

Research paper

Kaolinite stabilized paraffin composite phase change materials for thermal energy storage

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ABSTRACT

Three kinds of kaolinites (platelet, PKaol; layered, LKaol; and rod, RKaol) were used to stabilize paraffin to prepare PKaol/paraffin, LKaol/paraffin, and RKaol/paraffin composites. The effects of kaolinite microstructure on the thermal storage properties of the composites were investigated in detail. It was found that the crystallinity of the paraffin in the composites increased when the proportion of kaolinite pores that are smaller than 5 nm decreased; the pore size also affected the transfer of the heat within the paraffin in the region near the kaolinite. The paraffin in LKaol/paraffin composite showed higher crystallinity (F_c , 98.4%) and greater effective energy storage per unit mass (E_{ef} , $215.6 \text{ J} \cdot \text{g}^{-1}$) than that in the two other composites, indicating that most of the paraffin can contribute to energy storage. This is probably because the LKaol pore structure is more suitable for supporting phase change materials (PCM). This also led to less phonon scattering and therefore a larger phonon mean free path for paraffin in this composite, and a higher thermal conductivity ($0.78 \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$). Furthermore, the effect of nanopore confinement within the composites was elucidated at the atomic level. The as-prepared PCM have potential for application in solar thermal energy storage and solar heating.

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1. Introduction

Solar thermal energy has aroused worldwide research interest as abundant, cheap, effective, and clean energy (Anisur et al., 2013). However, the use of solar thermal energy is limited by the inherent challenge of intermittency (Moth-Poulsen et al., 2012). That is to say, the absence of the sunlight at night or scarcity on a gloomy day is challenging for continuous solar thermal energy. Because of this, strategies are needed to store this vast amount of energy for use in the absence of sunlight (Roeb et al., 2011; Romero and Steinfeld, 2012). Thermal energy storage system using phase change materials (PCM) could be a good option to reduce the impact of two major problems: the rapid depletion of available fossil fuels and the environmental impacts of global warming. PCM can be classified as inorganic and organic. Inorganic PCM include crystalline hydrous salts, molten salts, metals, and alloys, while organic PCM are usually paraffins and fatty acids (Li and Wu, 2012). Commercial paraffin and fatty acid have many advantages, such as low price, high storage density, wide melting range, chemical

inertness, and lack of corrosiveness and supercooling (Rozanna et al., 2005; Sharma et al., 2009; Rahman et al., 2012; Sarier and Onder, 2012). Nevertheless, these organic PCM still have some disadvantages, including leakage in the molten state and lower thermal conductivity (λ , around $0.2 \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$), which restricts their wide application (Arteconi et al., 2012; Li et al., 2013a). Consequently, the incorporation of organic PCM into form-stable composite PCM is the most promising practical method to overcome these problems (Kenisarin and Kenisarina, 2012; Wang et al., 2013; Zhou et al., 2013; Zhang et al., 2014).

Clay minerals have been extensively used as supports (Zhang et al., 2010) to stabilize organic PCM and prepare form-stable composite PCM because of their natural porosity, thermal stability, and low price. Diatomite was used to stabilize xylitol penta palmitate, xylitol penta stearate, and polyethylene glycol to successfully prepare form-stable composites (Karaman et al., 2011; Sari and Biçer, 2012). They reported latent heats of $77\text{--}87 \text{ J} \cdot \text{g}^{-1}$ and thermal conductivities of $0.10\text{--}0.32 \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$ for their composites. Li et al. used palygorskite to absorb capric-palmitic acid for the first time and synthesized form-stable composites with storage capacity of $48 \text{ J} \cdot \text{g}^{-1}$ (Li et al., 2011a). Researchers prepared form-stable composites by using halloysite as an adsorbent for capric acid, paraffin, and stearic acid (Mei et al., 2011a,b; Zhang et al., 2012). Opal was exploited as a desirable carrier of organic PCM (i.e., binary paraffin blends) for thermal energy storage in

