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Utilization of Form-Stable Paraffin/Nano-Silica Phase Change Materials for Thermal Energy Storage Enhancement in Mortar

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***Keywords*— Cement-Based Materials, Paraffin, Silica, Thermal Conductivity, Thermal Control Effect Test.**

Abstract— *This research explores the utilization of form-stable paraffin/nano-silica phase change materials (PCMs) to improve thermal energy storage in mortar. The composite PCM, comprising paraffin enclosed within a nano-silica framework, offers enhanced stability and compatibility with mortar compared to traditional PCM systems. Experimental investigations were conducted to assess the thermal performance and mechanical properties of mortar specimens incorporating varying concentrations of form-stable PCM. Thermal conductivity, heat storage capacity, and mechanical strength were analysed through thermal cycling tests and mechanical testing methods to understand the impact of PCM content. Results demonstrate that integrating form-stable paraffin and nano-silica PCMs enhances thermal energy storage capabilities in mortar while minimally affecting mechanical strength. Moreover, the nano-silica matrix facilitates better dispersion and adhesion of paraffin particles within the mortar matrix, addressing concerns related to PCM leakage and separation during hydration. This study underscores the viability and efficacy of employing form-stable paraffin and nano-silica PCMs to enhance thermal energy storage in mortar, offering a promising avenue for sustainable and energy-efficient construction materials.*

I. INTRODUCTION

Paraffin hydrocarbons are saturated hydrocarbons with the formula C_nH_{2n+2}, where 'n' represents an integer, 'C' stands for carbon, and 'H' stands for hydrogen. Incorporating phase change materials (PCM) into building materials offers numerous benefits. PCM integration enhances energy storage in walls, ceilings, and floors, thereby improving a building's thermal performance. Additionally, PCM can increase thermal inertia and indoor thermal stability, promote the use of renewable energy sources, facilitate passive cooling strategies, and offer various other advantages **[1,2, 3]**. PCM materials come in diverse compositions and properties, typically classified as organic, inorganic, and eutectic materials **[4,5,6]**. Various methods **[7]**, including direct application, are employed for integrating PCM into building materials.

This study focuses on the mechanical and physical behaviour of protected phase change material (PCM) incorporated into Portland cement mortars. The PCM consists of dry dust microparticles conforming to a silica-based matrix with a poly-nucleus of paraffin. Portland cement mortars were selected for this investigation due to their ability to accommodate paraffin particles and effectively interact with the silica matrix. This particular type of PCM offers several advantages for mortar applications. It enhances the mortar's heat storage capacity, reduces retraction, and mitigates issues related to thermal conductivity, thereby making it well-suited for thermal applications. However, a significant drawback of this PCM

is the potential for PCM leakage when employing direct methods for its incorporation into mortar mixtures. This leakage issue has been identified as a primary reason for the limited research studies on this type of PCM integration **[8]**.

Numerous research studies have explored the incorporation of phase change materials (PCM) into mortar and concrete mixtures using various methods such as direct mixing, immersion, and impregnation in porous aggregates **[9]**. Here are some examples: PCM impregnation in light sands **[10]**, PCM impregnation in concrete blocks **[11]**, and the utilization of burnt clay aggregate to enhance the thermal properties of concrete panels **[12]**. Investigation into the impact of light sand impregnated with PCM on the thermal behaviour of concrete during setting and freeze conditions **[13]** has also been conducted. Moreover, significant advancements have been made in PCM technology, leading to the development of innovative materials like cement-based composite phase change materials (CCPCMs). These materials leverage the hydraulic properties of cement and the water solubility of substances like polyethylene glycol (PEG) **[14]** to enhance thermal performance. Additionally, PCM coatings have been developed, such as salt hydrate/diatomite PCM coated with polyurethane acrylate, which exhibit improved thermal stability **[15]**.

