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Research Article

Effect of phase change material on mechanical and thermal properties of cementitious composites

Erdinç Halis ALAKARA [1,2*], İlhami DEMİR [3], Cemil ALKAN [4]

[1]Institute of Science, 71451 and Kırıkkale, TURKIYE
[2]Department of Civil Engineering, Tokat Gaziosmanpasa University, 60150 and Tokat, TURKIYE
[3]Department of Civil Engineering, Kırıkkale University, 71451 and Kırıkkale, TURKIYE
[4]Department of Chemistry, Tokat Gaziosmanpaşa University, 60150 and Tokat, TURKIYE

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Abstract

Cleaning the snow/ice formed on the pavement surface during winter months causes great time loss and high labor costs. In this context, phase change materials (PCM) can be considered as an effective alternative for cleaning the snow/ice on the pavement surface. In this study, microencapsulated PCM (mPCM) was added to cement mortars at 0%, 2%, 4%, 6%, 8% and 10% by weight of cement. The prepared cement mortars' flexural strength, compressive strength, and ultrasonic pulse velocity were investigated. In addition, to observe the effect of mPCM on thermal properties, reference, and 6% mPCM, added mortar specimens of 100 mm × 100 mm × 20 mm were produced, and thermocouples were placed inside them. As a result, it was observed that the mechanical properties of cement mortars decreased as the mPCM ratio increased. However, the mechanical strength of cement mortars remained sufficient for many structural applications. Thermal property tests showed that mPCM-added mortars heated and cooled down slower than the reference mortar. This is important, especially in regions with a very high day-night temperature difference. Cracking, one of the biggest problems seen in cement-based pavements and caused by the day-night temperature difference, can also be reduced with mPCM.

Keywords

Phase change materials, Thermal energy storage, Mechanical properties, Thermal properties, Cementitious composites, Freezing-thawing cycle

**** Corresponding Author: erdinchalis.alakara@gop.edu.tr***

1. Introduction

Safety and efficiency of transportation are important everywhere, and therefore, roads need protection from climatic hazards. Since concrete pavements constructed under cold climate conditions are exposed to frequent freezing and thawing, their mechanical and durability properties decrease, and their service life decreases accordingly. This situation causes concrete pavements to be renewed frequently and causes labor and time loss. On the other hand, snow and ice accumulated on road surfaces in winter months cause traffic accidents and significant economic losses. Due to the conditions such as the continuous occurrence of all these negativities, increasing the service life of concrete pavements, and ensuring safe traffic intervals, it is essential to intervene in the snow and ice in time and melt the pavement by removing it (Bednarska & Koniorczyk, 2020; Chi et al., 2019; Ding et al., 2023; Farnam et al., 2017; Lai et al., 2014; Wang et al., 2022; Yeon & Kim, 2018).

Snow and ice are frequently cleared from the pavements to protect people and vehicles in traffic during wintertime. Traditional methods such as snow shoveling, de-icing chemicals, and salts are generally preferred for clearing snow and ice from the pavement surface. However, these methods have many disadvantages, such as being costly, requiring intensive labor, not being environmentally friendly, and causing severe damage to the pavement surface (Farnam et al., 2015, 2017; Shi et al., 2010). Currently, the preferred method for melting snow and ice is de-icing salts. Salts sprinkled on the pavement surface penetrate the pavement by moving through the cracks on the pavement surface together with the melting snow/ice water. This causes flaking on the pavement surface in the later stages and corrosion of metal parts in the pavement and infrastructure (Carmona et al., 2015; Chi et al., 2019; Xu & Tan, 2012). To overcome these problems, scientists have proposed different cleaning methods with heating power to remove snow and ice from the pavement surface, such as the heating cable method, the high-temperature gas cleaning method, the geothermal dissolving method, and the electrothermal dissolving method, conductive concrete (Daniels & Heymsfield, 2020; Gao et al., 2016; Liu et al., 2019; Tumidajski et al., 2003). However, because the whole world is struggling with an energy crisis and significant amounts of energy are consumed in heating and cleaning methods, it is seen that these methods have some disadvantages in meeting the current demand. Moreover, there are problems in applying these methods (Gao et al., 2016; Zhang et al., 2009).

In recent years, phase change materials (PCMs) have been popular as thermal energy storage systems in built environments (Çaktı et al., 2022; Dai et al., 2021; Guardia et al., 2020; Jeon et al., 2013; Z. Li & Yuan, 2021). PCMs are materials that can store and release latent heat energy isothermally. PCMs having solid-liquid phase change properties at ambient temperatures, store energy during melting, and release it back into the environment during solidification (Gbekou et al., 2022). PCMs added to cement-based composites can be used to reduce damage caused by freeze-thaw in cement-based materials (Bentz & Turpin, 2007), relieve internal stresses in concrete structures by reducing temperature fluctuations (Sakulich & Bentz, 2012), melt snow and ice on pavement surfaces (Farnam et al., 2017; Sakulich & Bentz, 2012), and control thermal changes resulting from hydration (Arora et al., 2017). Using PCM in road pavements can prevent snow and ice formation on the pavement surface. Moreover, by reducing the use of de-icing salt and chemicals, the service life of the pavement and infrastructure parts can be increased. Significant savings are made in the labor and time required for snow removal. In addition, a more environmentally friendly solution can be offered due to the reduced de-icing of salt and chemicals (Acıkök et al., 2023; Farnam et al., 2016, 2017; Yu et al., 2023).

