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Structure and thermal properties of octadecane/expanded graphite composites as shape-stabilized phase change materials



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ABSTRACT

A series of shape-stabilized phase change materials (PCMs), composed of octadecane (OD) and expanded graphite (WEPG) (OD/WEPG), were prepared by using a kneader mixing technique. Chemical and morphological structure, shape-stability, thermal properties, and thermal stability of the OD/WEPG composites were investigated and characterized by Fourier transform infrared spectroscopy (FTIR), Bru nauer–Emmet–Teller (BET) analysis, scanning electron microscopy (SEM), differential scanning calorimetry (DSC), and thermogravimetric analysis (TGA). Composite properties were strongly dependent on the weight ratio of OD to WEPG. SEM and BET analysis showed that OD was adsorbed into the WEPG pores and uniformly covered the surfaces of WEPG, which helped to prevent leakage of melted OD during phase change from solid to liquid. Among the OD/WEPG composites, the OD/WEPG composite with 30% WEPG content showed good thermal properties and shape-stability. Based on these results, we anticipate that the OD/WEPG composites can be used in packaging materials to counteract the negative effects induced by unwanted temperature changes.

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1. Introduction

Thermal energy storage using phase change materials (PCMs) has attracted considerable interest [1-4]. PCMs are temperatureresponsive substances that absorb and release large amount of latent heat energy during temperature-driven phase changes [2,3]. The use of PCMs allows maintenance of a constant temperature in response to unwanted outer temperature changes, and PCMs have a higher thermal storage density than sensible heat storage materials [2,5,6]. Therefore, PCMs have been applied in various hot and cold energy storage systems such as building materials and packaging materials [1–6]. In the packaging area specifically, temperature is one of the most important factors that affects the quality of perishable and frozen foods, beverages, and pharmaceutical products during storage and distribution [5,6]. To maintain the quality of the product and minimize product and quality loss due to unwanted temperature changes, various PCMs have been used in containers to transport blood and organs [1], isothermal water bottles, and ice cream panels and containers [1,5].

PCMs used as thermal energy storage substances in packaging materials can be categorized as inorganic salt hydrates, organic paraffin waxes, and eutectic mixtures [1,3,5]. Among organic PCMs, octadecane (OD) has a large latent heat storage capacity, low melting/freezing phase change temperature, is non-toxic, and is readily available [7,8]. However, direct utilization of OD for heat storage is limited by its low thermal conductivity and leakage during the solid-liquid phase change process [7,8]. To solve these problems, researchers have investigated shape-stabilized PCM composites that have a melting temperature in the desired operating temperature range, a high latent heat of fusion per unit mass, and improved chemical and physical stability and thermal conductivity due to their ability to change phase from solid to liquid [1–9]. There are three general methods used to generate shape-stabilized PCM composites: (1) encapsulation of OD in shell materials [7]; (2) creation of composites with polymer [10]; (3) impregnation of OD into inorganic materials with a porous and layered structure, such as expanded graphite (WEPG) [8,11,12]. The first and second methods have some disadvantages such as low thermal conductivity, incongruent melting and freezing, latent heat capacities, complex manufacturing processes, and high cost [7,10,11]. In contrast, the third method is a simple, low-cost process. Furthermore, the PCM composites obtained using this method are shape stable over repeated phase changes, which prevents PCM leakage from the surface of PCM composites and maintains thermal conductivity and latent

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heat capacity [8,13–16]. The several studies have been performed to use the superior performances of OD, WEPG and OD/WEPG composites. Zhang et al. and Huang et al. investigated OD/WEPG composites prepared by vacuum impregnation of OD in WEPG [8,17]. Fang et al. did OD/WEPG composites prepared by absorbing OD in WEPG using magnetic stirring [18] However, because the method they used involved only absorption of fixed PCM into materials with a porous and layered structure to apply building materials, there was no homogeneous mixing, which made it difficult to control the thermal properties of the resultant materials. Furthermore, no systematic investigations relating the effects of WEPG loading on the thermal properties and shape-stability of the OD/WEPG composites have been conducted.

However, we applied a melt mixing method using a kneader with a two-shaft rotating blade to obtain OD-impregnated WEPG. After adding OD to WEPG, the kneader was used to obtain homogeneous mixing as well as impregnation of OD into the pores and layered surfaces of WEPG. This method is simple and practical, time effective, and low cost, and therefore has the potential to be used in mass production of shape-stabilized PCM composites. The chemical and morphological structure, thermal properties, thermal stability and shape stability of the OD/WEPG composites were thoroughly investigated as a function of WEPG content.

2. Experimental

2.1. Materials

Expandable graphite powders (EPG, ES 350 F5, carbon content >99%) were purchased from Samjung C&G Co., Ltd. (Daegu, Korea). OD with a density of 0.78 g/cm³, melting point of 26–30 °C and boiling point of 317 °C was purchased from Sigma Aldrich Co., Ltd. (Yongin, Korea). All materials in this study were used without further purification.

