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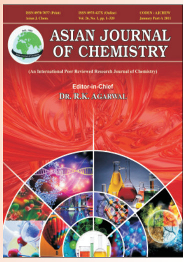

# Preparation and Performance Research of Gypsum Based Phase Change Materials†

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Expanded perlite and ceramsite were used as carriers, expanded perlite-paraffin (EP) and ceramsite-paraffin (CP) composite phase change materials (PCMs) were prepared by vacuum impregnation method and the optimal adsorbance were tested; and then gypsum based PCMs were prepared. The influences of two kinds of composite PCMs on thermal performance and mechanical performance of gypsum based materials were studied. The results showed that with the increase of the proportion of composite PCMs, heating and cooling rate of gypsum based PCMs were slowed down and it took longer to reach equilibrium temperature. Thermal inertia was increased by addition of composite PCMs; The composite effect of expanded perlite-paraffin and gypsum was better than ceramsite-paraffin and gypsum. The compressive strength of the sample with 20 % of expanded perlite-paraffin was 12.7 % higher than that of the sample with expanded perlite in the same volume of expanded perlite-paraffin.

**Keywords: PCMs, Gypsum based, Paraffin, Thermal property, Mechanical performance.**

## INTRODUCTION

Phase change materials (PCMs) are materials that can store (or release) thermal energy and adjust temperature by storing and releasing large amounts of latent heat during the phase change process. It can be used to solve the problem that energy supply and demand does not match in time and space, increase energy efficiency and also regulate the ambient temperature own to their constant temperature during heat storage/release[1]. Phase change materials have been a major topic of building materials in resent years. Though thousands of PCMs have been found, few of them were applicable for several reasons[2]. Compounding method was used to prepare PCMs with appropriate melting points and high latent heat[3,4]. Microencapsulation method shows a great potential for solving the problems existing in the utilization of solid-liquid PCMs, such as prevent PCMs from leaking out and control the volume change[5,6]. The methods for preparing microencapsulated PCMs have been developed, for instance, interfacial polymerization, spray drying and *in situ* polymerization[6]. The utilizations of PCMs in gypsum based system[7] and cement based system[8] have been studied and obtained a few of research findings.

Gypsum is a kind of green material with characteristics of respiration function, thermal and acoustic insulation and controlling indoor humidity, *etc*. Paraffin has some desirable properties such as high latent heat, suitable melting/freezing point, low vapor pressure and easy availability[9]. In this paper, paraffin and expanded perlite/ceramsite was used as PCM and carrier to prepare expanded perlite-paraffin (EP) and ceramsite-paraffin (CP) composite materials, respectively. Subsequently, gypsum based PCMs were prepared and the thermal performance and mechanical performance of them were studied.

## EXPERIMENTAL

Paraffin was supplied by Shanghai Yonghua paraffin Co., Ltd., China and the melting point was from 46-48 ºC. Building gypsum was obtained from Hefei Hongsong Decoration Co., Ltd, China. Expanded perlite (maximum particle sized ≤ 2.5 mm) and ceramsite (maximum particle sized ≤ 4.5 mm) were selected as carriers in this paper, which has 75 and 800 kg/m$^3$ bulk density and thermal conductivity of 0.04 and 0.40 w/(m K), respectively.

**Preparation of gypsum based phase change materials:** The composite PCMs were prepared using a vacuum absorption method. Firstly, clastic paraffin and expanded perlite/ceramsite were mixed with different proportions in beakers, placed in a vacuum oven at 65 ºC for 4 h and then cooled to ambient temperature out of the vacuum oven. 50 g of EP and CP were spread on self-made device, respectively. Heating at 65 ºC for 1 h and cooling to room temperature for two times.

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The optimal proportions of paraffin to expanded perlite/ceramsite with good thermal stability and high adsorption of paraffin were evaluated by mass loss rate of each samples.

