



# Novel light-driven CF/PEG/SiO<sub>2</sub> composite phase change materials with high thermal conductivity



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## ABSTRACT

Light-driven carbon fiber (CF)/polyethylene glycol (PEG)/silica (SiO<sub>2</sub>) composite phase change materials (PCMs) with high thermal conductivity, and at low cost were successfully prepared through a sol-gel process. Structural and thermal property were characterized by scanning electron microscopy (SEM), Fourier transform infrared (FT-IR) analysis, X-ray diffraction (XRD), differential scanning calorimetry (DSC), thermogravimetry analysis (TGA), and differential thermogravimetry (DTG). The CF/PEG/SiO<sub>2</sub> composite PCMs showed amazing fusion and crystallization enthalpies of 142.6 and 154.4 J/g at CF content of 3%, and the thermal conductivity coefficient reached 0.45 W/(m·K). The prepared PCMs also showed excellent light-thermal energy conversion capability, and a potential for wide application in improving the utilization efficiency of solar energy.

## 1. Introduction

The current energy crisis has become more critical because of the decreasing in fossil fuel and the increasing in greenhouse emissions [1–9]. Under these circumstances, renewable energy has been presented due to its cleanliness and renewability. Solar energy as an inexhaustible energy shows a wide application prospect because of the outstanding advantage of universal, innocuousness, generous quantity and permanency. Consequently, conversion, storage, and application of solar energy has caused considerable attention [4,5,10]. However, the intermittence of time and space of solar irradiation seriously limits the application of solar energy. Moreover, almost 44% of visible light cannot be applied effectively and directly due to low thermal efficiency at the present stage [11]. Therefore, development of light-driven PCMs for storing solar energy has become a popular research topic [6].

Our research group has introduced a new kind of organic dye which could absorb and convert it to heat energy to improve the utilization efficiency of solar energy [12,13]. The utilization efficiency of visible light is more than 0.90 under single-band irradiation. However, the thermal conductivity of phase change materials (PCMs) still not satisfied the requirements of application. Therefore, Our and other research groups introduced carbon nanotubes and graphene into the phase change system to improve the thermal conductivity [14–24]. A new form-stable PCMs with the latent heats of 63.76 J/g and 64.89 J/g is prepared by vacuum impregnation of paraffin within grapheme oxide

(GO) sheets. The GO as a modifier is highly improved the thermal conductivity from 0.305–0.985 W/(m·K) [25]. The expanded perlite (Exp)/n-eicosane (C20)/Carbon nano tubes (CNTs) composite PCMs has been prepared by doping the C20 into the structure of Exp/CNTs matrix. The result PCMs showed latent heat of 157.43 J/g, and in this case, the thermal conductivity has enhanced considerably as 113.3% cause by the addition of CNTs [26]. Comparing with the unmodified PCMs, the modified PCMs showed more excellent absorption of visible light, light-thermal energy conversion, phase change thermal storage, and thermal conductivity. The rate of thermal loading was significantly improved with the introduction of carbon nanotubes and graphene, but the high cost still severely limits the application of such materials.

Carbon fiber (CF) is a graphite-like material which prepared by carbonation and graphitization with organic fibers. Each CF is composed of thousands of tiny fibers which is stacked with carbon atoms arranged in a hexagonal form at the atomic level. CF showed almost the same thermal conductivity, corrosion resistance, durability, and light-thermal energy conversion capability with carbon nanotubes and grapheme, but at a more cheap cost [27,28]. Therefore, CF as an additive for improving the property of composite materials has attracted much attention [29–32].

In this work, CF was introduced to a polyethylene glycol (PEG)/silica (SiO<sub>2</sub>) phase change system through in situ doping, and prepared novel CF/PEG/SiO<sub>2</sub> composite PCMs by a sol-gel process. The prepared PCMs showed outstanding phase change enthalpy (fusion enthalpy of

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142.6 J/g and crystallization enthalpy of 154.4 J/g), superior thermal conductivity coefficient (0.45 W/(m·K)), and excellent light–thermal energy conversion. The utilization of solar energy was significantly improved caused by the enhancement of thermal conductivity. Therefore, the CF/PEG/SiO<sub>2</sub> composite PCMs could be used for thermal energy and solar storage, exterior thermal insulation materials and solar water heater, etc.

