Effect of Boron Nitride on the Heat Transfer and Heat Storage of Poly(ethylene glycol)/Expanded Vermiculite Composite Phase-Change Materials

Heng Wang,† Jie Lei,† Taofen Wu,† Dan Wu, Hai Liu, Yong Deng,* and Fuzhong Wu

ABSTRACT: In this work, the expanded vermiculite/poly(ethylene glycol)–boron nitride (E/PB-X) shape-stabilized composite phase-change materials with the encapsulation capacity of ∼66.16 wt % were prepared by a typical vacuum impregnation method to overcome liquid leakage during phase transition and poor thermal conductivity during heat transfer of poly(ethylene glycol). It was found that the boron nitride showed a great influence on the heat transfer and heat storage of E/PB-X. The thermal conductivity of E/PB-X was 0.45–0.49 W/(m·K), indicating that the heat transfer of E/PB-X was significantly enhanced by the dispersed boron nitride fillers, which was mainly attributed to the reduction of interfacial thermal resistance and the formation of rapid thermally conductive channels. However, the latent heat (∼55.76 J/g) of E/PB-X decreased with the increase in the boron nitride content, revealing that the heat storage behavior of E/PB-X was strongly affected by the confinement of surface interactions of boron nitride and expanded vermiculite, which was consistent with the crystallization behavior determined by X-ray diffractometer (XRD) results. Moreover, the spectroscopy (FT-IR) and thermogravimetric analyzer (TGA) results confirmed that E/PB-X exhibited excellent chemical compatibility and thermal stability, respectively, which were conducive to practical heat storage applications.

INTRODUCTION

Phase-change materials (PCMs) are latent heat storage substances.3 Due to high energy storage density and nearly isothermal behaviors during phase transition, the PCMs have been widely used in solar energy utilization, building energy efficiency, waste heat recovery, battery thermal management, etc.4–6 Poly(ethylene glycol) (PEG), a promising organic PCM, shows excellent thermophysical properties, such as adjustable phase-change temperature, large latent heat, good chemical stability and phase-change reversibility, and no corrosiveness and phase separation, and receives great attention.5–6 However, the drawbacks of liquid leakage during phase transition and poor thermal conductivity during heat transfer limit its heat storage applications because of poor mechanical behavior and slow heat storage rate.9,10

To simultaneously overcome the above-mentioned drawbacks, the strategy of constructing shape-stabilized composite phase-change materials (ss-CPCMs) composed of encapsulation media, PEG, and thermal conductivity enhancement fillers was adopted.11–13 Recently, the thermal properties of ss-CPCMs containing porous minerals/ceramics/scaffold, PEG, and carbon nanomaterials/metallic nanomaterials have been reported.14–16 It was found that the dimension, concentration, and surface properties of fillers significantly affected the heat transfer and heat storage of ss-CPCMs.17 Usually, the thermal conductivity increased while the latent heat decreased.18 Therefore, the selection of appropriate fillers plays an
important role in adjusting or improving the thermal properties of ss-CPCMs. Boron nitride (BN) has the advantages of excellent thermal conductivity (up to 400 W/(m·K)), stable chemical property, and good thermal stability for heat storage application.19 The excellent physical and chemical properties of BN determine that it can greatly improve the poor heat transfer of PEG. Therefore, the nano BN may be a promising filler to enhance the thermal conductivity of PEG ss-CPCMs. The effect of nano BN on the heat transfer and heat storage of PEG ss-CPCMs is seldom reported. Expanded vermiculite (EVM), a hydrous phyllosilicate mineral, shows a unique pore structure, high porosity, good chemical inertness, and excellent thermal stability, which is conducive to the encapsulation and shape stability of PEG. 20 Consequently, the EVM was employed as a promising and suitable encapsulation medium to prepare ss-CPCMs in this study.

In this work, the EVM/PEG-BN (E/PB-X) ss-CPCMs were prepared and their micro-morphology, chemical compatibility, crystallization behavior, thermal conductivity, heat storage property, and thermal stability were analyzed in detail. Particularly, the mechanism of heat transfer enhancement was clearly clarified.

■ RESULTS AND DISCUSSION

Morphology of E/PB-X. The scanning electron microscope (SEM) images of E/PB-6 with different magnifications are shown in Figure 1a–c. It was found that the compound of PEG and BN (the BN was enwrapped by abundant PEG) was uniformly encapsulated into the multilayered porous structures and adsorbed on the surfaces of EVM, which was further confirmed by the energy-dispersive X-ray (EDX) mapping of characteristic elements of PEG (C), BN (N), and EVM (Mg) (Figure 1d–f). The obtained morphology was conducive to the shape stability and thermal conductivity enhancement of E/PB-X but might have an impact on the phase-change behavior and heat storage performance because of the existence of surface interaction among PEG, BN, and EVM.