The thermal properties of cement-based materials modified with phase change materials (PCM) for building construction have been extensively investigated both experimentally and numerically, as highlighted in a study [16]. Additionally, a recent review **[17]** has examined the potential of microencapsulated PCM for energy savings in buildings. Studies on the application of PCM, particularly those based on paraffin nuclei and polymeric shells, to Portland cement mortars have revealed significant changes in the mixtures. These changes include a decrease in resistance. The incorporation of PCM into cement mortars has been associated with a decrease in mechanical strength or resistance and an increase in water content. PCM addition often leads to an increase in the water content of cement mortars and a decrease in thermal conductivity. PCM-modified mortars typically exhibit lower thermal conductivity, which contributes to improved thermal insulation properties and an increase in heat capacity. The presence of PCM in cement mortars enhances their heat storage capacity, enabling them to absorb and release heat more effectively **[18,19, 20,21]**. The study conducted by Cunha et al. **[22]** focused on the direct addition of phase change material (PCM) particles to mortars, demonstrating that mortar mixtures exhibit favourable physical and mechanical characteristics. Similar positive outcomes have been observed when PCM is combined with other substances such as silica **[23,24]**, graphite **[25]**, or diatomite **[26,27]**. Furthermore, investigations have explored the impact of microencapsulated phase change materials (MPCM) on Portland cement concretes (PCC) and their microstructure **[28]**. The results revealed that increasing the amount of MPCM led to a decrease in the compressive strength of PCC. Recently, forest biomass ashes (FBA) have been studied in cement-based mortars **[29]**. The findings indicated a slight reduction in mechanical strength but an increase in ductility. Additionally, it has been suggested that the use of these mortars is not hindered by the addition of FBA waste.

This study distinguishes between two types of phase change materials (PCM) that may be incorporated into Portland cement mortars: protected PCM and non-protected PCM. Protected PCM, characterized by a stable shell **[30]**, is created using a silica-based matrix with poly-nuclei of paraffin from dry dust microparticles. However, a significant drawback of protected PCM is the potential for microparticle leakage during mixing due to the separation of silica and paraffin upon contact with water. Some authors **[31]** have proposed solutions to this issue by modifying PCM using different types of nano silica. On the other hand, non-protected PCM lacks such a protective shell. Despite the risk of filtration during mixing or application to porous materials, non-protected PCM offers advantages such as higher heat-storage capacity compared to inorganic PCM, attributable to the absence of a shell **[32]**. Furthermore, direct incorporation of non-encapsulated PCM avoids the need for complicated incorporation techniques, leading to cost reduction. Consequently, direct incorporation of non-encapsulated PCM represents an innovative and promising approach to significantly enhancing the energy efficiency of buildings **[33–34]**.

In this study, various mixtures were developed with different Phase Change Material (PCM) contents, water-to-cement ratios (w/c), and two types of cement to analyse the impact of PCM addition to Portland cement mortars. Key properties such as apparent density, water absorption, open porosity, compressive strength, air content, and behaviour analysis after exposure to 35°C were investigated. The inclusion of compressive strengths, types of cement, water/cement ratios, and air content in the study provides valuable insights directly applicable to the mixing design process for Portland cement mortars. A notable aspect of the study was the implementation of cooling methods during the mixing process to prevent paraffin leaks. This approach differs from previous studies and ensures that the integrity of the PCM is maintained throughout the mixing process, thereby avoiding potential issues related to PCM leakage.

II. MATERIALS

Raw Materials:

In the study, Portland cement PO 42.5 produced by Taiyuan Co. Ltd., in accordance with relevant Chinese standards, was utilized. The polycarboxylate superplasticizer, with a solid content of 20%, was custom-made by the manufacturer for the experiments. Standard sand meeting the Chinese ISO sand standard GB/T17671 was sourced from Xiamen Aisio Standard Sand Co. Ltd. Tap water served as the primary solvent for the experimental procedures.

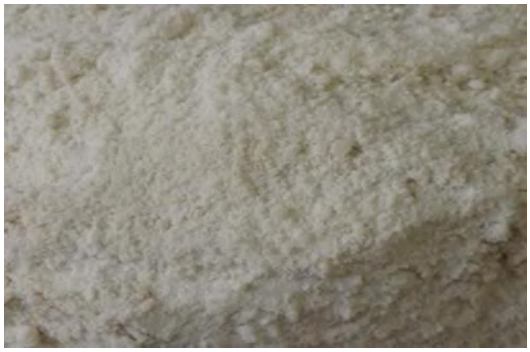


Fig.1: Photograph of paraffin.

III. EXPERIMENT

Cement mortar workability test and mix ratio selection:

A workability test was conducted following the guidelines outlined in GB/T2419-2005. The test employed a vibrating table to assess the consistency and workability of the newly formulated mortar. The content of polycarboxylate superplasticizer in the cement mortar was determined through this fluidity test, which provided insights into the mortar's flow characteristics and the effectiveness of the superplasticizer in enhancing workability.

