

PREPARATION AND CHARACTERIZATION OF PHASE CHANGE ENERGY STORAGE GYPSUM

by

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The thermophysical properties of binary phase change materials with different ratios of capric acid and palmitic acid were studied by step cooling curve method and differential scanning calorimetry in this paper. Furthermore, the best adsorption materials and coating materials were selected by testing their mass adsorption rate and mass loss rate. Finally, the specific heat capacity, thermal conductivity coefficient, and compressive strength of phase change energy storage gypsum was determined, respectively, and the energy-saving effect of the phase change energy storage gypsum in the wall is evaluated. The results show that the binary phase change materials can form a eutectic system. When the mass ratio of capric acid to palmitic acid is 7:3, the low eutectic point of the binary system is formed, and the crystallization temperature of system is 26 °C. The adsorption capacity of expanded perlite is much larger than that of ceramsite, and the mass loss rate of the material coated by styrene acrylic emulsion is lower than that of ethylene vinyl acetate. The specific heat capacity of phase change energy storage gypsum is about twice that of ordinary gypsum. With the addition of phase change materials, the thermal conductivity coefficient of phase change energy storage gypsum decreases gradually, and the compressive strength of phase change energy storage gypsum decreases gradually at the same time. Compared with ordinary gypsum, phase change energy storage gypsum has better energy-saving performance.

Key words: *binary phase change materials, differential scanning calorimetry, thermal conductivity coefficient, compressive strength*

Introduction

The temperature of phase change materials remains unchanged or stable in a certain temperature range in the process of storing and releasing energy [1]. Improving the thermal inertia of building walls by utilizing the heat storage and release characteristics of phase change materials is an effective way to realize building energy saving [2]. As the most widely used phase change material, paraffin wax has the disadvantage of super-cooling in the process of phase change. In addition, there are few kinds of phase change materials available in the field of low temperature energy storage, and it is difficult to meet the phase change temperature in use [3].

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In recent years, multi-component composite phase change materials have gradually become a research hotspot of phase change materials. Yang *et al.* [4] measured the thermal stability of capric acid-lauric acid binary phase change material after thermal cycles by differential scanning calorimetry. The results showed that the thermal stability of binary phase change materials was inversely proportional to the number of thermal cycles. Cai *et al.* [5] tested the thermophysical properties of lauric acid and tetradecanol by step cooling curve and other test methods. The results showed that lauric acid and tetradecanol reached the eutectic point at 23.1 °C, and maintained good thermal stability after 300 thermal cycles at 0-60 °C. Liu *et al.* [6] studied the thermophysical properties of capric acid-lauric acid binary phase change materials used in wall materials. The results showed that the binary system had the lowest eutectic point at 19 °C.

When the external temperature rises to the phase change temperature of phase change material, the phase change material will be transformed into liquid. If the phase change material is directly added to the building material, it will not only fail to play the role of energy storage, but also greatly reduce the stability of building material [7]. Therefore, some micro-structures (*e.g.* inorganic porous materials) are used to prepare shape-stabilized phase change materials to avoid liquid leakage in the process of phase transition [8]. Li *et al.* used expanded perlite as the encapsulating material. The results showed that the adsorption effect was the best when the mass fraction of lauryl alcohol was 40%. Shape-stabilized phase change materials were prepared by physical adsorption method, and the structures and properties were tested by XRD and other testing methods [9]. Liu *et al.* [10] studied the properties of expanded graphite based phase change materials. It was concluded that the content of graphite was the main factor affecting the thermal stability of expanded graphite based phase change materials.

In addition, the research of phase change materials has not only achieved some results in materials preparation, but also has a wide range of applications in the field of building energy saving [11]. The incorporation of shape-stabilized phase change materials into building materials cannot only improve the thermal insulation performance of buildings, but also adjust the indoor temperature of buildings [12]. Phase change energy storage gypsum (PCESG) can be prepared by using gypsum as cementitious materials. Xiao *et al.* [13] prepared dodecanol-bentonite composite phase change material by ultrasonic vibration and liquid phase intercalation. The results showed that the PCESG board had good thermal insulation performance. The PCESG was prepared by direct mixing method, and the performance difference between PCESG and ordinary gypsum was compared by testing [14]. Hu and Xiao [15] mixed phase change paraffin with gypsum to prepare PCESG, and its thermal performance was simulated by finite element software. Kong *et al.* [16] established a mathematical model and analyzed the influencing factors of heat transfer performance of phase change gypsum wall. Corresponding to the climate characteristics in summer, the heat storage performance was more obvious when phase change materials were set on the inner surface of the wall.

