Synthesis and characterization of PCM based insulated concrete for thermal energy storage

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Abstract

Phase change materials (PCMs) are an innovative solution in a thermal energy storage system that can contribute efficiently to the improvement of the energy performance in the building. The phase change is the latent heat of storage materials that can store a large amount of thermal energy in its phase change from solid to liquid and vice versa. The heat storage/release mechanism of PCM is due to its sensitivity to melting/solidifying processes. Optimized ratios of PCM in concrete is essential for effective thermal storage system and strengthen physical characteristic. In this research work, dip coating of phase change materials of melting temperature 17 °C to 50 °C was done for coarse aggregate in the ratios via 25%, 50%, 75%, and 100 v/v% with the cement. The compressive strength of PCM-based coated coarse aggregated 0%, 25%, 50%, 75%, and 100% was investigated after 7, 14, and 28 days' ages. Encapsulated coarse aggregates (ECA) of different ratios were compared for thermal energy conductance while keeping compressive strength as a key parameter. Temperature differences of 12.35, 15.25, 16.35, 17.5, and 20.3 °C were observed for ECA-100, ECA-75, ECA-50, ECA-25, and NCA, respectively in 12 h. The encapsulated PCM-based coarse aggregate concrete was characterized for physical, thermal conductance, and surface morphology. It was found that ECA-50 offers optimum storage of latent heat and reasonable compressive strength of concrete. However, an adverse impact was found regarding compressive strength as the PCM ratio increases above 50% v/v%, which can be maintained by bonding additives CNTs and GO.

1. Introduction

Energy demand has been increased due to rapid economic growth which leads to depletion of fossil and renewable energy resources, that upon usage results in the emission of harmful gases [1, 2]. To cope with such a demanding situation and to reduce the demand for purchased energy, energy resources need to be used efficiently and new techniques of energy storage should be introduced [3–5]. As a result, researchers are paying more attention to the introduction and innovation of green buildings materials having energy-saving capabilities for the construction of the building industry [6]. Studies showed that the building sector utilizes a 40% share of the total energy consumption. The use of concrete for bridges and building purposes has become so general, as a thorough investigation of its various properties has been carried out for many years [7]. Some of the properties of concrete such as strength, permeability, and durability have been given much attention, while others have been studied little, one of the properties that have not been the subject of in-depth investigation is the insulating behavior of concrete [7, 8]. In recent years, there has been an increasing interest in improving the thermo-mechanical properties of concrete. Improvements to the strength, and in a parallel pathway, thermal insulation criterion are the major keys to improve the performance of concrete [9]. The strength characteristic represents the load-bearing capability of concrete to support the applied load without failure, whilst, the insulation criterion refers to the ability of a structural element to maintain its separate function when it is subjected to elevated temperatures [10]. The orientation mentioned above has also associated with
environmental aspects in the construction processes. From that point, the idea of environmentally friendly construction has grown up during the last few decades [8, 11, 12]. Although recent developments in the field of construction materials have emphasized the importance of improving thermo-mechanical behavior of concrete elements, without compromising on mechanical properties of concrete, consequently raise the need for more research in this aspect [13].

Phase Change Materials (PCMs) are the latent thermal storage material that can store a large amount of thermal energy during its phase change from solid to liquid and vice versa [14]. The focus was be given to the preparation of macro encapsulated phase change materials (ME-PCMs) for the development of thermal energy storage Normal aggregate concrete (NAC) with appreciable structural properties, and capable of storing thermal energy within the human thermal comfort zone (20 °C–32 °C). Researchers found that ME-PCMs would be used for the development of thermal energy storage concrete in sustainable construction but physical properties effect due to low adhesion forces [15]. To fairly deal with all such situations and save a portion of energy in buildings, enhancing the energy storage capacity of concrete is required through some mechanism for example incorporation of phase change material (PCM) into concrete mixtures through dip coating is the viable option in thermal energy management system [5, 10, 16, 17]. Furthermore, a reasonable investigation has been carried out on both the organic and inorganic PCMs coating which offer reasonable and strong physical properties [4].

Inorganic PCM has this promising nature of high latent heat of storage, reasonable thermal conductivity, non-flammable and economical [18]. However, their usage gets restricted due to some of its undesirable characteristics for example corrosive nature, phase decomposition, and supercooling. In contrast, organic PCMs are chemically stable, free from phase decomposition & supercooling, non-corrosive, non-toxic, and have high latent heat of melting and freezing [19]. The novelty of this research work is also due to PCMs dip coating on concrete which offer high strength and high thermal conductance.

Figure 1. Locally available coarse aggregate.

