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Tetradecanol/expanded graphite composite form-stable phase change material for thermal energy storage

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ABSTRACT

Natural flake graphite was chemically intercalated to prepare expandable graphite. The expandable graphite was then expanded by means of microwave irradiation to obtain expanded graphite (EG). Tetradecanol (TD)/EG composite form-stable phase change materials (PCMs) were prepared by mixing TD with EG through an autoclave method. The highest loading of TD in the composite form-stable PCMs with good form-stability was 93 wt%. The composite form-stable PCMs exhibited excellent thermal energy storage capacity. The melting enthalpy (ΔH_m) and crystallization enthalpy (ΔH_c) of the composite form-stable PCM containing 93 wt% TD were 202.6 and 201.2 J/g, respectively. However, the solid–solid phase transition of TD in the composite form-stable PCMs was hindered as TD was strongly absorbed by EG. As a result, the ΔH_m and ΔH_c of the composite form-stable PCMs were slightly lower than the theoretical value. The thermal conductivity of the composite form-stable PCMs was greatly enhanced by EG and was increased with the increasing of EG loading. The thermal conductivity of the composite form-stable PCM containing 7 wt% EG attained 2.76 W/m K. Besides, effects of the prepared EG on properties of the composite form-stable PCMs were compared with those of EG derived from commercially obtained expandable graphite.

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1. Introduction

Thermal energy storage technology, which can solve the problem in time and spatial mismatch between thermal energy supply and demand, is very important in solar thermal energy applications [1,2]. Thermal energy storage materials are the core of thermal energy storage technology. Phase change materials (PCMs) can absorb or release large amounts of thermal energy by changing their phase from one to another [3] and thus possess merits of high energy storage density in small temperature intervals [4]. Hence, PCMs are the most important latent heat energy storage materials in solar thermal energy applications [1,2]. Solid–solid PCMs and solid–liquid PCMs are the two common kinds of PCMs when the changing of volume is concerned. However, the solid–liquid PCMs possess the advantage of better phase change dynamics properties than those of solid–solid PCMs [1]. Solid–liquid PCMs can be divided into organic and inorganic PCMs. Organic solid–liquid PCMs possess advantages of high thermal energy storage density, low or no undercooling, chemical and thermal

stability and no corrosives [1]. However, the main drawback that hinders the application of organic solid–liquid PCMs is their unacceptably low thermal conductivity [1]. Lower thermal conductivity would result in slower thermal energy storing or releasing speed and lower thermal energy utilization efficiency. The thermal conductivity of PCMs is important in the field of solar thermal energy applications, since solar radiation is varied seasonally and daily. Hence, high thermal conductivity is appreciated when PCMs are applied to store thermal energy that comes from solar radiation during sun shining period. As a result, the thermal conductivity of organic solid–liquid PCMs has to be enhanced [5].

The most popular method to enhance the thermal conductivity of solid–liquid PCMs is adding high conductivity fillers such as powder [6], metal foam [7], carbon fibers [8], carbon nanofibers [9], carbon nanotubes [10], exfoliated graphite nanoplatelets [11], expanded graphite (EG) [12], etc. Among them, EG exhibits various effects on the improvement of the thermal conductivity of PCMs. It has been reported that the apparent thermal conductivity of nitrates/10 wt% EG mixture PCM was increased by about 30–40% [13]. The thermal conductivity of SiO₂/paraffin/EG composite was 94.7% higher than that of paraffin [14] and the thermal conductivity of palmitic acid was improved to 0.60 W/m K by 20 wt% of EG [15]. On the other hand, the thermal conductivity

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of stearic acid (SA) (0.30 W/m K) could be increased by 266.6% by adding 10 wt% of EG [16]. The thermal diffusivity of SA/EG composites containing 20 wt% EG was 10 times higher than that of pure SA [12]. The thermal conductivity of $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ containing 50 wt% EG (8.796 W/m K) was 14 times higher than that of pure $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ (0.596 W/m K) [17]. Furthermore, the thermal conductivity of graphite matrix/paraffin composite PCM was 20–130 times greater than that of pure paraffin (~ 0.2 W/m K) [18]. Based on the reported results, we believe that the thermal conductivity enhancement of EG-doped composite PCMs is affected by properties of EG, the preparation procedure of the composite PCMs and the interactions between pure PCMs and EG. As a result, the investigation of the thermal conductivity enhancement of EG on solid–liquid PCMs is a hot topic. Furthermore, to the best of our knowledge, the thermal conductivity of fatty alcohol has not been enhanced by EG. Fatty alcohol is a kind of important organic solid–liquid PCM with high thermal energy storage density. The polarity of fatty alcohol is weaker than that of fatty acid. Furthermore, fatty alcohols exhibit a solid–solid phase transition which is close to the solid–liquid phase transition [19]. The solid–solid phase transition is also beneficial to thermal energy storage but its transition properties may be affected by fillers with high specific area. Hence, it is worthwhile to investigate the phase change properties and the thermal conductivity of fatty alcohol/EG composite PCMs. In this paper, EG was prepared from natural flake graphite and effects of the preparation procedure on the expansion ratio were investigated. Tetradecanol (TD) was selected as solid–liquid PCM and TD/EG composite form-stable PCMs were prepared via an autoclave method. The structure and the thermal properties of the prepared composite form-stable PCMs were investigated and results are reported here. Besides, effects of the prepared EG on the thermal energy storage properties and thermal conductivity of form-stable PCMs were compared with those of EG derived from commercially obtained expandable graphite.

