



Preparation and properties of lauric acid/diatomite composites as novel form-stable phase change materials for thermal energy storage



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ABSTRACT

The lauric acid (LA)/diatomite composite phase change materials (PCMs) were prepared using a direct impregnation method, in which LA was chosen as PCM. The chemical structure, chemical compatibility, element composition, crystalline structure, thermal properties and thermal stability of LA/diatomite composite were extensively investigated by Fourier transform infrared (FTIR) spectroscopy, energy dispersive spectroscopy, X-ray diffraction (XRD), differential scanning calorimetry and thermal gravimetric analysis. FTIR and XRD results indicate that there are only physical interactions between LA and diatomite in LA/diatomite composite. The decrease of XRD intensity of composite PCM confirms the reduction of the crystalline size of LA in LA/diatomite composite. The melting and freezing temperatures of composite PCM are 40.9 °C and 38.7 °C respectively. The latent heat of composite PCM is 57.2 J/g. LA/diatomite composite does not decompose when the surrounding temperature is lower than 157 °C, indicating that the composite PCM has a good thermal stability.

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1. Introduction

In the last two decades, the increasing attention has been paid to latent heat energy storage (LHES) based on phase change materials (PCMs) [1–3]. Among the PCMs, the fatty acid such as lauric acid (LA) is of much importance due to the proper phase transition temperature (PTT) for a given application in building field and textile industries [4–6]. They have the ability to absorb and release abundant heat during their phase transition and retain the thermal inertia at their PTT. It is considered that the thermal energy storage problem such as thermal insulation can be solved by LHES based on PCMs [7]. The thermal comfort can be enhanced by reducing the internal temperature fluctuation when the PCMs are utilized in the fields of the buildings and the textile products [8]. The PCMs will be converted into liquid above the corresponding PTT. Therefore, the effective capsulation of PCMs has to be employed to prevent leaking-out once melted [9–11].

Impregnation composite is a facile and robust technology, which has been used in such fields as building materials and textile industries [12]. Due to the lower thermal conductivity of fatty acid [13] compared with inorganic substance, the papers about fatty acid/inorganic substance composite increase in the last

few years [14]. Karaman's group [10] studied the polyethylene glycol/diatomite composites as form-stable PCMs. The relevant melting temperature, freezing temperature and latent heat were 27.7 °C, 32.2 °C and 87.09 J/g, respectively. Li and coworkers [15] revealed that the paraffin as the PCM was absorbed into the layered bentonite by using solution intercalation method. The melting temperature and latent heat of the paraffin/bentonite composite were 41.7 °C and 39.84 J/g, respectively. Li et al. [16] prepared binary fatty acid/diatomite composites as form-stable PCMs based on the eutectic caprice-lauric acid blends. 57 wt.% of caprice-lauric acid blends was absorbed into the porous diatomite and the latent heat of binary acid/diatomite composite reached 66.81 J/g. Xu et al. [17] studied the preparation and thermal properties of paraffin/diatomite composites as form-stable PCMs. The maximum latent heat of paraffin/diatomite composites was found as 70.51 J/g without the leakage of melted paraffin. He's group [18] reported the preparation and properties of shape-stabilized n-alkanes/silica composites as PCMs which were synthesized in a sol-gel process using sodium silicate precursor.

From what was mentioned above, diatomite was selected as the supporting material in many papers [19,20] resulting from its light weight, high porosity, high absorption capability, rigidity and inertness [10]. For the chemical composition and physical structure, the diatomite was suitable in lots of fields such as building materials and sound insulator [21]. Therefore, diatomite was considered to be used as the available supporting material of fatty

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Table 1
Chemical composition of the pure diatomite/%.

Series	MgO	Al ₂ O ₃	SiO ₂	K ₂ O	CaO	Fe ₂ O ₃	TiO ₂
Diatomite	6.93	12.36	69.84	3.43	2.80	4.21	0.43

acid/diatomite composites for thermal energy storage [22]. To the best of our knowledge, no studies have been conducted to develop the lauric acid/diatomite composites as form-stable PCMs to potentially improve the thermal comfort of a given field such as building materials due to the proper PTT (41–48 °C).

In this study, the LA/diatomite mixtures were prepared as novel form-stable composite PCMs using a direct impregnation method without vacuum treatment. The diatomite as inorganic supporting materials has good compatibility with concrete as important building materials [23]. Given this, the LA/diatomite composite form-stable PCM has a large potential application in building fields by the direct bending of the resulting form-stable PCM and concrete, enhancing the thermal energy storage capability of the building wall [14,24]. The chemical compatibility and impregnation morphology of prepared composite PCMs were investigated by Fourier transform infrared (FTIR) spectroscopy, X-ray diffraction (XRD) and scanning electron microscope (SEM), respectively. The thermal properties and thermal stability were also studied by differential scanning calorimetry (DSC) and thermal gravimetric analysis (TGA), respectively.