In this research work, phase change materials (CH₄(CH₂)nCH₃) of different ratios 0%, 25%, 50%, 75%, and 100% v/v with cement were dip-coated on coarse aggregate for a specific time to investigate its thermal properties to produce sustainable building constructing materials. The PCM-based coated encapsulated coarse aggregates were physically and thermally characterized in concrete. Physical investigation such as density, UPV, air contents, specific gravity, and compressive strength of samples was done according to structural strength. A thermal analyzer box with mounted thermocouple sensors was used for thermal conduction analysis.
2. Materials

2.1. Coarse aggregate
In a concrete cementing phase, aggregate is inert materials and filler dispersed. In this research work, Coarse aggregate was obtained from the local quarry of Peshawar, Pakistan. The sample was taken from the base quarry in Peshawar near Hayatabad as shown in figure 1. The specific gravity of coarse aggregates is assessed in the bulk dry, saturated-surface dry, and apparent conditions. The specific gravity was found to be 2.68 in conformity to ASSHTO T 85 and ASTM-C-127. The water absorption recorded for the coarse aggregate sample was 1.23%. The fineness modulus of fine aggregate was found to be 2.78 in conformity to ASTMC-136–96a. The reason for indigenous selection of coarse aggregate was based ideal for both cold and hot climate of Peshawar with economical transportation expenses.

2.2. Phase change material
Phase change materials (PCMs) are those materials that utilize the latent heat of fusion to absorb thermal energy. Among others, Paraffin wax is a commercially available organic phase change material in the form of CH₃(CH₂)nCH₃. PCM-based coated coarse aggregate is shown in figure 2.

Physical characterization of paraffin wax (Phase Change Materials) is shown in table 1. It is cheap and has a reasonable thermal storage energy density of 120 kJ kg⁻¹ up to 210 kJ kg⁻¹. Moreover, paraffin wax is effectively used in building construction due to its low thermal conductivity up to 0.2 W m⁻¹ K. The novelty of this research work is also due to PCM coating on concrete which offers high strength and high thermal conductivity. The epoxy layer was coated above phase change materials to reduce the sticky nature of phase change materials with cement.

Selection of CH₃(CH₂)nCH₃ was done on melting point higher than human comfort level range and mechanically layer stability. PCM epoxy layer brought from the local market was coated through dip methodology. It was produced by combining its two ingredients; one part was resin (transparent) and the other was hardener (yellowish) with a mix ratio of 1:1. Encapsulated coarse aggregates (ECA) with phase change materials ratios of 25%, 50%, 75%, and 100 v/v% with the cement were represent form ECA–25,50,75, 100 respectively.

Table 1. Physical properties of epoxy (Chemdur-300).

| Parameters                  | Values                        |
|-----------------------------|-------------------------------|
| Color                       | Grey                          |
| Density                     | 1.31 kg l⁻¹                   |
| Open time                   | 35 °C for 30 min              |
| Viscosity                   | Pasty does not flow           |
| Application temperature     | +15 °C to 55 °C               |
| Curing                      | 7 days, 23 °C                 |
| Tensile strength            | /mm 2                         |

Figure 2. Coated and uncoated PCM-based coarse aggregate.
2.3. Development of macro encapsulated phase change materials

The first step in the preparation of NCA-PCMs is to absorb paraffin into the pores of normal coarse aggregate. Sample immersion of NCA in water results from the absorption of 1.23% in 1 h because the pores of NCA are blocked with air. PCM absorption through simple immersion would be even much lesser than water because of its high viscosity. The effective and enhanced absorption of PCM into NCA was made possible through a vacuum impregnation rig facility as shown in the figure. At first, washed and oven-dried NCA was kept inside the vacuum chamber for 30 min to remove air from porous as shown in figure 3. The melted paraffin was then allowed into the vacuum chamber, and maintaining a pressure of $-0.1$ MPa the melted paraffin and NCA were further vacuumed for 60 min. During this whole process vacuum chamber was kept in a water bath maintaining the temperature of 50°C–60°C to keep the paraffin in melted form. In the end, NCA impregnated with paraffin was taken out and kept in a refrigerator below 4°C to ensure that paraffin gets solidified within pores of NCA.

2.4. Development of aggregate concrete for thermal energy storage and release

Concrete was prepared from PCM-based coated coarse aggregated of different ratios with cement and water. Five different concrete test specimens of size $16 \times 16 \times 2$ inches were prepared for further analysis as per standard of ACI 211.1; method for exploration of specimens at various ages i.e., 7, 14, and 28 days. Constant
water to binder ratio of 0.45 was used in this research work. Normal Coarse aggregates were pre-washed and oven-dried for 24 h before encapsulation.

