier with exceptionally high deflection capacity, all mixtures have a similar deflection capacity at a certain age. The deflection capacity increases from 1 to 3 days, then further increases at 7 days, but shows a decrease at 28 days. This is probably a consequence of decrease in the elastic modulus of the matrix: a

stiffer material will deflect less under load compared to a less stiff specimen. This is a well known phenomenon in SHCC. It should be noted that the deflection capacity could further decrease after 28 days due to the relatively slow hydraulic reaction of blast furnace slag [38].

3.3 Temperature evolution

Figure 17 shows the measured temperature as a function of time for all specimens. In addition, the imposed chamber temperature during the cycle is plotted as a function of time.

A small difference between the reference specimen and specimens incorporating microencapsulated PCMs can be seen. For the 10% PCM and the 20% PCM specimens, the difference seems rather small. The onset of temperature rise in these specimens is rather similar to the reference specimen, just somewhat delayed: this could be either due to somewhat better insulation of these specimens compared to the reference, or their relative position in the (large)

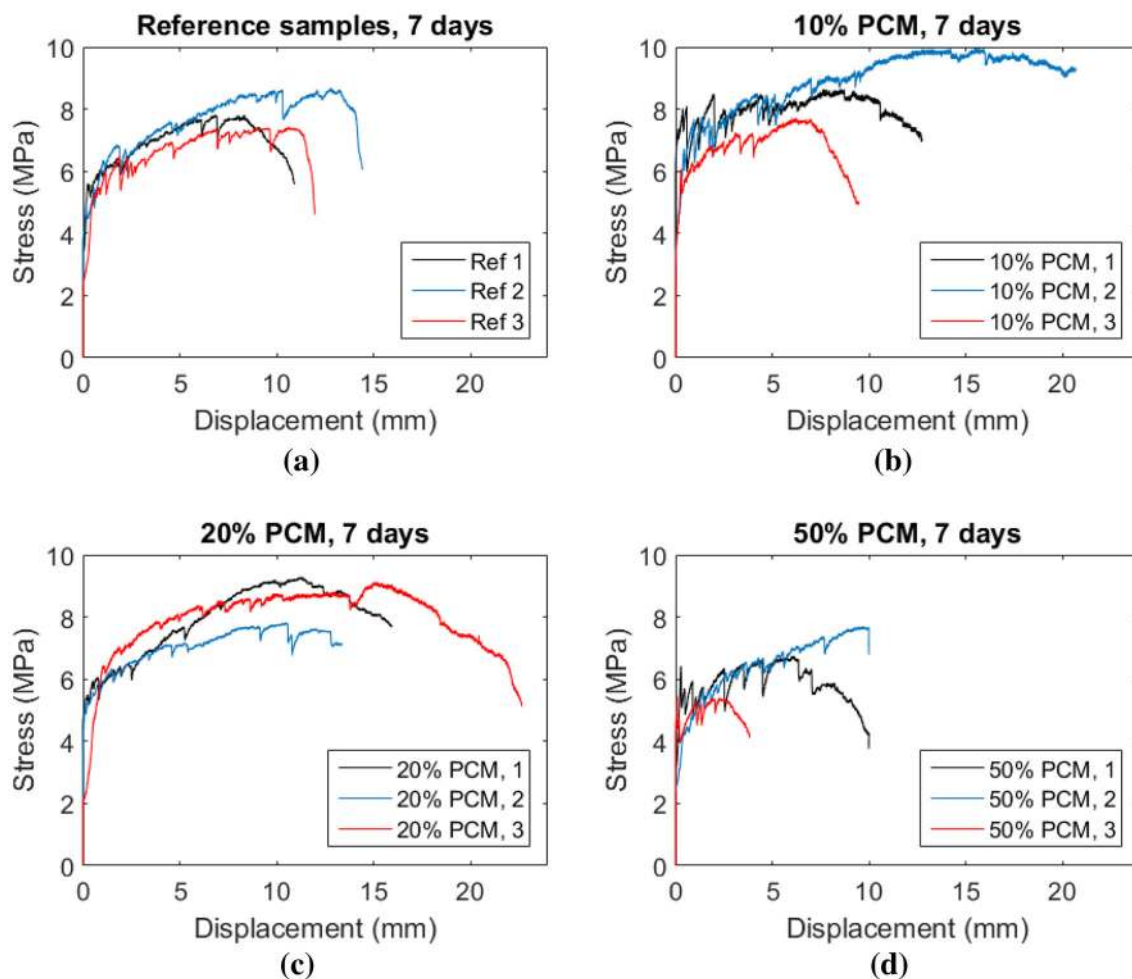


Fig. 13 Flexural load-displacement curves of different mixtures tested after 7 days