Preparation of cement mortar samples:

Cement mortar samples containing varying amounts of paraffin **(Fig. 1)** were prepared according to the mixing proportions outlined in **Table 1**. The water-to-cement ratio was maintained at 0.4, and the cement-to-sand ratio was kept at 1:2 across all samples. To ensure consistent fluidity for each cement mortar sample, the dosage of the water reducer was adjusted proportionally with the increase in paraffin dosage. Sample M represents plain cement mortar without the addition of paraffin.

The cement mortar was prepared as follows:

1. A mixing pot was utilized, into which the paraffin-mixed solution and cement were added. The pot was then positioned on a fixed frame, and the mixture was stirred at low speed for 30 seconds. Following the initial 30 seconds of mixing, standard sand was evenly introduced into the mixing pot via a mixing funnel. Subsequently, the mixture was stirred at a high speed for an additional 30 seconds.

2. After the mixing process ceased, any material adhering to the sides of the mixing bowl was promptly scraped down into the batch. The mixer enclosure was then closed, or alternatively, the bowl was covered with a lid. The paste was allowed to stand undisturbed for a duration of 90 seconds.

3. Following the resting period, the mixture underwent further mixing for 60 seconds at high speed.

4. The freshly prepared cement mortar was poured into steel molds and compacted using a standard vibrating table. The molds were subsequently sealed with polyethylene nanosheets to prevent moisture loss. After a curing period of 24 hours, the samples were demolded and subjected to further curing in a saturated lime-water bath maintained at 20°C for specific aging durations of 3, 7, and 28 days.

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

Cement(g)	Sand(g)	Water(g)	Paraffin(g)	SiO2(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

A steel mold measuring 40mm × 40mm × 160mm was chosen for the flexural and compressive strength tests. The flexural and compressive strengths of the specimens were assessed in accordance with the guidelines outlined in GB/T17671-1999 at three different aging durations: 3, 7, and 28 days. For each series, three specimens were subjected to testing to determine their strength characteristics. The flexural strength test was conducted utilizing a three-point bending test apparatus with a loading rate of 0.06 N/s. This test method provides insights into the ability of the specimens to resist bending forces. On the other hand, the compressive strength test involved subjecting the specimens to compression using a loading rate of 2.4 kN/s. This test evaluates the ability of the specimens to withstand compressive forces.

SEM characterization of hydration products:

In this study, the scanning electron microscope (SEM) model ZEISS Gemini SEM 300 was employed to directly

observe the microscopic morphology of the materials by imaging the surface properties of the sample. The working principle of SEM involves using an electron lens to reduce a single electron beam spot to a nanoscale size. Subsequently, a deflection system is utilized to raster scan the high-energy electron beam across the surface of the sample, exciting secondary electrons and other physical information. These signals are collected by detectors and converted into an image for display. The SEM equipment used in this study offers a scanning speed ranging from 20 nanoseconds to 10 milliseconds per pixel and a magnification capability ranging from 1 to 1 million times. Figure 2 depicts the SEM equipment utilized.

Furthermore, it is imperative that the samples for SEM testing are dry. Therefore, prior to SEM observation, cement hydration suspension treatment is necessary due to the continuous hydration process of cement. Suspension of hydration and drying can be carried out simultaneously. In this experiment, a combination of solvent substitution and vacuum drying was employed. Solvent substitution involves using organic solvents to displace water from the sample. Specifically, the samples were soaked in absolute ethanol for 24 hours. Subsequently, the samples were placed in a vacuum drying oven set at 60°C for 6 hours to facilitate the volatilization of absolute ethanol from the samples.

Fig.2: Photograph of Scanning electron microscope (SEM)

V. RESULT AND DISCUSSION

Effects of the paraffin on the workability of cement paste:

The fluidity **(Fig. 3)** of cement mortar decreases as the phase-change materials (PCMs)/paraffin content increase. Specifically, compared to cement mortar M, the fluidity decreases by 10.9% when the phase-change materials (PCMs)/paraffin content is 15% and by 18.48% when the phase-change materials (PCMs)/paraffin content is 20%. This reduction in fluidity is likely due to the distribution and orientation of the phase-change materials (PCMs)/paraffin materials within the mortar. To ensure consistency in fluidity values and enable the comparison of strengths, a water-reducing agent was added to the samples. This addition helps maintain consistent fluidity across the samples, facilitating an accurate comparison of their strengths. **Figure 3** illustrates the observed trends in fluidity as the phase-change materials (PCMs)/paraffin content vary, providing a visual representation of the relationship between phase-change materials (PCMs)/paraffin content and fluidity in cement mortar samples.