Chemical Compatibility of E/PB-X. Excellent chemical compatibility of E/PB-X was the prerequisite for maintaining high latent heats release. The FT-IR spectra of EVM, PEG, and E/PB-X are shown in Figure 2. The PEG and EVM showed characteristic absorption peaks consistent with those reported in the literature.6,21,22 In the spectra of E/PB-X, the peaks of PEG and EVM could be clearly observed at the same wavenumber. The peaks at 798 and 1389 cm−1 corresponded to the B–N bending vibration and stretching vibration, respectively.23 Particularly, the peak at 1720 cm−1 was attributed to the oxidation of PEG in the preparation of E/PB-X. In addition, no other new significant functional groups appeared, indicating that the interaction among PEG, BN, and EVM was physical, confirming that E/PB-X showed excellent chemical compatibility.

Crystallization Behavior of E/PB-X. Figure 3 displays the XRD patterns of EVM, PEG, and E/PB-X. Two obvious diffraction peaks at 19.2 and 23.4° were observed for PEG, indicating that PEG as PCM exhibited good crystallization behavior. After PB-X was encapsulated in EVM, the diffraction peaks of EVM were clearly observed, but the intensity of the diffraction peaks of PEG was significantly reduced (Figure 3b), meaning that the crystallization behavior of E/PB-X was obviously restrained because of surface interaction, which might be detrimental to the heat storage property of E/PB-X.

Thermal Conductivity of E/PB-X. The change in thermal conductivity of E/PB-X with increasing BN content is shown in Figure 4. It was found that the thermal conductivity of E/PB-3, E/PB-6, E/PB-9, and E/PB-12 was 0.45, 0.47, 0.48, and 0.49 W/(m·K), respectively, indicating that the BN fillers effectively improved the heat transfer rate of E/PB-X. An approximately linear fit result between the thermal conductivity of E/PB-X (y) and BN content (x) is described by

\[ y = 0.0059x + 0.4412 \quad (R^2 = 0.9422) \]  

When compared with that of PEG (0.25 W/(m·K)), the thermal conductivity enhancement ratios of E/PB-3, E/PB-6, E/PB-9, and E/PB-12 were 1.84, 1.92, 2.08, and 2.2, respectively.
E/PB-9, and E/PB-12 were 80.0, 88.0, 92.0, and 96.0%, respectively. Based on the SEM results, due to the introduction of BN nanosheets, the close interfacial combination among PEG, BN, and EVM could efficiently decrease the interfacial thermal resistance, resulting in the formation of rapid thermally conductive channels in/among the phase-change layers, thus increasing the phonon mean free path of PEG (Figure 5), which was responsible for the thermal conductivity enhancement of E/PB-X.

**Heat Storage Property of E/PB-X.** The DSC curves of PEG and E/PB-X during the melting and solidification processes are shown in Figure 6, and the corresponding phase-change parameters are listed in Table 1. It was found that all of the E/PB-X showed obvious endothermic and exothermic peaks, indicating good heat storage and release characteristics. The difference between the melting and solidification temperatures of E/PB-X was ascribed to the supercooling behavior of PEG.24 When compared with the DSC curves of PEG, E/PB-X showed a huge difference in the phase-change temperature, revealing that the heat storage behavior of E/PB-X was strongly affected by the confinement of surface interactions of BN and EVM. The reduced phase-change temperature of E/PB-X for both melting and solidification processes could be explained by the Gibbs-Thomson equation and physical surface interaction.25,26 However, it should be noted that the solidification temperature

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![Figure 3. XRD patterns of (a) EVM, PEG, and E/PB-X. (b) Partial XRD patterns of E/PB-X.](image)

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**Figure 4.** Thermal conductivity of E/PB-X and the corresponding linear fitting curve.

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![Figure 5. Schematic diagram of thermal conductivity enhancement of E/PB-X.](image)

**Figure 5.** Schematic diagram of thermal conductivity enhancement of E/PB-X.
of E/PB-X decreased with the increasing BN content, indicating that the surface interaction between PEG and BN was enhanced. Further, the latent heat of E/PB-X (∼55.76 J/g) decreased with the increasing BN content, which was closely related to the restricted phase-change behavior caused by surface interaction (see XRD results). Consequently, the thermodynamic and dynamic characteristics of PEG were changed and PEG molecular chains on the surface of EVM and BN were amorphous or mesomorphic phase so that the PEG could not easily and freely undergo a phase change and formed a “nonphase change layer”, which could hardly contribute to the latent heat. In summary, the heat storage of E/PB-X was degraded while the heat transfer was enhanced, so it was important to balance the relationship between the two.

Notably, the latent heat (∼55.76 J/g) and thermal conductivity (0.45–0.49 W/(m·K)) were simultaneously maintained. When considering the comprehensive thermal performances, the E/PB-X showed great application potential in the fields of building energy efficiency, solar energy utilization, and battery thermal management.