The effect of microencapsulated PCM (mPCM) additive on the physical, mechanical and microstructure properties and thermal behavior of cementitious composites has been investigated with great interest in recent years. However, it is thought that this issue still needs to be investigated. In this study, an mPCM that has the ability to melt snow/ice and has a phase transition temperature of around +4 C was used. The mPCM used was used in a previous study. However, the amount of mixing water was kept constant in that study. In the study presented, the amount of mixing water was reduced by keeping the spreading diameter of the mortars constant. In this way, it was aimed at reducing the decreases in mechanical properties. The aim of the study is to investigate the effect of mPCM on the mechanical and thermal properties of cementitious composites. mPCM was added to the mortar mixtures at the rates of 0%, 2%, 4%, 6%, 8%, and 10% of the cement weight. To determine the fresh and hardened properties of the produced composites, the spreading diameter, flexural and compressive strength, and ultrasonic pulse velocity (*UPV*) tests were performed. In addition, to investigate the effect of mPCM additives on cementitious composites' thermal properties, the mortar specimens' internal temperature was monitored using a freeze-thaw chamber. The findings obtained from the study showed that mPCM can be used to regulate the temperature fluctuations of cementitious composites.

1.1. Symbols and Abbreviations

phase change materials: PCMs
microencapsulated PCM: mPCM
Portland Cement: PC
Ultrasonic pulse velocity: *UPV*
differential scanning calorimetry: DSC

2. Materials and method

2.1. Materials

CEN standard sand was used as aggregate in cement mortars. Tap water was used for preparations. CEM I 42.5 R type Portland Cement (PC) (provided by ADOCIM Cement factory) conforming to Turkish standards (TS EN 197-1, 2012) was used as a binder. The physical and chemical properties of the used PC are given in Table 1.

Table 1. Physical and Chemical Properties of Cement.

Chemical composition	**(%)**
SiO_2	20.85
Al_2O_3	5.97
Fe_2O_3	4.02
CaO	62.21
MgO	1.10
Na_2O	0.16
K_2O	0.29
SO_3	3.21
Physical properties	
Specific gravity (unitless)	3.12
Blaine fineness (cm^2/g)	3460
Loss on ignition	1.38

The mPCM used in the study is paraffin wax supplied by MikroCaps Company. The technical information of mPCM provided by the company is shown in Table 2. mPCM is supplied as a liquid solution. In this way, it is distributed more homogeneously in cement mortars. The heat storage properties of mPCM because of DSC analysis are given in Table 3. mPCM added to cement mortars will significantly reduce the temperature changes of mortars during freezing-thawing and will melt snow/ice while releasing the stored energy.

Table 2. Technical Specifications of mPCM

Classification	Phase Change Materials Microcapsule dispersion
Type of membrane	Melamine-formaldehyde
Type of PCM	Paraffin wax
PCM content in the dispersion	25 – 30%
PCM content in dry capsule	75–80%
Dry content in the dispersion	35–38%
pH	7.0–9.0
Density	900–970 g/L
Viscosity (at 25 °C)	10–1000 cPs
Appearance	White slurry

Table 3. Thermal Performance Properties of mPCM After DSC Analysis.

	Initial Temperature (°C)	**Final Temperature (°C)**	**Enthalpy (J/g)**
Melting Point	4.3	17.3	170.1
Freezing Point	1.3	− 12.2	− 169.3

2.2. Preparation of mixtures

Mortar mixtures were prepared according to TS EN 196-1 (TS EN 196-1, 2016) standard. During the mixing process, PC and water were first mixed. Then, sand was added to the mixture and mixed at high speed. The mixer was restarted, and mPCM was added to the mixture, and mixing continued until a homogeneous mixture was obtained. Adding mPCM to the mixture was last to reduce the risk of damaging the microcapsules. The freshly produced mortars were placed in molds with dimensions of 40 mm × 40 mm × 160 mm to test the mechanical properties. Mortar specimens with dimensions of 100 mm × 100 mm × 20 mm were produced to observe the effect of mPCM on thermal properties. Thermocouples were placed inside these specimens, and the internal temperature changes of the mortars were observed during the freezing-thawing test. The mortars were removed from the molds after 24 hours and cured in 20 ± 2

°C water until the test day (3, 7, and 28 days). The mixture content of the mortars is given in Table 4. The PCM dispersion ratio was adjusted according to the required amount of mPCM, assuming that the PCM content in the dispersion was 27.5% (varied from 25% to 30% by weight (Table 2)). mPCM was added to the mortar mixtures at 2, 4, 6, 8 and 10% by weight of cement. While determining these ratios, studies in the literature were taken into consideration (Acıkök et al., 2023; Jayalath et al., 2016).

