



Preparation of paraffin/expanded vermiculite with enhanced thermal conductivity by implanting network carbon in vermiculite layers



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HIGHLIGHTS

- Expanded vermiculite/carbon (EVMC) was prepared by in-situ sucrose carbonization.
- The form-stable composite PCM of paraffin/EVMC were fabricated.
- The maximum absorption of paraffin in the paraffin/EVMC is 53.2 wt.%.
- We developed a new way to enhance the thermal conductivity of the organic PCMs.

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ABSTRACT

Paraffin/expanded vermiculite with modified porous carbon layer was prepared in the study. In the composite phase change materials (PCM), expanded vermiculite was impregnated with sucrose in the layer and then carbonated in situ in order to enhance thermal conductivity and improve the adsorption capacity of expanded vermiculite, which acted as a carrier material in the preparation of the form-stable PCM. SEM images showed the morphology of expanded vermiculite had been changed after carbonation. A series of micro-pores had been formed among the layers of expanded vermiculite with the diameter ranging from several microns to 120 μm . Thermal properties of the composite PCM were determined by differential scanning calorimeter (DSC) analysis. According to DSC analysis results, when the paraffin adsorption reached 53.2 wt.%, latent heats of the composite PCM were respectively 101.14 J/g at the freezing temperature of 48.85 $^{\circ}\text{C}$ and 103 J/g at the melting temperature of 53.1 $^{\circ}\text{C}$. TGA results showed that the form-stable composite PCM presented good thermal stability. Due to the formation of carbon structure in the layer of expanded vermiculite, the thermal conductivity of the supporting materials was improved, thus slightly decreasing the extrapolated onset melting temperature of pure paraffin. Furthermore, the results of FT-IR analysis and thermal cycling tests showed that the form-stable composite PCM had good chemical stability and thermal reliability after 200 melting/freezing cycles. The form-stable composite PCM with good thermal properties, thermal reliability, and chemical stability is the promising PCM for the low-temperature thermal energy storage applications.

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1. Introduction

Phase change materials (PCM) for LHTES have become an attractive option due to their high energy storage density, high heat recovery with small temperature drop, constant heat source temperature, and repeatable utilization property [1–2]. And PCM have been applied in many fields, such as solar energy storage, waste heat recovery, air-conditioning systems, building energy conservation, and insulation clothing [3].

In recently years, a lot of PCMs, including salt hydrates, PEG, paraffin, fatty acids and their mixtures, have been extensively

investigated and are almost commercially applied in energy-saving building materials [4]. Building materials containing PCM allows the latent heat storage, thus achieving the temporal and spatial transfer of energy. When the ambient temperature rises, the PCM absorbs heat by melting. On the contrary, when the temperature declines, the PCM turns back to its original state by releasing the stored heat. In this way, the PCM can moderate the indoor temperature to swing within a certain comfort range. However, the leakage of organic PCM and heat transfer between PCM and the ambient environment are two difficulties in the development of building materials containing PCM. As a result, most of PCM are impregnated in traditional building materials (gypsum board, plaster, concrete, and other wall covering

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materials) or enclosed in metallic or polymeric capsules [5]. Although the immersion of conventional building materials into molten PCM is the simple and cheap process, they will become inflammable because of the leakage of the PCM to the surfaces, especially after the PCM experiences several melting/freezing cycles. The leakage of the PCM will not only cause the decline of heat storage capacity but also affect the appearance and security of buildings. The integration method of microencapsulated PCM with conventional building materials is complicated and expensive [6]. In recent years, the form-stable PCM have been extensively studied. The form-stable PCM can be prepared by blending organic PCMs with supporting materials, such as vermiculite, diatomite, expanded perlite, silica fume, expanded graphite, bentonite, and clay mineral [7–13].

As a mixture of various straight-chain n-alkanes, Paraffin has suitable thermo-physical properties, such as good stability, large latent heat and non-toxicity and becomes one of the most widely used PCM. Paraffin can be mixed with porous building materials easily and retained in pores by surface tension and capillary forces. Paraffin has the latent heat storage capacity of 200–250 J/g a wide melting temperature range, and high thermal stability at 250 °C [14]. However, the low thermal conductivity (below 0.4 W/(m·k)) of paraffin poses a challenge for its application. Therefore, it is necessary to enhance the thermal conductivity of paraffin. [15–16].