3. Methodology

Selection of coarse and fine aggregate was done on the basis of moisture contents, water absorption, specific gravity, dry rodded density, fineness modulus, and sieve analysis as per ASSHTO T 84 and ASTM C-128 standards for further analysis. The saturated paraffin was mixed with cement and sand to cast samples for testing at 7, 14, and 28 days' cylindrical strength of control and modified concrete employing percentages of 0%, 25%, 50%, 75%, and 100% of encapsulated normal coarse aggregate. Phase change materials were coated in different ratios on coarse aggregate after mixing and molded in the slab plate as shown in figure 4. The experiments were performed in three phases. In the first phase, an increase in temperature was recorded after casting PCM-based coated concrete. The temperature deviation was observed to decline after getting its peak values. The test was terminated when the inclined temperature difference attains the maximum value in all 5 mixes. In the second phase, besides the compressive strength test ultrasonic pulse velocity (UPV) was measured at 7, 14, and 28 days of moist curing. The compressive strength behavior and UPV values were also recorded on the concrete slab plate. Meanwhile, the chemical analyses for all the specimens were conducted using XRF, TGA/DTA, SEM, and XRD analysis. In the third phase, a study was conducted to develop a temperature prediction exponential model, based on the temperature conduction equation. Surface analysis was done through Scanning Electron Microscope (SEM), and thermal conductance was analyzed in the temperature sensor room as shown in figure 5. The ratio of the density of a material to the density of distilled water at a given temperature, where the value being dimensionless is considered as the specific gravity of an aggregate. The specific gravity of coarse aggregates is assessed in the bulk dry, saturated-surface dry, and apparent conditions. The specific gravity test was performed in conformity to ASSHTO T 84 and ASTM C-128, the results obtained were 2.67. The water absorption recorded for the fine aggregate sample was 0.60.

The thermal analyzer system contains four sensors namely C1, C2, C3, and C4 as shown in figure 5. C1 is a thermal sensor that shows atmosphere temperature, while C2 is at a 5-inch distance from the heating source inside the thermal analyzer box with a controlled temperature at 50 °C while C2 and C3 are at a 5-inch distance from each other. Similarly, C3 and C4 are separated by a PCM-based coated concrete plate with a thickness of 2 inches. The thermal analyzer box is perfectly heat-insulated from all the surrounding sides by 6 inches of thermoform materials. Heat sources were 50-watt tungsten bulb was used at inside thermal analyzer system with constant voltage.
4. Results and discussions

4.1. XRF analysis of Sand, Coarse aggregate, and cement

Table 2 shows the percentage analysis of encapsulated coarse aggregate (ECA) ingredients such as sand, cement, and coarse aggregate. Mass (%) ratios show that SiO₂ and Al₂O₃.

| Chemical composition | Sand (mass %) | Coarse aggregate (mass %) | Cement (OPC) (mass %) |
|----------------------|--------------|---------------------------|----------------------|
| SiO₂                 | 54.98        | 68.31                     | 22.51                |
| Al₂O₃                | 17.54        | 14.45                     | 6.25                 |
| Fe₂O₃                | 5.33         | 4.68                      | 1.14                 |
| CaO                  | 5.40         | 3.48                      | 65                   |
| MgO                  | 0.20         | 1.87                      | 1.07                 |
| MnO                  | 0.00         | —                         | 0.02                 |
| Na₂O                 | 0.95         | 2.41                      | 0.13                 |
| K₂O                  | 0.09         | 3.07                      | 1.3                  |
| P₂O₅                 | —            | —                         | 0.06                 |
| TiO₂                 | —            | —                         | 0.14                 |
| L.O.I.               | 14.50        | 1.81                      | 2.44                 |

Figure 6. Thermal differences between C3 and C4 sensor.

Figure 7. Compressive strength of ECA (0, 25, 50, 75, and 100%) after 7, 14, and 28 days with beam test.

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The aggregate percentage of silica and alumina oxide was found from sand, cement, and coarse aggregate in the range of 2.0 to 3.0 which is sufficient for further synthesis processes to achieve the desired physical properties of concrete. Furthermore, Fe₂O₃, CaO, and MgO were also found in the ranges, which is helpful in strong bonding reactions during mixing.

