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Analysis of Thermo-Mechanical Properties in Cement Mortar Using Synthesized Silicate Microencapsulation of Phase Change Materials by Sol-Gel Process

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Abstract

This investigation delves into improving building thermal comfort using microencapsulated phase change materials (PCM@SiO2) in cement mortar. The encapsulation process, achieved via a sol-gel method, envelops PCM in a silicate shell Differential Scanning Calorimetry (DSC) and Fourier-Transform Infrared Spectroscopy (FTIR) were used to confirm the thermal properties and integrity of encapsulated PCM, which showed an encapsulation efficiency of 92.7% and a thermal storage capacity of 99.7%. The study involved the integration of PCM@SiO_2 into cement mortar using a binder-to-sand ratio of 1:3. The PCM@SiO_2 was varied in concentrations ranging from 0% to 20%, with increments of 5%. The mechanical strength and thermal conductivity of these PCM@SiO_2 cement mortar mixes were meticulously evaluated. Findings revealed that while the addition of PCM@SiO_2 marginally compromised the mortar's mechanical strength, it substantially boosted thermal performance. This enhancement underscores PCM@SiO_2's utility as a multifunctional building material, harmonizing structural resilience with augmented thermal energy storage capabilities, showcasing its potential to significantly elevate the energy efficiency and comfort of modern buildings.

Keywords: Synthesis PCM, mechanical strength, thermal comfort, building material, energy efficient

INTRODUCTION

In the pursuit of sustainable energy solutions, the exploration of renewable energy sources and the development of innovative materials for energy storage have become imperative. One such promising technology is the latent heat storage of phase change materials (PCMs), which utilize a solid-liquid phase transition. PCMs, with their high latent heat storage capacity and narrow operating temperature range, offer an efficient means of storing thermal energy. Their applications range from smart textiles for body temperature regulation to solar energy devices for energy storage, underscoring their versatility and effectiveness[1]. [2]. [3]. [4].

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Organic PCMs, particularly N-alkanes like paraffin waxes, have garnered significant attention due to their excellent phase change performance. These materials are chemically inert, ecologically benign, non-corrosive, economical, and readily available, making them ideal for various applications. Octadecane, an alkane hydrocarbon, exemplifies this with a phase change temperature in the thermal comfort range of humans, thus finding usage in garments and building products[5]. [6]. [7].

In the realm of building materials, cement-based materials have traditionally served structural

purposes but are now increasingly recognized for their potential in thermal energy storage. Incorporating PCMs into building envelopes is a straightforward and effective strategy to enhance the energy efficiency of buildings[8]. However, challenges such as the inherently low thermal conductivity of paraffin, which affects the rate of heat storage and retrieval, have led to the exploration of additives like carbon fibers and expanded graphite to improve heat diffusion in PCMs [9].

The application of microencapsulation technology in this context is a strategic measure to mitigate the leakage of phase change material (PCM) during phase transitions. This technique involves the encapsulation of PCM particles within a shell of high-molecular-weight polymer. Such encapsulation serves a dual purpose: it provides resistance to volume changes inherent in phase-change processes and enhances the efficiency of heat transfer, attributed to the increased surface area per unit volume offered by the microcapsules. However, a significant challenge persists in the form of the inherently low thermal conductivity of the organic polymers commonly used in creating microencapsulation shells, as well as the PCM (typically paraffin) itself. This limitation in thermal conductivity is a critical factor that impedes the effective transfer of heat, thus affecting the overall efficiency of the system [10].

This study aims to address these challenges by exploring the use of inorganics with high thermal conductivity for the preparation of organic–inorganic hybrid microencapsulated PCM. Building on the research by Xuan et al [11]. and Yin et al.[12]. who utilized iron nanoparticles and SiO_2 respectively to enhance the thermal conductivity of MPCMs, this research delves into the synthesis of PCM with a SiO_2 shell. The focus is on improving the thermal performance of cement mortar integrated with PCM@SiO_2, for enhanced energy storage efficiency in construction materials.

MATERIALS REQUIRED

For Synthesizing PCM

Tetraethyl silicate (TS) as precursor, anhydrous ethanol (AE) as solvent, distilled waste as solvent, hydrochloric acid (HA) as activator, organic paraffin (PCM) as core material and sodium dodecyl sulphate (SDS) as oil water emulsifier.

For Preparation of Cement Mortar

Ordinary Portland cement (OPC), river sand water and superplasticizer are used as binder, fine aggregate, mixing and additive respectively for mortar preparation.