freeze-thaw chamber, which was somewhat more distant from the heaters in the chamber. The influence of microencapsulated PCMs on heating rate in these two specimens seems quite limited. This can probably be attributed to relatively low content of PCMs: 27.8 and 55.6 kg/m³ for the 10 and 20% mixtures, respectively. In their study, Sakulich and Bentz suggested PCM addition levels of 50–120 kg/m³ of concrete, as the optimum and the maximum quantity, respectively [39]. Farnam et al. [27] used 150 kg/m³ of PCM in their study. Note that both of these studies focused on PCMs impregnated in light-weight aggregates, not microencapsulated PCMs. For microencapsulated PCMs, Hunger et al. [30] used 23–113 kg/m³ with satisfactory results. This is, of course, also related to the heat of fusion of PCMs: the higher the heat of fusion, the lower the quantity of PCMs needed [40].

In Fig. 18, a part of the plot shown in Fig. 17 is zoomed-in and shown for the two extreme cases, i.e. the reference specimen and the specimen 50% PCM specimen. A clear difference can be seen in terms of temperature rise. Around the phase change temperature, it can be seen that the

temperature increase is slower as the PCMs melt and absorb some of the heat. Therefore, the quantity of microencapsulated PCMs in this case (139 kg/m³) is probably more practical.

It is therefore necessary to make a balance between desired mechanical properties (compressive strength) and functionality: higher PCM additions do provide more capacity for heat capture, but with a reduced compressive strength as a consequence.

4 Conclusions

This paper presented a development of ductile cementitious composites that incorporate microencapsulated phase change materials (PCMs) as functional additions. The PCMs themselves were characterized in terms of their particle size distribution, phase change temperature, heat of fusion, and microstructure. They were used to replace a part of the limestone filler in a commonly used SHCC mixture. Newly developed SHCC mixtures were