However, the thermal performance of PCESG was not demonstrated, which is available in the field of low temperature. This paper will introduce how to prepare shape-stabilized phase change materials and how to mix the materials with desulfurization gypsum for building walls. Firstly, the thermophysical properties of binary phase change materials with different ratios of capric acid and palmitic acid were studied by step cooling curve method and differential scanning calorimetry, and the optimum ratio of binary phase change materials was selected. Furthermore, porous materials were used as encapsulating materials to prepare capric acid-palmitic acid form-stabilized phase change materials. Finally, PCESG was prepared with desul-

furization gypsum as cementitious material, and the specific heat capacity, thermal conductivity coefficient and compressive strength were tested, respectively, and the energy-saving effect of the PCESG in the wall is evaluated.

Experimental

Raw materials

– Phase change materials

Palmitic acid, produced by Shanghai Maikun Chemical Co., Ltd., analytical purity. Capric acid, produced by Shenzhen Runyou Chemical Co., Ltd., chemical purity. The basic properties of raw materials were shown in tab. 1.

Table 1. Basic properties of palmitic acid and capric acid

Materials	Appearance	Purity	Latent heat [Jg ⁻¹]	Phase transition temperature [°C]	Thermal conductivity coefficient [Wm ⁻¹ K ⁻¹]
Palmitic acid	White pearlescent scale	99%	185	62.5-64	0.162
Capric acid	Colorless crystal	98%	132	31-32.5	0.149

– Encapsulating materials

The encapsulating materials used in this study were expanded perlite from Henan Baihui perlite factory and medium-sized ceramsite from Hongda ceramsite factory in Zhongkai high-tech zone of Huizhou. The basic properties of expanded perlite and ceramsite were shown in tab. 2.

Table 2. Basic properties of expanded perlite and ceramsite

Materials	Density [kgm ⁻³]	Water absorption [%]	Thermal conductivity coefficient [Wm ⁻¹ K ⁻¹]	Particle size mm	Cylindrical compressive strength [MPa]
Expanded perlite	120	30	0.38	0.5-1.5	36
Ceramsite	480	8	0.18	15-20	4

– Coating materials

The coating materials were styrene acrylic emulsion and ethylene vinyl acetate (EVA) emulsion. Styrene acrylic emulsion has the advantages of high bonding strength, good water resistance. The minimum film forming temperature is 20 °C. The EVA emulsion has the advantages of high bonding strength, curing at room temperature, and good toughness and durability.

– Cementitious materials

Gypsum is a monoclinic mineral, and its main chemical composition is calcium sulfate hydrate. Desulfurization gypsum was selected as cementitious materials. The basic properties of desulfurization gypsum were shown in tab. 3.

Table 3. Basic properties of desulfurization gypsum

Materials	Initial setting time [hour]	Final setting time [hour]	Water retaining rate [%]	Flexural strength [MPa]	Compressive strength [MPa]	Thermal conductivity coefficient [Wm ⁻¹ K ⁻¹]
Desulfurization gypsum	1.2	6.8	92	3.4	6.6	0.325

Experimental method

Step-cooling curve

The mass mixing ratios of capric acid and palmitic acid were 7:3, 6:4, 5:5, 4:6, and 3:7, respectively. Samples of binary phase change materials with different mixing ratios were put into test tubes, then heated in a water bath pot, and then stopped heating when the temperature rise to 70 °C. After heating, the test tubes containing the samples were quickly put into the cold water bath at 20 °C. During this process, the temperature and corresponding cooling time were recorded with a portable thermometer. The test time interval was 20 seconds, and the test was stopped until the mixture had completely solidified.

