

Application of form-stable paraffin/nano-silica phase change materials for thermal energy Storage in mortar

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Abstract: In this study, PCM based on paraffin's adhered to a silica-based matrix appear to be a suitable option in the search for techniques for incorporating Phase Change Materials (PCM) into Portland cement mortar mixtures. When water is added, though, paraffin particles have been seen to elude the silica matrix. There is a paucity of information on how the use of such PCM impacts mortar behavior. Portland mortar mixtures with 15%, 20%, 25% and 30% PCM content were developed using Portland cement in order to assess the impact of this PCM addition. Both fresh and dry samples were examined for physical characteristics like concentration, accessible pore volume, relative density, and water absorption. According to the results, more cement and water are needed in the PCM-Silica added mixtures compared to regular mortar mixtures to achieve comparable compressive strengths & flexural strength. Here is 20% PCM mixtures had a highest tensile strength of 13.45 MPa, whereas 25% PCM mixtures had values 10.99 MPa. As a result, the proposed study broadens our knowledge of PCM cement mortar mixture behavior and proportioning.

Keywords: Cement-Based Materials, PCM, Thermal Conductivity, Paraffin, Silica.

I. INTRODUCTION

Paraffin hydrocarbons are any saturated hydrocarbons with the general formula C_nH_{2n+2} , where C is a carbon atom, H is a hydrogen atom, and n is an integer. By incorporating phase change materials (PCM) into building materials, it is possible to increase the energy storage in walls, ceilings, and floors. These PCM can improve a building's overall thermal performance, increase thermal inertia and indoor thermal stability, increase the use of renewable energy, enable passive cooling, and more [1,2,3]. Depending on their composition and properties, the wide variety of PCM that are currently available are categorized as organic, inorganic, and eutectic materials [4, 5, 6]. Different methods [7] such as direct application.

There has been a lot of research done on PCM addition to mortar and concrete mixtures. They can be used by direct mixing, immersion, and impregnation in porous aggregates [9]. The research on PCM impregnation in light sands [10], the works on PCM impregnation in concrete blocks [11], the use of burnt clay aggregate to improve the thermal properties of a concrete panel [12], the research on the impact of light sand impregnated with PCM over the thermal behavior of concrete during setting and freeze conditions [13], are a few examples. The creation of cement-based composite phase change materials (CCPCMs) based on the hydraulicity of cement and the water solubility of polyethylene glycol (PEG) [14] and the creation of salt hydrate/diatomite PCM coated with polyurethane acrylate that has improved thermal stability [15].

On the other hand, the thermal properties of cement-based materials modified with PCM for use in building construction have been studied experimentally and numerically [16], and more recently [17] a review of the potential of microencapsulated PCM for energy savings in buildings has been made. Applications of PCM based on paraffin nucleus and polymeric shells to Portland cement mortars have so far shown changes in the mixtures, including a decrease in resistance, an increase in water content, a decrease in thermal conductivity, and an increase in heat capacity [18, 19, 20, and 21].

A study was conducted on the direct addition of PCM particles to mortars by Cunha et al. [22], confirming that mortar mixtures perform well in terms of their physical and mechanical characteristics. This also occurs when PCM are mixed with other substances like silica [23, 24], graphite [25], or diatomite [26, 27]. It has also been investigated how Portland cement concretes (PCC) and microstructure are affected by microencapsulated phase change materials (MPCM) [28]. The results indicated that as the amount of MPCM increased, the compressive strength of PCC decreased. Forest biomass ashes (FBA) have recently been studied in cement-based mortars [29]. The findings indicated a slight reduction in mechanical strengths and an increase in ductility. Moreover, it has been claimed that the use of these mortars is not limited by the addition of FBA waste.

Portland cement mortars may contain one of two types of PCM: protected PCM, which is protected by a stable shell, or non-protected PCM, which is not protected by this kind of shell [30]. In this study, a silica-based matrix with poly-nuclei of paraffin was used to create protected PCM from dry dust micro particles. They have a serious drawback that can cause micro particle leakage during mixing: silica and paraffin separate when they come in contact with water. Alternative solutions to this problem have been put forth by some authors [31], who modified the PCM using three different types of nano-silica. The main disadvantage of non-protected PCM is that they can be filtered either during the mixing process or when applied to porous materials. This type of PCM also has the advantage of having a higher heat-storage capacity than inorganic PCM due to the absence of a shell [32]. Due to the lack of a PCM encapsulation process and the consequent avoidance of the need for complicated incorporation techniques, the direct incorporation of non-encapsulated PCM also enables a cost reduction, and thus represents an innovative and promising way to significantly improve the energy efficiency of buildings [33, 34].