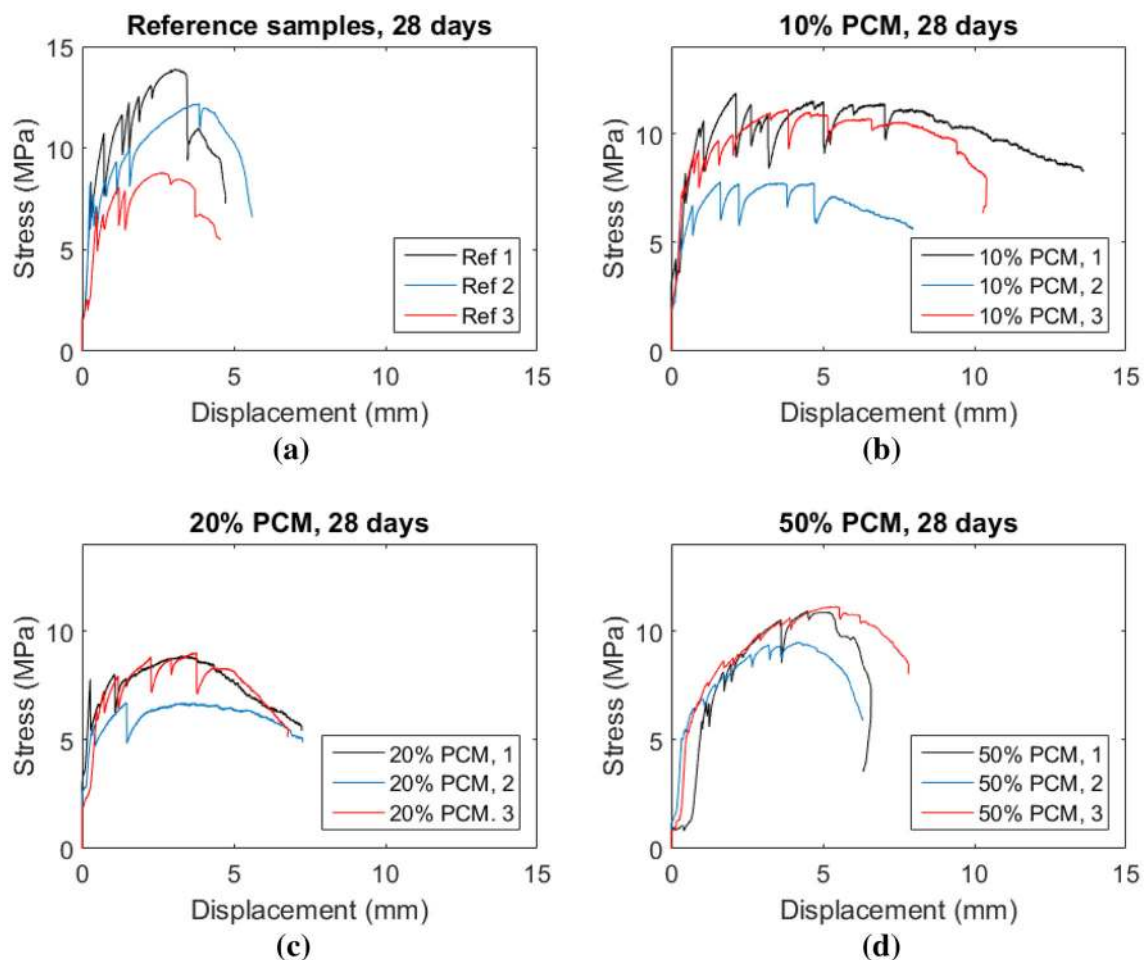


Fig. 14 Flexural load-displacement curves of different mixtures tested after 28 days

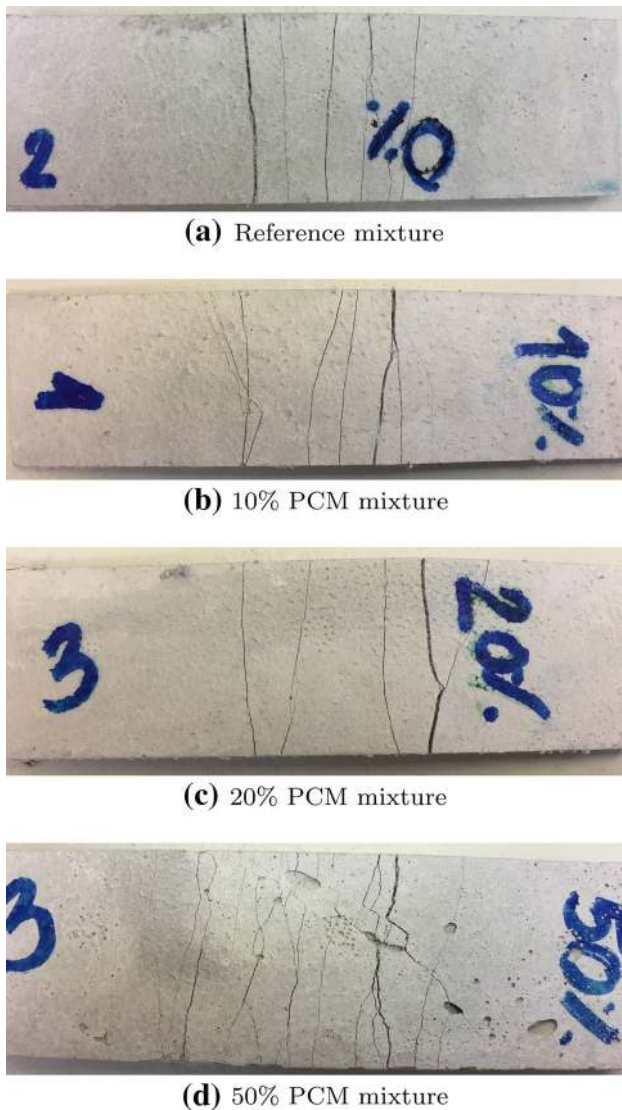


Fig. 15 Cracks at the tension side of SHCC specimens with different PCM additions occurring after testing at 28 days. **a** Reference mixture. **b** 10% PCM mixture. **c** 20% PCM mixture. **d** 50% PCM mixture