METHODOLOGY

Synthesis of PCM With Sio_2 Shell

The microencapsulation of Phase Change Material (PCM) with a SiO_2 shell was accomplished through a sol-gel synthesis process. Initially, 20 grams of PCM and 0.5 grams of Sodium Dodecyl Sulphate (SDS) were homogenized in 200 ml of distilled H2O within a beaker. The resultant solution was subjected to mechanical stirring at a rotational speed of 1000 revolutions per minute (rpm) for a duration of 2 hours while being maintained at a temperature of 70°C on a magnetic hot plate, leading to the formation of an oil-in-water (O/W) emulsion. Subsequently, a separate mixture was prepared by dissolving 20 grams of Tetraethyl Orthosilicate (TS), 20 grams of Ammonium Ethoxide (AE), and 0.5 grams of Hexadecylamine (HA) into 100 ml of distilled H_2O within a conical flask. This solution was agitated at a lower rotational speed of 500 rpm and a temperature of 60°C for 30 minutes, resulting in a homogenous solution. The TS solution was then incrementally integrated into the previously prepared PCM solution, employing a dropwise addition technique while continuously stirring the mixture for an additional 4 hours at 70°C on a magnetic hot plate. This step facilitated the formation of a SiO_2 shell around the PCM core. Post-formation of the shell structure, the polymerization was allowed to proceed to completion. The synthesized PCM@SiO_2 material was then separated through filtration and subsequently washed with distilled H_2O and ethanol to eliminate any residual reactants or byproducts. The final product was then placed in an oven and dried at 50°C for 24 hours. The entire sol-gel process, instrumental in the synthesis of the microencapsulated PCM with a SiO_2 shell, is graphically depicted in Figure 1.