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buildings due to its special porous structure and adsorption capacity (Sun et al., 2013). Perlite can be expanded up to 10–20 times its original volume when heated rapidly at 700–1200 °C to give a highly porous material known as expanded perlite. Sari and Karaipekli reported systematic studies of perlite-based form-stable composite PCM with ΔH and λ in the range of 85–98 J·g⁻¹ and 0.05–0.09 W·m⁻¹·K⁻¹, respectively (Karaipekli and Sari, 2008; Sari and Karaipekli, 2008; Karaipekli et al., 2009; Sari et al., 2009). Similar to perlite, highly porous expanded vermiculite was obtained when vermiculite was briefly heated at high temperature (~900 °C). Karaipekli and Sari (2009, 2010) also used pristine vermiculite and expanded vermiculite to prepare form-stable composites with λ values of 0.05–0.09 W·m⁻¹·K⁻¹. The obtained composite with good thermal conductivity by using vermiculite that had been transformed into a phlogopite structure has been described in the previous work (Li and Yang, 2013). Montmorillonite and bentonite have also been employed as supports for PCM for thermal energy storage (Li et al., 2011b; Sarier et al., 2011), and the interfaces between PCM and support were investigated (Li et al., 2013a). At present, much interest has been focused on the preparation and characterization of clay mineral-based composite PCM. However, the effects of the microstructures of the clay minerals on thermal storage properties of the final form-stable composites are still poorly understood, and have not been extensively reported in the scientific literatures.

In this paper, three different forms of kaolinite (platelet, layered, and rod) were selected to stabilize paraffin, to investigate the effects of the forms of kaolinite and their pore structures on the thermal storage properties. The relationships between kaolinite microstructures and thermal storage performance were examined at an atomic level.

2. Experimental

Three kinds of kaolinites were obtained from different deposits in China: Maoming (platelet kaolinite, PKaol), Hanpu (layered kaolinite, LKaol) and Longyan (rod kaolinite, RKaol). Paraffin (brand 52[#]) was supplied by Changsha Tianquan Chemical Materials Co., LTD, Hunan, China. It had a thermal conductivity of 0.25 W·m⁻¹·K⁻¹, a melting temperature of 51.5 °C, and a latent heat of melting of 219.1 J·g⁻¹. In order to homogenize the surfaces of the different kaolinites, the three

kinds of kaolinites were pretreated with hydrochloric acid (Analytical reagent, obtained from Zhuzhou Research Institute of Chemical Industry, Hunan, China) under microwave irradiation (Korichi et al., 2009, 2012): 100 g kaolinite was added to 150 g HCl solution (8 wt.%); the mixture was then microwave irradiated at 700 W for 5 min, washed with distilled water several times to remove Cl⁻, and dried at 105 °C. The kaolinite/paraffin composites were prepared via vacuum coating (Fig. S1): 4.0 g pretreated kaolinite and 6.0 g paraffin were placed inside a conical flask, which was connected to a vacuum pump via a backflow prevention device. The conical flask, evacuated to -0.1 MPa for 10 min, was placed in a thermostatic water bath at 95 °C for 20 min. The vacuum pump was then turned off and air entered the flask again, while the flask was ultrasonically heated at 80 °C for 5 min. The reactants were cooled, and then thermally filtered at 80 °C to produce the final kaolinite/paraffin composites. The maximum loadings of paraffin for PKaol/paraffin, LKaol/paraffin and RKaol/paraffin, determined from the loss on ignition (Li et al., 2013a), were 50.9 wt.%, 44.0 wt.%, and 43.9 wt.%, respectively.