2.2. Preparation of WEPG

The process used to prepare WEPG has been described in our previous studies [8,17]. In this study, WEPG was prepared using EPG as the main raw material by microwave treatment (700 W). First, 0.5 g EPG powder was put into a quartz crucible (100 ml). The quartz crucible was then pushed into the center of a microwave (700 W) and reacted for 150 s. The quartz crucible was then taken out of the microwave into the air for rapid cooling. Finally, WEPG powders were obtained.

2.3. Preparation of the OD/WEPG composites

The OD/WEPG composites were prepared with composition ratios (%) of 100:0, 100:10, 100:15, 100:20, 100:30, 100:50, and 100:70 using a kneader mixing technique (TO-350, TEST ONE. Co., Ltd., Gwangmyeong, Korea). Before mixing, WEPG powders were dried in a vacuum oven to evacuate air and water at 105 °C for 24 h [3,18]. Liquid OD was obtained by heating it to its melting temperature, after which it was added to WEPG. For thorough mixing and penetration of liquid OD into the porous and layered structure of WEPG, the OD/WEPG mixtures were mixed at a processing temperature of 80 °C and screw speed of 30 rpm for 1 h and cooled at room temperature. Finally, powder-type OD/WEPG composites were obtained.

2.4. Characterization

Adsorption-desorption properties were used to analyze the Bru nauer-Emmet-Teller (BET) specific surface area and total pore volume of WEPG and OD/WEPG composites. Nitrogen adsorption was recorded using a ASAP 2010 (Micromeritics Co. Ltd., Norcross, GA, USA).

SEM images of WEPG and the impregnation and combination states of WEPG particles in the OD matrix were obtained by a Quanta FEG250 scanning electron microscope (FEI Co. Ltd., Hillsboro, OR, USA). Prior to examination, all samples were coated with a thin layer of platinum (Pt)/palladium (Pd).

To characterize the functional groups of pure OD and OD/WEPG composites, Fourier transform infrared spectra (FTIR) were recorded between 4000 and 400 cm⁻¹ at a resolution of 32 cm⁻¹ using a Spectrum 65 FTIR spectrometer (PerkinElmer Co. Ltd., Wal-tham, MA, USA) in attenuated total reflection (ATR) mode.

To investigate the shape-stability of the OD/WEPG composites, the OD/WEPG composites were placed in an oven at 80 °C for 1 h. Leakage was checked and then enthalpy changes for melting and crystallization were investigated with a DSC method [2].

To investigate the effect of WEPG loading on the heat transfer of OD and OD/WEPG composites, melt analysis were conducted using the water bath method, as shown in Fig. 1 [12,19–21]. Pure OD and OD/WEPG composites were placed in 30 ml glass test tubes. Samples were placed in a water bath at 10 °C and the temperature was increased to 50 °C. Temperature changes of pure OD and OD/WEPG composites were monitored automatically as a function of time using a Testo 176T4 data logger with an accuracy of 0.3 °C (Testo Co. Ltd., Seoul, Korea).

The temperatures and enthalpies during melting and crystallization of pure OD and OD/WEPG composites were analyzed using a Q10 differential scanning calorimeter (TA Instrument Co. Ltd., New Castle, DE, USA) under a nitrogen atmosphere. Samples were heated from 0 °C to 40 °C at a heating rate of 3 °C/min. Thermogravimetric analysis of OD, WEPG, and OD/WEPG composites was carried out at a heating rate of 10 °C/min from 30 °C to 600 °C under a nitrogen atmosphere using a TGA 4000 thermogravimetric analyzer (PerkinElmer Co. Ltd., Waltham, MA, USA).

3. Results and discussion

3.1. Preparation of the OD/WEPG composites

To investigate how OD and WEPG combined physically, BET and SEM analyses were conducted. To be specific, these analyses can be used to determine whether organic materials are effectively intercalated into materials or not [2,14,17,20]. BET surface area and pore volume, which are strongly dependent on the shape and size of pores [22], can be obtained by investigating how much porous material interacts with N₂ gas. As shown in Fig. 2, the hysteresis loops of the adsorption and desorption in WEPG and OD/WEPG composites were indicated by black dot and white dot, respectively. As shown in Fig. 2(a), less N₂ gas adsorbed in WEPG and OD/WEPG composites at low relative pressure and sudden increase of adsorption occurs at high relative pressure. A bigger pore volume with larger pore diameter can be found from the desorption curves than those from the adsorption curves. This hysteresis is well consistent with the type V based on IUPAC definitions [14,17,21]. Therefore, we surmised that WEPG and OD/WEPG composite have ink-bottle pore structure with open pores with narrow pore-mouth (open side) and large pore body (closed side). In contrast to the sharp increase in the amount of gas adsorbed by WEPG, the amount of gas adsorbed decreased in the OD/WEPG composites with 30-70% WEPG loading. Furthermore, the BET surface area of the OD/WEPG composites decreased significantly from 37.6 to $2.0 \text{ m}^2/\text{g}$ compared with that of WEPG. The decrease in pore volume and BET surface area of the OD/WEPG composites was likely due to adsorption of OD and filling of the interconnected open

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