Differential scanning calorimetry

The mass mixing ratios of capric acid and palmitic acid were 7:3, 6:4, 5:5, 4:6, and 3:7, respectively. The capric acid and palmitic acid were mixed in a water bath pot and heated to the maximum of the melting points. After the samples were cooled and solidified, 10 mg of capric acid and palmitic acid were put into the crucible pot. Then the crucible with the sample and the blank crucible were heated simultaneously. The device was connected closely with the thermal sensor. The samples were cooled to -10 °C by nitrogen. The heating device was started and the heating rate was controlled to 5 °C per minute. The test was completed when the temperature rise to 70 °C.

Porous matrix adsorption test

The principle of adsorption is to adsorb phase change materials into the pore by capillary force. Expanded perlite and ceramsite were selected as porous matrix in this paper. The test method was capric acid-palmitic acid binary phase change material was heated in a constant temperature, and then stirred until it was completely mixed, so that the mixed phase change material remained in liquid phase. Expanded perlite and ceramsite were immersed in phase change materials, respectively, and the adsorption rates of the porous matrix to phase change materials were measured in the natural conditions. The adsorption rates were tested every 1 hour and last for 14 hours.

Thermal stability test

Due to the change of ambient temperature, the solidifying-melting cycle of phase change materials stored in porous matrix occurs continuously during the use of shape-stabilized phase change materials. The test method was EVA emulsion and styrene acrylic emulsion were added to the shape-stabilized phase change material. The shape-stabilized phase change material and the coating material were stirred evenly by the glass rod, so that the surface of the shape-stabilized phase change material was completely coated by the emulsion. Non-coated materials and coated materials were heated in an electrothermal thermostat at 40 °C for 20 minutes, and the specimens were placed in the natural environment for heat release. The test intervals of thermal cycle were 10 times. The mass of the shape-stabilized phase change materials were tested after thermal cycles.

Specific heat capacity test

The specific heat capacity of PCESG can reflect its heat storage capacity. The larger the specific heat capacity, the stronger the heat storage capacity. In this paper, the specific heat capacity of PCESG is measured according to the principle of energy conservation, shown in

eq. (1). Water was selected as the reference (the specific heat capacity of pure water is $4.2 \cdot 10^3$ J/kg). The test method was PCESG specimen and common gypsum specimen were heated in an electrothermal thermostat to 40 °C, while the surface temperature of gypsum specimens was recorded by thermocouple. Then water was taken to immerse the gypsum specimens, and the initial mass and temperature of water were recorded. Then the specimens were quickly put into the water (ignoring the heat loss of the sample in the air), and the temperature of water was monitored by thermocouple until the temperature did not change.

$$m_s C_s (t_w - t) = m_w C_w (t - t_s) \quad (1)$$

where m_s , m_w are the mass of gypsum and water, respectively, C_s , C_w – the specific heat capacity of gypsum and water, respectively, t_s , t_w – the initial temperature of gypsum and water, and t – the equilibrium temperature of water.

Thermal conductivity coefficient test

The curing temperature of the specimen was about 20 °C, and the humidity was not less than 90%. The specimens were cured at room temperature for 7 days [17, 18]. The target temperature of the hot plate was set as 35 °C, and the target temperature of the cold plate was set as 10 °C, and the thickness of the specimen was 30 mm, and the length of the hot plate opening was 300 mm, and the width of the hot plate opening was 300 mm. The test method was the common gypsum specimen and three groups of PCESG specimens were recorded as G_0 , G_1 , G_2 , and G_3 , in which G_1 , G_2 , and G_3 represented three different mix ratios of 4:6, 5:5, and 6:4. The size of the specimen was 300 × 300 × 30 mm.

Compressive strength test

Compressive strength refers to the strength limit under the external pressure. While improving the thermal performance of PCESG, its compressive strength should be guaranteed in a reliable range. The test method was: the common gypsum specimen and three groups of PCESG specimens were recorded as P_0 , P_1 , P_2 , and P_3 , in which P_1 , P_2 , and P_3 represented three different mix ratios of 4:6, 5:5, and 6:4. The size of the specimen was 100 × 100 × 100 mm.