In Portland cement mortars, dry dust micro particles are conformed by a silica-based matrix with poly-nucleus of paraffin. This paper focuses on the mechanical and physical behavior of protected PCM based on these dry dust micro particles. These mortars were chosen because they could accommodate paraffin particles and worked well with the silica matrix. This type of PCM gives mortars the advantage of increasing their capacity to store heat, having a lower retraction, and avoiding issues with thermal conductivity, making them suitable for thermal applications. On the other hand, their primary drawback is the potential PCM leak when using a direct method to be incorporated into the mixture, which has been proposed as the cause of the dearth of research studies [8].

Different mixtures with various PCM contents, water to cement ratios (w/c), and two types of cement were developed with the intention of analyzing the impact of PCM addition to Portland cement mortars. Properties such as apparent density, water absorption, open porosity, compressive strength, air content, and behavior analysis after exposure to 35 °C were studied. Since compressive strengths, types of cement, water/cement ratios, and air content are all studied, the obtained results have a direct application in the mixing design process. In addition, cooling methods were used, in contrast to other studies, to prevent paraffin leaks during the mixing process.

II. MATERIALS

Raw Materials: Portland PO 42.5 cement produced by Taiyuan Co.Ltd.based on the relevant Chinese standard was used. The polycarboxylate superplasticizer (solid content of 20%) was customized by the manufacturer. Standard sand, which conformed to the Chinese ISO sand standard GB/T17671, was obtained from Xiamen Aisio Standard Sand Co.Ltd. Tap water was used for the experiments.

III. EXPERIMENT



Figure:1 Photograph of paraffin

Cement mortar fluidity test and mix ratio selection:

A fluidity test was performed in accordance with GB/T2419-2005 using a jumping table to measure the consistency and work ability of newly formulated mortar. The poly-carboxylate superplasticizer content in the cement mortar was determined by performing a fluidity test.

Preparation of cement mortar samples:

Cement mortar samples with different contents paraffin were prepared. The mixing proportions of the samples are listed in **Table 1**; the water: cement ratio is 0.4, and the cement: sand ratio is 1:2. The dosage of the water reducer was increased with an increase in the dosage of paraffin to a consistent fluidity for each cement mortar sample. Sample M is plain cement mortar.

The cement mortar was prepared as follows:

–The carbon-fiber mixed solution and cement were added to a mixing pot, and the pot was placed on a fixed frame. The mixture was mixed at a low speed for 30s. Standard sand was evenly added via a mixing funnel after 30s, and the mixture was then mixed at a high speed for 30s.

–After the mixer stopped, any material that collected on the side of the bowl was immediately scraped down into the batch. Thereafter, the mixer enclosure was closed, or the bowl was covered with a lid, and the paste was left to stand for 90s.

–The mixture was mixed for 60 s at a high speed.

–The fresh cement mortar was poured into a steel mold and compacted using a standard vibrating table. The molds were then sealed with polyethylene Nano-sheets to prevent the loss of moisture. After 24h, the samples were demolded and cured in a saturated lime-water bath at 20°C for specific aging durations (3, 7, and 28 d).

Table 1: Mixing proportions of paraffin/silica cement mortar samples

Cement(g)	Sand(g)	Water(g)	Paraffin(g)	SiO ₂ (g)	PC(g)
468	1200	240	120	12	5
501	1200	240	90	9	4
435	1200	240	150	15	6.5
402	1200	240	180	18	8.5

IV. MECHANICAL TEST

The flexural and compressive strengths of the specimens were determined according to GB/T17671-1999 after 3, 7, and 28 d. For each series, three specimens were tested to determine their strength. The flexural strength test was performed in a three-point bending test apparatus with a loading rate of 0.06N/s. The loading rate for the compression strength test was 2.4KN/s.

SEM characterization of hydration products:

Scanning electron microscope (SEM) is a method that can directly observe the microscopic morphology of the material by imaging the material properties on the surface of the sample. The working principle of SEM is to use an electron lens to reduce a single electron beam spot to a nanoscale size, and then use a deflection system to make the high-energy electron beam raster scan on the surface of the sample to be tested, thereby exciting secondary electrons and other various physical information. It is collected and converted into a signal by the detector, and then the image is displayed. In this study, the scanning electron microscope model ZEISS Gemini SEM 300 is used, and its scanning speed is 20ns-10ms per pixel, and the magnification is 1-1 million times. The equipment is shown in **Figure 2**. In addition, the samples for SEM testing must be dry, and due to the continuity of cement hydration, cement hydration suspension treatment is required before SEM observation. Suspension of hydration and drying can generally be carried out together. In this experiment, a combination of solvent substitution and vacuum drying was used. Solvent substitution refers to the use of mutual solubility between water and organic solvents to displace water from the sample. In this experiment, the samples were soaked in absolute ethanol for 24 hours, and then the samples were placed in a vacuum drying oven at 60 °C for 6 hours to make the absolute ethanol volatilize from the samples.



