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Polyethylene glycol based shape-stabilized phase change material for thermal energy storage with ultra-low content of graphene oxide



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ABSTRACT

Graphene oxide (GO) sheets are introduced to stabilize the shape of the most widely studied phase change material for thermal storage, polyethylene glycol (PEG) during the solid–liquid phase change process. In this composite, a maximum weight percentage of PEG as high as 96% without any leakage up to a temperature as high as 150 °C, far above the melting temperature of PEG, has been achieved. Such a weight percentage of PEG is the highest value among PEG based shape-stabilized PCMs to the best of author's knowledge. The shape-stable PCM presents a high heat storage capacity of 142.8 J g⁻¹ and excellent thermal reliability within at least 200 melting/freezing cycles. The thermal properties of the PEG/GO composite PCMs with various GO contents are also investigated. The present study provides a highly potential shape-stabilized composite PCM with a high content of PEG for thermal energy storage as well as a way to enhance the heat storage density of PEG based shape-stabilized PCM.

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1. Introduction

The continuous increase in greenhouse gas emissions and the depletion of the fossil fuels are the main driving forces behind efforts to more effectively utilize various sources of renewable energy. Energy storage systems not only improve the performance and reliability of energy systems, but also provide the potential to attain energy savings and reduce the mismatch between supply and demand of energy, which in turn reduce the impact on environment. Latent heat thermal energy storage (LHTES) realized using phase change materials (PCMs) is one of the most effective techniques of thermal energy storage because of its advantages such as high storage density, isothermal characteristics and small temperature difference between heat storing and releasing [1–8]. As advanced energy saving materials, PCMs, which store and release latent heat as they undergo a phase change, can be used as controlled thermal storage candidates for thermal management of computers, electrical engines, solar power plants, and for thermal protection of electronic devices [1,9,10].

Polyethylene glycol (PEG) is a promising solid–liquid organic PCM owing to its desirable characteristics, including high phase change enthalpy, chemical and thermal stabilities, biodegradation, non-toxicity, non-corrosiveness, low vapor pressure and suitable melting temperature which can be tuned by its molecular weight

[11–14]. However, the leakage of liquid phase above the melting temperature (T_m) is the main drawback of PEG, which must be encapsulated in specially sealed containers. Microencapsulation of PCMs using polymer shells may be a solution, but it causes some problems such as improved cost to encapsulate the PCM, improved heat resistance caused by a capsule shell and the leakage of the melted PCM when a capsule shell was damaged [12–15].

Shape-stabilized PCMs composed of a working substance and supporting materials were developed in the late 1990s, which could keep shape of materials and prevent leakage throughout the phase change process [16]. Several reports have focused on the preparation and thermal energy storage of the shape-stabilized PCMs based on the PEG [2,13,17,18]. Karaman et al. [13] prepared a novel form-stable composite PCM by incorporating PEG into the pores of diatomite. The PEG could be retained by 50 wt% in the pores of the diatomite without leakage of melted PEG from the composite and the phase change temperature and latent heat of the composite PCM were measured as 27.70 °C and 87.09 J g⁻¹, respectively. Wang et al. [17] prepared shape-stabilized PCMs by a blending and impregnation method with PEG as thermal storage materials and graphene oxide (GO) as a supporting substance. The highest stabilized PEG content was 90 wt% in the composites, resulting in a heat storage capacity as high as 156.9 J g⁻¹. By introducing expanded graphite into PEG, Wang et al. [18] prepared shape-stabilized PCMs and the maximum weight percentage of PEG was as high as 90% without any leakage during the melting period, the latent heat was 161.2 J g⁻¹ and T_m was 61.46 °C. It is worth noting that although these PCMs can avoid leaking, the

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maximum weight percentage of PEG was not more than 90%. For application, high latent heat of PCMs is required to provide a higher thermal storage per unit weight, which is essentially determined by the weight percentage of PEG in the PCMs. It is expected that the phase change enthalpy would be lower than that of pure PEG because some of the PEG would be replaced by the supporting materials that do not undergo phase change in the operating temperature range. Therefore, it is of great significance to maximize the content of PEG without any seepage above T_m of PEG. Meanwhile, the cost from the supporting material can also be lowered by reducing its content.

As a rising star in the carbon family, graphene has attracted a great deal of attention in recent years because of its remarkable properties including high thermal conductivity, superior mechanical properties and excellent electronic properties [19–21]. GO derived from graphene is a layer-structured material with oxygen-containing functional groups on the basal planes and edges, such as hydroxyl, carbonyl, epoxide and carboxyl [22,23]. The presence of these functional groups implies that hydrogen bonding may exist between GO and other organic materials such as PEG when they are synthesized into composites [17]. The interaction caused by the hydrogen bonding, combined with the high porosity and surface area of GO, should be advantageous for shape-stabilization during the solid–liquid transition of PCMs because it helps to grasp the liquid by surface tension and capillary forces [24].