The SEM test

The SEM is used to observe the surface morphology of samples by secondary electron signal imaging. In this paper, the microstructure of PCESG with different proportions was characterized by SEM of HITACHI SU8020.

Energy-saving effect test

The PCESG is applied to the interior surface of wall. Taking general plaster as control specimen, the energy-saving effect of PCESG was tested. The test process is: put the model into the refrigerator at –10 °C until the internal temperature of the model drops to about 10 °C, and then move it into the box with the constant temperature 40 °C and measure the test points by thermocouple, respectively. Observe the temperature change trend of each part, and record the temperature change every 10 minutes.

Test results and analysis

Analysis of step-cooling curve test results

The step-cooling curves of capric acid-palmitic acid binary phase change materials were tested at the mass ratios of 7:3, 6:4, 5:5, 4:6, and 3:7. Mixed samples of capric acid and palmitic acid with different mix proportion were shown in fig. 1. The experimental results were shown in fig. 2. It can be seen from fig. 2 that the eutectic point does not occur in the horizontal section, but at the turning point when the mass ratio of binary phase change materials deviate from that of the lowest eutectic point. In addition, the temperature should continue to decrease after crystallization in theory, but as can be seen from fig. 2, the temperature remains constant for a long time. The reasons are the mixture near the tube surface is cooled first during the cooling process, and then it is cooled to the center of the tube. The portable thermometer is placed in the center of the mixture during the test, so the temperature of the mixture detected by the thermometer remains unchanged for a long time. The turning points of step-cooling curve are taken as the change transition temperature. The phase transition temperatures of different ratios are shown in tab. 4. From tab. 4, it can be seen that low eutectic system can be formed by capric acid and palmitic acid. When the mass ratio of capric acid to palmitic acid is 7:3, the low eutectic point of the binary phase change material is formed, and the crystallization temperature of the system is 26 °C.

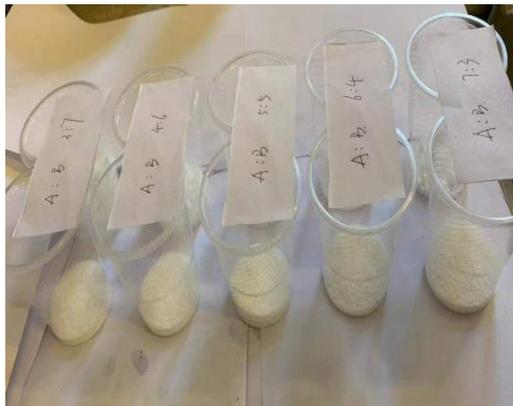


Figure 1. Mixed samples of capric acid and palmitic acid with different mix proportion

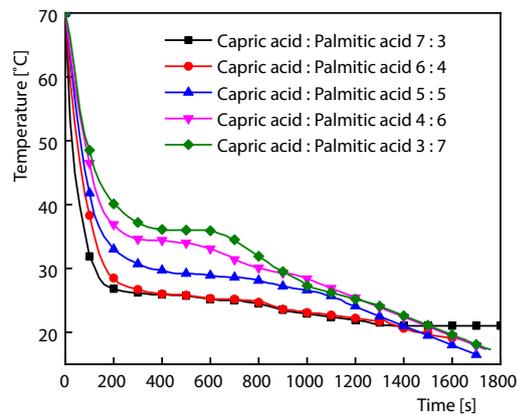


Figure 2. Step-cooling curve of binary phase change materials

Table 4. Phase transition temperature of different ratios

Mass ratio of capric acid and palmitic acid	7:3	6:4	5:5	4:6	3:7
Phase change temperature [°C]	26	27	32	37	44