Table 4. Mixture Ratios of Mortars

Sample Codes	Cement (g)	Sand (g)	Water (g)	mPCM Slurry (g)	mPCM ratio (% by weight of cement)
Ref	450	1350	225	0	0
mPCM_2	450	1350	215	32.7	2
mPCM_4	450	1350	200	65.5	4
mPCM_6	450	1350	190	98.2	6
mPCM_8	450	1350	180	130.9	8
mPCM_10	450	1350	170	163.6	10

2.3. Tests of physical and mechanical properties of mortars

The workability of fresh mortars was measured by TS EN 1015-3 (TS EN 1015-3, 2000). *UPV* test of hardened mortars was carried out by TS EN 12504-4 (TS EN 12504-4, 2021). Flexural strengths were measured using 40 mm × 40 mm × 160 mm specimens. Compressive strengths were measured using specimens split into two after flexural strength. Flexural and compressive strength tests were carried out by TS EN 196-1 (TS EN 196-1, 2016), and the loading speeds of the specimens were 50 N/s and 2.4 kN/s, respectively. To investigate the effect of mPCM additive on the thermal properties of cement mortars, prismatic mortar specimens with dimensions of 100 mm × 100 mm × 20 mm were produced with mixtures coded Ref and mPCM_6. Temperature measurements of these mortars were carried out in a freeze-thaw cabinet. Temperature measurements were taken with thermocouples placed inside the mortar specimens every 1 minute with a precision of ±0.1 °C and recorded with a data logger. The photograph of the device in which the experiment was performed is given in Figure 1.

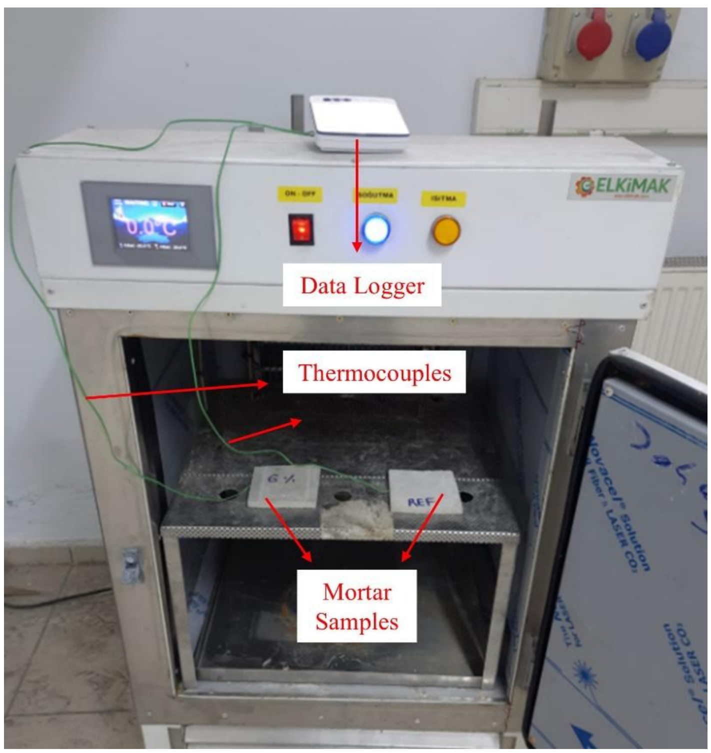

Figure 1. Experimental Setup Used To Monitor The Internal Temperature Changes of Mortars

3. Results and Discussions

3.1. Workability

The workability test of fresh mortars was determined according to the flow table method. mPCM in solution contains 62-65% liquid. The average weight of the solution containing mPCM is 27.5%. When calculating the mPCM ratios in mortars, these data were considered, and mortars were prepared using the mixture ratios in Table 4. When the mPCM solution is added to cement mortars, the workability of the mortar increases due to the liquid content. Since this situation would cause strength decreases, the mixing water in the mortars would be reduced, and the workability would be kept constant. The workability of the reference mortar was measured as 150 mm, and the mixing water amounts were determined by the trial-and-error method so that the workability of the mPCM added mortars would also be 150 mm.

3.2. Flexural strength

The flexural strength results of cement mortars after 3, 7, and 28 days of curing are shown in Figure 2. The flexural strength was carried out on three specimens for each mortar code. The percentage decrease rates of the flexural strengths of mortars with mPCM additive compared to the reference mortar are shown in Figure 3.