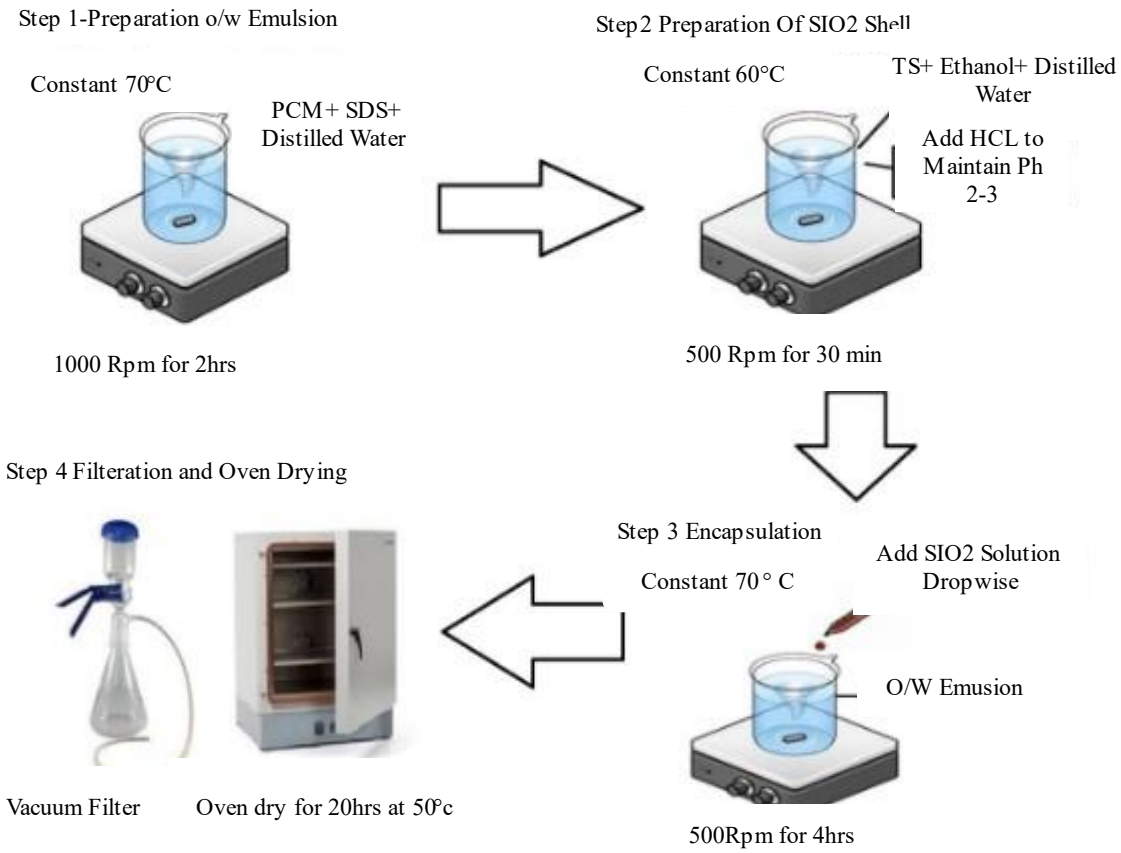

Figure 1. Sol-gel process for synthetization of microencapsulated PCM with a SiO_2 shell

Mix Design for Cement Mortar

In the experimental setup of this study, cement mortar specimens were formulated with a volumetric ratio of 1:3, representing cement to sand. The formulation involved the integration of encapsulated PCM@SiO_2, which was varied across a range from 0% to 20% in increments of 5%, in the mixture. To adhere to a specific water-to-binder ratio of 0.35, the dosage of superplasticizer was adjusted within a range of 0.5% to 0.8% by weight of the cement. This adjustment was critical to ensure the flowability of the mortar mixture, targeted to be within the range of 180-220 mm, thereby maintaining the workability of the mortar while incorporating varying percentages of PCM@SiO_2. Table 1 shows the mixing details for PCM@SiO_2 cement mortar mixes.

Table 1. Mixing details for PCM@SiO_2 cement mortar mixes

Mix ID	Cement	Sand	PCM@SiO_2	Water	Admixture
PCM0	320	1290	-	112	0.50
PCM5	320	1290	16	112	0.62
PCM10	320	1290	32	112	0.68
PCM15	320	1290	48	112	0.74
PCM20	320	1290	64	112	0.80

RESULTS AND DISCUSSION

Properties of PCM@SiO_2

Thermal behaviours of PCM encapsulated with SiO_2 is evaluated by differential scanning calorimeter test. Outcomes of DCS test shows that shifting of 1 °C is observed for melting and freezing temperature. Liquid specific heat (J/g) of encapsulated PCM with SiO_2 is decreased from 235 J/g to 218 J/g compared

with PCM. Similarly, Solid specific heat of PCM@SiO_2 is decreased from 202 J/g to 186 J/g compared with PCM. Efficiency of encapsulation of PCM is examined by encapsulation ratio and thermal storage capacity which are 92.7% and 99.7% respectively.