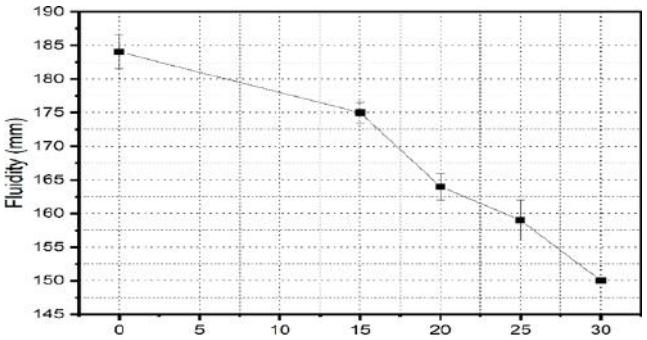


Fig.3: Workability test compared to Ordinary mortar and paraffin composite

Flexural Strength

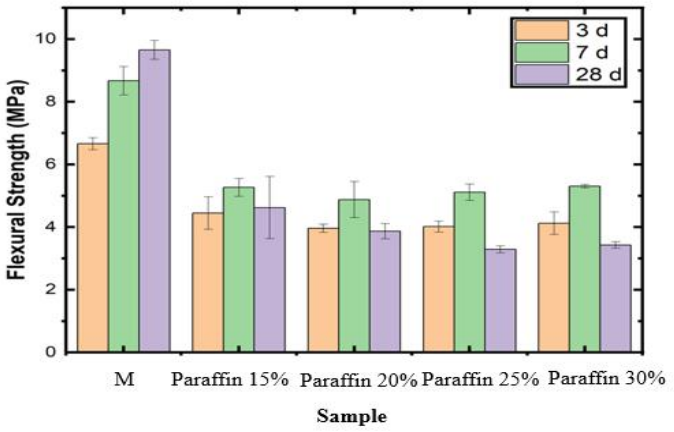


Fig.4: Photograph of Flexural Strength

Figure 4 illustrates the flexural strength of cement mortar mixed with varying amounts of paraffin at 3, 7, and 28 days. Initially, the flexural strength of the cement mortar increases gradually with an increase in the paraffin content. However, beyond an optimal paraffin content, the flexural strength begins to decrease. The flexural strength of the cement mortar mixed with paraffin is highest when the paraffin content is 15%, 20%, 25%, or 30%. At curing ages of 3 days and 7 days, the flexural strength of sample

paraffin increases by 5%, 4.5%, 5.2%, and 5.5%, respectively, compared to that of plain cement mortar sample M. This increase in flexural strength is attributed to the high strength of paraffin, which enhances the mechanical properties of the cement mortar.

Compressive Strength

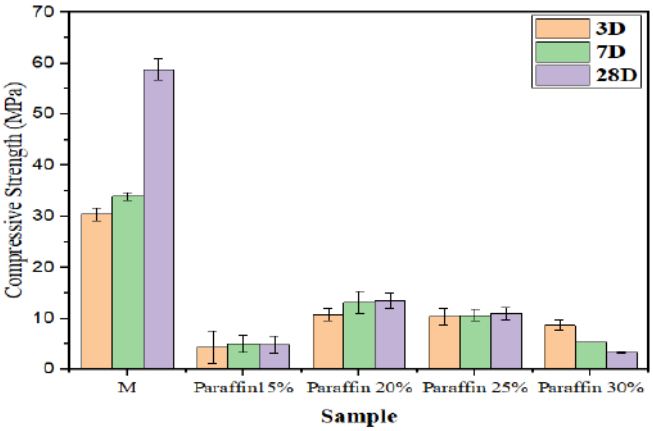
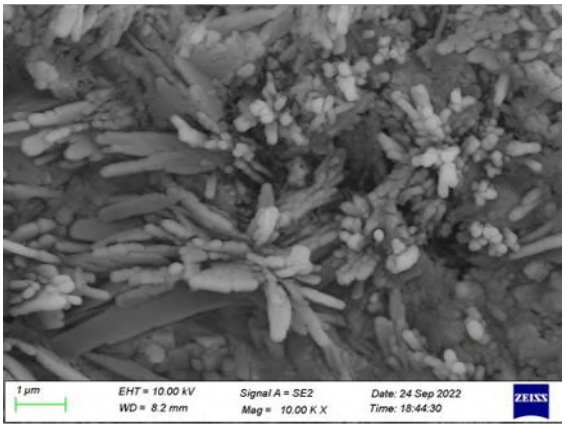
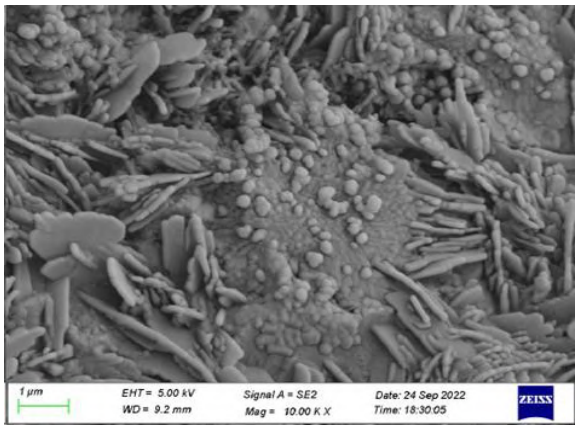