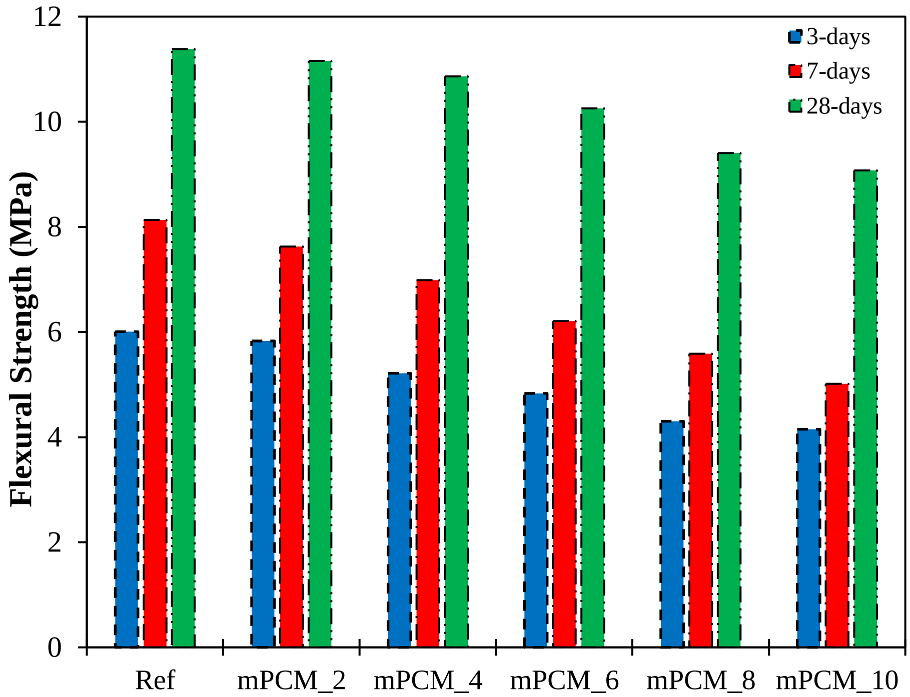

Figure 2. Flexural Strength Results

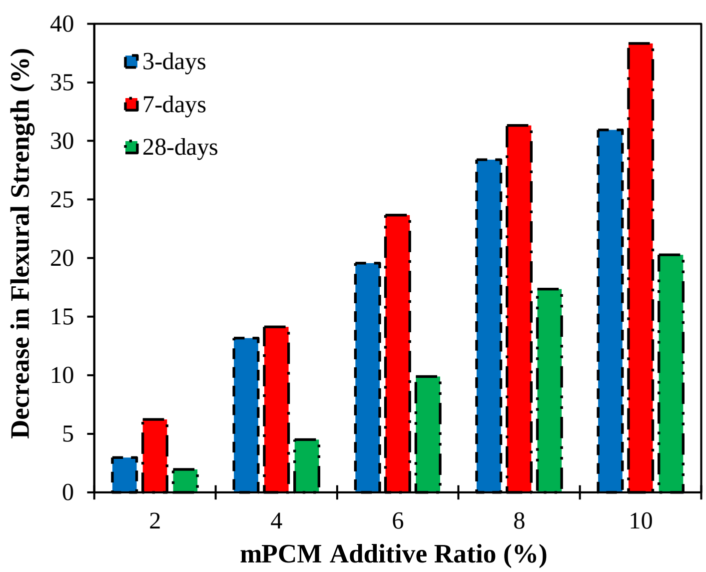

Figure 3. Percentage Decrease Rates In Flexural Strengths

When the flexural strength findings in Figures 2 and 3 are examined, the flexural strengths decreased as the mPCM amount in the mortars increased. It is obvious that an increase in flexural strengths is observed as the curing period increases (Demir, 2022). The percentage decrease rates in flexural strengths of mortars cured for 28 days are lower than others. The decrease in flexural strengths can be attributed to the increased porosity due to the increased mPCM content (Alkan et al., 2023; Zhang et al., 2016). The flexural strength results were similar to the studies presented in the literature (Meshgin et al., 2012; Meshgin & Xi, 2012). Zhang et al. (2016) added 10, 20, 25, and 30% mPCM to cementitious composites, and the flexural strengths decreased as the mPCM ratio increased. It was found that the flexural strength of the cementitious composite with 30% mPCM added decreased by 34.9%. Cui et al. (2015) produced mortars with 5, 10, 15, and 20% mPCM and reported that their flexural strength decreased by 13.2%, 14.9%, 17.5%, and 33.3%, respectively, compared to the reference mortar.

3.3. Compressive strength

The results of the compressive strength test performed on six specimens obtained after the flexural strength test are shown in Figure 4 as the percentage decrease rates of the compressive strengths of mPCM-added mortars compared to the reference mortar are shown in Figure 5.