4.2. Thermal conductance
The sensitivity of phase change materials for latent heat release and absorption is due to its transition from liquid to solid and vice versa. The synthesized PCM-based concrete of different ratios was placed between sensor C3 and C4 in the thermal analyzer box as shown in figure 5. It was observed that the heat storage capacity of concrete is significantly increased when PCM was applied on aggregate. Effectiveness in thermal conductance of phase change materials was observed in the range of 25 to 75%, while high thermal conductance was observed at 100%, with the compressive strength of 1700 psi which is not suitable for further application as per ASTM-standards as shown in figure 6. In thermal conductance of 12 h, the temperature difference of 12.35, 15.25, 16.35, 17.5, and 20.3 °C was observed for ECA-100, ECA-75, ECA-50- ECA-25, and NCA, respectively. ECA-100 and ECA-75 offer high thermal conductance but low compressive strength, therefore, ECA-50 was considered suitable for further analysis and application of thermal storage.

4.3. Compressive strength
Compressive strength is one of the fundamental properties of concrete which can be found by the maximum load applied on test specimen before failure [2]. The innovation in this research work is the optimization of PCM-based concrete with compressive strength and thermal energy management. Many researchers reported that the addition of PCM reduced significantly the overall compressive strength of concrete. Panayiotou et al., [9] reported that the innovated PCM-concrete was found to achieve a compressive strength of over 25 MPa and
tensile splitting strength of over 6 MPa (after 28 days) which are appropriate levels for some structural application purposes. However, in our case, compressive strength analysis was done in the ratio of phase change materials employing PCM-based aggregate varying from 0%, 25%, 50%, 75%, and 100% at 7, 14, 28 days as shown in figure 7. For compressive strength, all the samples were fabricated having 4 × 4 × 8 cm dimensions for the ‘INSTRON’ mechanical press. Results show that ratio of phase change materials in PCM based concrete increase, compressive strength decrease. It was found that the compressive strength of 50% PCM-based concrete remains the same and suitable for structural application. However, a high ratio of PCM-based concrete reduced the compressive strength below 2000 psi which is not suitable for building construction. The decrease in compressive strength is due to limited dispersion of phase change materials which causes leakage. Other researchers used the same methodology for compressive strength analysis but the reason for the decrease was reported on the incorporation of Phase-change material instead of coating technique with epoxy layer.

4.4. Scanning electron microscope
Surface analysis of PCM-based concrete was done through visualization of scanning electron microscopy (SEM) with the magnification of 20 Kv, 50 μm for pore size, porosity, and surface cracks morphology [2]. SEM micrograph shows clearly the coated PCM in the mixed concrete for thermal storage and release. Further, no pine hole cracks were observed in encapsulated PCM-based concrete and all are equally dispersed in the concrete matrix, which represents a homogeneous concrete mix. No apparent pine hole cracks show that PCM did not affect the overall bonding mechanism as shown in figure 8.

4.5. UPV correlation with compressive strength
Ultrasonic pulse velocity (UPV) test is an in situ, non-destructive test to investigate the quality of mixed concrete [18]. In this test, the strength and quality of concrete were assessed by measuring the velocity of an ultrasonic pulse passing through a concrete structure. This test is conducted by passing a pulse of an ultrasonic pulse through concrete to be tested and measuring the time taken by the pulse to get through the specimen. Higher velocity indicates good quality and continuity of the material, while slower velocity may indicate concrete with many cracks or voids. In our research work, UPV correlation was done for compressive strength as shown in figure 9.

To investigate the post-construction performance of PCM-based concrete, the investigation was carried out for developing the relationship between the ultrasonic pulse velocity (UPV) and the compressive strength analysis of 50 v/v% PCM-based concrete after 28 days as shown in figure 9. Results show that 50 v/v% PCM-based concrete for 28 days has pulse velocity ranges from 3.00 to 4.50 km sec−1 which verifies the suitability for stressed applications. Mahure et al [14] reported that UPV and compressive strength correlation are the key characterizations to monitor the post-construction performance of concrete.

5. Conclusion
Thermal energy storage through phase change materials is a sustainable technology that aims to improve building insulation and renewable energy conservation. This research found that a 50 v/v PCM-based concrete provides a reliable physical strength as well as optimal heat conduction in all applications. ECA-100 and ECA-75 encapsulated coarse aggregates have a high sensitivity for thermal energy conduction but a low compressive strength, making them unsuitable for use in higher stress environments. It was also discovered that a high ratio of PCM reduced the compressive strength and other physical properties of concrete while increasing the percentage thermal conductance. It was concluded that PCM-based concrete makes the indoor environment comfortable with a ratio of 50% in the concrete. However, other parts of this study effort include the inclusion of carbon nanotubes (CNTs) to improve compressive strength without affecting heat conductance.

Data availability statement
All data that support the findings of this study are included within the article (and any supplementary files).

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