Fig.5: Photograph of Compressive Strength



6.A



6.B

Fig.6: Photograph of SEM image (A&B)

In **Figure 5**, the compressive strength of cement mortar mixed with different amounts of paraffin at 3, 7, and 28 days is depicted. When the curing ages are 3, 7, and 28 days and the paraffin content is 20% and 25%, the compressive strengths of the cement mortar are 10.5% and 10.4% higher than those of the plain cement mortar, respectively. However, with a further increase in paraffin content, the compressive strength of the cement mortar does not increase and is lower than that of the plain cement mortar. This phenomenon is attributed to the uneven distribution of the paraffin within the mortar and the increased porosity. At a curing age of 28 days, the compressive strength of all the mixed cement mortar samples doped with paraffin is lower than that of the plain cement mortar. This could be due to the uneven dispersion of paraffin, which traps the flow of free water in the cement slurry, thereby reducing the degree of hydration.

SEM results: From **Figures 6 (a) and (b)**, it can be seen that the paraffin wax almost fills the circular sieve or long cylinder with a porous structure, indicating that the paraffin has been fully impregnated. The above demonstrates that this experiment successfully loaded paraffin into the expanded perlite using high-temperature and decompression adsorption methods.

DSC Curve analysis of paraffin samples:

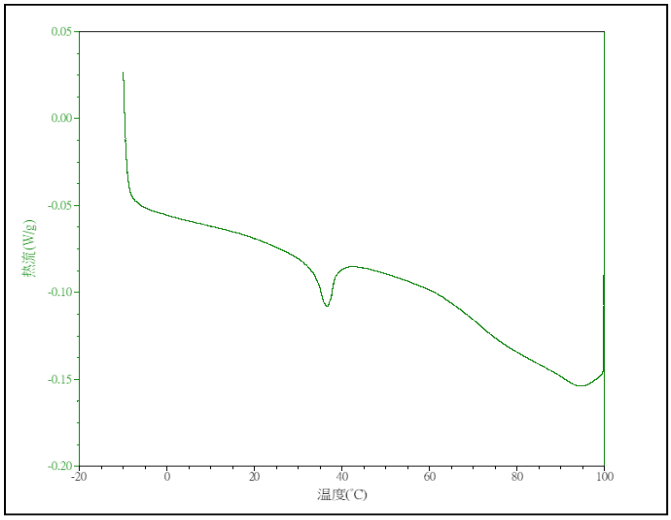


Fig.7: Photograph of DSC Curve

The determination of the latent heat of the phase change of the sample involved setting the experimental temperature range from -20°C to 100°C. Liquid nitrogen was used to cool the sample to -20°C initially, followed by increasing the temperature at a rate of 5°C/min. The system automatically recorded the sample's curves and data on heat flow versus temperature. It was observed that peak temperatures and the overall curve shape of the phase-changing process were magnified by DSC testing

with higher heating and cooling rates. As the rate of heating or cooling rises, the hysteretic reaction is also accentuated. The melting point of paraffin was 39°C. The DSC curve of solid paraffin mortar is shown in **Figure 7**.