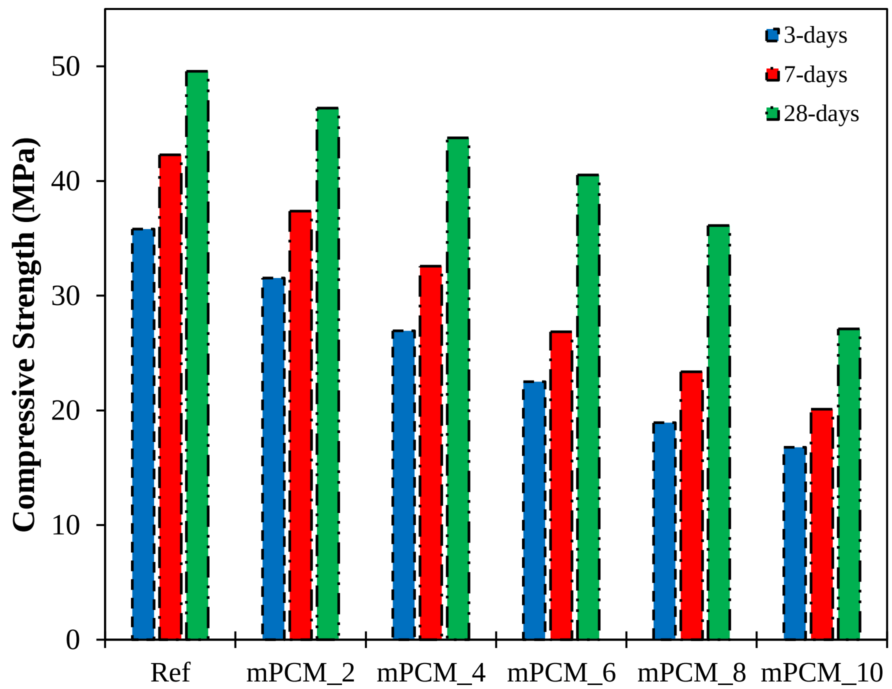

Figure 4. Compressive Strength Results

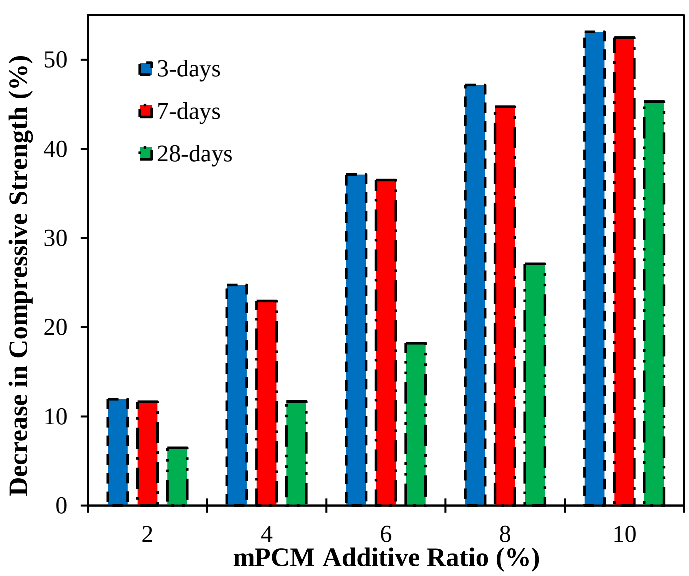

Figure 5. Percentage Decrease Rates In Compressive Strengths

When the compressive strength findings in Figures 4 and 5 are examined, a similar trend in flexural strengths is obtained. The compressive strengths decrease as the amount of mPCM in the mortars increases. As the curing period extended, compressive strength was increased due to hydration development. The decrease in compressive strengths can be explained by the pores caused by mPCM particles (Cui et al., 2015). When Figure 5 is examined, it is seen that the 28-day compressive strengths of mortars with 2, 4, 6, 8, and 10% mPCM added decreased by 6.5%, 11.7%, 18.2%, 27.1%, and 45.3%, respectively, compared to the reference mortar. Cui et al. (2018) reported that the weak interfacial transition zone between the microcapsule and the cement matrix is a factor that reduces the compressive strength. The compressive strength results are similar to the studies presented in the literature (Acıkök et al., 2023; Alkan et al., 2023). Cui et al. (2018) added 10% and 20% mPCM to alkali-activated slag composites and found that their compressive strength decreased by 27.6% and 41.9%, respectively. Yeon & Kim, (2018) added 10% and 20% mPCM to cement mortars and found that the 28-day compressive strength decreased by 27.7% and 46.9%, respectively, compared to the control sample. When the mechanical properties are evaluated, it is seen that there are decreases as the mPCM ratio increases. It was decided that mPCM can be used at a maximum rate of 6%.

3.4. Ultrasonic pulse velocity (*UPV*)

The *UPV* test was carried out on three specimens with dimensions of 40 mm × 40 mm × 160 mm, and the results are shown in Figure 6. The percentage decrease in *UPV* values of mortars with mPCM admixture compared to the reference mortar is shown in Figure 7.