Porous building materials have been applied in the construction field in recent years. Vermiculite is the 2:1 type layered silicate mineral. When vermiculite is heated to about 800 °C, vermiculite can expand to 8–30 times of its original volume. Expanded vermiculite (EVM) has good physical properties, such as fire resistance, the porous structure, non-toxicity and low density. Besides it is usually used as a lightweight aggregate for plaster, concrete compounds and the component of wallboard filler. In addition, it has good chemical compatibility with organic PCMs, such as alkanes and their mixtures. Considering the above factors, the EVM is believed as one of the most suitable and economical candidate materials for preparing the form-stable composite PCM for energy storage in buildings. However, the low thermal conductivity of the EVM limits its applications in LHTES. In order to solve this problem, many methods have been developed. Especially, adding a material with high thermal conductivity into EVM was the most promising method [7,17].

In the paper, we proposed a new method to enhance the thermal conductivity of the form-stable composite PCM of paraffin/EVM (EVMP). The supporting material, expanded vermiculite/carbon (EVMC), was prepared firstly by in-situ carbonizing sucrose which had been absorbed in the layer of EVM. Then, the pure paraffin was absorbed in EVMC via a method of vacuum impregnation. Finally, a form-stable composite PCM (EVMCP) with enhanced thermal properties was obtained. The results indicated that the prepared composite PCM was a promising candidate material for thermal energy storage applications.

2. Experimental

2.1. Materials

Paraffin (CP, mp: 50~52 °C) was purchased from Xilong Chemical Reagent Beijing Co., Ltd. EVM was obtained from Hebei Lingshou and the chemical composition of EVM was provided in Table 1. The particle size of the EVM was about 1~2 mm and the

EVM samples were dried at 105 °C for 24 h for use. Sucrose (AR), which used to enhance the thermal conductivity of the form-stable composite PCM, was supplied by Xilong Chemical Reagent Beijing Co., Ltd.

2.2. Preparation of the composite EVMC

Firstly, the sucrose solution was prepared. Then, the composite EVM/sucrose was prepared by the impregnation method. The composite EVM/sucrose was dried at 105 °C and then calcined at 600 °C for 3 h in an atmosphere containing 10% H₂ and 90% N₂ (volume percent). The carbonization result of the composite EVMC was explored by FT-IR and SEM. Fig. 1 shows the micro-structure of EVM, the molecular structure of sucrose and the whole carbonization process.

2.3. Preparation of the form-stable composite PCM

The composite PCM were prepared by the vacuum impregnation method. Fig. 2 shows the preparation process of the composite PCM by vacuum impregnation. Solid paraffin and composite EVMC were placed in an enclosed glass container. The vacuum pump was connected to the container to evacuate air. Then the container was heated under the vacuum condition in order to melt solid paraffin. Paraffin was easily absorbed under the vacuum condition. The initial weight of the composite EVMC was placed in enclosed glass container and recorded as m_0 . After vacuum impregnation, the composite EVMC filled with paraffin were taken out from liquid PCM with a stainless mesh. Then placed the composite PCM on a filter paper and put into the oven at a temperature of 80 °C, for removing liquid PCM captured by the surface of composite or not supported in pore. Continuous substituting the filter paper in the whole process until the paraffin was no longer leaking. The weight of the final form-stable composite PCM was recorded as m . Then the mass percentage of retained paraffin was calculated by the Eq. (1):

$$\eta = \frac{m - m_0}{m} \quad (1)$$

The calculation results by the above method was corresponded with the result which calculated according to Eq. (2).

$$\eta = \frac{H_{theo}}{H_{PCM}} \quad (2)$$

Where the value of η is the mass fraction of paraffin; H_{PCM} denotes the latent heat of the pristine paraffin; H_{theo} represents the theoretical phase change enthalpy of the composite PCM.

Finally, 53.2 wt.% paraffin could be retained in composite EVMC and 64.6 wt.% paraffin could be retained in EVM without leakage. Therefore, the final products were considered as the form-stable PCM.

2.4. Characterization

The morphology of the EVM, composite EVMC and the form-stable composite PCM were observed by scanning electron microscopy (SEM, Hitachi S4800). The thermal reliability of the form-stable composite PCM was analyzed by thermal cycling test and Fourier transform infrared spectroscopy (FT-IR, SHIMADZU

Table 1
Chemical composition of the expanded vermiculite (wt.%).

Components	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	CaO	K ₂ O	H ₂ O	Others	Total
Percentages (wt.%)	43.2	12.68	4.56	24.2	0.96	5.95	7.6	0.85	100

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