Fast Thermal Response of Shape-Stabilized Thermal Storage Materials: The Case of Interconnected Netlike Graphene/Hexadecane/HDPE Composites

Li Xu, Jixiang Zhang,* Cui Liu, Nian Li, Liqing Chen, Shudong Zhang,* and Zhenyang Wang*

ABSTRACT: Fast thermal response and sensitivity of organic latent heat storage materials with inherent low thermal conductivity to external temperatures is still a challenge in their practical applications. Herein, a shape-stable heat storage material composite, composed of interconnected netlike graphene, hexadecane, and commonly used high-density polyethylene (HDPE), was obtained using a convenient melt blending method. The start melting temperature, melting temperature, and end melting temperature of the obtained shape-stabilized thermal storage materials are presented, indicating that the response rate of the composites to ambient temperature could be indeed increased. This is because the interconnected netlike graphene with high conductivity intercalated into the HDPE matrix provided many conductive pathways for heat transfer. Therefore, the thermal conductivity of the composites is increased to 0.67 W/(m·K), which is about 123% higher than that of pure hexadecane. Meanwhile, the structure of polyethylene and hexadecane is similar, and hexadecane is uniformly dispersed in polyethylene and perfectly combined with polyethylene, which can effectively prevent the leakage of hexadecane during the phase transition. Thus, the obtained composites may play an important role in thermal management applications such as heat collection, transportation, thermal power conversion, and so forth.

1. INTRODUCTION

With the continuous development of human industrialization, the demand for energy is dramatically increasing worldwide. Therefore, energy conversion and conservation have become increasingly important. Thus, energy storage systems play a significant role in resolving the mismatch between energy utilization and supply gap; latent heat storage materials using phase change materials (PCMs) can greatly improve heat storage efficiency of systems. PCMs can store a large amount of heat through solid–solid or solid–liquid phase changes at a constant temperature. Among many kinds of PCMs, organic PCMs such as paraffin are a better choice because of their relatively stable properties, large range of practical melting points, moderate thermal storage density, low price, and so forth. However, one factor hindering organic PCMs (OPCMs) from practical application is the leakage of liquid-state PCMs. Therefore, some good and more effective methods such as microencapsulation, electrospinning and postfilling PCM into porous media have been developed to prevent leakage and have shown great potential. However, because these methods are complex and time consuming, it is not convenient to meet the practical application demands of large-scale production. From the perspective of large-scale production, the development of shape-stable PCM (SSPCM) is a very effective way to solve these problems. Stereotyped PCMs combine PCMs with polymers, which not only facilitates large-scale production of latent heat storage materials but also avoids the problem of leakage of solid–liquid phase change. Now, high melting point polymers are often used as supporting materials of SSPCMs. OPCMs are dispersed in the three-dimensional structure of the polymer to form a macroscopic solid with certain support and mechanical properties. However, another inevitable disadvantage of organic SSPCMs is their inherent low thermal conductivity, which will hinder their fast thermal response and sensitivity to external temperatures resulting in low-energy utilization in their practical applications. Combining high thermal conductivity materials with SSPCMs is the most direct and effective way to solve the problem of low thermal conductivity.
Herein, a shape-stable heat storage material composite, composed of interconnected netlike graphene, hexadecane, and common high-density polyethylene (HDPE), was conveniently obtained using the melt blending method. The interconnected netlike graphene with high conductivity intercalated into the HDPE matrix provided many conductive pathways for heat transfer. Therefore, the thermal conductivity of the composites (1.5% of graphene) could increase to 0.67 W/(m·K), which is about 123% higher than that of free hexadecane. The start melting temperature (SMT) of the composites is lower than pure hexadecane, such as the SMT of the composite is 12.4 °C when the content of graphene is 1.5%, which is 3.0 °C lower than that of pure hexadecane; the SMT is increased by 19%. Meanwhile, the structure of polyethylene and hexadecane is similar, and hexadecane is uniformly dispersed in polyethylene and perfectly combined with polyethylene, which can effectively prevent the leakage of hexadecane during the phase change. The maximum phase change enthalpy of the complex is 140.75 J/g, which ensures efficient heat storage. Thus, the obtained composites may play an important role in thermal management applications such as heat collection, transportation, thermal power conversion, and so forth.