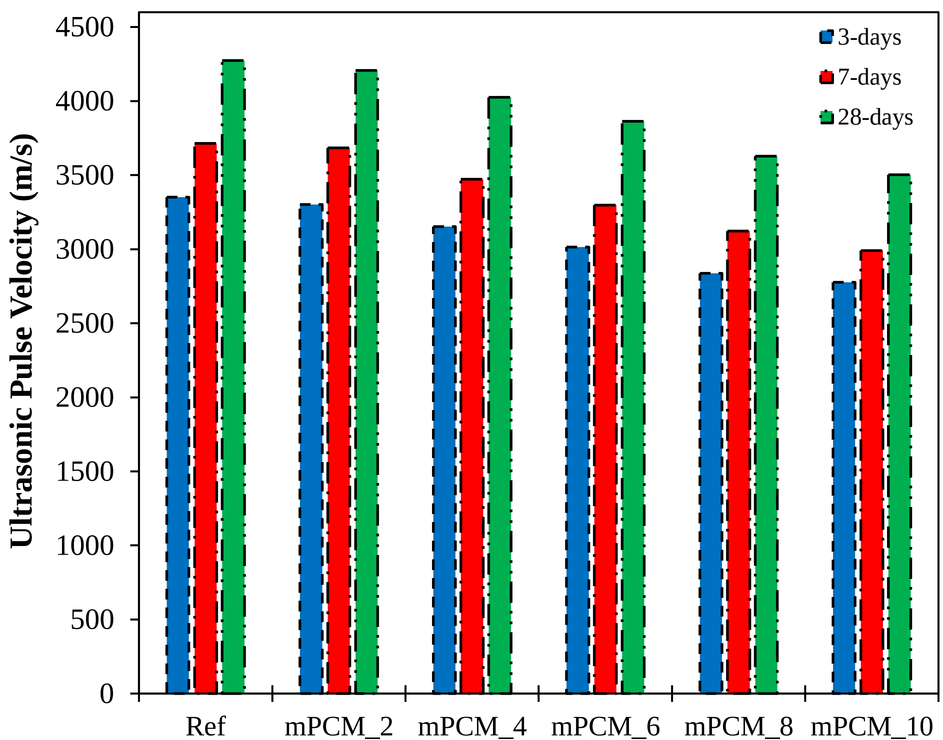

Figure 6. *UPV* Results of Mortars

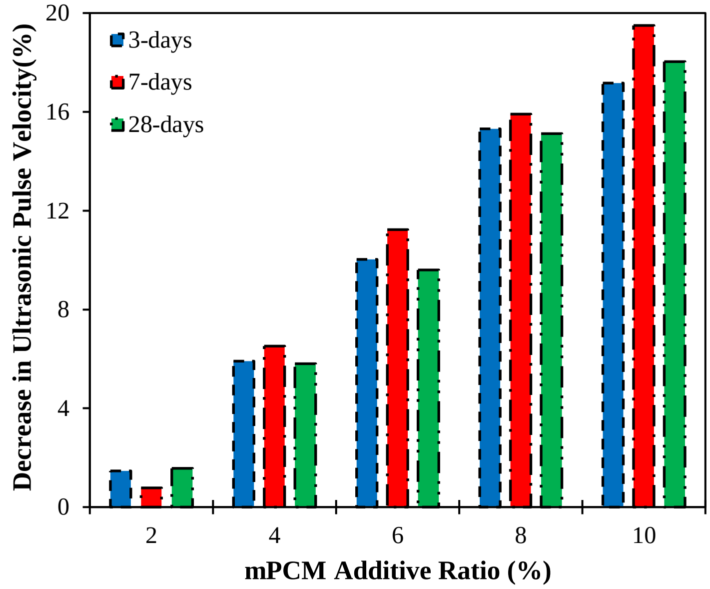

Figure 7. Percentage Decrease Rates In *UPV* Values

When the UPV findings given in Figure 6 are examined, it is seen that UPV values increase as the curing time elapses. As in the flexural and compressive strength findings, there are decreases in UPV values as the mPCM ratio in the mortars increases. The findings obtained from the UPV results confirm the mechanical properties (Gümüş et al., 2020). This is due to the fact that mPCM additive increases the porous structure of cement mortars. (Cui et al., 2015). When Figure 7 is examined, it is seen that the 28-day UPV values of the mortars with 2, 4, 6, 8, and 10% mPCM additives show decreases of 1.6%, 5.8%, 9.6%, 15.1%, and 18.0%, respectively, compared to the reference mortar. According to the Whitehurst, (1951) classification, the mortars cured for 28 days are in the "good" class. It is seen that the mortars coded Ref and mPCM_2 cured for 7 days are in the "good" class, and the mortars coded mPCM_4, mPCM_6, mPCM_8, and mPCM_10 are in the "doubtful" class. Mortars with codes Ref, mPCM_2, mPCM_4, and mPCM_6, which were cured for 3 days, were in the "doubtful" class, while mortars with codes mPCM_8 and mPCM_10 were in the "poor" class.

Correlations between flexural and compressive strength values and UPV values are given in Figures 8 and 9. Linear regression was used to relate flexural and compressive strength values to UPV values. The obtained equations are shown in the graphs in Figures 8 and 9. The R^2 values given in the figure mean a good correlation (Alakara, 2022; Alakara et al., 2022; Kubba et al., 2018).