Expanded perlite-paraffin (EP) and ceramsite-paraffin (CP) were added at 0, 5, 15 and 20 % by weight of gypsum, individually and then EP-gypsum based PCMs (EP-system) and CP-gypsum based PCMs (CP-system) were prepared. 40 mm × 40 mm × 160 mm specimens were prepared for testing compressive strength. Resistance thermometers of RT-1 type were placed in the cylindrical specimens with the Φ 60 mm × H90 mm for thermal properties testing.

**Performance testing:** The samples were dried in a drying oven at 60 ºC to constant weight after curing for 3d. Compressive strength of samples was tested by hydraulic testing machine at 2 mm/min load rate. The as-prepared cylindrical specimens were placed in a constant temperature water-bath at 65 ºC (Fig. 1), then took out after reaching equilibrium temperature. The temperatures of the samples were tested by multiple temperature recorder (MCU-32, Nanjing Genan Industrial Co., Ltd, China) every 3 minutes and then the thermal properties of gypsum based PCMs were evaluated by the change of temperature.

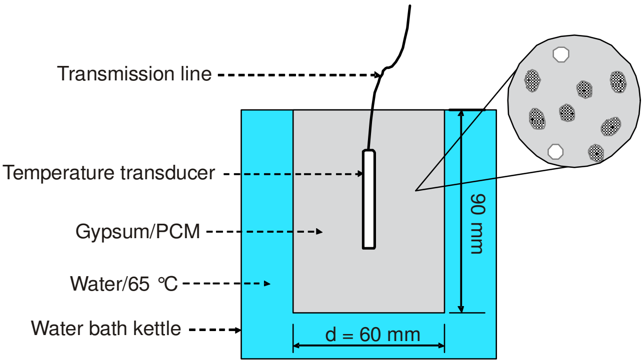
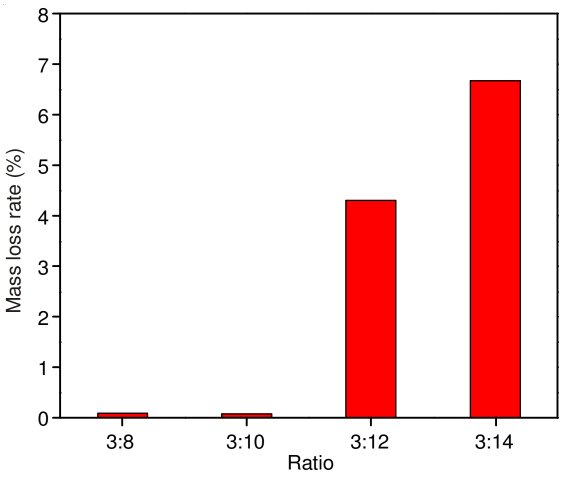
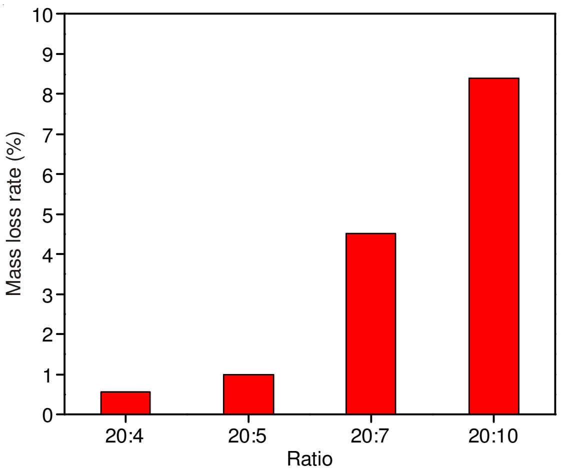


Fig. 1. Thermal properties testing unit of gypsum based PCMs



(a) Expanded perlite-paraffin system



(b) Ceramsite-paraffin system

Fig. 2. Mass loss rate of composite PCMS in different systems after 2 times thermal cycles