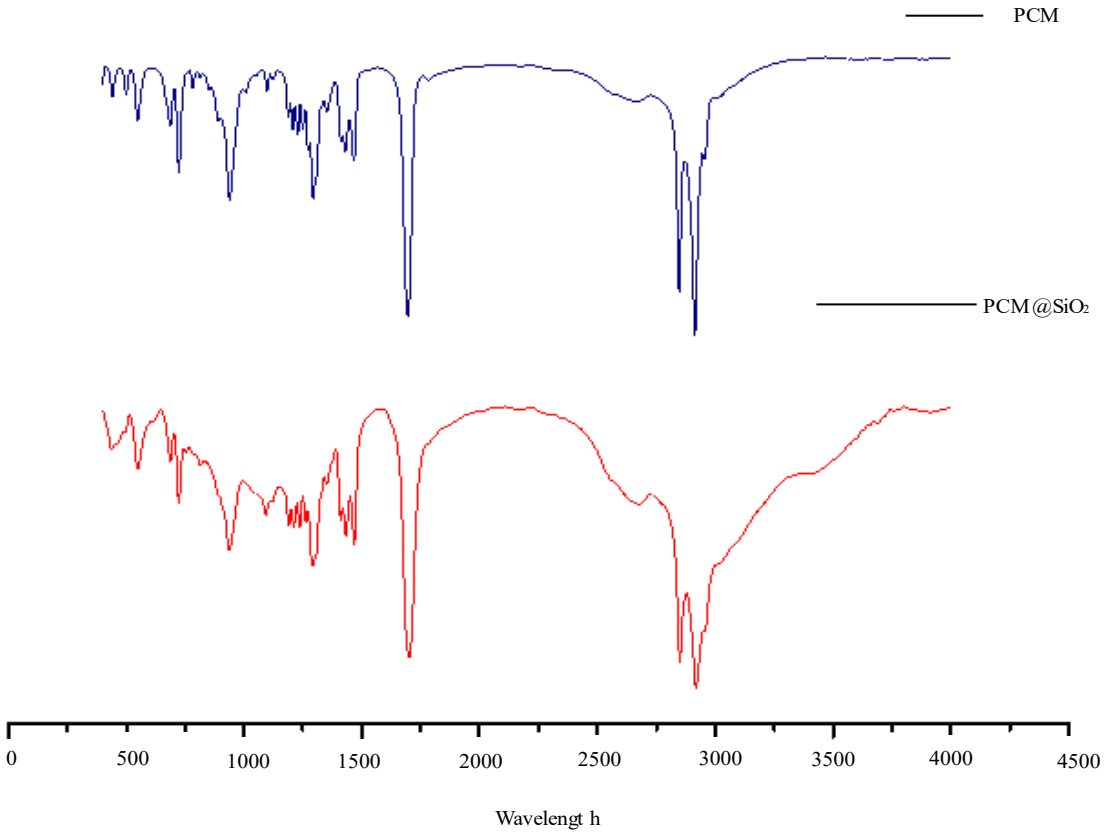

Figure 2. FTIR spectra for PCM and PCM@SiO_2

To make sure that the phase change material (PCM), which stores and releases thermal energy, was properly enclosed within a protective silica shell without any chemical reaction taking place, we used a tool called Fourier-transform infrared spectroscopy (FTIR). This tool helps us see if there are any specific types of bonds or groups in a material based on the material's molecular structure. We focused on n-octadecane, a type of paraffin and the main ingredient in our PCM, which is made up of chains of carbon and hydrogen atoms. The FTIR technique showed in

Figure 2 the signature peaks or spots where these chains vibrate. For the raw, unencapsulated PCM, we found these spots at certain numbers (2914, 2847, and 1351 cm-1 for the methyl groups, and 939.4 and 723.8 cm-1 for the methylene groups), which slightly changed when the PCM was encapsulated in SiO2 (2918, 2850, and 1351.1 cm-1 for methyl groups, and 938.3 and 723.5 cm-1 for methylene groups). There were also specific peaks for the silica shell itself, seen at 1211 and 1092 cm-1. Importantly, we didn't see any new peaks that would suggest a chemical reaction had occurred between the PCM and the silica shell. This tells us that the PCM was successfully wrapped in silica without chemically bonding to it, just as intended.

Mechanical Properties of Mortar

In the context of evaluating the mechanical properties of cement mortar composites incorporated with encapsulated PCM with SiO_2, an analysis was conducted to ascertain the influence of PCM@SiO_2 on the 28-day compressive and flexural strengths. The experimental design was structured to systematically vary the PCM@SiO_2 concentration within the mortar matrix, with the intent to observe the consequent variations in its mechanical robustness.

Figure 3 and Figure 4 shows data elucidated a negative correlation between PCM@SiO_2 content and the mechanical strength parameters. The decremental trend in both compressive and flexural strengths can be attributed to the inherent material characteristics of PCM@SiO_2, which exhibits suboptimal compressive attributes, and the induction of porosity within the composite matrix due to the integration of PCM@SiO_2 particles. Figure 5 shows interfacial analysis via microstructural examination would likely reveal a pronounced disparity at the juncture of PCM@SiO_2 and the cementitious matrix, characterized by a discernible interfacial gap.

This surface modification aims to enhance the interfacial adhesion between the PCM@SiO_2 microcapsules and the inorganic cementitious binder, thereby fortifying the compressive strength of the resultant composite.

Experimental evidence from the study indicates that the inclusion of PCM@SiO_2 exerts a deleterious effect on the compressive and flexural strengths of cement mortar. A 5% PCM@SiO_2 admixture precipitated an initial strength reduction of 3.95% in compressive strength and 12.3% in flexural strength. With further augmentation of PCM@SiO_2 concentration to 20%, the flexural strength exhibited a diminution of 36.13%, while compressive strength registered a 16.41% decrement. Nevertheless, it is noteworthy that the 20% PCM@SiO_2-infused mortar still achieved compressive and flexural strengths of 6.98 MPa and 2.44 MPa, respectively, which are congruent with the Indian standards for structural applications.

The Figure 6. Error! Unknown switch argument**.** also presents a linear regression model articulating the relationship between compressive and flexural strengths, delineated by the equation $y=0.8661x-3.5645$ with a coefficient of determination, $R2 = 0.8985$. This high R2 value denotes a strong predictive capacity of the regression model, underscoring a substantial correlation between the compressive and flexural strengths of the PCM@SiO_2 cement mortar composites.