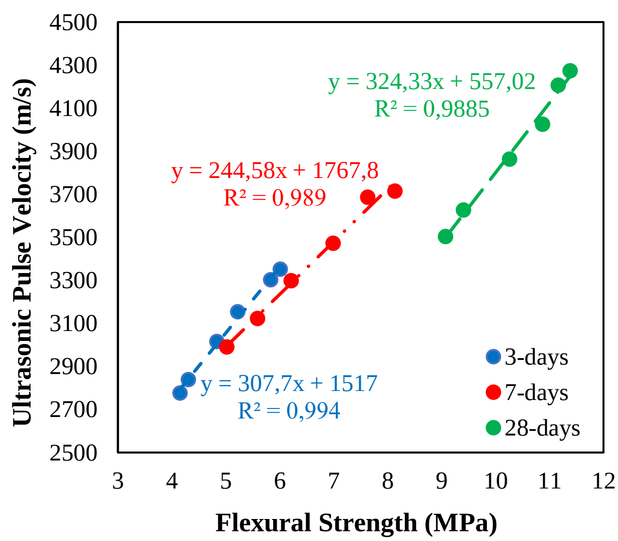

Figure 8. Relationship Between Flexural Strength – *UPV*

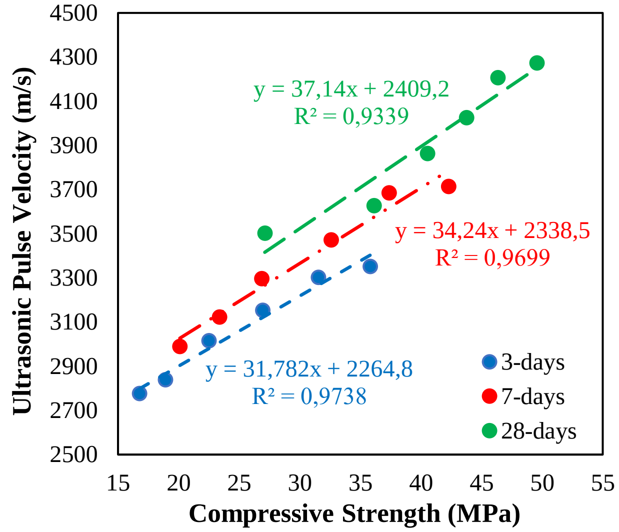

Figure 9. Relationship Between Compressive Strength – *UPV*

3.5. Results of periodic freezing and thawing tests on the mortar specimens

When the mechanical properties were evaluated, it was decided that the optimum mPCM ratio was 6%. Therefore, temperature measurements were carried out on the Ref and mPCM_6 added mortar. To examine the temperature changes of the mortars coded Ref and mPCM_6, the setup shown in Figure 1 was used. Freezing-thawing cycles were applied to the mortar specimens between "+20 °C" and "-20 °C" temperatures in this setup. The specimens were kept at the lowest and highest temperatures for 3 hours. The graph obtained from the temperature measurements is given in Figure 10.

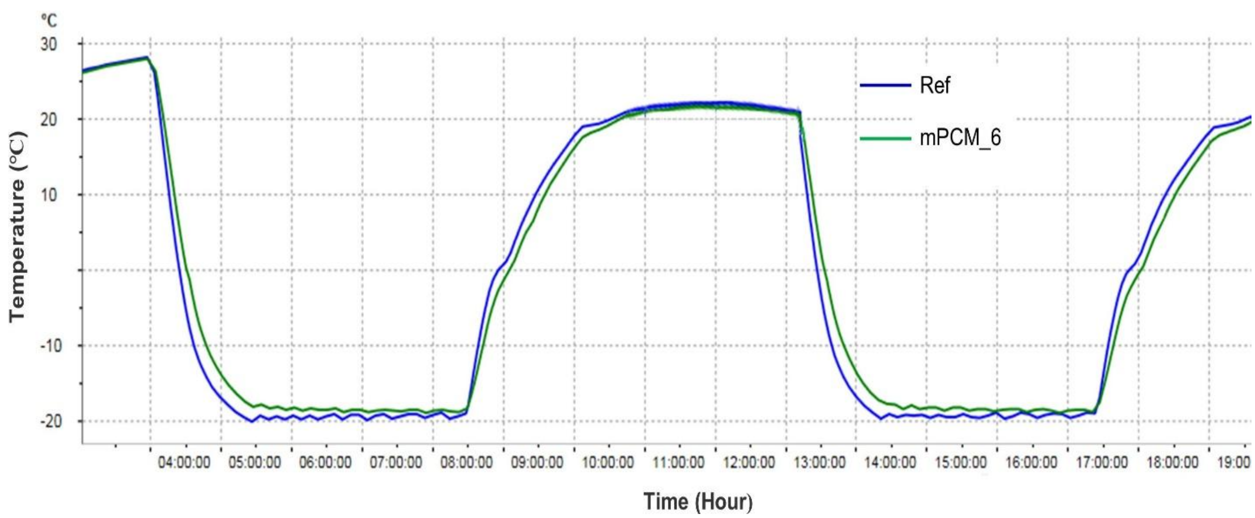

Figure 10. Internal Temperatures of Mortars After The Freezing-Thawing Cycle

As shown in the curves, there are significant temperature differences between the Ref and mPCM_6 coded mortars. This situation is caused by the energy storage feature of mPCM. During the cooling process, the sample with the mPCM additive had a temperature decrease slightly as compared to the reference sample. This situation is because the PCM absorbs heat during the melting process and releases it back as the temperature decreases, thus preventing the sudden decrease in temperature. While the temperature of the reference sample reaches -20 °C levels more quickly, the sample with PCM additive drops to these levels over a long time. This is an important contribution of PCM to temperature stabilization. During the cooling stage, the maximum temperature difference between the Ref and mPCM_6 coded specimens was 6.40 °C at 04:32. At this hour, the internal temperature of the Ref-coded mortar was measured as -7.70 °C. In contrast, the internal temperature of the mPCM_6 coded mortar was measured as -1.30 °C.

