



Preparation, characterization and thermal properties of fatty acid eutectics/bentonite/expanded graphite composites as novel form-stable thermal energy storage materials



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ABSTRACT

In this work, bentonite-based composite phase change materials (CPCMs) were fabricated by the impregnation of fatty acid eutectics into bentonite clay. In the composites, the palmitic acid (PA)–stearic acid (SA) eutectics mixtures were undertaken as phase change materials (PCMs) for thermal energy storage, and the bentonite were performed as the supporting material. Expanded graphite (EG) was employed for helping restrain the eutectic mixtures from leakage as well as improving thermal conductivity of the CPCMs. The differential scanning calorimetry (DSC) was adopted to assess the thermal properties of the composites, the results showed that the CPCMs have suitable melting temperature of around 54 °C with latent heat capacity of 89.12–163.72 kJ/kg. Fourier transformation infrared (FT–IR) and X-ray diffractometer (XRD) were utilized to test the chemical structure and crystalline phase of the CPCMs. The scanning electron microscope (SEM) images revealed that the organic PCMs homogeneously spread to the surface and interior of the bentonite. The thermal gravimetric analyzer (TGA) detected that the CPCMs were provided with good thermal stability. As the content of the EG increased, the leakage of the PA–SA eutectics reduced considerably. The results from the thermal conductivity meter (TCM) showed that the thermal conductivity of the CPCM with content of 5% EG reached to 1.51 W/(m K) in liquid state and 1.66 W/(m K) in solid state, which was nearly 5.6 times and 4.9 times higher than that of the CPCM without the EG. Experiments displayed that the thermal storage and release rates were noticeably enhanced by combining the EG into original CPCMs. The CPCMs maintained thermal properties after 50 heating–cooling cycling. It is envisioned that the satisfactory CPCMs maintain considerable prospects in thermal energy storage.

1. Introduction

Thermal utilization technology contributes to energy conservation and environmental regulations [1–4]. Thermal energy storage system (TESS), in its various forms like latent heat storage, sensible heat storage and thermo chemical storage systems has been widely adopted in dealing with fluctuations in energy demand and supply gap [5]. In particular, latent heat thermal energy storage (LHTES) is considered as the best way to achieve higher thermal performance from passive and active solar heating systems. LHTES is also regarded as phase change energy storage system, which is carried out by absorbing and releasing thermal energy during phase change process. LHTES is superior to sensible heat storage in energy storage density, suitable phase change temperature and contribution to environment-friendly energy use [6,7]. In today's world, the application of the LHTES has expanded over many fields like heating–cooling of buildings [8–10], greenhouse

[11,12], textile [13–15], medicine [16], food conserving [17,18] and thermal management of electronic equipment [19] etc.

Phase change materials (PCMs) absorb and release energy during phase change process within a small range or at almost constant temperature [20]. Typically, PCMs can be categorized into two fundamental types according to their chemical composition: inorganic and organic [21]. Compared to inorganic PCMs like water and salt hydrates, organic PCMs are now in widespread use on account of their cost-effectiveness, stable chemical and thermal properties, wide range of temperatures and moderate phase change enthalpies [22,23]. Attributing to the preferable energy efficiency, PCMs are widely used in thermal management of buildings. Normally, PCMs are integrated with building masonry [24] and act as thermal energy storage materials for solar water heating [25]. Besides, PCMs are macro-encapsulated into ventilated facade for room heating [26] and free cooling of buildings [27,28]. Moreover, PCMs are applied to lightweight thermally

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activated ceiling panels [29], curtains integrated with thermally valid windows [30], hot water storage with stratification [31], tiles for building [32] and floor supply air conditioning system [33]. PCMs are also used in air conditioning systems for lessening fluctuations in the daily cooling load and applied to refrigeration systems to save energy [34].

Fatty acids along with their eutectics have extended potentials as PCMs owing to their large storage capability, non-toxicity, non-corrosiveness and low supercooling [35–38]. However, fatty acids and their eutectics still exist some disadvantages. For one thing, the thermal conductivity of these PCMs in general is low; for another, liquid PCM will leak when phase transition takes place from solid to liquid state. These deficiencies limit their applications in reality. In these circumstances, various substances have been combined into fatty acid and their eutectics, for example, expanded perlite, graphite and vermiculite [39,41], carbon micro/nanofibers [42,43] and carbon nanotubes [44]. To prevent the leakage, PCMs have been encapsulated or supported by graphite, expanded perlite and vermiculite [40,41,45–47], carbon nanotubes [48], silicon dioxide [49,50,62], polymers [51] and diatomite [52]. These PCMs are named as form-stable PCMs and privileged in terms of cost-effectiveness, no requirements for excess encapsulation and stability in shape.

The form-stable PCMs consisting fatty acids and their eutectics can be fabricated by methods like sol-gel [49,50], ultraviolet curing dispersion polymerization and self-polymerization [51,53], physical adsorption and vacuum impregnation [54] and electrospinning [55]. However, the mixed fabrication method is unable to defeat the problem of leakage during the solid-liquid phase change process. Although it is simple to fill PCM into the net structure of organics, the flammability of organics limits the CPCMs's practical application. Therefore, it is well worth producing a novel way of composition of the PCM to optimize its properties and practicability [56].

