Research Article

Preparation and Property Modification on Novel Energy Storage Material: *n*-Octadecane PCMs/Expanded Perlite Composite Gypsum Board

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Phase change materials (PCMs) have been widely used to improve the thermal energy storage capacity of building materials. In this study, the *n*-octadecane (OD)/expanded perlite (EP) composite PCM, which was prepared by incorporation of liquid *n*-octadecane into EP using the vacuum impregnation method, was used to fabricate the gypsum board. The microscopic, thermal, and mechanical properties were studied. The SEM results showed that OD could be absorbed into the pores of EP uniformly. The FT-IR results showed that OD and EP have good chemical stability. It was found that the gypsum board has best heat transfer delay when the volume fraction of OD/EP was 20% (v/v). The mechanical property of the gypsum board with OD/EP decreased. To deal with the problem, the effect of nano- Al_2O_3 on the gypsum board was also studied. The results showed that the mechanical properties of the gypsum board were effectively increased when the dosage of nano- Al_2O_3 was 0.5 wt.%, and the gypsum board had the best thermal insulation effect when the nano- Al_2O_3 content was 0.3 wt.%. Considering the cost and the comprehensive property, it was suggested that the optimal addition content of nano- Al_2O_3 was 0.3 wt.%.

1. Introduction

Energy shortage has become a common problem in the world with the fast economic growth and the large consumption of conventional energy. In recent years, more and more fossil fuels had been consumed to improve internal building comfort [1, 2]. A lot of work has been done in terms of reducing building energy consumption such as thermal energy storage (TES) system, and the preparation of a PCM was most important in this system [3, 4]. PCM is a class of energy storage material which can store or release energy in a phase transition at a certain temperature [5]. Diana and Liu [6, 7] used phase change microcapsules to improve the heat transfer performance of plaster. The results showed that the addition of phase change microcapsules leads to good heat storage property of plaster. OD is one of the most used organic PCMs in the fields of the TES system and building energy conservation, because it has appropriate temperature and high energy storage, shows small volumetric change between solid and liquid phases and good reversibility, and is nontoxic and

noncorrosive [8–10]. However, there are also some drawbacks that limit the applications of OD, such as reduced mechanical properties and leakage in the melting state.

Although the addition of OD would reduce the strength, there are many possible ways to improve this performance. Many studies had shown that the addition of appropriate kinds and amounts of nanomaterials could significantly reduce porosity in the gypsum board, thereby increasing the strength [11–13]. Nanoalumina was used as a cement-based reinforcement material due to the effect of filling and nucleation [14], but the effect of nanoalumina in plaster has not been studied. In this study, nanoalumina was added to improve the mechanical properties.

To solve the problem of leakage in the melting state, a shape-stabilized PCM developed by microencapsulation of OD with a core-shell structure was prepared. In the existing research studies, the main shell materials are silica [9, 15, 16], *n*-butyl methacrylate [17], calcium carbonate [18], and some others [19, 20]. Wang [9] et al. prepared silica encapsulation of OD via the sol-gel process to enhance thermal

conductivity and phase change performance. Two other methods for microencapsulation of OD with silica were proposed: one is that microcapsules are synthesized through interfacial polycondensation [21], and the other is that microcapsules are prepared via the sol-gel process using sodium silicate instead of tetraethyl orthosilicate to produce the silica shell. Recently, calcium carbonate-encapsulated *n*-octadecane was prepared through a self-assembly method, and this was a new method different from other inorganic microencapsulated PCMs reported by literatures [18]. But, complicated technology and high cost limited the applications of encapsulated PCMs. EP is a kind of porous material. In recent years, there are many studies on adsorbing different PCMs with EP, such as sodium nitrate [22], fatty acid [23–25], and paraffin [26–28]. Filling the pores of EP with OD is an easier and cheaper way to produce form-stable PCMs compared with the encapsulation technology.

In this study, first, preparation of OD/EP composite materials by filling EP pores with OD by the means of the vacuum impregnation method (VA method) was proposed. Then, the gypsum board was prepared by mixing the prepared OD/EP materials and plaster together. Third, the mechanical properties, chemical compatibility, and thermal property of the gypsum board with OD/EP materials were investigated. At last, because of the decreased mechanical properties of the gypsum board, this research also investigated the modification of mechanical properties by adding nano- Al_2O_3 .