Figure:2 Scanning electron microscope (SEM)

V. RESULT & DISCUSSION

Effects of the paraffin on the workability of cement paste:

The fluidity of the thermochroic materials Compared with cement mortar M, when the thermochroic content is 15%, the fluidity of cement mortar decreases by 10.9%. When the thermochroic content is 20%, the fluidity of the cement mortar decreases by 18.48%. The fluidity of cement mortar decreases with an increase in the thermochroic content, possibly owing to the distribution and orientation of the thermochroic materials. Therefore, the addition of a water-reducing agent to the samples ensures consistency in their fluidity values and enables the comparison of their strengths. Below shows Figure:3.

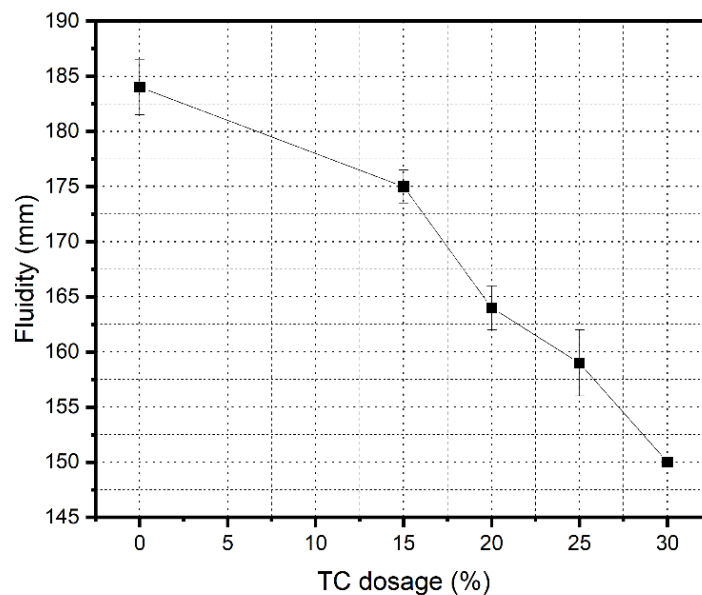


Figure:3 Fluidity of the cement mortar samples mechanical strength of paraffin -modified cement mortar

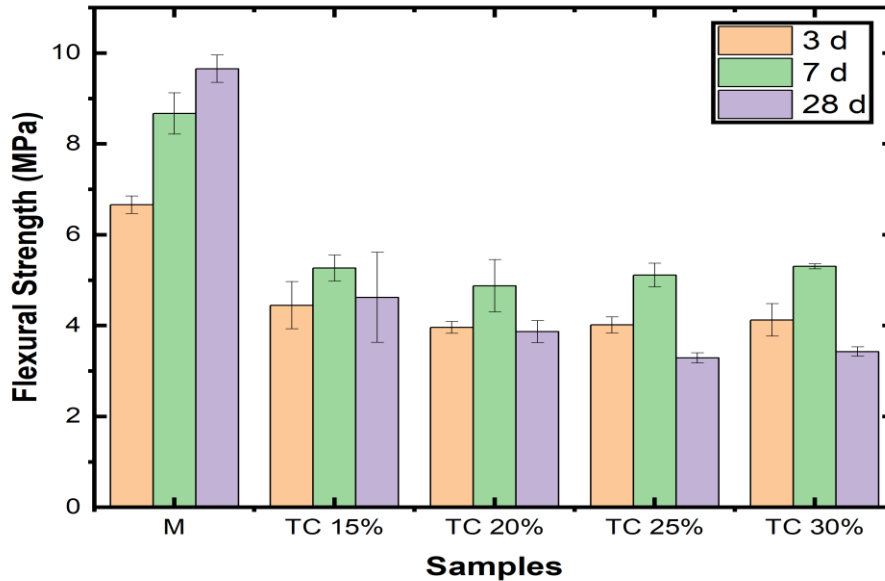


Figure: 4 Flexural Strength

Figure:4 shows the flexural strength of the paraffin mixed with different amounts of paraffin at 3, 7, and 28d. The flexural strength of cement mortar gradually increases with an increase in the paraffin content. However, after an optimal paraffin content, the flexural strength starts decreasing. When the paraffin content is 15%,20%,25%,30%, the flexural strength of the cement mortar mixed with paraffin is at the highest. At curing ages of 3d & 7d, the flexural strength of Sample TC increases by 5%,4.5%, and 5.2%, 5.5%, respectively, compared with that of plain cement mortar Sample M because the high strength of paraffin improves the mechanical properties of the cement mortar.