When the temperature values of both specimens reach around -20 °C, irregular fluctuations are observed in the reference sample. In contrast, the mPCM_6 sample maintains stability in temperature during this period. This is caused by the latent heat released during the crystallization of PCM.

During the heating process, the temperature of the reference sample increases rapidly, while the temperatures of the PCM-added specimens increase very slightly and rationally with the mPCM additive. This delay is caused by PCM absorbing energy from the environment while phase changing. This balances temperature fluctuations in the application field. These two processes (freezing-thawing) prove that the PCM-added mortar sample regulates the temperature. Although both curves in the graph generally follow a similar trend, the effect of the mPCM_6 additive in slowing down and regulating the temperature change is observed. During the heating phase, the maximum temperature difference between the Ref and mPCM_6 coded specimens was measured as 3.70 ℃ at 17:42. At this time, the internal temperature of the Ref-coded mortar was measured as -3.40 ℃. In contrast, the internal temperature of the mPCM_6 coded mortar was measured as -7.10 ℃.

The graph shows that the mPCM additive has a significant thermal regulating effect on the mortar sample. The phase change properties of PCM brought about temperature fluctuations that were slowed down, and a more stable temperature profile was obtained. A similar effect of PCM on the thermal behavior of mortars has also been seen in the literature. (Yeon & Kim, 2018; Yousefi et al., 2021; Yu et al., 2023). The latent heat capacity of PCM reduces thermal stresses that may occur in concrete by slowing down temperature fluctuations during freeze-thaw cycles. In this way, it can prevent microcracks and premature aging of concrete caused by freeze-thaw cycles. In addition, PCM ensures that the concrete surface remains free of snow and ice for extended periods and increases surface safety (Li et al., 2023; Nayak et al., 2019; Paswan & Das, 2024a, 2024b). Since it helps melt snow and ice, it can also reduce the slipperiness on concrete surfaces.

4. Conclusion

The study investigated the effect of mPCM on the mechanical and thermal properties of cementitious composites. PCM was added to the mixtures at the rates of 2%, 4%, 6%, 8%, and 10% by weight of cement. The following results were obtained from the study:

- It was found that there was a continuous decrease in flexural strength as the mPCM additive ratio increased. It was determined that there were decreases in 3, 7, and 28 days flexural strengths at the ranges of 2.98% to 30.95%, 6.22% to 38.33%, and 1.95% to 20.28%, respectively.
- Compressive strength results were like flexural strengths, which decreased as the mPCM ratio increased. The highest compressive strength decrease rates were determined in mixtures containing 10% PCM. These rates were 53.13%, 52.46%, and 45.30% for 3, 7, and 28 days specimens, respectively.
- As in the flexural and compressive strength results, *UPV* values decreased as the mPCM additive content increased. According to the Whitehurst, (1951) classification, it was determined that all the mortars cured for 28 days were in the "good" class.
- It was found that the relationships between the parameters “Flexural strength – *UPV*” and “Compressive strength – *UPV*” exhibited a high coefficient of determination (R^2).
- The results obtained from periodic freezing-thawing tests proved that mPCM can improve the thermal properties of cementitious composites. It was determined that the reference mortar heated up and cooled down faster than the mPCM additive. During the freezing-thawing processes, the internal temperature difference of the mortars coded Ref and mPCM_6 was measured as 3.70 ℃ and 6.40 ℃, respectively.

When the results above are evaluated in general, mPCM additives have the potential to improve the thermal properties of cement composites while reducing their mechanical properties. PCM additive makes cementitious composites more resistant to thermal shocks by balancing temperature fluctuations. PCM used within the scope of the study will contribute to the melting of snow and ice on the surface of cement-based building materials. In this way, pedestrian safety may be increased by reducing the slipperiness that may occur on concrete surfaces, and vehicle accidents may be significantly prevented. Such mPCMs have great potential, especially in projects operating in cold weather conditions (roads, bridges, pavements), when the material compositions are carefully determined. As a result of the study, it is suggested that the mPCM additive can be used up to 6%. Because there are serious strength losses after 6%. Researchers who will work on this subject in the future can examine how the losses in the mechanical properties of the mPCM additive can be minimized. For example, flexural strength can be increased by adding different types and different rates of fiber. Compressive strength properties can be increased by using plasticizer or superplasticizer chemicals.

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