2. Materials and Methods

2.1. Materials. *n*-Octadecane (OD, 90% pure) and nano- Al_2O_3 (α phase, 99.9% pure) were purchased from Aladdin Industrial Corporation. EP was purchased from Xinyang Perlite Plant, China. The chemical composition of EP is shown in Table 1. Plaster (β -hemihydrate, 99.9% pure) was purchased from Henan Qiangnai New Materials Co., Ltd, China.

2.2. Preparation of the Gypsum Board with OD/EP. In this study, the OD/EP composite material was prepared by the VA method [29]. The EP was dried at 105°C for 24 h, before manufacturing the composite material. Secondly, the melted OD was added into the flask to cover the EP, which was placed at the bottom of the flask, and stirred evenly. Third, the mixture was placed in a vacuum oven, and it took 2 h to reach 60°C after evacuation and then was maintained in a stable vacuum for 1 h, in order to allow air to enter the flask again and force the liquid OD to penetrate into the porous structure of EP. It was found that the maximum mass fraction of OD/EP was 60 wt.% without the leakage of the melted PCM.

To investigate the effect of OD/EP on the thermal conductivity and heat transfer property of the plaster, the OD/EP was incorporated into the plaster with the replacement at 10% (v/v), 20% (v/v), and 30% (v/v) by volume. Based on the gypsum board with 20% (v/v) OD/EP which showed the best thermal conductivity and heat transfer property, different contents of nano- Al_2O_3 was then added to investigate the effect of the nano- Al_2O_3 on thermal conductivity of the plaster.

TABLE 1: Chemical composition of EP.

Composition	SiO_2	Al_2O_3	Na_2O	K_2O	CaO	H_2O
wt. %	70.7	15.8	5.6	5.2	1.3	0.3

2.3. Characterization. The morphology and microstructure of the specimens were observed with an SEM (JSM-6390LV) at the acceleration voltage of 15 kV under low vacuum. The chemical compatibility analyses of the samples were carried out by using an FT-IR (vertex 70). The FT-IR spectra were recorded in the frequency range of 4,000–400 cm^{-1} .

The mechanical properties of the plaster with OD/EP materials were evaluated by measuring 2 h compressive strength and flexural strength. The specimen size used for this experiment was 40 mm $\times$ 40 mm $\times$ 160 mm. All of the samples were dried at room temperature before testing.

The thermal performance of the plaster with OD/EP materials was evaluated by analyzing thermal conductivity and heat transfer property. The specimen with 300 mm $\times$ 300 mm $\times$ 50 mm size was used for the heat transfer property test, and the specimen with 200 mm $\times$ 200 mm $\times$ 15 mm size was used for the thermal conductivity test. The heat transfer property was tested by a self-made design shown in Figure 1. The thermal conductivity of the OD/EP gypsum board was tested in the temperature range of 20°C to 50°C, which included the phase transition temperature.

3. Results and Discussion

3.1. Microstructure of OD/EP Materials. The morphology and microstructure of the EP and the OD/EP composite were observed by SEM and are shown in Figure 2. Figure 2(a) shows that the EP had many rough porous structures, and the scaly pore diameter was mostly in the range of 5 μm –150 μm . These porous structures could absorb OD and prevent the leakage of the melted PCMs by capillary force and surface tension. It could be seen from Figure 2(b) that OD had been uniformly absorbed in the pores of the EP, and part of the pore structure in the EP was not filled by OD, which provided space for the expansion during the phase change from solid to liquid.

3.2. Chemical Compatibility of OD/EP Materials. The chemical compatibility of EP, OD, and OD/EP samples were characterized by FT-IR spectroscopy. The FT-IR spectra of EP, OD and OD/EP are presented in Figure 3. The absorption peaks of 2920 cm^{-1} , 2850 cm^{-1} , 1465 cm^{-1} , 1377 cm^{-1} , and 720 cm^{-1} were produced by the stretching vibration of the functional groups $-\text{CH}_2$ and $-\text{CH}_3$. It could be found that the OD had $-\text{CH}_2$ bonding and $-\text{CH}_3$ bonding, EP had $-\text{OSi}$ bonding, and the absorption peak was 1020 cm^{-1} –1095 cm^{-1} . In addition, all the characteristic absorption peaks of the OD/EP composite appeared in the spectrum of both EP and OD, and there were no any new peaks generated by the OD/EP composite. This phenomenon indicated that there was physical cross-linking between EP and OD, not chemical reaction. So, the OD/EP material has favorable chemical stability.

