



Synthesis, characterization and properties of palmitic acid/high density polyethylene/graphene nanoplatelets composites as form-stable phase change materials



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ABSTRACT

In this work, form-stable phase change materials (FSPCM) consisting of palmitic acid (PA) and high density polyethylene (HDPE) were modified by graphene nanoplatelets (GNP). In the FSPCM, PA was used as a solid–liquid phase change material (PCM) for thermal energy storage, HDPE was a supporting material to prevent the leakage of the melted PA, and GNP were added for improving thermal conductivity and shape stabilized of the FSPCM. Thermal properties and shape stability of the composites vary with their different mixture ratios. According to the results of Fourier transformation infrared spectroscopy (FT-IR) and X-ray diffractometer (XRD), the composites have advantages like stabilized chemical structure and crystalline phase. The differential scanning calorimeter (DSC) results show that the FSPCM has a constant melting temperature of around 62 °C and a high latent heat of at least 155.8 J/g. With the help of scanning electronic microscope (SEM), a layer structure and uniform dispersion of the PA is observed in the modified FSPCM. The thermos-gravimetric analyzer (TGA) and thermal cycling test results indicate that the modified FSPCM has a good thermal reliability, and the leakage of the PA drops significantly with the assistance of the GNP. The thermal conductivity of the FSPCM was measured by thermal conductivity meter and it increased to $0.8219 \text{ W (m K)}^{-1}$ which is nearly 2.5 times as high as that of the pure FSPCM, when the mass fraction of the GNP is 4%. It is anticipated that the modified FSPCM possess a potential application in solar energy and building heating systems.

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1. Introduction

Improving energy efficiency and developing renewable energy are two crucial solutions to mitigate the green-house effect and solve the energy crisis. In recent years, thermal energy storage system (TESS) in forms of latent heat, sensible heat and reversible thermochemical reactions has been put into various applications for realizing energy redistribution and energy efficiency on short-term or long-term basis [1]. The ordinary applications include building-energy management [2,3] and solar-thermal conversion [4,5], in which storing and releasing energy is time dependent. Among the different forms of TESS, latent heat storage method has a big role to play in optimizing energy efficiency and energy management because the materials used in latent heat storage system, especially phase change materials (PCM), have the advantages of high enthalpy change, constant operating temperature, stable chemical structure, low cost, etc. [6].

Solid–liquid PCM are the most common materials for thermal energy storage. They store thermal energy during melting process and release thermal energy during solidifying process. The solid–liquid PCM are usually divided into three types: organic PCM (OPCM), inorganic PCM and their eutectics [7]. In addition to the normal benefits of the PCM, OPCMs, such as fatty acid, n-alkanes and esters etc, are economically available, non-toxic, and have characteristics like low degree of subcooling and appropriate phase change temperature [8]. However, pure OPCM has major shortcomings like liquid leakage in melting state, low thermal conductivity and high volume variation which limit their application in TESS [9].

Previously, encapsulating the OPCM into special storage devices was an alternative approach to preventing the leakage of the OPCM, but this way of resolution increases cost and the thermal resistance between storage devices and the OPCM [10]. Nowadays, this defect can be overcome by absorbing OPCM into supporting materials to form shape-stabilized PCM which are called form-stable phase change materials (FSPCM) [11]. Supporting materials involve polymers, such as high density polyethylene (HDPE) [12–

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16], low density polyethylene (LDPE) [17], polypyrrole [18,19], poly (methyl methacrylate) [20–22], polyurethane [23], eudragit [24], cross-linked poly (acrylonitrile-co-itaconate) [25], poly (vinyl chloride) [26], styrene maleic anhydride copolymer [27], and inorganic frameworks, such as TiO₂ foam [28], gypsum [29,30], expanded vermiculite and perlite [31,32]. Among them, the HDPE is an arresting matrix for synthesis of the FSPCM owing to the merits of high insulation resistance, acid and alkali resistance, organic solvent resistance, strong intermolecular forces, etc. Mu et al. [33] prepared shape stabilized phase change materials based on HDPE/paraffin waxes using twin-screw extrusion and the results indicate that the waxes are uniformly dispersed and distributed in the HDPE and have a strong plasticizing effect on the HDPE. Chen and Wolcott [34] developed the stabilized paraffin waxes with three different polyethylene (HDPE, LDPE and linear low density polyethylene (LLDPE)) and the result shows the paraffin leakage of paraffin/HDPE performs best among the three polyethylene and the blends develop a co-continuous structure which is responsible for the control of leakage behavior.