2. Experimental

2.1. Materials

Lauric acid (LA, 98.5% pure) was purchased from Chengdu Kelong Chemical Reagent Company (Chengdu, China). The diatomite sample was supplied from China's Pharmaceutical Industry Co., Ltd (Beijing, China). The chemical composition is given in Table 1. The diatomite samples were dried at 80 °C for 8 h to remove existing water.

2.2. Preparation of form-stable composite PCMs

The LA/diatomite composite PCMs were prepared using a direct impregnation method without vacuum treatment [25]. Diatomite was added into six 15 ml beakers with 2 g for each beaker, and then LA of different mass was put into the six beakers respectively. A series of weight ratio of LA and diatomite was set as 0.3, 0.4, 0.5, 0.6, 0.7 and 0.8. After LA and diatomite homogenized by stirring, the beakers were sealed with the plastic wrap and put in the oven at 80 °C for 12 h, aiming for saturated adsorption in thermodynamics. Meanwhile, the sample in each beaker was stirred every 4 h during the adsorption process. Finally, the samples were cooled down to 25 °C. The formula and appearance of the form-stable composite PCMs before and after composite are shown in Table 2.

2.3. Characterization

The particle size and distribution of diatomite were measured by MASTERIZER 2000 (Malvern Instruments Ltd, England) at 25 °C.

Table 2
Formula and appearances of the form-stable LA/diatomite composites before and after composite.

Ratio of LA/diatomite	0.3:1	0.4:1	0.5:1	0.6:1	0.7:1	0.8:1
LA (g)	0.6	0.8	1.0	1.2	1.4	1.6
Diatomite (g)	2.0	2.0	2.0	2.0	2.0	2.0
Appearance before composite	Powder	Powder	Powder	Powder	Powder	Powder
Appearance after composite	Powder	Powder	Powder	Powder	Bulk	Bulk

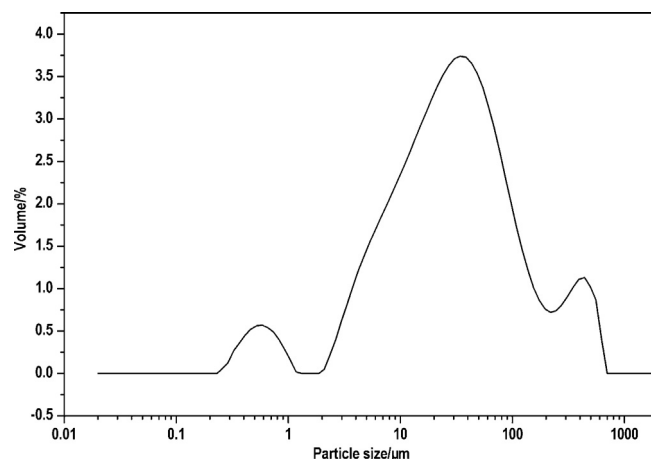


Fig. 1. Particle size and distribution of diatomite.

The testing particle size ranges are from 0.02 to 2000 μm. Before the testing process, the diatomite sample was treated with ultrasound for 5 min to obtain a better dispersion in water. The structures of LA, diatomite and LA/diatomite composite were characterized using a Nicolet-560 Fourier transform infrared (FTIR) spectrometer (USA) with a resolution setting of 4 cm⁻¹. The scanning range was changed from 4000 to 400 cm⁻¹. The crystalline structures of diatomite, LA and LA/diatomite composite were investigated using the X-ray diffraction (X'Pert pro MPD, Netherlands). The melting and freezing temperatures as well as latent heat of LA and LA/diatomite composite were studied by a differential scanning calorimeter (DSC 204 F1, German) at 10 °C/min under nitrogen atmosphere. Thermal gravimetric analysis was carried out in a TA Instrument SDT-Q600 thermal analyzer (USA) from 25 to 600 °C under a nitrogen atmosphere with a heating rate of 10 °C/min and about 10 mg of each sample. The obtained TGA results were analyzed by a TA universal analysis program. The micrographs of diatomite and LA/diatomite composite PCMs were observed using a JEOL JSM-5900LV SEM (Japan) with an accelerated voltage of 20 kV. The SEM samples were gold-sputtered before the observation.

3. Results and discussion

3.1. Characterization of the diatomite

The diatomite was employed as inorganic supporting materials. Considering the impact on absorption, it is indispensable to investigate the particle size and distribution of the pure diatomite before the LA/diatomite composites were prepared. Fig. 1 reveals the particle size and distribution of diatomite and the particle size parameters of diatomite are listed in Table 3. From Fig. 1, the particle size shows the trimodal distribution and the peak particle sizes are 0.58, 34.15 and 441.02 μm respectively. The integral area of three peaks is very asymmetric and the 34.15 μm is corresponding to the main peak. In Table 3, the D[50] is only 30.85 μm and the specific surface of diatomite is 0.836 m²/g which is helpful to adsorbing the melting LA.

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