FIGURE 1: Schematic and design of the instrument used for testing the heat transfer property.

(a)

(b)

FIGURE 2: SEM images of EP (a) and OD/EP (b).

FIGURE 3: FT-IR spectra of EP, OD, and OD/EP.

3.3. Mechanical Properties of Gypsum with Different OD/EP Contents. In order to identify the effect of the OD/EP material content on mechanical properties of gypsum, the compressive strength and flexural strength of gypsum with different contents of OD/EP were tested and are shown in Figure 4. The results revealed that the compressive strength and flexural strength decreased with the increase of the OD/EP material. The phenomenon could be attributed to a

variety of reasons. First, the strength of the OD/EP material was obviously lower than gypsum's, and the OD/EP material contained more defects, resulting in lower strength; secondly, the vulnerable interfacial bond between the OD/EP material and plaster also decreased the strength.

3.4. Heat Conduction of Gypsum with Different OD/EP Volumes. In this study, the steady-state plate method was used to measure the thermal conductivity of gypsum boards with OD/EP materials, and the results are presented in Figure 5. It showed that the thermal conductivity increased with the increasing of the OD/EP material volume until 20% (v/v), and then it decreased when the OD/EP material volume increased continuously. Therefore, the composite PCM had positive and negative effect on the heat transfer property of the gypsum board. This could be explained from the following factors. On the one hand, a certain amount of water was adsorbed due to the porous structure of the EP, and the thermal conductivity of gypsum boards increased with the increase in moisture content [30, 31]. On the other hand, both of the EP and OD had a poor thermal conductivity, and the addition of OD/EP materials also increased the internal defects of the plaster. All of the above reasons led to decrease in the thermal conductivity of gypsum.

FIGURE 4: Mechanical properties of gypsum boards with OD/EP.

FIGURE 5: Thermal conductivity of gypsum boards with OD/EP materials.

FIGURE 6: The center of the cubicle system temperature variation curve.

The heat storage property of the gypsum with OD/EP materials was mainly evaluated by its heat storage capacity and efficiency. In this study, the heat storage property of the gypsum board with different volume fractions of OD/EP materials was investigated by measuring the temperature variation regularity of the inside center of the cubicle system. The results are shown in Figure 6. The internal temperature of the blank gypsum board rose to 45°C after 107 minutes, while the time was postponed to 132 min, 153 min, and 138 min for the samples with 10% (v/v), 20% (v/v), 3 and 0% (v/v) OD/EP in the gypsum board, respectively. It meant that it took 1.23, 1.43, and 1.29 times longer to raise the temperature of the gypsum board to 45°C respectively, compared to the blank gypsum board. The ability to adjust the temperature increased first and then decreased with the increase in the content of the OD/EP material. The cause of this phenomenon was that the thermal storage capacity of the gypsum board was increased with the volume fraction of the OD/EP materials at the early stage. Therefore, the heat storage performance should be

better with the increase of the volume fraction, and the ability to adjust the temperature was stronger. But, the utilization of the OD/EP materials was too low while the volume was more than 20% (v/v) because the high volume fraction of the OD/EP materials made the gypsum board had low thermal conductivity. This could be explained from two factors. First, when the temperature reached the phase change temperature, the PCM in the gypsum board would absorb the heat, and the heat transfer process was prevented to a certain degree. On the other hand, the internal structure defect of the gypsum board increased with the incorporation of the composite PCM. The result was that the convection effect within the gypsum board had been reinforced.

