



A capric–palmitic–stearic acid ternary eutectic mixture/expanded graphite composite phase change material for thermal energy storage



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ABSTRACT

This paper demonstrated a capric acid–palmitic acid–stearic acid ternary eutectic mixture/expanded graphite (CA–PA–SA/EG) composite phase change material (PCM) for low-temperature heat storage. The CA–PA–SA ternary eutectic mixture with a mass ratio of CA:PA:SA = 79.3:14.7:6.0 was prepared firstly, and its mass ratio in the CA–PA–SA/EG composite can reach as high as 90%. The melting and freezing temperatures of CA–PA–SA/EG composite were 21.33 °C and 19.01 °C, and the corresponding latent heat were 131.7 kJ kg⁻¹ and 127.2 kJ kg⁻¹. The CA–PA–SA/EG composite powders can be formed into round blocks by dry pressing easily, with much higher thermal conductivity than CA–PA–SA. Thermal performance test showed that the increasing thermal conductivity of CA–PA–SA could obviously decrease the melting/cooling time. Thermal property characterizations after 500 heating/cooling cycles test indicated that CA–PA–SA/EG composite PCM had excellent thermal reliability. Based on all these results, CA–PA–SA/EG composite PCM is a promising material for low-temperature thermal energy storage applications.

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1. Introduction

Phase change materials (PCMs) refer to materials that can store or release heat energy during their phase transition process at a nearly constant temperature. Numerous efforts have been made to explore PCMs for realizing the control of environmental temperature and matching the energy supply and demand in time and space due to their high energy storage density. PCMs have a heat storage capacity, which is about 5–14 times higher than the conventional (sensible) thermal storage materials such as water, rock and masonry. A large number of PCMs are known to melt with a heat of fusion in any required range [1]. Therefore, PCMs have been explored in many applications such as building energy conservation, solar energy storage, low temperature refrigeration and indoor temperature controlling.

Among the numerous investigated PCMs, fatty acids have been widely employed by virtue of their following desirable features, high heat capacity, good thermal and chemical stability, small volume change, self-nucleating behavior, non-toxicity, non-corrosiveness, low vapor pressure, negligible supercooling and cost-effectiveness [2]. However, the individual fatty acids are

restricted in some practical applications for their fixed phase change temperatures. Fortunately, the phase transition temperatures can be tailored into a suitable range by preparing binary or ternary eutectic mixtures of fatty acids [3]. Capric acid (CA), lauric acid (LA), myristic acid (MA) and stearic acid (SA) were selected to prepare binary fatty acid eutectic (CA–LA, CA–MA, CA–SA and LA–MA) by Wang and Meng [4], and phase change temperatures of the composites CA–LA/PMMA, CA–MA/PMMA, CA–SA/PMMA and LA–MA/PMMA (50/50 wt.%) were adjusted to a suitable range for building energy conservation. Sari [5] prepared three binary eutectic mixtures including LA–MA, LA–PA and MA–SA with a melting temperature of 34.2 °C, 35.2 °C and 44.1 °C, which are lower than the individual fatty acids. Cai et al. [6] prepared five binary fatty acid eutectics LA–MA, LA–PA, MA–PA, MA–SA and PA–SA through Schrader equation, whose melting temperatures ranged from 33.27 °C to 53.69 °C, and freezing temperatures ranged from 33.71 °C to 53.45 °C. Then, an innovative type of electrospun binary fatty acid eutectics/PET ultrafine composite fibers for latent heat storage in the field of building energy conservation were successfully fabricated as form-stable PCMs.

Nonetheless, fatty acids and their eutectic mixtures have a notable drawback of low thermal conductivity, which seriously limits the heat transfer rates during phase transition. In order to overcome the problem, the combination of PCMs with porous

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matrix materials to prepare form-stable PCMs has been proven to be an effective way [7]. Furthermore, the leakage during solid–liquid phase transition processes can be prevented due to the capillary and surface tension forces of the porous matrix materials [8]. The form-stable PCMs containing fatty acids and/or their eutectics can be prepared through various methods such as physical adsorption [9], sol–gel [10,11], microencapsulation [12] and electrospinning [13]. Among the investigated methods, directly incorporating PCMs into porous materials is a simple, convenient, low cost and effective technique. Since previous reported research, expanded graphite (EG) has been verified to be the ideal porous matrix material owing to its advantages of high thermal conductivity, large pore volume, low density and good compatibility [14].

The recent studies are mainly focused on fatty acids and/or their binary eutectics, investigations on ternary fatty acid eutectic mixtures as PCMs have rarely been reported. Zhang et al. [15] prepared LA–MA–PA/EG composite PCM, Liu et al. [16] prepared LA–MA–SA/EG composite PCM and Yang et al. [17] prepared MA–PA–SA/EG composite PCM with the phase change temperature of 30.94 °C, 29.05 °C and 41.64 °C. To the best of our knowledge, the investigation of CA–PA–SA ternary eutectic mixture as PCM has not been reported. Moreover, compared with medium-temperature and high-temperature applications have been well developed, low-temperature thermal applications such as building energy conservation, low temperature refrigeration and indoor temperature controlling have dropped behind but show great potential in the future, especially in heat storage systems on the use of PCMs with the phase change temperatures below 25 °C [18]. Hence, this research is aimed at preparing a form-stable PCM for low-temperature latent heat storage applications. Ternary eutectic mixture of CA–PA–SA with a melting temperature of about 20 °C was prepared firstly. Then, the supporting material EG was prepared through microwave method [7]. Finally, CA–PA–SA were absorbed into EG to fabricate a form-stable CA–PA–SA/EG composite PCM. The microstructure, thermal properties and stability of CA–PA–SA/EG composite PCM were characterized by Scanning electron microscopy (SEM), X-ray diffractometer (XRD), Fourier transfer infrared (FT-IR), Differential scanning calorimeter (DSC) and thermogravimetric analysis (TGA). The thermal cycling test was conducted to determine the thermal reliability of CA–PA–SA/EG composite. Afterward, the influences of EG on thermal conductivity and thermal energy storage/retrieval rates of CA–PA–SA/EG composite PCM were also investigated.