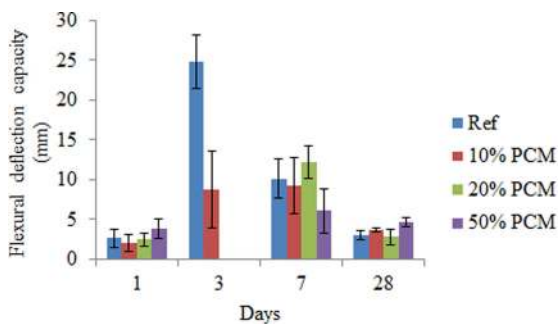


Fig. 16 Deflection capacity in four-point bending as a function of time for SHCC mixtures containing different amounts of microencapsulated PCM (error bars indicate standard deviation)

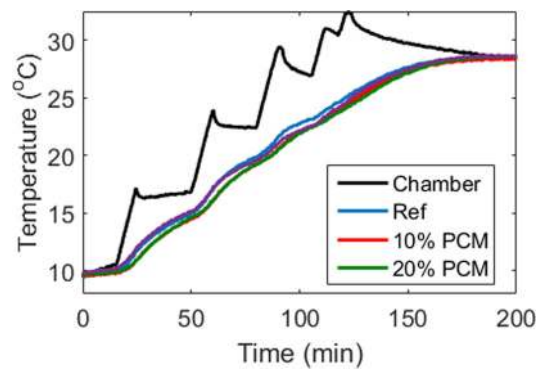


Fig. 17 Measured temperature in all specimens during the heating cycle. Rectangle marks the zoomed-in area shown in Fig. 18

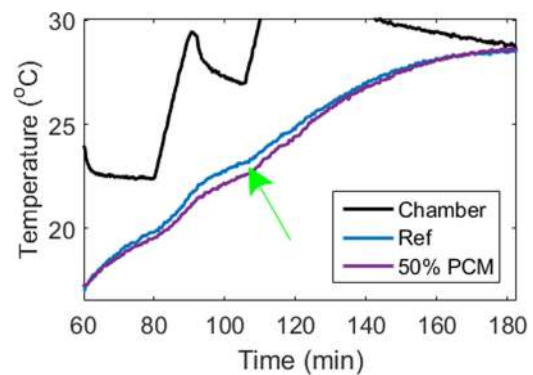


Fig. 18 Measured temperature in reference and 50% PCM sample at a point of phase change. Green arrow marks the temperature difference caused by PCMs

characterized in terms of microstructure, mechanical properties (compressive strength, bending strength, deflection capacity), and functionality in terms of heat capturing capacity during heating.

Microencapsulated PCMs used in this study have a mean particle size of 17 μm , which is smaller than the mean particle size of the limestone powder used (26 μm). Therefore, the workability of the mixture is affected when limestone is replaced by PCMs, so the amount of superplasticizer was increased for all mixtures compared to the original mixture developed by Zhou et al. [10] (by approx. a factor of four). Microcapsules are roughly spherical in shape, and do not agglomerate, which is beneficial for their dispersion in the matrix.

Based on the presented results, it can be concluded that, as expected, a partial replacement of hard filler phase (limestone) with soft inclusions (microencapsulated PCMs) has a negative effect on the compressive strength of the composite. The decrease in compressive strength is proportional to the PCM addition, and is present for all ages tested. On the contrary, flexural strength is not much

affected by the PCM addition, being reduced by no more than 15% for the highest replacement ratio at any age tested. Flexural deflection capacity of the mixtures is also unaffected by PCM incorporation. All mixtures do retain deflection hardening behaviour and multiple cracking capability. In terms of heat capture due to PCM incorporation, only the mixture with the highest amount of PCMs showed a significant difference compared to the reference. The amounts of PCMs used in this study can, therefore, be considered conservative. As the most important mechanical properties of SHCC have been completely preserved (i.e. ductility and multiple cracking capacity) in mixtures with PCM incorporation, further studies will focus on developing mixtures with higher levels of microencapsulated PCMs (up to 100% limestone replacement by PCM) in order to capture more heat and increase the functionality. It is expected, however, that compressive strength will be even more affected in this case.

Microencapsulated PCMs used in this study had a phase change temperature of around 22 °C, which makes them applicable for promoting thermal comfort in buildings or reducing early age temperature rise in concrete. If, however, the phase change material would be changed, for example by having a phase change temperature around 2–5 °C, this type of SHCC material could be used in bridge overlays to reduce freeze-thaw damage and possibly even melt snow. SHCC mixtures with incorporating such low temperature PCMs will form a part of a future study.

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