4. Modification Effect of Nano- Al_2O_3 on Gypsum Board

4.1. Effect of Nano- Al_2O_3 on Mechanical Properties of OD/EP Gypsum Sample. For materials such as cement and ceramics,

nano- Al_2O_3 had the function of modification and reinforcement, which could optimize the pore structure of the material and enhance the mechanical properties [32–34]. In this paper, the influence of nano- Al_2O_3 on the performance of the gypsum board was investigated. In this experiment, the content of nano- Al_2O_3 was set to from 0 to 0.6 wt.% of the mass of the plaster, and the volume fraction of the OD/EP material was 20% (v/v). Figure 7 shows the test results of the mechanical properties. The results revealed that the compressive strength of the gypsum board increased first and then decreased with the increase of the content of nano- Al_2O_3 . This could be attributed to a variety of reasons. First, the nano- Al_2O_3 acted as an ultrafine aggregate to fill the voids and bubbles around the plaster particles, making the structure more compact and enhancing strength. Second, when the amount of nano- Al_2O_3 was too high, the water requirement of the nanomaterial would increase due to the large specific surface area of the nanomaterial, which would make it difficult to achieve uniform dispersion of the nanomaterial, thereby causing agglomeration and wrapping the plaster particles, hindering the hydration reaction of the plaster particles. At the same time, excessive microbubbles were caused by the increase of water demand during the process of stirring the plaster slurry and increased the number of harmful pores of the plaster slurry, resulting in a decrease in strength. And the compressive strength of gypsum samples cured for 28 d decreased by 12.9% after 50 cycles of phase change [35], but the compressive strength still reached 8.4 MPa. Therefore, when the amount of nano- Al_2O_3 was 0.5 wt.%, the mechanical properties of the gypsum board were the best.

The XRD pattern of the hydration product of gypsum is shown in Figure 8. It was revealed that the hydration product of the plaster after adding the nano- Al_2O_3 was $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$, the same as the blank sample. No new hydration products formed. There were two main reasons for nano- Al_2O_3 improving the mechanical properties. On the one hand, the increase in the strength of the gypsum board was due to the filling action of the nanomaterial. On the other hand, the surface of the nanomaterial had more active bonds, which could be used as a ready-made nucleus, allowing the hydration product to grow on the nanomaterial to form a dense structure [36, 37].

4.2. Effect of Nano- Al_2O_3 on Thermal Conductivity of OD/EP Gypsum Board. Nano- Al_2O_3 had good heat resistance and stable crystal form, which could significantly improve the compactness of the slurry. Figure 9 shows the test results of the thermal conductivity of the gypsum board. The results revealed that the thermal conductivity of the gypsum board appeared to decrease first and then increase with the increase in the content of nano- Al_2O_3 . When the content of nano- Al_2O_3 was 0.3 wt.%, the thermal conductivity of the gypsum board was the lowest, indicating that the heat transfer performance of the gypsum board was poor and the thermal insulation effect was good. Figure 10 illustrates the test results of heat transfer performance, wherein the amount of nano- Al_2O_3 was 0.3 wt.% to 0.6 wt.% of the mass of the plaster. When the heat source was heated for 2 h and then it was turned off, the regularity of the space temperature of the

FIGURE 7: Compressive strength of the OD/EP gypsum sample with nano- Al_2O_3 .

FIGURE 8: XRD pattern of hydration products of the OD/EP gypsum sample.

FIGURE 9: Effect of nano- Al_2O_3 to thermal conductivity of gypsum boards with the OD/EP material.

gypsum board was tested with time. It could be seen from Figure 10 that the temperature in the test device was approximately 36°C after heating for two hours, and the

FIGURE 10: The center of the cubicle system temperature variation curve.

temperature of the gypsum board with high thermal conductivity in the test device was low after the heat source was turned off. The results demonstrated the space temperature of the gypsum board with higher thermal conductivity was lower, indicating that the thermal insulation effect was not good. When the content of nano- Al_2O_3 was 0.3 wt.%, the OD/EP gypsum board had the best thermal insulation effect.

5. Conclusions

In this study, a novel energy storage material, plaster with OD/EP materials, was prepared, and the properties were investigated. In addition, the modification effect of nano- Al_2O_3 on the properties of OD/EP gypsum board was investigated. The obtained conclusions can be described as follows:

- (i) The OD/EP composite material could be prepared, and OD could be uniformly absorbed in the pores of EP. The results showed that the optimum volume fraction of the OD/EP material was 20% (v/v), and the maximum temperature was delayed by 43% compared to the sample of blank gypsum.
- (ii) With the addition of OD/EP materials, the compressive strength and flexural strength of the gypsum board decreased with the increase in the volume of the OD/EP materials.
- (iii) The strength of the OD/EP gypsum board increased first and then decreased with the increase in the content of nano- Al_2O_3 . When the content was 0.5 wt.%, the mechanical property showed the highest value. The thermal conductivity of the gypsum board was decreased after adding 0.3 wt.% nano- Al_2O_3 , and the gypsum board had the best thermal insulation effect. Considering the cost and the comprehensive property, it was suggested that the optimal addition content of nano- Al_2O_3 was 0.3 wt.%.

Data Availability

All data used to support the findings of this study are included in the Results and Discussion section.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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