2. Experimental

2.1. Materials

Capric acid (CA, $\text{CH}_3(\text{CH}_2)_8\text{COOH}$, 98%, Chemical Pure), palmitic acid (PA, $\text{CH}_3(\text{CH}_2)_{14}\text{COOH}$, 98%, Chemical Pure), stearic acid (SA, $\text{CH}_3(\text{CH}_2)_{16}\text{COOH}$, 98%, Analytical Reagent) were supplied by Shanghai Lingfeng Chemical Reagents Co. (Shanghai, China). Expandable graphite (mesh 32, expansion ration: 300 ml g^{-1}) was purchased from Qingdao Graphite Co. Ltd., China. The chemicals were used as received without further purification.

2.2. Preparation of CA–PA–SA ternary eutectic mixture

Based on the lowest eutectic point theory, fatty acids can be blended together with an eutectic ratio to achieve the eutectic temperature, which is lower than those of individual fatty acids. The eutectic ratios of fatty acid mixture were calculated by the Schrader equation [3,15,17].

$$T = 1 / (1/T_i - R \ln X_i / \Delta H_i) \quad (1)$$

where T_i and ΔH_i are the phase change temperature and latent heat of the i th fatty acid, T is the phase change temperature of the eutectic mixture, X_i is the content of the i th substance in the eutectic mixture, and R is ideal gas constant.

Through the above equation, the mass ratio of CA–PA binary eutectic mixture was calculated first and was acted as a kind of “single” fatty acid, and then the mass ratio of single fatty acid (i.e., CA–PA) and SA was calculated again. Finally, the mass ratios for CA–PA–SA ternary eutectic mixture were determined at 79.3:14.7:6.0 and the calculated melting point of the mixture was 25.71 °C.

The CA–PA–SA ternary eutectic mixture was prepared through melt-blending. Three fatty acids (CA, PA and SA) with the calculated eutectic mass ratio were first mixed in a sealed beaker, then placed into an oven at 70 °C for 2 h until totally melted. Subsequently, the molten mixture was stirred in a magnetic stirrer for 2 h at 300 r min^{-1} to ensure the homogeneity of the mixture, and then slowly cooled down to ambient temperature.

2.3. Preparation of CA–PA–SA/EG composite PCM

The CA–PA–SA/EG composite PCMs containing 85 wt.%, 90 wt.%, and 95 wt.% CA–PA–SA were prepared by absorbing different amounts of liquid CA–PA–SA into the pores of EG. To determine the maximum mass fraction of CA–PA–SA that can be absorbed in the composite PCM, the leakage test was carried out as follows. Three CA–PA–SA/EG composite PCM samples containing 85 wt.%, 90 wt.%, and 95 wt.% CA–PA–SA were placed on three pieces of filter paper, and then inserted into an oven at a temperature of 40 °C (above the melting point of CA–PA–SA) for 8 h to make sure the phase change of CA–PA–SA from solid to liquid takes place. After cooling to ambient temperature, the samples were removed from the corresponding filter paper. Finally, a careful examination was given to each piece of filter paper to find out whether there were any traces of liquid CA–PA–SA left on it. Furthermore, the weights of the samples were compared before and after the thermal treatment to further judge whether the leakage happened.

2.4. Fabrication of compressed CA–PA–SA/EG composite PCM

The CA–PA–SA/EG composite PCM powders were formed into several round blocks by dry pressing with a cylindrical mold (4 cm inside diameter and 1 cm height) under the pressure of 100 kg cm^{-2} . Actual packing densities were calculated by the actual masses to volumes of the formed blocks, which were 435.7, 526.7, 615.4, 710.4 and 743.3 kg m^{-3} , respectively. Hot disk thermal constant analyzer (TPS2500, Hot Disk Inc., Sweden) was applied to measure the thermal conductivities of the prepared round blocks with different packing densities, with a type 5501 probe acting as both heat source and sensor. A transient plane source method, in which two samples (round blocks) of the test materials with a similar thickness were required to be placed in contact with the probe and heated at constant power for a setting scanning time, was selected for these measurements. The type of the probe, heating power and scanning time for these tests were chosen based upon the diameter, thickness and range of thermal conductivity of each sample.

2.5. Characterization

The phase change temperatures and latent heat of CA, PA, SA, CA–PA–SA and CA–PA–SA/EG composite PCM were analyzed by DSC (DSC Q20, TA Instruments, USA) under nitrogen atmosphere at a flow rate of 50 mL min^{-1} . The heating/cooling rate was set at 5 °C min^{-1} . Crystalline phases of EG, CA–PA–SA and CA–PA–SA/EG composite PCM were characterized by an X-ray diffractometer

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