2. RESULTS AND DISCUSSION

The preparation progress of graphene/hexadecane/HDPE composites is convenient and is shown in Scheme 1. Figure 1a,b shows typical optical images of graphene/hexadecane/HDPE composites with different shapes, formed by pouring into a variety of structural molds, showing their excellent structural stability. Graphene provides many conductive pathways for heat transfer, and the corresponding scanning electronic microscopy (SEM) image (Figure 1c) indicates that graphene has a disordered structure with a networking framework. Figure 1d is the Fourier transform infrared (FT-IR) spectrum group of hexadecane, HDPE, graphene, and graphene/hexadecane/HDPE composites. In the FT-IR spectrum, the main absorption peaks of HDPE are 2930, 1627, 1100, and 804 cm⁻¹, among which 2930 cm⁻¹ is the stretching vibration of the −CH2 group of alkanes.32 The absorption peaks at 1627 cm⁻¹ is the stretching vibration of C=C. The obtained peaks at 1100 and 804 cm⁻¹ are caused by the stretching vibration of C−C and the oscillating vibration of the methylene surface, respectively. The infrared absorption peak of hexadecane is similar to HDPE, indicating structural similarity and good compatibility between hexadecane and HDPE; the absorption peaks at 722 cm⁻¹ is caused by the swing vibration of −(CH2)4.33 For graphene, the absorption peak at 3436 cm⁻¹ is attributed to the stretching vibration of −OH. For graphene/hexadecane/HDPE, the absorption peaks of pure hexadecane, graphene, and HDPE can be found in the composite, indicating that there is no chemical cross-linking between the substances. Figure 1e shows the X-ray diffraction (XRD) pattern of the HDPE, graphene, and PCM, which can be used to analyze the crystal structure. For HDPE, the main absorption peaks at 21.7 and
24.1° are attributed to the (110) and the (200) basal planes of the orthorhombic crystal form of HDPE. From graphene/hexadecane/HDPE composites, the main absorption peak at 26.5° of graphene is weakened, which is attributed to the low content of graphene being added. Nevertheless, hexadecane is the only PCM that can release and store thermal energy, the addition of graphene and HDPE has no effect on the crystalline phase and crystallinity of hexadecane, and the crystal structure of the composite is stable. The above-mentioned results show that the shape-stabilized thermal storage materials are simply physical mixtures and did not form a new chemical structure.

Fast thermal response and sensitivity of organic latent heat storage materials with inherent low thermal conductivity to external temperatures is of importance in practical thermal storage applications. Obviously, the SMT, melting temperature (MT), and end melting temperature (EMT) of PCMs indicate thermal response and sensitivity of organic latent heat storage materials to external temperatures. DSC is very suitable and convenient technique to study phase transformation of PCMs, including the initial phase transition temperature, latent heat, and so forth. In order to better understand the thermal response and sensitivity of the shape-stabilized thermal storage materials, DSC data of the shape-stabilized thermal storage materials were studied from three aspects: the SMT, MT, and EMT. Figure 2a–c shows the SMT, MT, and EMT of graphene/hexadecane/HDPE composites with different graphene contents of 0.5, 1, and 1.5%, respectively. According to the DSC curves, the SMT of pure hexadecane is 15.4 °C (Figure S3). The SMT of graphene/hexadecane/HDPE composites with different graphene contents of 0.5, 1, and 1.5% is 14.3, 13.9, and 12.4 °C, respectively. As compared with pure hexadecane, the SMT of the composites is lower. For example, the MT and EMT of the composite are 5 and 2.7 °C lower than that of pure hexadecane when the graphene content is 1.5%; the MT and EMT are increased by 20.7 and 9.7%, respectively. The abovementioned DSC results (Table 1) demonstrated that the thermal response and sensitivity of obtained graphene/hexadecane/HDPE composites after the addition of graphene were obviously faster than that of the pure hexadecane. There is no doubt that the addition of graphene is beneficial to improve the thermal response rate of obtained graphene/hexadecane/HDPE composites.

As well known, the inherent low thermal conductivity of OPCMs deteriorates their thermal response rate. When the phase change latent heat is absorbed and released, heat transfer will be reduced, and energy storage performance will be affected. Thermal conductivity is an important index to characterize the rate of thermal response. The thermal conductivity of the composites is shown in Figure 2d. It can be seen that the thermal conductivity of pure hexadecane is 0.24 (W/m·K). With the increase of the graphene content, the thermal conductivity of the composites first increased linearly.

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Table 1. Phase Change Properties of Graphene/Hexadecane/HDPE Composites with Different Graphene Contents and Pure Hexadecane

| Sample                | SMT (°C) | MT (°C) | EMT (°C) | ΔH (J/g) | η (%) |
|-----------------------|----------|---------|----------|----------|-------|
| 70% hexadecane        | 14.3     | 24.5    | 28.3     | 140.75   | 62    |
| 0.5% graphene         | 13.9     | 20.7    | 25.8     | 135.2    | 59    |
| 1% graphene           | 12.4     | 19.1    | 25.0     | 138.5    | 60    |
| 1.5% graphene         | 15.4     | 24.1    | 27.7     | 227      | 100   |

*SMT, MT, EMT, total latent heats of melting (ΔH), and thermal storage capability rate (η).*

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When the graphene content increased to 1.5%, the maximum thermal conductivity of the graphene/hexadecane/HDPE composite reached 0.67 (W/m·K), which increases by 123%. This is because the interconnected netlike graphene with high conductivity intercalated into the HDPE matrix provides many conductive pathways for heat transfer.