In summation, the research delineates that while the introduction of PCM@SiO_2 attenuates the mechanical strengths of cement mortar, strategic compositional adjustments and surface treatments can ensure compliance with requisite structural standards.

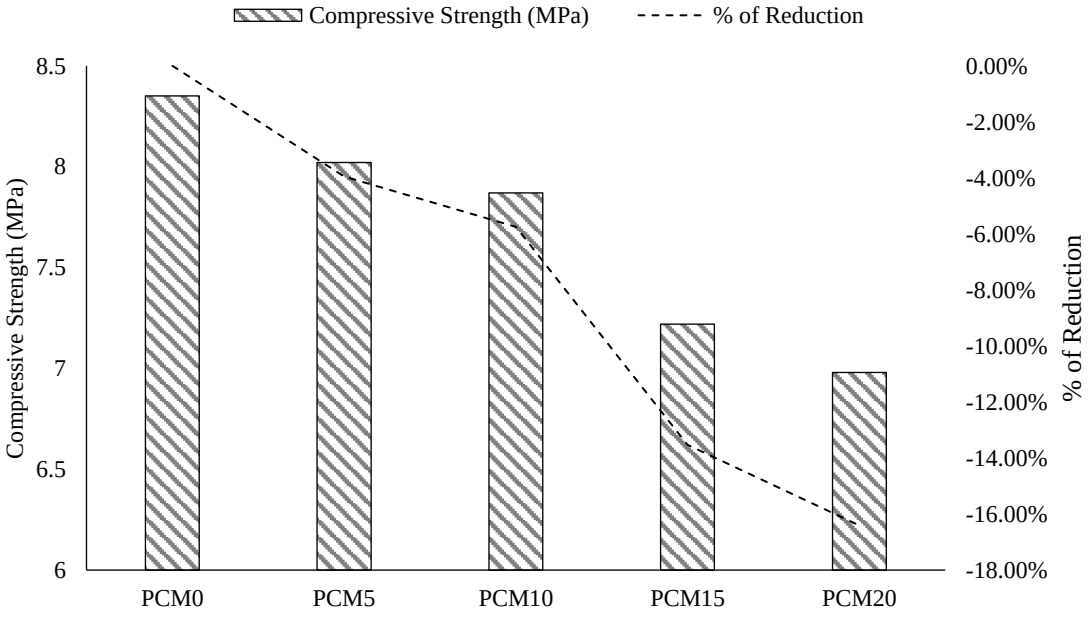

Figure 3. Compressive Strength of PCM@SiO_2 mixed cement mortar

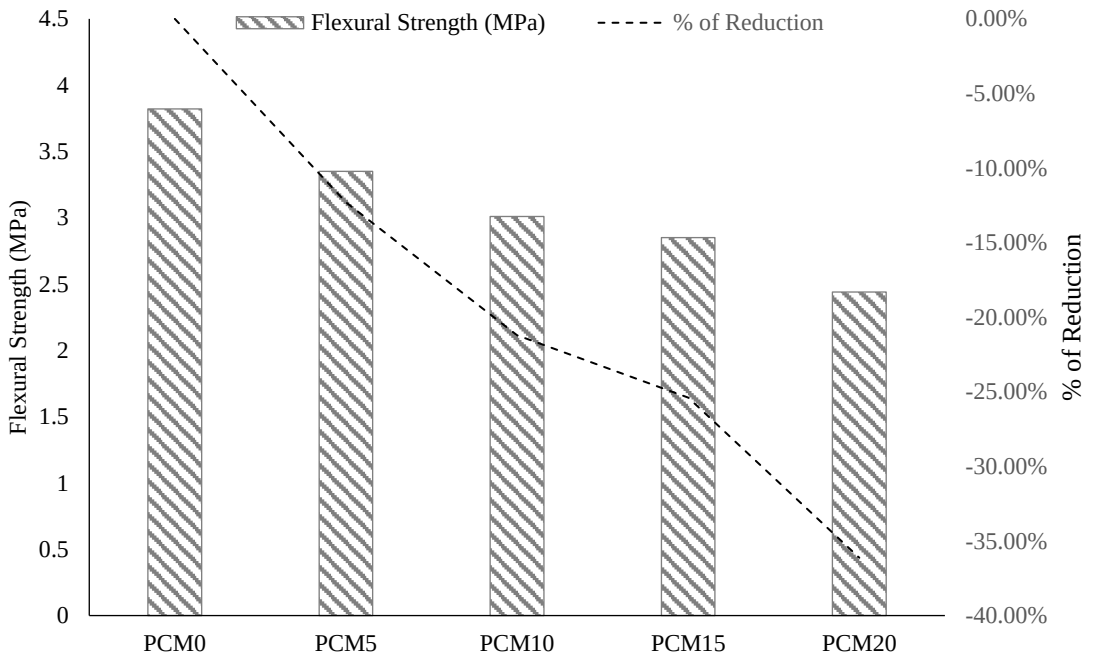

Figure 4. Flexural Strength of PCM@SiO_2 mixed cement mortar