Analysis of DSC test results

The DSC curves of capric acid-palmitic acid binary phase change materials were tested at the mass ratios of 7:3, 6:4, 5:5, 4:6, and 3:7, respectively. The experimental results were shown in figs. 3-7. It can be seen from figs. 6 and 7 that the change of phase change peak tends to be bimodal when capric acid and palmitic acid mix at 4:6 and 3:7. It shows that the phase change temperature of the composites is unstable when the mixture ratio deviates from that of the lowest eutectic point. The phase change temperature and enthalpy of capric acid-palmitic acid binary phase change materials of different mass ratios can be obtained from DSC curves,

as shown in tab. 5. it can be seen that the binary phase change material has the lowest eutectic point, and the temperature is about 25-27 °C, which is consistent with the step cooling curve test results. By comparing the DSC curves of different mixing ratios, the temperature range of binary phase change materials increases from the mass ratio of 7:3 to 3:7. That is, the more the ratio of binary phase change materials deviates from the ratio of low eutectic point, the lower the accuracy of phase change temperature. Combined with the previous analysis, it is shown that the optimum mixing ratio of binary phase change materials is the lowest eutectic point.

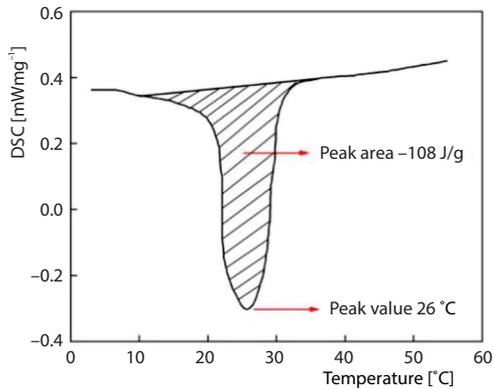


Figure 3. The DSC curve with mass ratio of 7:3

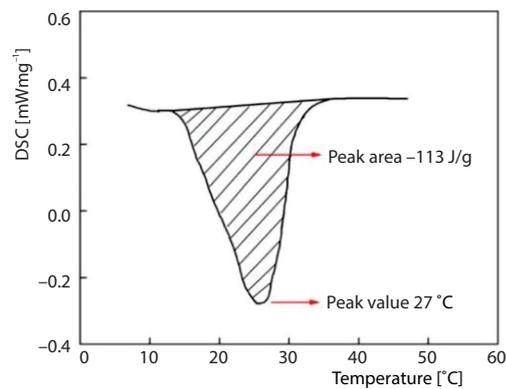


Figure 4. The DSC curve with mass ratio of 6:4

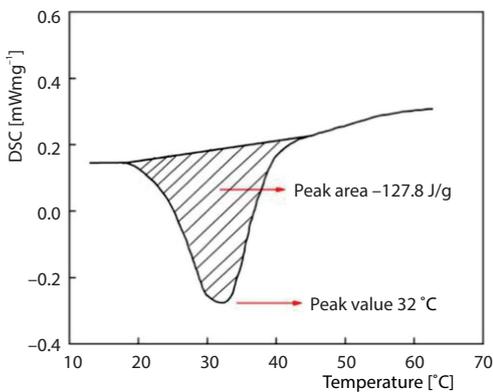


Figure 5. The DSC curve with mass ratio of 5:5

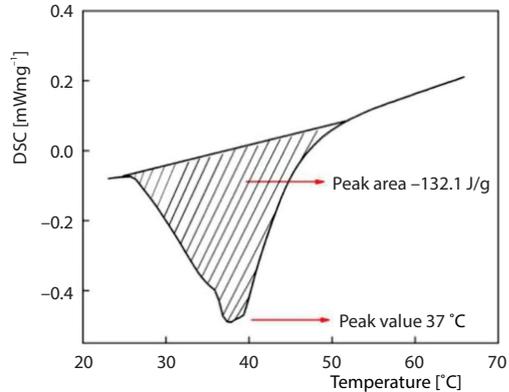


Figure 6. The DSC curve with mass ratio of 4:6

Table 5. Phase change temperature and enthalpy of different mass ratios

Mass ratio of capric acid and palmitic acid	7:3	6:4	5:5	4:6	3:7
Phase change temperature [°C]	25-27	28-31	31-33	36-40	42-46
Phase change enthalpy [Jg ⁻¹]	108.0	113.0	127.8	132.1	129.7