In the current work, we prepared PEG based shape-stabilized PCMs with GO as a supporting material by a simple blending and impregnation process. The phase change behavior and performance of the composite materials were investigated. The highest PEG content was 96 wt% without any leakage up to a temperature as high as 150 °C, far above the melting temperature of PEG. Such a weight percentage of PEG is the highest value among the shape-stabilized PCMs as far as we know. A small reduction in the phase change enthalpy coupled with the superior thermal energy storage stability was achieved. The demonstrated performance of the shape-stabilized PCM by the addition of only a very small amount of GO is promising for LHTES applications.

2. Experimental

2.1. Materials

Natural flake graphite (NG) with an average particle size of 200 mesh and a purity of over 99.9% was purchased from Shenghua Research Institute (Changsha, China). PEG (Mn=6000) was purchased from Aladdin Reagent (Shanghai, China). Sodium nitrate (NaNO_3), potassium permanganate (KMnO_4), concentrated sulfuric acid (H_2SO_4), hydrochloric acid (HCl), and hydrogen peroxide (H_2O_2) were purchased from Chengdu Kelong Chemical Reagent Factory (Chengdu, China). The materials were used as received without further purification.

2.2. Synthesis of GO

GO was synthesized from NG powder by modified Hummer's method [25]. In this method, concentrated H_2SO_4 (70 mL) was added to a mixture of NG (3.0 g) and NaNO_3 (1.5 g), and the mixture was cooled using an ice bath to 0 °C. KMnO_4 (9.0 g) was added slowly in portions with stirring to keep the reaction temperature below 20 °C. The reaction was then kept at 35 °C and stirred for 7 h. Additional KMnO_4 (9.0 g) was then added in one portion, and the reaction was stirred for 12 h at 35 °C. Next the reaction mixture was cooled to room temperature and poured onto ice (400 mL) with 30% H_2O_2 (3 mL). The color of the mixture became bright yellow. The GO was repeatedly rinsed and

redispersed in a 5% solution of HCl three times. It was then washed continually with deionized water to completely remove the metal ions and acids until the pH was neutral. At last, the brown fluffy GO product was obtained by freeze-drying the GO solutions.

2.3. Fabrication of composite PCMs

PEG/GO nanocomposites were prepared by a physical blending and impregnation method. PEG was melted at 80 °C in a three-neck flask and GO powders were dispersed in deionized water with 1 h ultrasonication (KQ-400KDB, 40 kHz, 400 W) to form a homogeneous suspension. Then the GO aqueous suspension was dripped into melted liquid PEG during stirring followed by vigorous stirring for 5 h. Finally, the dispersion was dried to constant weight at 80 °C. Pure PEG was prepared with the same procedure. The GO content in the PEG/GO PCMs varied from 0.5 to 8 wt% (0.5, 1, 2, 4, 6 and 8 wt%) to seek for the maximum ratio of PEG without any leakage during the melting period. The obtained pure PEG and nanocomposites samples were labeled as PEG, PG0.5, PG1, PG2, PG4, PG6, and PG8, correspondingly.

2.4. Characterization

A JOEL JSM-5900LV field-emission SEM instrument and a Tecnai G2 F20S-TWIN transmission electron microscopy (TEM) instrument were used to visually characterize the morphology of the bulk fillers and nanocomposites with accelerating voltages of 20 kV and 200 kV, respectively. AFM images were taken using the Nanoscope Multimode and Explore atomic force microscopy (Veeco Instruments, USA), in the tapping mode of operation using rectangular cantilevers with spring constant of $\sim 40 \text{ N m}^{-1}$ and typical resonance frequencies between 250 and 300 kHz. Wide-angle X-ray diffraction (XRD) pattern of the samples was obtained using a DX-1000 diffractometer with $\text{Cu K}\alpha$ radiation ($\lambda=0.154 \text{ nm}$) under a voltage of 40 kV and a current of 40 mA. Samples were scanned in the range of diffraction angle $2\theta=2\text{--}45^\circ$, with a scan speed of $3^\circ/\text{min}$ at room temperature. Fourier-transform infrared (FTIR) spectroscopy was performed on a Nicolet 6700 FTIR spectrometer (Nicolet Instrument Company, USA) with the resolution of 4 cm^{-1} in the transmission mode. Dynamic rheological measurements were performed on a stress-controlled rheometer (AR 2000, TA Instruments) equipped with parallel-plate geometry (diameter of 25 mm). The frequency sweep was conducted in a frequency range of 0.01–100 Hz with a strain of 0.1%. Disk-shaped samples with the diameter of 25 mm and the thickness of 1.5 mm were used. The test temperature was 80 °C and the gap between two plates was 1.2 mm. The dynamic rheometer was also utilized to perform the shape-stabilization test. Temperature ramp was recorded at a rate of 2° min^{-1} from 30 °C to 150 °C and a constant normal force was set to 5 N. To maintain the normal force at the set value, the gap value will adjust sensitively and automatically during the temperature rising process. The change in gap value represents the dimension change of samples which can reflect the shape-stability. If the sample has a poor shape-stability and is easy to leak above the melting point of PEG, the gap value will decrease with increasing temperature; while if it is shape-stabilized, the gap value will basically remain invariable. For differential scanning calorimeter (DSC) measurements, the samples were sealed in an aluminum pan for characterization by the TA Q20 instrument with a heating/cooling rate of $10^\circ \text{ C min}^{-1}$.

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