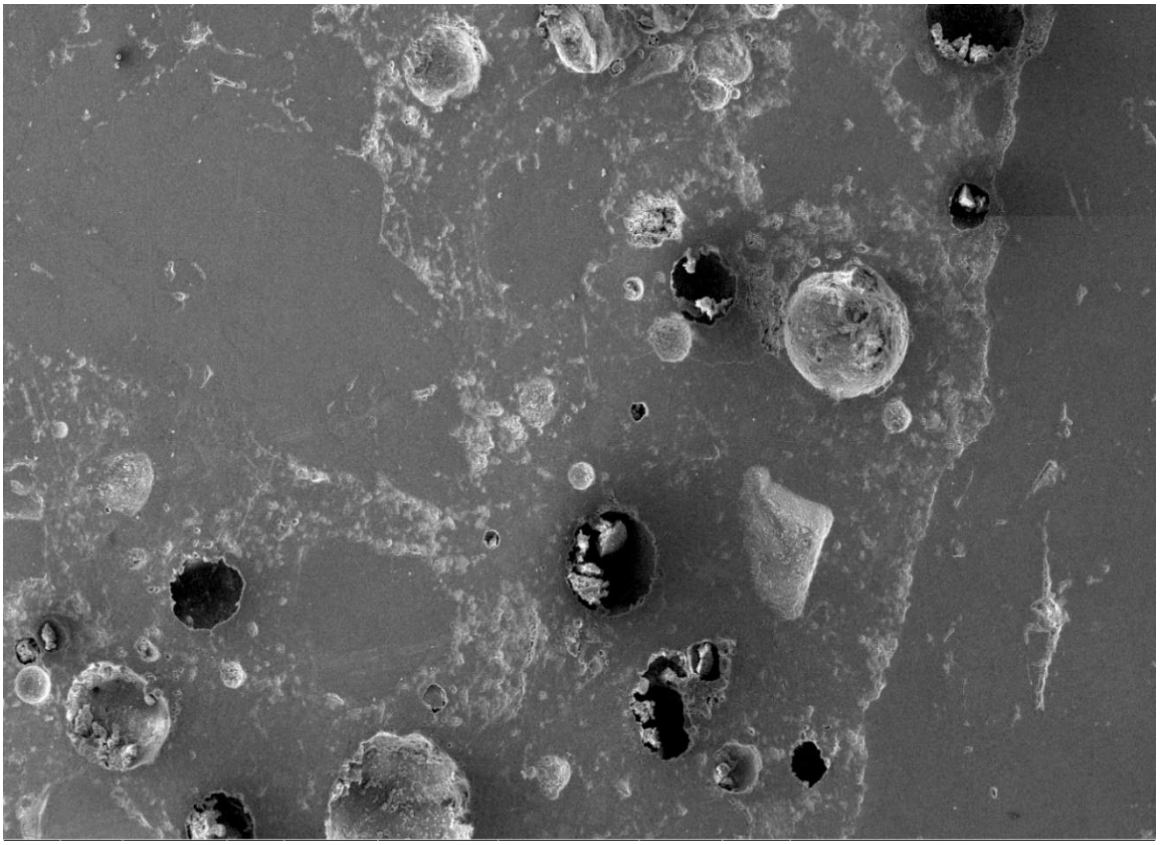

Figure 5. Microscopic image PCM@SiO_2 mixed cement mortar

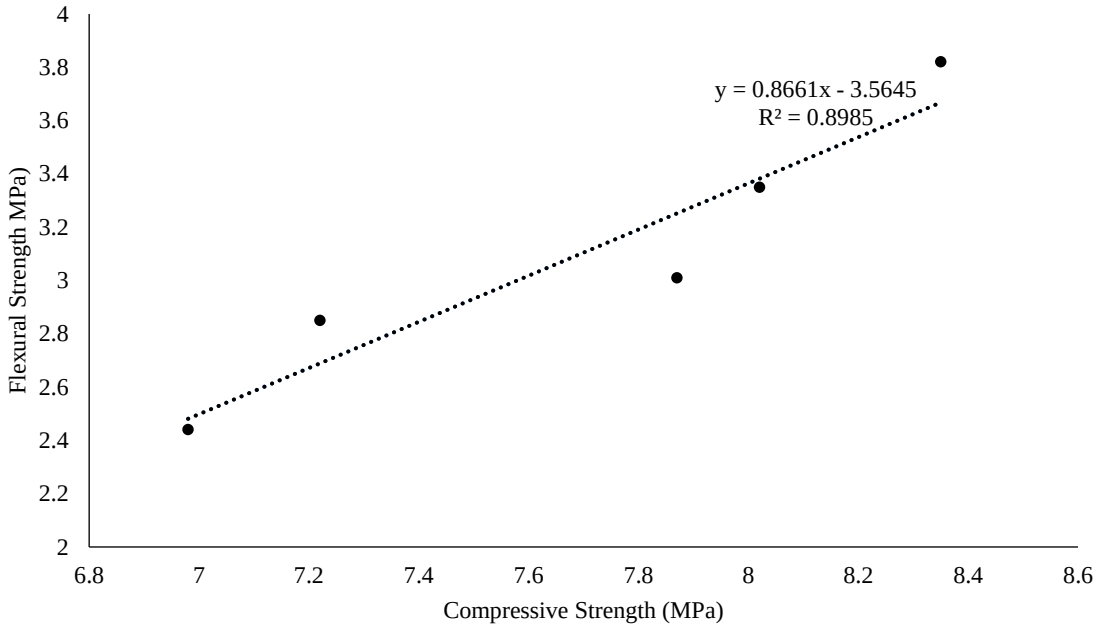

Figure 6. Error! Unknown switch argument. Corelation between mechanical Strength of PCM@SiO_2 mixed cement mortar

Thermal Conductivity Of PCM@Sio_2 Mortar Mixes

In the context of evaluating the thermal performance of cement mortar for potential use in thermal energy storage systems, the current investigation quantitatively assesses the thermal conductivity of cement mortar composites integrated with phase change materials (PCM) embedded in silica dioxide. This property is paramount, given its critical role in the efficiency of thermal energy storage and retrieval. Measurements were conducted across three distinct thermal states of the PCM: solid (at average temperatures of 42-44 °C), phase-change (48–53 °C), and liquid (56-58 °C).