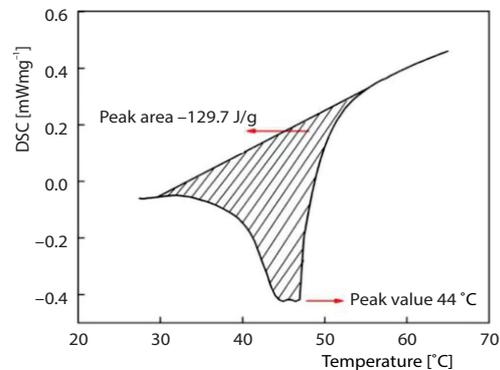


Figure 7. The DSC curve with mass ratio of 3:7

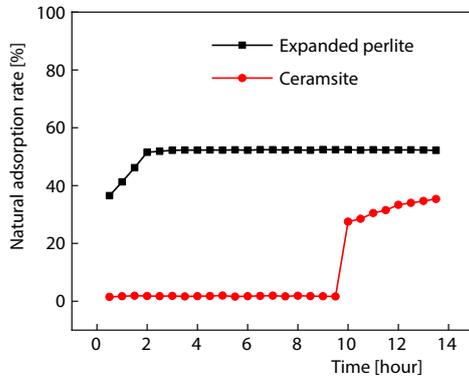


Figure 8. Natural adsorption rate of expanded perlite and ceramsite

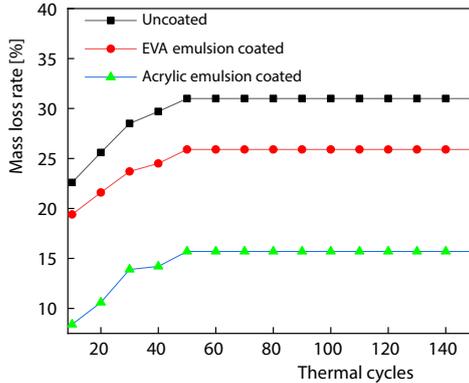


Figure 9. Mass loss rate of shape-stabilized phase change materials

with the increase of the number of thermal cycles, which indicates that the thermal stability gradually increases. Therefore, styrene acrylic emulsion is chosen as the coating material.

Analysis of specific heat capacity test results

The specific heat capacity of ordinary gypsum specimens and PCESG specimens are tested, respectively. The test results are shown in tab. 6. From tab. 6, it can be seen that the specific heat capacity of PCESG and ordinary gypsum are $1.41 \cdot 10^3$ J/kg and $0.69 \cdot 10^3$ J/kg, respectively, which means that the specific heat of PCESG is twice that of ordinary gypsum. When the temperature drops to its phase change temperature range, the phase change material in PCESG undergoes phase change, resulting in a large amount of latent heat of phase change. The specific heat capacity can well reflect the performance of its heat storage capacity. The larger the specific heat, the stronger the heat storage capacity. Therefore, PCESG has better heat storage performance compared with ordinary gypsum.

Table 6. Test results of specific heat capacity

Specimens	m_s [kg]	m_w [kg]	t_w [°C]	t_s [°C]	t [°C]	C_s [Jkg ⁻¹]
PCESG	2.345	2.513	18.6	40.0	23.7	$1.41 \cdot 10^3$
Ordinary gypsum	1.975	2.209	21.6	37.9	23.7	$0.69 \cdot 10^3$

Analysis of porous matrix adsorption test result

Natural adsorption rate of expanded perlite and ceramsite in the natural conditions are shown in fig. 8. It can be seen from fig. 8 that the adsorption rate of expanded perlite is greater than that of ceramsite. The adsorption rate of expanded perlite does not change significantly after three hours, which indicates that expanded perlite can encapsulate the binary phase change materials completely within three hours. The adsorption rate of ceramsite is basically 0 in the first 10 hours. The heat storage capacity of expanded perlite is higher than that of ceramsite. Therefore, expanded perlite is finally selected as the encapsulating materials.