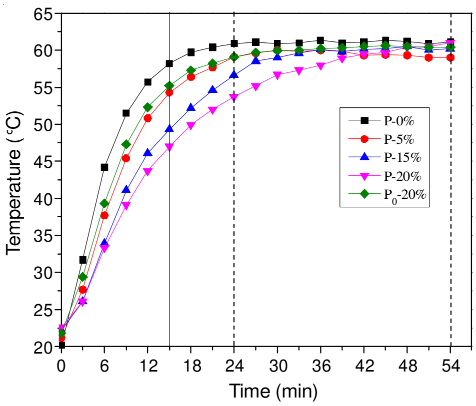
## RESULTS AND DISCUSSION

**Optimal proportion of composite PCMs:** Fig. 2 shows the mass loss rate of composite PCMs in different systems after 2 times thermal cycles. When the proportion for expanded perlite:paraffin ≤ 3:10, ceramsite:paraffin ≤ 20:5, the mass loss rate is below 0.08 and 1 %, respectively, no obvious leakage of paraffin and none agglomeration phenomenon were observed. The optimal proportions of paraffin were 77 and 20 % in EP and CP by weight, respectively. Microstructure of EP was determined by optical microscope (OM). The results showed that paraffin penetrated to the honeycomb microstructures of expanded perlite and as-prepared EP composite PCMs could maintain its form perfectly and no leakage of paraffin was observed due to the effect of capillary force and surface tension. However, the pores on ceramsite were so large that it would be easy to leakage at phase-transition temperature, so the adsorption and stability of CP was lower than EP.

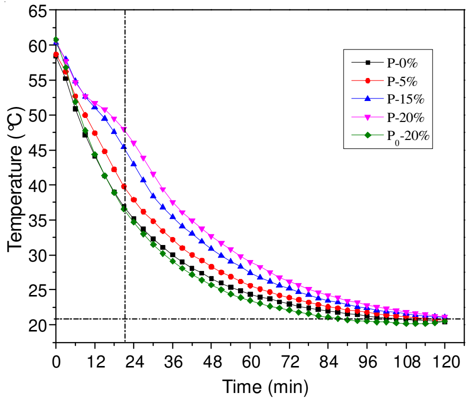
**Thermal properties of gypsum based PCMs:** Figs. 3 and 4 shows the temperature-time curves of EP-system and CP-system for heating and cooling process, respectively. Linear fitting results of the temperature within 0-15 min in heating process and 0-21 min in cooling process are shown in Table-1.

TABLE-1
FITTING RESULTS OF TEMPERATURE-TIME CURVES OF GYPSUM BASED MATERIALS IN HEATING AND COOLING PROCESS

| No. | System | Addition (%) | Heating (X = 0-15 min) | Cooling (X = 0-21 min) |
|---|---|---|---|---|
| 1 | Gypsum | 0 | $Y = 2.56X + 24.35, R^2 = 0.915$ | $Y = -1.04X + 57.60, R^2 = 0.985$ |
| 2 | EP + G | 5 | $Y = 2.31X + 22.20, R^2 = 0.973$ | $Y = -0.91X + 58.50, R^2 = 0.998$ |
| 3 | | 15 | $Y = 1.93X + 21.97, R^2 = 0.980$ | $Y = -0.69X + 59.65, R^2 = 0.986$ |
| 4 | | 20 | $Y = 1.72X + 22.40, R^2 = 0.984$ | $Y = -0.56X + 59.17, R^2 = 0.939$ |
| 5 | E + G | Sub for 20 EP in volume | $Y = 2.32X + 23.48, R^2 = 0.962$ | $Y = -1.17X + 59.57, R^2 = 0.980$ |
| 6 | CP + G | 5 | $Y = 2.44X + 21.58, R^2 = 0.962$ | $Y = -1.00X + 59.29, R^2 = 0.989$ |
| 7 | | 15 | $Y = 2.39X + 21.93, R^2 = 0.964$ | $Y = -1.00X + 59.60, R^2 = 0.989$ |
| 8 | | 20 | $Y = 2.40X + 20.65, R^2 = 0.979$ | $Y = -0.97X + 59.47, R^2 = 0.996$ |
| 9 | C + G | Sub for 20 CP in volume | $Y = 2.53X + 25.83, R^2 = 0.912$ | $Y = -1.23X + 25.83, R^2 = 0.983$ |