3. CONCLUSIONS

In summary, a shape-stable heat storage material composite, composed of interconnected netlike graphene, hexadecane, and HDPE was conveniently prepared using the melt blending method. The thermal conductivity of the composites increased to 0.67 W/(m·K), which is about 123% higher than that of free hexadecane, attributing to the interconnected netlike graphene with high conductivity intercalated into the HDPE matrix affording many conductive pathways for heat transfer. Therefore, the SMT, MT, and EMT of the composites are lower when compared with the pure hexadecane. Meanwhile, similar structures of polyethylene and hexadecane can make hexadecane uniformly disperse into the polyethylene matrix and perfectly combine with polyethylene, which can effectively prevent the leakage of hexadecane during the phase change. Thus, the obtained composites may play an important role not only in thermal management applications but also in thermal power conversion of heat collection, transportation, and supply.

4. EXPERIMENTAL SECTION

4.1. Materials. Graphene (specific surface area, 50–200 m²/g) and hexadecane (C₁₆H₃₄, chemically pure) for thermal energy storage were purchased from Hefei Baierdí Chemical Technology Co. Ltd. HDPE (softening point: 125–135 °C and density: 0.940–0.976 g/cm³, Industrial pure) was obtained from Dongguan Huangjiang Co. Ltd.

4.2. Preparation of the Interconnected Netlike Graphene/Hexadecane/HDPE Composites. The interconnected netlike graphene/hexadecane/HDPE composites were prepared using the melt blending method. In a typical preparation process, first, 3 g of HDPE was completely melted in oil bath at 170 °C. Second, a different amount of graphene (0.05, 0.1, 0.15, and 0.2 g, respectively) was subsequently dispersed into hexadecane (7 g) at room temperature. Then, the abovementioned mixture was poured into the fused HDPE under magnetic stirring to make the graphene/hexadecane/HDPE composites with graphene ages of 0.5, 1, 1.5, and 2%, respectively. In order to form a dispersed suspension, the abovementioned mixture was stirred at 170 °C for 8 h. Finally, the mixture was transferred to the wide-mouth molds of different shapes to form a shape-stabilized structure through natural cooling. Meanwhile, using the same method, we prepared other different proportions of HDPE and hexadecane except the content of graphene was 1.5%, respectively.

4.3. Characterization. The microstructure and functional groups of HDPE, hexadecane, graphene, and graphene/hexadecane/HDPE composites were characterized by the Nicolet 8700 FT-IR spectrometer produced by Thermo Nicolet instrument company, USA. During the test, the sample to be tested was mixed and ground with KBr, the mixture for testing is pressed into sheets by the mold. HDPE, hexadecane, graphene, and graphene/hexadecane/HDPE composites were characterized by X’pert powder XRD. By XRD characterization, information such as material composition, structure,
and morphology of atoms or molecules inside the material can be obtained. Morphologies of the graphene/hexadecane/HDPE composites were observed and analyzed by using a scanning electronic microscope (CamScan Apollo 300).

DSC was used to characterize the heat storage capacity of all samples. During the test, the sample size was about 10 mg, the test temperature was 0–100 °C, and the heating rate was 10 °C/min. The test was carried out in a nitrogen atmosphere (50 mL/min). Cycling DSC scans were conducted on the representative composite in the temperature range of 0–100 °C, and their thermal reliability was evaluated during the repeated melting/solidifying processes. The thermal conductivity of the composites was measured with a TC3000 high precision thermal conductivity meter. The sample is split into two identical rectangular samples of length 40 mm and width 40 mm each, and the sensor is placed between the two flat samples. During the measurement, the sample was completely covered on both sides of the metal wire and compacted by 50 g weight. Each sample was tested six times, and each test was repeated 10 times. The thermal stability of the synthesized composite PCMs was characterized by the thermogravimetric analysis of pure hexadecane; and TGA curves of HDPE, hexadecane, and the graphene/hexadecane/HDPE composites with different hexadecane contents (PDF)

**ASSOCIATED CONTENT**

1. Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsomega.0c01183.

Raman spectra of graphene, TEM of graphene; DSC curve of pure hexadecane; and TGA curves of HDPE, hexadecane, and the graphene/hexadecane/HDPE composites with different hexadecane contents (PDF)

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**Author Contributions**

L.X. and C.L. contributed equally. The manuscript was written through contributions of all authors. All authors have given approval to the final version of the manuscript.

**Notes**

The authors declare no competing financial interest.

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