2. Experimental

2.1. Materials

Natural flake graphite with average size of 300 μm was obtained from Qingdao Runba Graphite Co., Ltd. China. Expandable graphite with average particle size of 300 μm and expansion ratio of 200 mL/g was supplied by Qingdao Graphite Co., Ltd., China. All other reagents were of analytical grade and were used without further purification. Deionized water was used throughout the experimental process.

2.2. Preparation of EG

The EG was prepared according to reference [20] with some modification. In general, a certain amount of natural flake graphite was added to a solution of glacial acetic acid and concentrated nitric acid (65%) under stirring in a 35 °C water bath to form a well-distributed mixture. A certain amount of potassium permanganate was added to the mixture while the stirring was continued. The mixture was stirred at this temperature for a certain time. During this time, nitric acid/acetic acid/graphite intercalation compound (NA-GIC) was formed. Then the NA-GIC was filtered, washed thoroughly with water, and dried at 50 °C under vacuum to obtain dry expandable graphite. The obtained expandable graphite was then radiated for 60 s in a domestic microwave oven (WP750, GALANZ) to obtain EG. The expanded volume was applied as an evaluation standard to modify the ratio of raw materials and reaction time. The expansion ratio of EG was measured as follows: 0.20 g expandable graphite in a 50 mL beaker was irradiated for 60 s, and then the volume of the obtained EG was precisely read. The procedure was

repeated three times for each sample and the relative deviation was less than 6%. The obtained mean volume value divided by 0.20 g resulted in the expansion ratio of EG as mL/g.

2.3. Preparation of TD/EG composite form-stable PCMs

A certain amount of the EG, TD and anhydrous ethanol were mixed in an autoclave. The autoclave was sealed and heated at 120 °C for 24 h and then cooled to ambient temperature. The mixture was then taken out from the autoclave and heated at 50 °C under reduced pressure to evaporate the ethanol and was then dried under vacuum for 24 h to obtain TD/EG composite form-stable PCMs. The samples were designated as TD/EG-1–5, corresponding to TD/EG composite form-stable PCMs containing 93 wt%, 90 wt%, 85 wt%, 80 wt% and 60 wt% of TD, respectively.

In order to compare the effects of the prepared EG on the thermal energy storage properties and thermal conductivity of the form-stable PCMs with EG obtained from a known method (namely, k-EG), another series of TD/k-EG composite form-stable PCMs were prepared as follows. Expandable graphite was dried at 80 °C for 24 h and was then radiated for 60 s in the microwave oven to obtain k-EG. TD/k-EG composite form-stable PCMs were then obtained according to the preparation procedure of TD/EG composite form-stable PCMs. The samples were designated as TD/k-EG-1–4, corresponding to the TD/k-EG composite form-stable PCMs containing 90 wt%, 80 wt%, 70 wt% and 60 wt% of TD, respectively.

2.4. Characterization

FT-IR spectra were recorded on a FT-IR spectrometer (AVATAR-360) using KBr pellet (4000–400 cm^{-1}). The surface morphology investigation was carried out on a scanning electron microscope (SEM, JEOL JSM-6380). Before the SEM investigation, samples were sputtered with gold. Powder X-ray diffraction (XRD) experiments were performed on a Rigaku D/max-gb X-ray diffractometer with a monochromatic detector. Copper $\text{K}\alpha$ radiation was used, with a power setting of 30 kV and 30 mA, and a scanning rate of 5°/min.

The thermal stability of samples was characterized by means of thermogravimetry (TG)/differential thermal analysis (DTA) on a thermogravimetric analyzer (NETZSCH STA 409 PG/PC) from room temperature to 800 °C at the heating rate of 10 °C/min and with N_2 as carrier gas. The analyzer was calibrated using $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$ (99.9%) prior to the experiment. Differential scanning calorimetry (DSC, TA, Q2000) was used to investigate the thermal energy storage properties of the prepared composite form-stable PCMs over the temperature of 10–80 °C with the heating and cooling rate of 10 °C/min in nitrogen atmosphere. Prior to the DSC experiments, the instrument was calibrated using indium (99.999%) as the standard material. The thermal conductivity of samples at room temperature was measured by means of steady-state heat flow method using a thermal conductivity tester (DRX-II-RW, Xiangtan Huafeng Instrument Manufacturing Co., Ltd, China). Before the measurement, samples were grounded and pressed under 10 MPa to obtain disks ($\Phi = 30$ mm). The hot plate of the tester was set at 35 °C and the cold plate was cooled by water of 15 °C. A disk was mounted between the two plates. The thermal conductivity of the disk was measured when the temperature of the two plates reached stable values for more than 1 h.

3. Results and discussions

3.1. Preparation of EG

The preparation of EG was conducted according to reported procedure [20] with some modifications. The modifications were

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