Bentonite has multi-layer structure, it is a common industrial clay comprising clay minerals of smectite group. It is mostly put into use in drilling mud, ceramic bodies and as a binder [57,58]. Bentonite has advantages of low price, outstanding absorbability and instant availability with cement and concrete. It is suitable to produce form-stable bentonite CPCMs due to its universality and chemical inertness [59]. Sari [59] carried out an experiment on bentonite-based form-stable CPCMs impregnated with capric acid (CA), polyethylene glycol (PEG600), dodecanol (DD) and heptadecane (HD). The DSC analyzed that the composites comprising 40 wt% CA, 43 wt% PEG600, 32 wt% DD and 18 wt% HD had favorable phase change temperature from 4 to 30 °C, the latent heat capacity was 38–74 J/g respectively under heating and cooling conditions. Li et al. [56] investigated the fabrication and thermal performances of paraffin/bentonite composite prepared by a solution intercalation process. Analysis manifested that the CPCMs with 44.4% paraffin melted at 41.7 °C and froze at 43.4 °C, which was close to that of paraffin, the latent heat capacity was 39.84 J/g. Besides, the heat transfer rate was improved due to the addition of bentonite. A novel mineral-based CPCMs was prepared by microwave-acid treatment of graphite (G) and bentonite (B) under vacuum conditions [60]. Latent heats for melting and freezing were 48.43 and 47.13 J/g of SA/B sample, which were lower than that of 84.64 and 84.14 J/g of SA/GBm sample. The thermal conductivity of SA/GBm composite reached to 0.77 W/(m K), which was 31% higher than that of SA/B composite by adding the graphite. Chen et al. [61] relatively studied the morphology, crystalline and phase transition properties and heat storage/release performances of myristic acid (MA)/bentonite and MA/Eudragit L 100 CPCMs. The results revealed that the maximum MA content was 50 wt% and 70 wt% respectively.

Previous studies concerned the phase change temperature and latent heat capacity of the CPCMs, the performances were compared with different support materials in certain papers. However, fatty acid eutectics were rarely involved and few papers discussed thermal energy storage and release performances. In this work, synthesis of fatty acid

eutectics and bentonite-based composites as form-stable phase change materials are reported. The melting and solidification process of the samples were recorded as well to censor the thermal energy storage and release performances. The dosage of the bentonite was limited in former studies for the purpose of avoiding the leakage of the PCM, nevertheless, in this work extra EG was added to examine the influence it brought to preventing the PCMs from leakage due to its microstructure with numerous layers, in addition, the thermal conductivity of the CPCMs were obviously enhanced. The morphology and thermal properties of the CPCMs were also analyzed.

2. Experimental method

2.1. Materials

Palmitic acid ($C_{16}H_{32}O_2$, hexadecanoic acid, Analytical reagent, Sinopharm Chemical Reagent Co., Ltd.) and stearic acid (octadecanoic acid, $C_{18}H_{36}O_2$, Analytical reagent, Sinopharm Chemical Reagent Co., Ltd.) were used as PCMs. Bentonite (Chemically Pure, Sinopharm Chemical Reagent Co., Ltd.) was used as the supporting material. Expanded graphite was purchased from the Qingdao Graphite Co. Ltd.

2.2. Preparation of the PA-SA eutectics

Palmitic acid (PA) and stearic acid (SA) were blended together to constitute eutectic mixtures. The ratio can be approximately formulated by the Schrader equation [63,64]:

$$T = \frac{1}{\left(\frac{1}{T_A} - \frac{R \ln x_A}{\Delta H_A}\right)} \quad (1)$$

where T refers to the melting temperature of the mixture, T_A , x_A and ΔH_A respectively represent the melting temperature, the molar fraction and the enthalpy of the PA. For ease of calculation, the molar fraction has been converted into mass ratio. R is the gas constant. Based on the Eq. (1), the melting temperature of the PA-SA eutectics was measured with different PA mass ratio, the melting temperature and latent heat value of the PA was 62.21 °C and 210.25 J/g, for the SA the melting temperature and latent heat was 68.61 °C and 217.38 J/g. Since T_A , ΔH_A and R are known quantities, the Eq. (1) turns into the relationship of T and x_A . The eutectic is acquired once the mixture has the lowest melting temperature, in other words, x_A is determined when the minimum T is obtained. The calculated results are shown in Fig. 1.

For the purpose of achieving accurate mass ratio of the two ingredients in eutectics, a group of PA-SA mixtures was prepared by water bath with different mass ratio at 70 °C, Table 1 lists the mixtures composition. The samples were stirred by a magnetic stirrer spinning 500 times a minute for 30 min. The thermal properties of each sample

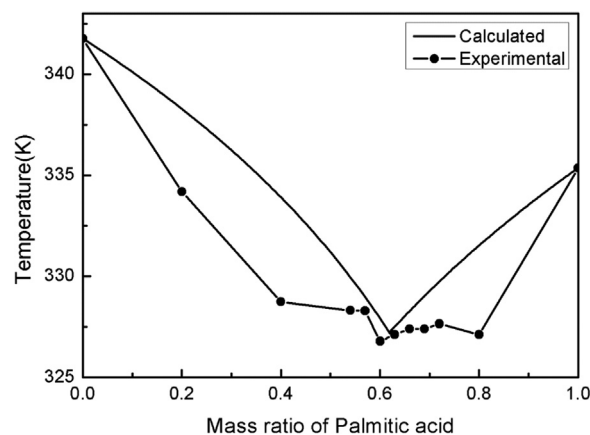


Fig. 1. Relationship of eutectic phase change temperature to mass ratio of PA-SA.

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