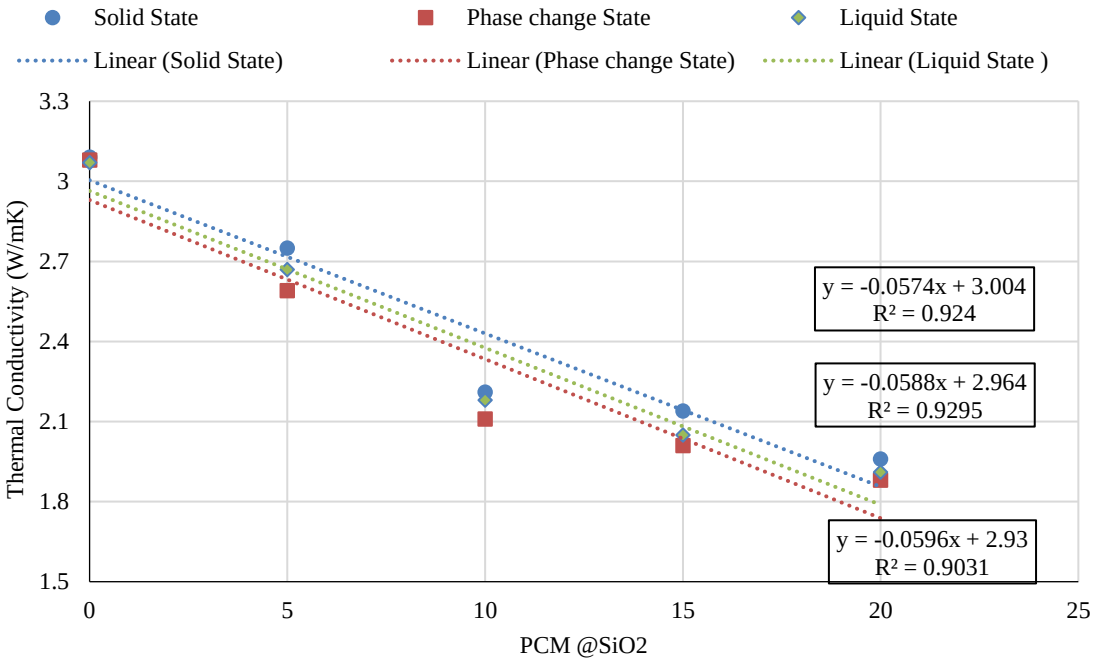

Figure 7. Thermal Conductivity of PCM@SiO_2 mixed cement mortar

The result data, as depicted in Figure 7, elucidates a inversely proportional relationship between the PCM@SiO_2 dosage within the cement mortar and its corresponding thermal conductivity. The incorporation of PCM@SiO_2 into the cement mortar matrix induces a notable decrement in thermal conductivity, attributable to the lower intrinsic thermal conductivity coefficient of the paraffin PCM (approximated at 0.4 W/m·K) as compared to that of the hardened cement mortar (averaging 3.08 W/m·K). The quantitative analysis reveals that the thermal conductivity coefficient diminishes by 36.6%, 39%, and 37.8% for a 20% PCM@SiO_2 concentration, corresponding to the PCM in solid, phase-change, and liquid states, respectively. This variation is consistent with extant literature, which suggests that the thermal conductivity of solid-state paraffin supersedes its liquid-state counterpart.

Furthermore, Figure 7 delineates a near-linear dependency of the cement mortar's thermal conductivity coefficient on the PCM@SiO_2 dosage, with regression equations formulated as $y=-0.0574x+3.004$, $y=-0.0596x+2.93$, and $y=-0.0588x+2.964$ for the solid, phase-change, and liquid states, respectively. The determination coefficients (R2) for these linear models are 0.924, 0.9031, and 0.9295, indicating a robust linear correlation and predictive reliability for the influence of PCM@SiO_2 dosage on thermal conductivity across the different states of PCM.

In summation, the empirical evidence firmly establishes the dosage of PCM@SiO_2 as a critical determinant of the thermal conductivity of PCM integrated cement mortar, with higher dosages resulting in a marked decrease in thermal conductivity, which is pertinent to the composite's application in thermal energy storage solutions.

CONCLUSION

This study comprehensively examined the thermal and mechanical properties of cement mortar integrated with microencapsulated phase change materials (PCM@SiO_2), synthesized via a sol-gel process. The thermal behavior of PCM@SiO_2 was characterized using DSC, revealing a shift in melting and freezing temperatures and a decrease in specific heat in both liquid and solid states compared to unencapsulated PCM. The efficiency of encapsulation was confirmed with high encapsulation ratios and thermal storage capacity.

FTIR analysis substantiated the physical encapsulation of PCM within SiO_2 shells, indicating no chemical interaction between core and shell materials. This encapsulation was critical for maintaining the structural integrity of the PCM while allowing for its effective use in thermal energy storage.

Mechanically, the inclusion of PCM@SiO_2 in cement mortar displayed a reduction in compressive and flexural strengths, with a notable correlation between PCM@SiO_2 content and mechanical strength reduction. Despite this, even at higher PCM@SiO_2 dosages, the mortar's strength remained within acceptable standards for structural applications. A linear regression model further elucidated the relationship between compressive and flexural strengths, indicating a predictable pattern of strength variation with PCM@SiO_2 concentration.

The thermal conductivity of the PCM@SiO_2 cement mortar composites demonstrated a significant decrease with increasing PCM@SiO_2 content, indicating an inversely proportional relationship. This reduction in thermal conductivity was more pronounced in the PCM's solid state, confirming the material's suitability for thermal energy storage applications.

In summary, the study highlights the potential of PCM@SiO_2 integrated cement mortar as a functional material for building structures, offering a balance between thermal regulation and mechanical strength. Despite the reduction in mechanical strength, the enhanced thermal performance of the mortar suggests its utility in improving the energy efficiency and thermal comfort of buildings. The findings pave the way for further exploration into the use of microencapsulated PCM in construction materials, aiming at sustainable and energy-efficient building solutions.

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