Differential scanning calorimetry (DSC) analysis of paraffin and composites was performed using a TA instruments DSC Q10 (V9.9 Build 303) (the accuracy of apparatus was ± 0.10 °C for phase change temperatures, and $\pm 1\%$ for latent heat capacities) at a heating rate of 5 °C/min under a constant stream of argon at atmospheric pressure. Liquid nitrogen was used to cool the sample during the freezing period. The testing process was performed as following. First, calibration of the measuring equipment was carried out. Second, the DSC scan was operated twice to ensure more accurate data acquisition. In the first scanning run, the sample was sealed in an aluminum crucible and heated to 79 °C, kept at this temperature for 5 min (improved the contact between the aluminum crucible and the sample), and cooled to 10 °C. In the second scanning run, the samples were heated and cooled again, and the second scan was recorded for determining the onset temperature and latent heat. X-ray diffraction (XRD) was carried out using a Rigaku D/max-rA analyzer (with monochromated Cu-K α , 1.5418480 Å) under the following conditions: voltage of 40 kV; current of 40 mA, scan range of 5–80° at a rate of 0.6°·min⁻¹, step size of 0.02°, counting time of 1 s, and 1° & 1° & 0.2 mm of slit widths. The samples were pressed into the sample holder with a smooth surface. The thermal conductivities were tested using a TPS 2500 Hot Disk Thermal Constant Analyzer.

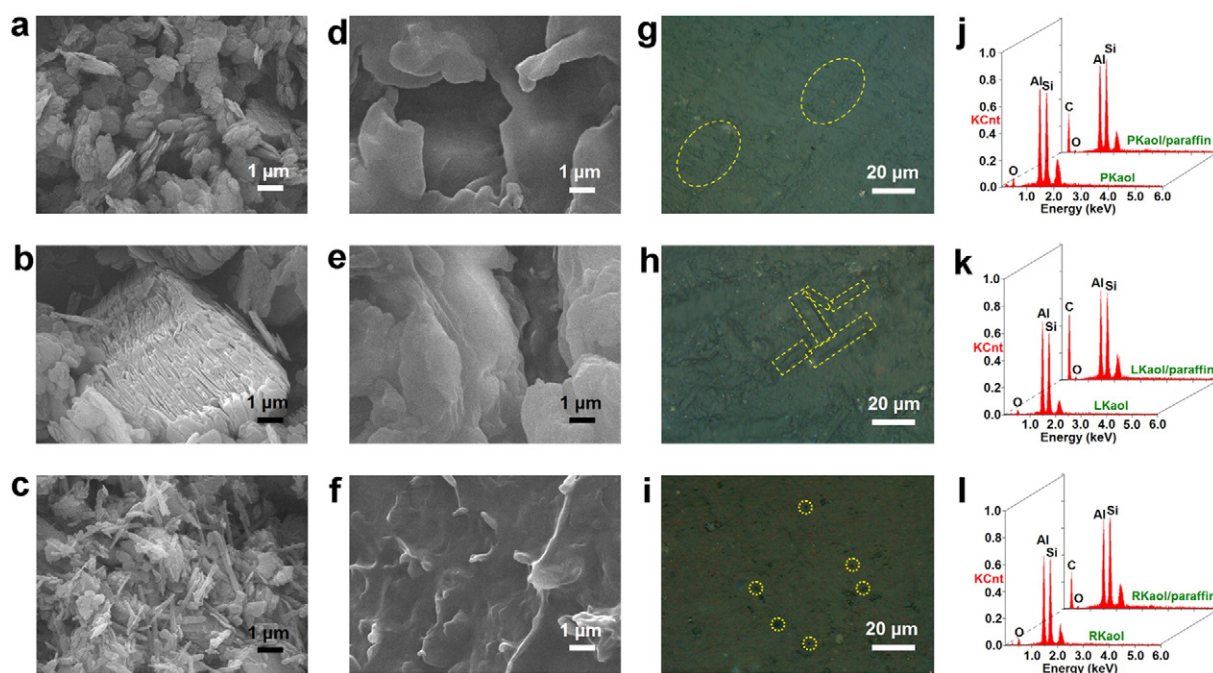


Fig. 1. SEM images of the (a) PKaol, (b) LKaol, (c) RKaol and the corresponding composites (d) PKaol/paraffin, (e) LKaol/paraffin, (f) RKaol/paraffin; reflected light microscopy images of (g) PKaol/paraffin, (h) LKaol/paraffin and (i) RKaol/paraffin; EDS patterns of (j) PKaol and PKaol/paraffin, (k) LKaol and LKaol/paraffin, and (l) RKaol and RKaol/paraffin.

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