**Thermal Stability of E/PB-X.** The thermal stability of E/PB-X was evaluated by TGA, and the corresponding curves are shown in Figure 7. As shown in Figure 7, the PEG shows a one-step complete degradation process when heated to 600 °C, mainly as a consequence of the thermal decomposition of PEG. The E/PB-X exhibited similar thermal stability characteristics with PEG, and the weight loss of E/PB-3, E/PB-6, E/PB-9, and E/PB-12 within ~600 °C was 70.7, 65.5, 64.0, and 63.9%, respectively, which was close to the encapsulation capacity of E/PB-X. Furthermore, the temperature at which the obvious weight loss began was above 100 °C for E/PB-X, which was much higher than its phase-change temperature. In addition, the weight loss of all of the E/PB-X was less than 1.67% within the designed working temperature range of 80 °C, indicating that E/PB-X exhibited excellent thermal stability.

**CONCLUSIONS**

In this work, we prepared a series of E/PB-X ss-CPCMs and studied the effect of BN on the heat transfer and heat storage of E/PB-X ss-CPCMs. The thermal conductivity (∼0.49 W/(m·K)) of E/PB-X was significantly enhanced by BN fillers while the latent heat (∼55.76 J/g) was decreased, which was mainly ascribed to the formation of rapid thermally conductive channels and surface confinement interactions of BN and EVM for the phase-change behavior of PEG. Moreover, the excellent chemical compatibility and thermal stability of E/PB-X were confirmed by the FT-IR and TGA results, respectively, which was an important guarantee for meeting critical heat storage applications.

**EXPERIMENTAL SECTION**

**Materials.** PEG-2000 (C.P.) with an average molecular weight of 2000 was purchased from Shanghai Macklin Biochemical Co., Ltd. BN (particle size: 50 nm) was purchased from Hefei ZhongHang Nanomater Technology Development Co., Ltd. EVM was obtained from Yuli, Xinjiang, China.

**Preparation of E/PB-X.** E/PB-X was prepared based on the typical vacuum impregnation method (Figure 8). First, the BN was dispersed in 70 mL of alcohol, stirred for 1.5 h at room temperature, and ultrasonicated for 0.5 h. Then, PEG was added to the above BN alcohol dispersion while stirring at 65 °C for 1.5 h to obtain the compound of PEG and BN (PB-X) (Table 2). Next, EVM was added to the above PB-X and impregnated by vacuum at 65 °C for 2 h. Finally, the impregnated samples were transferred onto filter paper at 80 °C. The filter paper was continuously replaced to remove the
leaking PEG. E/PB-X (E/PB-3, E/PB-6, E/PB-9, and E/PB-
12) was obtained until no liquid leakage trace was observed,
and the corresponding encapsulation capacity was 66.16,
65.56, 64.30, and 63.31 wt %, respectively.

Characterization. The micro-morphology of E/PB-X was
observed by a scanning electron microscope (SEM, Gemini
300, ZEISS, Germany). The chemical compatibility of E/PB-X
was analyzed by Fourier transform infrared spectroscopy (FT-
IR, Nicolet iS5, Thermo Fisher). All samples were ground
sufficiently and mixed with KBr. The testing wavenumber
range was 4000–400 cm\(^{-1}\). The diffraction patterns of PEG,
EVM, and E/PB-X were collected by X-ray diffractometer
(XRD, X'Pert Pro, PANalytical, Netherlands). The scan rate of
all samples was 5°/min in the 2θ range of 5–55°. The phase-
change parameters of PEG and E/PB-X during melting and
solidification were measured by a differential scanning
calorimeter (DSC, Q2000, TA). The testing temperature
range was −10 to 80 °C for the heating process and 80 to −10
°C for the cooling process at a rate of 5 °C/min in an N\(_2\)
atmosphere. The thermal conductivity of E/PB-X at 25 °C was
measured by a thermal conductivity meter (Hot Disk, TPS
2500S, Hot Disk AB, Switzerland). The thermal stability of
PEG and E/PB-X was carried out with a thermogravimetric
analyzer (TGA, TGA 5500, TA). All samples were heated from
25 to 600 °C at a rate of 10 °C/min in an N\(_2\) atmosphere.

| PB-X | PB-3 | PB-6 | PB-9 | PB-12 |
|------|------|------|------|------|
| PEG (wt %) | 97.0 | 94.0 | 91.0 | 88.0 |
| BN (wt %) | 3.0 | 6.0 | 9.0 | 12.0 |

Figure 8. Schematic diagram of the preparation process of E/PB-X.

Table 2. Components of PB-X

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T.W. performed the methodology and investigation. D.W.
performed the investigation and formal analysis. H.L.
performed the investigation and formal analysis. Y.D.
supervised, conceptualized, and validated the study and
wrote, reviewed, and edited the manuscript, and sought
funding acquisition. F.W. was responsible for project
administration.
Notes
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