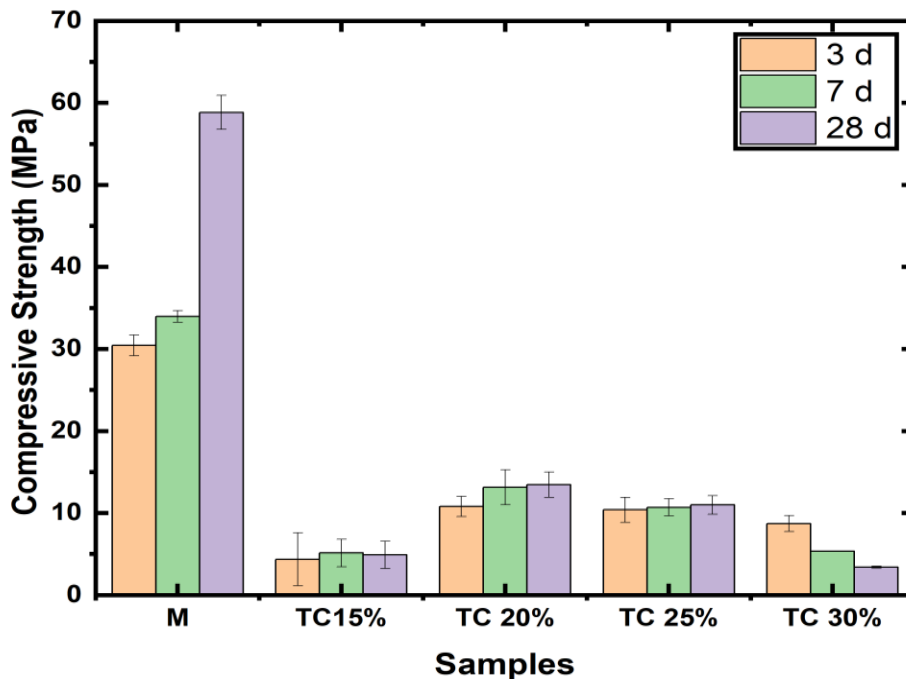


Figure: 5 Compressive Strength

Figure:5 shows the compressive strength of the cement mortar mixed with different amounts of paraffin at 3, 7, and 28 d. When the curing ages are 3, 7 & 28d and the paraffin content is 20% & 25% (Sample TC), the compressive strengths of the cement mortar are 10.5% and 10.4% higher than that of the plain cement mortar respectively. However, when the paraffin content continues to increase, the compressive strength of the cement mortar does not increase and is lower than that of the plain cement mortar. The reason for this phenomenon is the uneven distribution of the paraffin and the increased porosity. At a curing age of 28 d, the compressive strength of all the mixed cement mortar samples doped with paraffin is lower than that of the plain cement mortar. This maybe owing to the uneven dispersion of paraffin that trap the flow of free water in the cement slurry, thereby reducing the degree of hydration.

SEM results: SEM analysis was performed to investigate the paraffin distribution, adhesion, and failure mechanisms.

Figure:6 show the SEM images of the plain cement mortar, revealing microstructures of the ITZ. Cracks and pores are observed in the ITZ, and a direct combination of aggregates and hydration products is not visible. show the SEM images of cement mortar samples with 15% Paraffin. The Paraffin are distributed in the cement matrix in the confusion. More Paraffin are present in the 20% Polycarboxylate-Superplasticizer sample. Therefore, an increase in the Paraffin/ nano-silica content enhances the conduction of the cement mortar. However, when the Paraffin/ nano-silica content reaches a certain level, the mechanical properties of the cement mortar deteriorates. The nano-silica content has significant effects on the mechanical properties of cement mortar because the mechanical properties are sensitive to the aggregation of paraffin.