## 2. Experimental section

### 2.1. Materials and chemicals

Tetraethyl orthosilicate (TEOS, density in terms of silica is 28.4%, 208.33 g/mol, 0.932–0.936 g/mL (20 °C)) and polyethylene glycol-6000 (PEG-6000, 6000 g/mol, 60 ± 3 °C) was purchased from Xilong Chemical Co., Ltd and Sinopharm Chemical Reagent Co., Ltd, respectively. CF (7 μm) was produced by Hangzhou Hi-Tech Composite Material Co., Ltd. Hydrochloric acid (HCl) was received from Beijing Chemical Works. Sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>) and Aniline-2,5-disulfonic acid monosodium salt (C<sub>6</sub>H<sub>3</sub>NO<sub>6</sub>S<sub>2</sub>Na) was purchased from Tianjin Bodi Chemical Co., Ltd and Luoyang Tengyi Chemical Company. Deionized water was used in the whole experiment. All reagents were used without further purification.

### 2.2. Synthesis of CF/PEG/SiO<sub>2</sub> composite PCMs by sol–gel and in situ doping method

The in-situ generated diazonium cation medication was conducted by the diazotization of C<sub>6</sub>H<sub>3</sub>NO<sub>6</sub>S<sub>2</sub>Na. 10 mmol of C<sub>6</sub>H<sub>3</sub>NO<sub>6</sub>S<sub>2</sub>Na was first dispersed in 25 mL of deionized water, followed by the addition of Na<sub>2</sub>CO<sub>3</sub> solution (100 g/L) to adjust pH to 7. Afterwards, added excess sodium nitrite (1.03 times than C<sub>6</sub>H<sub>3</sub>NO<sub>6</sub>S<sub>2</sub>Na) into above mixture under stirring. Finally, added diluted HCl solution (5 mol/L) to the mixture and stirred in an ice-bath for 15 min to prepare aniline-2,5-disulfonic acid (BDSNH<sub>2</sub>) diazonium solution. Unmodified CF (1 g) was dispersed in 25 mL of deionized water under stirring at 60 °C, followed by the addition of 25 mL of prepared BDSNH<sub>2</sub> diazonium solution and stirred for 1 h. Subsequently, the resulting CF were centrifuged, washed and steamed, the product was surface-modified CF powder with high water-dispersity [14].

TEOS (7.8 g) was first mixed with water (the molar ratio of TEOS to water of 0.1) and stirred at room temperature for 5 min. Then, the HCl solution (0.5 mol/L) was added to adjust the pH to 2. Afterwards, stirred above mixture for 20–30 min for hydrolysis and obtained translucent silica sol. Then dispersed modified CF in deionized water with an ultrasonic oscillator. Afterward, added melted PEG-6000 (85 wt%) and above silica sol to the modified CF solution. Na<sub>2</sub>CO<sub>3</sub> solution was then added to adjust the pH to 7–8 to transform the sol to hydrogels. The hydrogels were dry at 50 °C to prepare the CF/PEG/SiO<sub>2</sub> composite PCMs. The resultant composite PCMs were denoted as CF (X)/PEG/SiO<sub>2</sub>, where X was the content of CF.

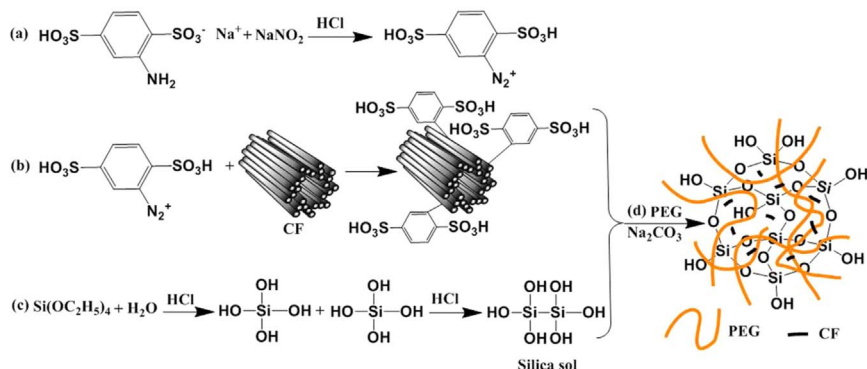


Fig. 1. Synthetic route of CF/PEG/SiO<sub>2</sub> composite PCMs.