Analysis of thermal stability test result

The EVA emulsion and styrene acrylic emulsion are selected as the coating materials. The mass loss rate of shape-stabilized phase change materials (uncoated, EVA emulsion coated, and styrene acrylic emulsion coated) after 150 thermal cycles is shown in fig. 9. It can be seen that the mass loss rate of the coated shape-stabilized phase change material is obviously smaller than that of the uncoated material, and the mass loss rate of the phase change material coated by styrene acrylic emulsion is less than that of EVA. As can be seen from fig. 9, the curves of each group gradually tend to be flat

Analysis of thermal conductivity coefficient test results

The smaller the thermal conductivity coefficient is, the stronger the thermal insulation performance is. The thermal conductivity coefficient of ordinary gypsum is generally 0.30-0.325 W/mK. The test results of thermal conductivity coefficient are shown in tab. 7. From tab. 7, it can be seen that the thermal conductivity coefficient of the specimens is decreasing with the addition of shape-stabilized phase change materials, indicating that the addition of shape-stabilized phase change materials will reduce the thermal conductivity coefficient of PCESG. When the mass fraction of shape-stabilized phase change materials increases from 40-50%, the thermal conductivity coefficient of PCESG decreases from 4.20-4.18 W/mK, which shows that the better thermal insulation can be achieved when the mass fraction of the shape-stabilized phase change materials reaches 40%.

Table 7. Test results of thermal conductivity coefficient

Specimens	G_0	G_1	G_2	G_3
Thermal conductivity coefficient [Wm ⁻¹ K ⁻¹]	0.325	0.263	0.216	0.214

Analysis of compressive strength test results

While improving the functionality of wall materials, the new materials must meet the requirements of wall materials (compressive strength should not be less than 3 MPa). The compressive strength was tested and the average value was obtained. The test results of compressive strength are shown in tab. 8. With the increase of the shape stabilized phase change materials, the voids in the composites increase, which leads to the decrease of the compressive strength of the composites, but the degree of reduction is not significant. The addition of shape-stabilized phase change materials does not significantly affect the mechanical properties of gypsum.

Table 8. Test results of compressive strength

Specimens	P_0	P_1	P_2	P_3
Compressive strength [MPa]	5.02	4.79	4.20	4.18

Analysis of SEM

The SEM images of four different proportions of PCESG are shown in figs. 10-13.

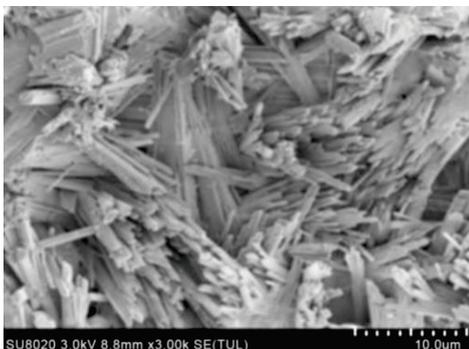


Figure 10. The SEM images of G_0

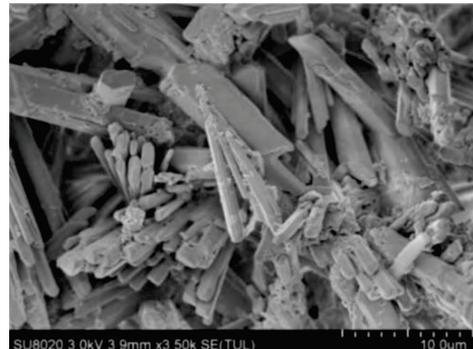
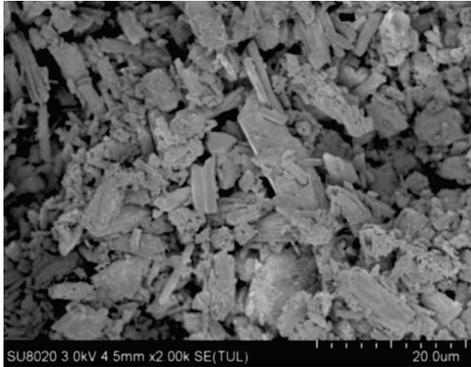
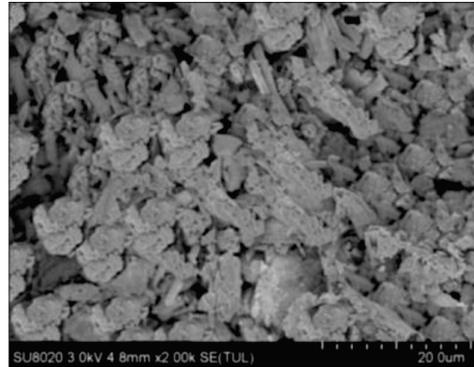