Thermal conductivity:

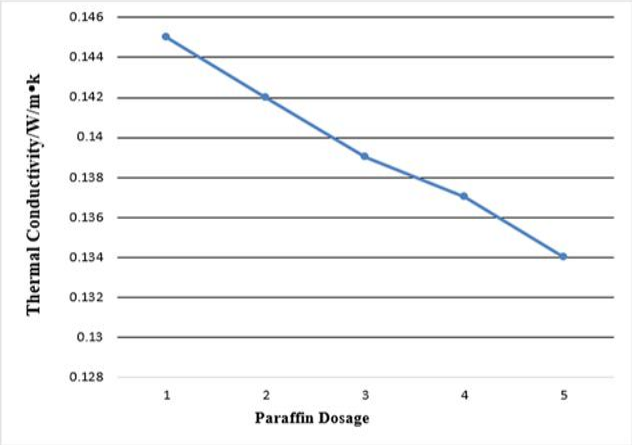


Fig.8: Photograph of Thermal conductivity of Paraffin sample

Figure 8 depicts that for thermal conductivity testing, a paraffin sample containing 20% paraffin was sent to the test centre. The specimen's dimensions were a diameter of 14.7 mm and a thickness of 2.32 mm. Data were collected at different temperatures. A material with low thermal conductivity does not easily transfer heat. Instead, it tends to insulate against heat flow, resisting the transfer of thermal energy. Materials with low thermal conductivity are often used as insulators to prevent heat loss or gain in various applications, such as building insulation, thermos flasks, and protective clothing. Low-thermal conductivity materials effectively resist heat transfer.

Thermal Control Effect Test:

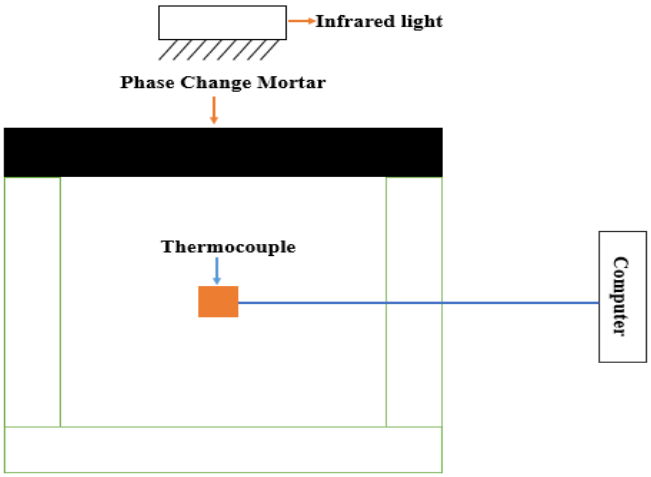


Figure-9: *Photograph of Thermal Control Effect test*

As depicted in Figure 9, the temperature adjustment performance test involves simulating the behaviour of phase-change materials under various environmental conditions to determine their effect on a house. To achieve this, a house model (Six panels are required to build a house or box) is constructed using mortar boards with panel top sides, each panel measuring *200*200*20* mm. The top side of the mortar board serves as the energy storage mortar board to be tested. High-efficiency infrared lamps are utilized for heating, while a thermometer records the temperature in the mortar model room. A sensor, consisting of a thermocouple, is placed at the centre of the house to sense temperature changes. The temperature-measuring thermocouple is connected to a computer via an ART Data Acquisition Module, DAM-3138, USB485/RS-422. Temperature software is used to control the infrared lamp heating process, and data on the indoor temperature control of the energy storage mortar is collected. Temperature data are collected every 10 minutes during the heating and cooling processes for graphing.

Temperature variation with Addition of Paraffin:

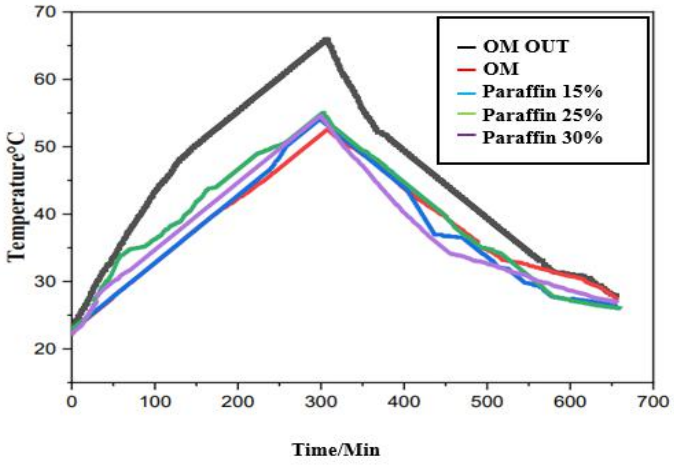


Fig.10: Photograph of Temperature variation with Addition of Paraffin

Figure 10 displays the quantity of paraffin composite phase change materials used in mortar rooms, along with a diagram illustrating the effect on the internal temperature of the house model. The temperature differences between paraffin-15%, paraffin-25%, and paraffin-30% are 19.69°C, 18.18°C, and 16.16°C, respectively, in comparison to the temperature of OM, while the temperature inside OM is 21°C relative to the outside temperature of OM.

Temperature Difference with Addition of Paraffin:

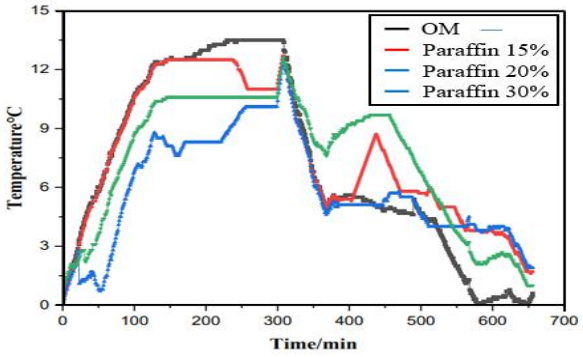


Fig.11: Photograph of Temperature Difference with Addition of Paraffin

Figure 11 depicts the temperature difference between ordinary mortar (OM) and paraffin composite phase change materials outside the house. The temperature differences for paraffin content of 15%, 25%, and 30% are 5.18°C, 7.4°C, and 9.6°C, respectively, while the temperature difference inside OM remains at 13.5°C.

VI. CONCLUSION

Based on the perspectives of energy saving, emission reduction, and building energy efficiency, this study examines paraffin. Its mechanical and thermal properties are tested, and the temperature adjustment performance test of the house model is determined. The effect of shape changes on the energy storage mortar board at room temperature is evaluated.

1. A paraffin mixture consisting of 20% and 25% was tested by DSC. It was found that the phase transition temperature of the paraffin wax mixture is 39°C, with a latent heat of phase transition of 111.5 J/g. Workability tests and thermal cycle tests indicate that the paraffin wax mixture exhibits good thermal stability and durability, rendering it suitable for use in building walls.

2. The expanded perlite, after acidification, exhibits numerous honeycomb pores capable of absorbing a significant amount of paraffin. Upon coating with calcium silicate powder, it demonstrates good thermal stability. Diatomite, after roasting and acidification treatment, primarily assumes a sieve-like and cylindrical shape, featuring high porosity and a large surface area. Additionally, it possesses nano-silica pores, making it an excellent porous adsorption material. Various porous materials can be utilized in the preparation of shape-fixed phase-change composites.

3. SEM results indicate that the expanded perlite experiences significant enhancement through heating and vacuum adsorption, leading to increased absorption of the paraffin mixture. Infrared test results demonstrate that the adsorption process for both paraffins is purely physical, with no chemical reactions occurring. Upon encapsulation of the paraffin or expanded perlite with calcium terephthalate powder, a low exudation rate is observed, accompanied by excellent thermal stability. Additionally, the surface tension exhibits strong resistance to paraffin, preventing leakage and ensuring the binding force of the paraffin wax. Consequently, the prepared paraffin/expanded perlite and earth-set phase transition material exhibit stable performance, making them suitable for application in the field of building materials.

4. The phase-change energy storage mortar was prepared using both the intercalation method and the direct mixing method. With increasing dosage, the compressive strength, dry density, and thermal conductivity of the phase-change mortar gradually decreased. The performance of the phase-change energy storage mortar prepared using the intercalation method was superior to that of the phase-change energy storage mortar prepared using the direct mixing method. Even when the content is increased to 20%, the strength remains higher than 13.45 MPa, meeting the mechanical requirements for actual construction. Additionally, its thermal conductivity coefficient is extremely low, while the heat storage coefficient maintains a certain level, demonstrating good heat preservation and storage effects. Intercalation is thus identified as the preferred preparation process for phase-change energy storage mortar.

5. For the thermal control effect test, we found that the temperature was significantly lower than ordinary mortar when using paraffin.

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