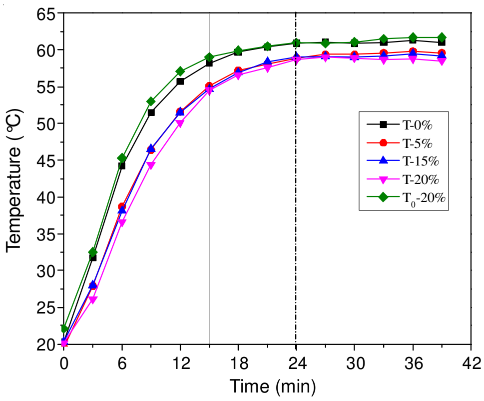


(a) Heating curves

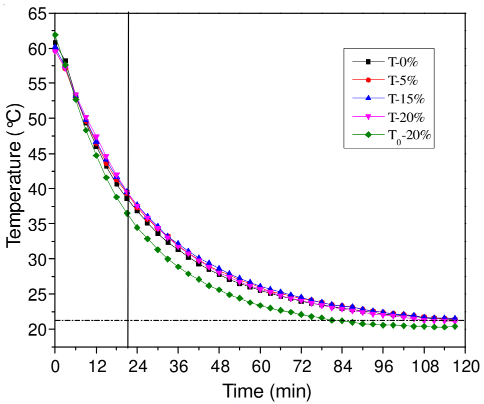


(b) Cooling curves

Fig. 3. Influence of EP/E on heating and cooling process of gypsum based PCMs



(c) Heating curves



(d) Cooling curves

Fig. 4. Influence of CP/C on heating and cooling process of gypsum based PCMs

It could be observed that the thermal conductivity of gypsum based materials reduced with an addition of expanded perlite in No. 1, 5 and 9. However, the thermal conductivity was improved with an addition of ceramsite in cooling process. It can be explained that there is a great gap on thermal conductivity between expanded perlite and ceramsite and it affects the rate of heating and cooling on gypsum based composite materials. With the increase of the proportion of EP, the slope of fitting lines decreased gradually in heating process, increased gradually in cooling process and temperature variation was getting slower. But the change rule of CP-system was more complex than EP-system. It could be explained that the thermal conductivity played a leading role in samples with low proportion of CP, while PCMs played a vital role with high proportion. Therefore, it could be concluded that thermal inertia was increased by the adding of EP and CP. It took 24, 30, 36 and 54 min for reaching equilibrium temperature by addition of EP with the proportion of 0, 5, 15 and 20 % in the heating process of EP-system, respectively. It was delayed by 25, 50 and 125 % on the basis of control sample, respectively. In CP-system, it took 24, 27, 27 and 30 min for reaching equilibrium temperature. The influence of EP on thermal performance was more obvious than CP.

In EP-system, temperature plateaus appeared near 52 ℃ in cooling curves and the higher of the adsorption of EP, the clearer of the plateaus. It may be caused by the releasing of latent heat in the process of solid-liquid phase change and it proves that the latent heat value of composite PCMs is directly related to the adsorption of paraffin. The theoretical latent heat value of composite PCMs can be calculated in accordance with formula as follows:

$$\Delta H_{\text{composite PCMs}} = \Delta H_{\text{paraffin}} \times \eta$$

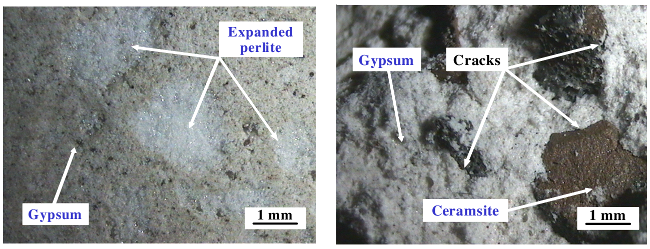
where $\Delta H_{\text{composite PCMs}}$ and $\Delta H_{\text{paraffin}}$ represent the latent heat of composite PCMs and paraffin, respectively. $\eta$ is the mass fraction of paraffin in composite PCMs.