Figure 11. The SEM images of G_1

Figure 12. The SEM images of G_2 Figure 13. The SEM images of G_3

It can be seen from fig. 10 that the gypsum matrix is needle like structure and the crystals are closely connected, and it can be seen from figs. 11-13 that the shape-stabilized phase change material is closely attached to the surface of the gypsum matrix. With the addition of shape-stabilized phase change material, the crystalline state of gypsum changes to a certain extent, and the bonding effect between crystals becomes loose. This is corresponding to the test results in tab. 8, that is, the change of crystalline state of gypsum will slightly reduce the strength of PCESG.

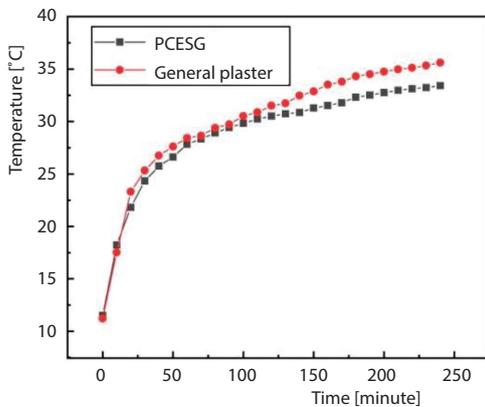


Figure 14. Temperature curves of PCESG and general plaster

outdoor heat transfer, slow down the rising rate of indoor temperature, and it can effectively slow down the indoor temperature fluctuation and improve the living comfort compared with the general plaster under the same temperature change.

Conclusions

In this paper, the thermophysical properties of binary phase change materials with different ratios of capric acid and palmitic acid were studied by step cooling curve method and differential scanning calorimetry, and porous materials were used as encapsulating materials to prepare form-stabilized phase change materials, and the specific heat capacity, thermal conductivity coefficient and compressive strength of PCESG were tested, respectively. The following conclusions are concluded.

Analysis of energy-saving effect test

The temperature curves of PCESG and general plaster are shown in fig. 14. When general plaster is used on the inner surface of the wall, the time taken for the internal temperature of the building model to rise from 22.32-29.86 °C is 90 minutes, and the internal temperature of the building model is 35.8 °C after 4 hours. When the PCESG is applied to the inner surface of the wall, the internal temperature of the building model increases from 22.06-29.64 °C for 130 minutes, and the internal temperature of the building model is 33.10 °C after 4 hours. This shows that the heat storage and release of PCESG can reduce the indoor and

- Capric acid-palmitic acid binary phase change material can form a eutectic system. When the mass ratio of capric acid to palmitic acid is 7:3, the low eutectic point of the binary system is formed. The crystallization temperature of the system is the lowest, which is 26 °C.
- The adsorption capacity of expanded perlite is much larger than that of ceramsite, and the mass loss rate of the material coated by styrene acrylic emulsion is lower than that of EVA. Expanded perlite and styrene acrylic emulsion are selected as encapsulating materials and coating materials, respectively.
- The specific heat capacity of PCESG is about twice that of ordinary gypsum. With the addition of phase change materials, the thermal conductivity coefficient of PCESG decreases gradually, and the compressive strength decreases gradually at the same time.
- The PCESG can effectively slow down the indoor temperature fluctuation and improve the living comfort compared with the general plaster under the same temperature change.

Acknowledgment

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Nomenclature

C_s	– specific heat capacity of gypsum
C_w	– specific heat capacity of water
m_s	– quality of gypsum
m_w	– quality of water
t	– equilibrium temperature
t_s	– initial temperature of gypsum
t_w	– initial temperature of water

Acronyms

EVA	– ethylene vinyl acetate
PCESG	– phase change energy storage gypsum

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