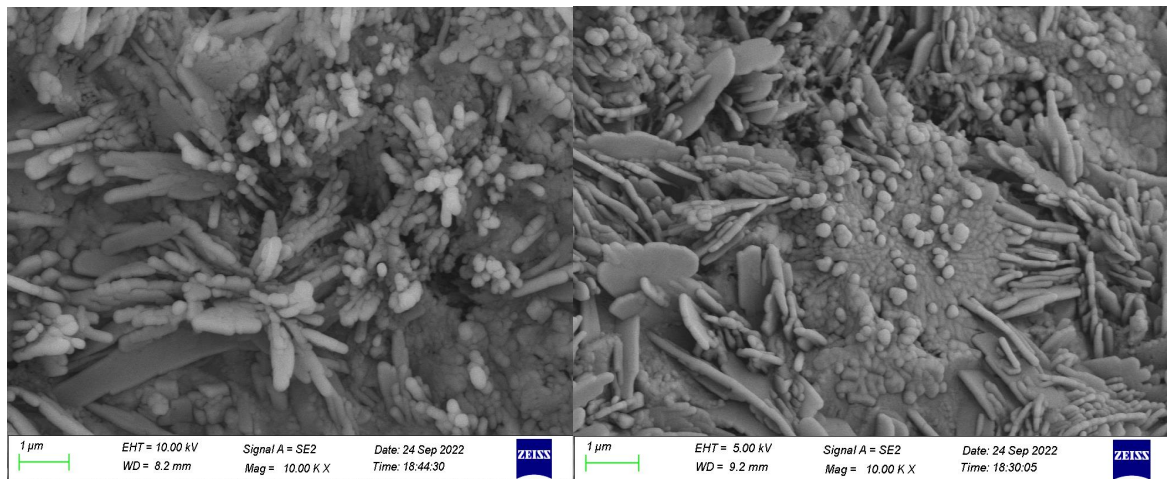


Figure:6 SEM image

Screening of paraffin samples:

In this experiment, differential scanning calorimetry (DSC) was used to test the thermophysical properties of a paraffin mixture. The phase transition temperature and latent heat of phase transition were determined by analysis software.

Calibrate the differential scanning calorimetry system. A nitrogen atmosphere was used in the test, high-purity nitrogen was used as a protective gas, and blowing with sweeping gas and liquid nitrogen cooling, the protective gas flow rate is 65 ml/min and the sweeping gas flow rate is 15 ml/min.

Weigh 5 mg of paraffin wax sample, put it in a small aluminum dry pan with a cover with a diameter of about 7 mm, and place it in a scanning calorimeter test system.

Determination of latent heat of phase change of the sample Set the experimental temperature range from -20 °C to 100 °C. Using liquid nitrogen, we first cooled the sample to -20 °C and then increased the temperature at a rate of 5 °C/min. The system automatically records the sample. Curves and data of heat flow versus temperature The phase transition latent heat and phase transition temperature of 20% paraffin samples with different solid-to-liquid ratios were measured in the experiment. The peak temperatures and overall curve shape of the phase-changing process were found to be magnified by DSC testing with higher heating/cooling rates. As the rate of heating or cooling rises, the hysterical reaction is also accentuated. The DSC of solid paraffin is shown in **Figure 7**.

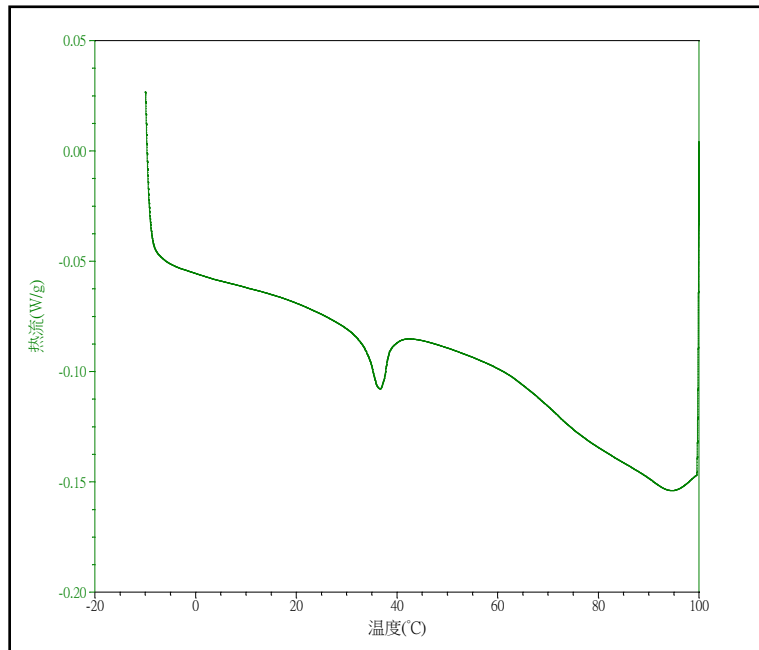


Figure:7 DSC Curve

Energy Performance:

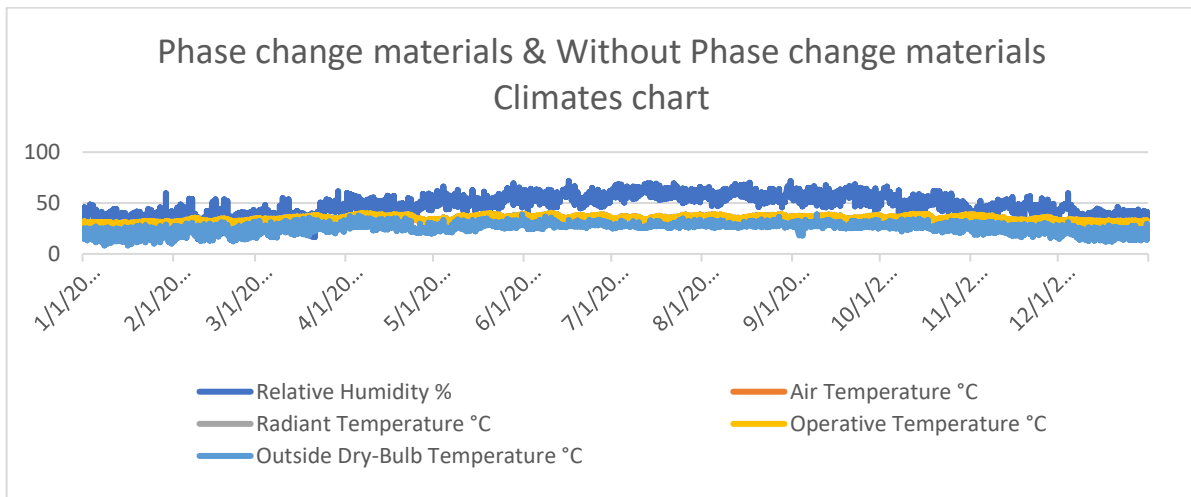
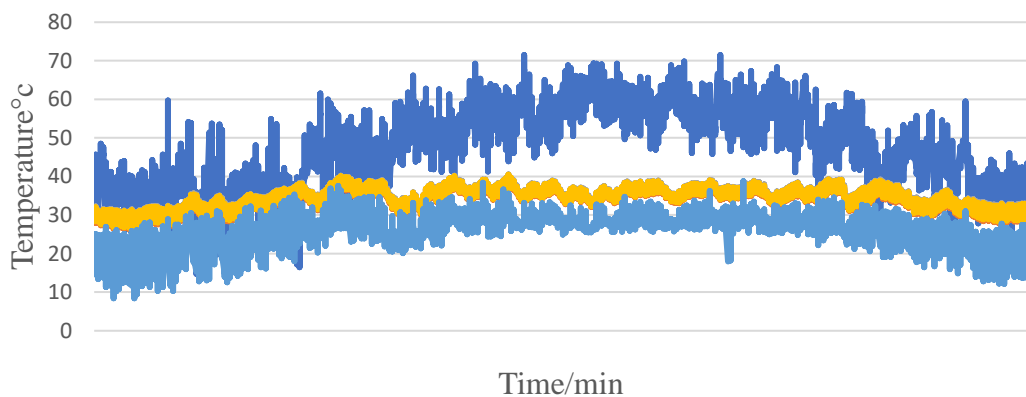