The synthetic route of the CF/PEG/SiO<sub>2</sub> composite PCMs is showed in Fig. 1. It was worth to mention that heterogeneous distribution of unmodified CF was observed due to its low water-solubility and large specific surface. Therefore, the BDSNH<sub>2</sub> diazonium was used to graft the sulphanyl groups on the surface of CF via free radical reaction (Fig. 1b) to improve the water-solubility. Silica sol was produced by hydrolysis of TEOS with addition of HCl (Fig. 1c). Then added Na<sub>2</sub>CO<sub>3</sub> to the silica sol including melted PEG and modified CF to adjust pH to 7 to obtain silica hydrogel (Fig. 1d). Finally, the gel was dried to prepare the CF/PEG/SiO<sub>2</sub> composite PCMs.

### 2.3. Characterization

PEG morphology in PCMs network was investigated by scanning electron micrograph (SEM, Phenom G1, FEI). The infrared spectra of PCMs was performed by Fourier transform infrared spectroscopy (FT/IR-430, JASCO) in the range of 400–4000 cm<sup>-1</sup> with 2 cm<sup>-1</sup> spectral resolution using KBr pellets. X-ray diffraction (XRD) was carried out using a Rigaku D/Max 2400 Advance X-ray diffractometer and a curved graphite crystal monochromator Cu Kα in the range of 5–80° with the scanning step of 0.02°. The phase change temperature and latent heat of the products was measured using differential scanning calorimeter (DSC, DSC 240, NETZSCH) in the range of 0–80 °C at a heating rate of 5 °C/min under nitrogen stream. Thermal gravimetric analysis (TGA) of samples was on a Mettler-Toledo TGA/SDTA851 thermal analyzer at a heating rate of 10 °C/min from room temperature to 700 °C in a nitrogen stream. All the UV–vis spectra of the composite PCMs were obtained by using Hitachi U-4100 spectrophotometer, which at a scan speed of 300 nm/min and with a slit width of 8.00 nm.

## 3. Results and discussion

### 3.1. The structure characterization of CF/PEG/SiO<sub>2</sub> composite PCMs

Characteristic bands of PEG were observed at 3430 cm<sup>-1</sup> (ν(OH)), 2917 cm<sup>-1</sup> (ν(CH<sub>3</sub>)), 2889 cm<sup>-1</sup> (ν(CH<sub>2</sub>)) and 1106 cm<sup>-1</sup> (ν(C–O–C)) (Fig. 2a). The ν(OH) of CF was observed at 3430 cm<sup>-1</sup> (Fig. 2c). The FT-IR spectrum of SiO<sub>2</sub> was added to compare to that of the prepared composite PCM, characteristic bands of SiO<sub>2</sub> were observed at 3430 cm<sup>-1</sup> (ν(OH)), 1105 cm<sup>-1</sup> (ν<sub>as</sub>(Si–O–Si)), 968 cm<sup>-1</sup> (Si–OH), and 798 cm<sup>-1</sup> (ν<sub>s</sub>(Si–O–Si)) (Fig. 2b). These results suggest that the SiO<sub>2</sub> framework was successfully prepared by hydrolytic condensation (Fig. 1c). Characteristic bands of CF/PEG/SiO<sub>2</sub> composite PCMs were obtained at 3430 cm<sup>-1</sup> (ν(OH)), 2917 cm<sup>-1</sup> (ν(CH<sub>3</sub>)), 2889 cm<sup>-1</sup> (ν(CH<sub>2</sub>)), 1106 cm<sup>-1</sup> (ν<sub>s</sub>(C–O–C)), 1105 cm<sup>-1</sup> (ν<sub>as</sub>(Si–O–Si)), 968 cm<sup>-1</sup> (Si–OH), and 798 cm<sup>-1</sup> (ν<sub>s</sub>(Si–O–Si)). Except for the same peaks as PEG (Fig. 2a), SiO<sub>2</sub> (Fig. 2b), and CF (Fig. 2c), no new characteristic peaks and peak shift were observed in the FT-IR curve of the CF/PEG/SiO<sub>2</sub> composite PCMs (Fig. 2d). Therefore, PEG was fixed on the porous structure of SiO<sub>2</sub> by physical adsorption because no new chemical groups were prepared during the entire

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