**Mechanical performance of gypsum based PCMs:** Mechanical properties of gypsum based materials are shown in Table-2. It is noted that compressive strength and specific strength of samples decreased with the increase of the proportion of composite PCMs in each system. When the proportion of EP and CP increased from 5 to 20 %, the compressive strength of the samples dropped significantly from 2.98 to 2.40 MPa, from 2.25 to 1.80 MPa, equal to dropped 19.5 and 20 %, respectively. In addition, the compressive strength and specific strength of EP-gypsum based PCMs were higher than CP-gypsum based PCMs at the same proportion of EP and CP. Fig. 5 shows the optical microscopy images of the sections of gypsum based PCMs in different systems. It could be observed that the interface between EP and gypsum was well and there was a meshing effect between gypsum and EP. However, there is a weak interface between CP and gypsum with some obvious cracks.

As shown in Table-2, the compressive strength of the sample with 20 % of EP is 12.7 % higher than that of the sample with expanded perlite in the same volume. It is mainly due to that the compactness of expanded perlite was improved by paraffin, so that the compressive strength of EP-gypsum based PCMs was increased. However, the compressive strength

TABLE-2
MECHANICAL PROPERTIES OF GYPSUM BASED MATERIALS

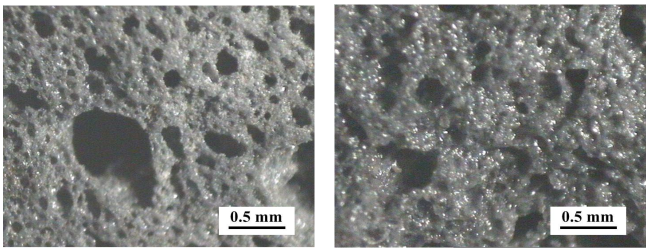
| No. | System | Addition (%) | Compressive strength (MPa) | Unit weight ($g/cm^3$) | Specific strength × $10^{-3}$ [$(N/m^2)/(Kg/m^2)$] |
|---|---|---|---|---|---|
| 1 | EP + G | 5 | 2.98 | 1.27 | 2.35 |
| 2 | | 15 | 2.45 | 1.16 | 2.11 |
| 3 | | 20 | 2.40 | 1.14 | 2.10 |
| 4 | E + G | Sub for 20 % EP in volume | 2.13 | 1.08 | 1.97 |
| 5 | CP + G | 5 | 2.25 | 1.30 | 1.73 |
| 6 | | 15 | 2.07 | 1.28 | 1.62 |
| 7 | | 20 | 1.80 | 1.26 | 1.43 |
| 8 | C + G | Sub for 20 % CP in volume | 1.99 | 1.24 | 1.61 |



(a) EP-gypsum based PCMs (b) CP-gypsum based PCMs

Fig. 5. Optical microscopy images of the sections of gypsum based PCMS in different systems

of the sample with 20 % of CP reduced by 9.5 % relative to that of the sample with ceramsite in the same volume. It can be explained that the surface of CP was adhered by paraffin (Fig. 6), which results in the decrease in interfacial strength of the samples. In addition, the difference in elasticity modulus of ceramsite and gypsum is the major cause of stress concentration along the interface.



(a) Ceramsite (b) Ceramsite + paraffin

Fig. 6. Optical microscopy images of the surface on ceramsite and ceramsite-paraffin composite PCMs

## Conclusion

- Expanded perlite and ceramsite were used as carriers, EP and CP composite PCMs were prepared by vacuum impregnation method. The optimal proportion with low leakage and high adsorption was 3:10 for expanded perlite: paraffin, 20:5 for ceramsite: paraffin, respectively.
- Thermal inertia was increased by the admixing of EP and CP. With the increase of proportion of composite PCMs, the heating and cooling rate of gypsum based materials was gradually declined and the time to equilibrium temperature was prolonged. The composite effect of EP and gypsum was better than CP and gypsum.
- Compressive strength of the sample with 20 % of EP was 12.7 % higher than that of adding expanded perlite in the same volume of 20 % of EP, while with 20 % of CP reduced by 9.5 % relative to adding ceramsite in the same volume.

## ACKNOWLEDGEMENTS

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