Figure:8a

Phase change materials (PCM) With Climates



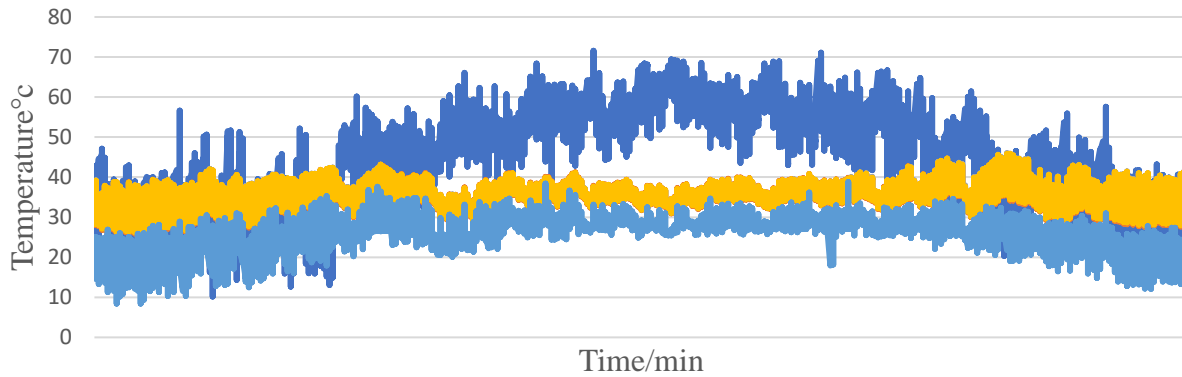


Figure:8b

Without Phase change materials (PCM) Climates

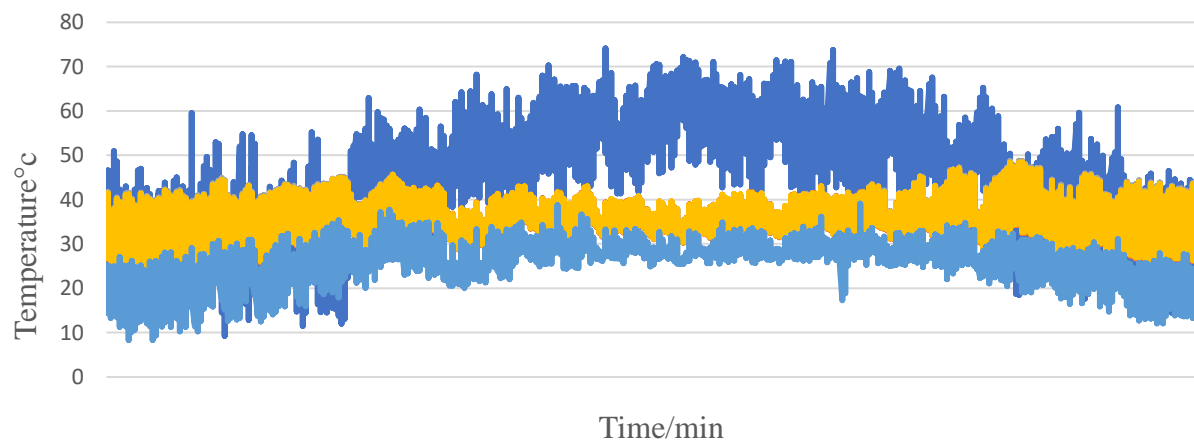
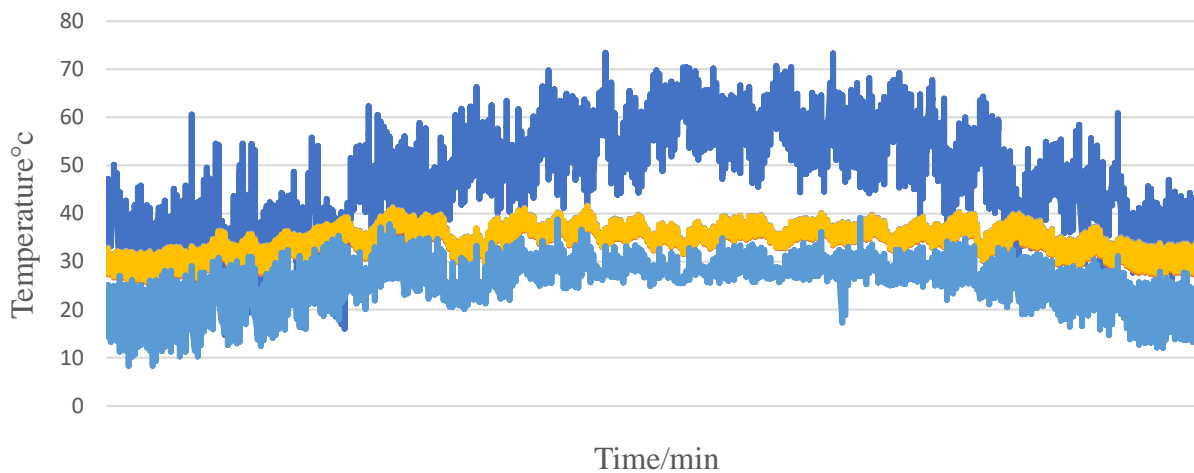


Figure:8c

Figure:8(a, b &c) Phase change materials Climates & Without Phase change materials Climates. Date/Time, Relative Humidity, Air Temperature, Radiant Temperature, Operative Temperature, Outside Dry-Bulb Temperature.