Low thermal conductivity is still the limit for the FSPCM based on the polymer matrixes, since high thermal conductivity is able to accelerate the speed of thermal energy storage and release, which can improve the energy efficiency. Many outstanding works have been done to improve thermal conductivity of the FSPCM [35,36]. A natural method against this defect is to add materials with high thermal conductivity into the FSPCM. Nanometer materials with extremely high thermal conductivity and high specific surface area are the best suited additives for thermal conductivity enhancement of the FSPCM. Deng et al. [37] embedded silver nanowire into polyethylene glycol/expanded vermiculite FSPCM, and the result showed that the thermal conductivity increased to 0.68 W (m K)⁻¹ with 19.3 wt% of silver nanowire and a theoretical model was introduced for describing the relationship between the thermal conductivity of the doped FSPCM and the loading of silver nanowire. However, few nano-additives are reported to be added into polymer-based FSPCM for thermal conductivity enhancement. Zeng et al. [38] found that the thermal conductivity of PA/polyaniline PCM doped with 7.87 wt% of exfoliated graphite nanoplatelets could reach 1.08 W (m K)⁻¹ which was 2.375 times higher than that of pure PA/polyaniline PCM. Silakhori et al. [19] prepared FSPCM consisting of PA, polypyrrole and graphene nanoplatelets (GNP) and discovered that the thermal conductivity and latent heat of modified FSPCM reached up to 0.43 W (m K)⁻¹ and 151 J/g by addition of 1.6 wt% GNP. Therefore, the carbon nanoplatelets are good nano-additives for thermal conductivity enhancement of polymer-based FSPCM. In addition, since the structure of the GNP is similar with that of expanded graphite (EP) and a few studies have confirmed that the GNP can prevent the leakage of the PCM, the GNP may be provided with the same characteristic [36]. In previous literatures, the effects of the carbon nanoplatelets on the thermal conductivity enhancement and leakage prevention are hardly investigated together, and this work will focus on the two effects.

In this paper, the PA and HDPE were selected as PCM and supporting materials, respectively. The GNP as nano-additives were added into PA/HDPE form-stable phase change materials for thermal conductivity enhancement. In order to prevent the leakage of the melted PCM, the loading of the HDPE in FSPCM should reach a certain level, which will limit the thermal enthalpy of the FSPCM as PCM is the only material to provide latent heat. If the GNP can significantly ameliorate the leakage behavior of the HDPE-based FSPCM, the loading of the PCM will increase thus the thermal enthalpy of the FSPCM will also increase. Therefore, the main purpose of this work is to investigate the influences of the GNP on the thermal conductivity and the leakage behavior of the PA/HDPE FSPCM. The FSPCM with different ratios of the GNP and

HDPE will be evaluated in terms of thermal properties, thermal conductivity, chemical stability, microstructure and thermal reliability. According to the results, the FSPCM modified by the GNP will have capacities of high thermal conductivity and thermal enthalpy, which ensure promising applications in solar energy and building heating systems.

2. Experimental

2.1. Materials

PA (C₁₆H₃₂O₂, hexadecanoic acid, Chemically Pure), which was used for thermal energy storage, was purchased from Jiangsu Huakang Chemical Reagent Company. HDPE (melt flow index: 20 g/10 min, density: 0.953 g/cm³, Vicat softening temperature: 126 °C) was obtained from Dongguan Huangjiang Co., Ltd. GNP (Thickness: 3–20 nm, Flake diameter: 5–10 μm, Specific surface area: > 30 m²/g, purity: 99.5%) were provided by Nanjing XFNA-NO Materials Co., Ltd.

2.2. Preparation of the PA/HDPE/GNP composite

In this work, in order to improve the thermal conductivity and leakage behavior of the PCM, a series of the PA/HDPE FSPCM modified by the GNP were prepared and the compositions of the PA/HDPE/GNP composites are listed in Table 1. HPCM1 and CPCM1 with same ratio of the PA to HDPE (9:1) are set for comparison to investigate the effect of the GNP on the leakage behavior of the PA, and so do HPCM2/CPCM2 (the ratio of PA to HDPE is 8:2) and HPCM3/CPCM3 (the ratio of PA to HDPE is 7:3). The pure PA/HDPE composites were blended by twin-screw agitator at a screw speed of 1000 rpm for 30 min, and the operating temperature remained at 160 °C which was below the PA decomposition temperature of 285.07 °C. The GNP doped FSPCM were prepared in different mass ratio of PA/HDPE/GNP. Firstly, the GNP were blended with melted PA by water bath at 65 °C and the blends were stirred by a magnetic stirrer at the rate of 1500 rpm for 30 min. Meanwhile, the HDPE was heated at 160 °C until the HDPE is reduced to a pulpy consistency in a stainless steel vessel. Finally, the preheated HDPE was mixed with the melted blends at 160 °C by twin-screw agitator at the speed of 1000 rpm for 30 min. Fig. 1 shows the photographs of the HPCM1–3 and CPCM1–6 at room temperature. The white samples were the pure PA/HDPE composites while the black samples were the GNP doped FSPCM. The effects of the GNP on thermal conductivity are analyzed by comparison of the CPCM2 and CPCM4–6 in which the mass ratios of the PA to HDPE are 8:2.

2.3. Characterization techniques

The chemical structures and crystalloid phase of the FSPCM modified by the GNP were analyzed by a fourier transformation

Table 1
The compositions of the PA/HDPE/GNP composites.

Samples	HDPE (g)	PA (g)	GNP (g)	Mass fraction of GNP (%)
HPCM1	5.00	45.00	0	0
HPCM2	10.00	40.00	0	0
HPCM3	15.00	35.00	0	0
CPCM1	4.95	44.55	0.5	1
CPCM2	9.90	39.60	0.5	1
CPCM3	14.85	34.65	0.5	1
CPCM4	9.80	39.20	1	2
CPCM5	9.70	38.80	1.5	3
CPCM6	9.60	38.40	2	4

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