Figure:8(a, b&c) shows daily temperature ranges for the present-day and morphed future climates in the 2022. Although the climates are generally getting warmer throughout the year, the increase in winter temperatures is more than that of the summer temperatures. Diurnal variations are generally greater during winter months (January to April) than summer (April to September) and monsoon (September to January) months in the present-day climate. This meteorological pattern is retained in future climates. However, with the increase in temperature, in particular in the 2022 June climate, the length

of winter season is shortened. The warming of the winter months is explored further in (March to November), which shows changes in the daily mean temperature in future climates from the present-day baseline climate. Polynomial curve-fits of the data highlight this trend in different climates with marked seasonal variations. Operative Temperature are with PCM or without PCM both billow in this year 2022. There are comparison between with PCM or without PCM.

VI. CONCLUSION

Based on the perspective of energy saving, emission reduction and building energy saving, this subject is tested for various types of paraffin, Fatty acid was investigated, and the 52# solid compound with a certain proportion suitable for use under normal temperature conditions was screened out. A mixture of paraffin and liquid paraffin is used as a phase change material, and expanded perlite and diatomaceous earth are selected as matrix materials. The material is prepared by heating and vacuum adsorption, and the shape-changing material is encapsulated. use system The obtained shape-changing phase change material is made into shape-setting phase change energy storage sand by direct mixing and intercalation methods. slurry, its mechanical properties and thermal properties are tested, and the temperature adjustment performance test of the house model is simulated and determined. The adjustment effect of shape change energy storage mortar board on room temperature.

(1) A paraffin mixture composed of 20% and 25%, tested by DSC It is found that the phase transition temperature of the paraffin wax mixture is 25 °C, the latent heat of phase transition is 111.5 J/g, and the thermo gravimetric Tests and thermal cycle tests show that the paraffin wax mixture has good thermal stability and durability, suitable for Used in building walls;

(2) The expanded perlite after acidification has a large number of honeycomb pores, which can absorb a large amount of paraffin. After coating with calcium silicate powder, the thermal stability is good; the diatomite after roasting and acidification treatment is mostly sieve-like and Cylindrical shape, large porosity, large surface area, nano- silica pore size, is an excellent porous adsorption material; All kinds of porous materials can be used in the preparation of shape-fixed phase change composites.

(3) SEM results show that expanded perlite and diatomite can be greatly enhanced by heating and vacuum adsorption. The amount of paraffin mixture is adsorbed, and the infrared test results show that the adsorption method of the two to paraffin is physical absorption. Attached, no chemical reaction occurs. After paraffin/expanded perlite is encapsulated by calcium terephthalate powder, the exudation rate is low, Good thermal stability; diatomaceous earth has a large number of nano-silica pores inside, and its surface tension has good resistance to paraffin The binding force of paraffin wax is not easy to seep out; the prepared paraffin/expanded perlite and paraffin/diatomaceous earth set the phase transition The material has stable performance and is suitable for the field of building materials.

(4) The phase change energy storage mortar was prepared by intercalation method and direct mixing method. With the increase of the dosage, the compressive strength, bulk density and thermal conductivity of the phase change mortar gradually decreased; the intercalation method The performance of the phase change energy storage mortar prepared is better than that of the phase change energy storage mortar prepared by the direct mixing method. When the content is increased to 20%, the strength is still higher than 13.45 MPa, the mechanical properties meet the actual construction requirements, and its thermal conductivity The coefficient is extremely low, and the heat storage coefficient still maintains a certain level, which has good heat preservation and heat storage effects. Intercalation It is a better preparation process of phase change energy storage mortar.

(5) The temperature adjustment performance test of the mortar board house model shows that under the same heating conditions, the PCM mortar The air temperature in the board house model is generally lower than that of the ordinary mortar board house model, and with the increase of PCM content, The temperature at each time point in the mortar board house model also decreased gradually, and when the dosage was 15 mm, the highest The air temperature dropped from 45 °C for plain mortar to 30.1 °C and 30.1 °C for EP-PCM mortar board house model respectively DP-PCM mortar board house model is 34.4 °C; during the cooling process, the temperature inside the mortar board house model The density increases with the increase of PCM content. This shows that the phase change energy storage mortar can pass through the phase change material Variable reaction absorbs and releases heat in the environment, adjusts the temperature of the environment, and improves the thermal insulation performance of the building structure and thermal insulation properties.

(6) Here energy performance result indicates that Phase change materials Climates & Without Phase change materials Climates. Date/Time, Relative Humidity, Air Temperature, Radiant Temperature, Operative Temperature, Outside Dry-Bulb Temperature in year 2022.

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