

Microencapsulated phase change material modified by graphene oxide with different degrees of oxidation for solar energy storage



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ABSTRACT

High thermal performance of microencapsulated phase change material (MEPCM) helps to achieve good solar energy storage behavior. Here, n-dodecanol/melamine resin composite microcapsules modified by graphene oxide (GO) with different oxidation degrees were synthesized, which was applied to study how the oxidation degree of GO influenced the thermal properties of MEPCM. The as-prepared microcapsules were all spherical particles with the average latent heat of 170 J/g. TEM analysis showed that MEPCMs with or without GO had obvious core-shell structure with uniform shell thickness. When the dosing of GO was about 0.6 wt%, the thermal conductivity of MEPCM/GO with the minimum oxidation degree was enhanced by 115%, while MEPCM/GO with maximum oxidation degree was increased by 72% in thermal conductivity, greatly improving the energy storage efficiency. The result suggested the as-prepared microcapsules with large energy storage capacity and high energy storage efficiency could be a potential material used for solar energy storage.

1. Introduction

Phase change storage technology is an effective way to make use of solar energy, geothermal and waste heat, which achieves the storage and release of energy by the melting and freezing of phase change material [1]. Having large energy storage density and being recyclable [2], latent thermal energy storage has recently become a hot topic and been extensively applied in many fields, for example building materials [3–6], solar thermal storage [7], electronic devices [8], etc. Despite of the above advantages, phase change materials have the shortcomings of leakage, corrosion, phase segregation when used directly, and micro-encapsulation can effectively solve these problems [9]. Organic materials are generally used as encapsulated shell because of better compatibility and easier preparation, for example PMMA [10,11], PC [12], MF [13,14], PU [15], etc. However, organic material has a low thermal conductivity that may reduce the storage and release rate of phase change materials.

Based on the low thermal conductivity and poor heat storage and release performance of microencapsulated phase change materials (MEPCM), high conductive fillers or inserts are employed to improve its thermal conductivity [16]. The common heat-conducting substances are divided into carbon materials and metal materials [17], such as carbon nanotube [18], carbon fiber [19], silver nanowires [20] and Al₂O₃ [21], Si₃N₄ [22]. Graphene has a high thermal conductivity of

about 5300 W/mK [23], thus Graphene is used as a filler to improve the thermal conductivity [24–26]. Yavari et al. [24] prepared graphene-modified 1-octadecanol and investigated its thermal properties. Harish et al. [25] analyzed the chemically functionalized GNPs to enhance the thermal conductivity of lauric acid.

Graphene oxide (GO) is a derivative of graphene and can also be used to improve thermal conductivity [27–30]. Mehrli et al. [27] prepared paraffin/GO composite containing 48.3 wt% of paraffin by vacuum impregnation, and its thermal conductivity was improved from 0.305 to 0.985 W/mK. Hajjar et al. [28] prepared the GO/water nanofluids and investigated how the content and temperature of GO influenced thermal conductivity of nanofluids. When the loading of GO was 0.25%, the thermal conductivity was enhanced by 33.9% at 20 °C and 47.5% at 40 °C. Fillers to improve thermal conductivity may be armed with the properties of low density, high thermal conductivity and good compatibility with matrix. GO is used as a thermal conductive filler because of the similar structure of graphene and large numbers of functional groups [31]. The former provides GO with relatively high thermal conductivity, while the latter makes GO compatible with the matrix that can reduce phonon scattering and decrease interfacial thermal resistance. The ratio of the two functional regions is relevant to preparation conditions [32], that is, degrees of oxidation. In addition, GO has low density that small amounts of GO can improve thermal conductivity, and the latent heat of MEPCM would not be reduced too

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much. Up to now, many studies have added GO to improve the thermal conductivity of polymer [33], PCM [27,29], and nanofluid [28], but it has only recently been used in MEPCM [14,34]. As far as I know, there are no studies investigating the effect of the structure of GO on thermal properties of MEPCM. Therefore, it is very meaningful to conduct a detailed research on the effects of oxidation degree of GO on thermal properties of MEPCM. It may help us understand the thermal conducting mechanism and appropriate GO or materials with similar structures could be selected according to the structural features of matrix.

In this work, we varied the oxidation degree of GO through changing oxidation time. GO with different oxidation degrees were added to MEPCM and we systematically studied the impacts of oxidation degree on the morphology and thermal properties of MEPCM.

2. Experimental section

2.1. Materials

Graphite powder (99.99%) was obtained from Nanjing XF Nano Materials Tech Co. Ltd. Dodecanol was purchased from Aladdin Industrial Corporation, USA. Styrene maleic acid copolymer sodium salt (SMA1000HNa) was provided by Sartomer, USA. Dodecanol was purchased from Aladdin. Other reagents were bought from Guangdong Chemical Reagent Corporation and all the reagents were analytical grade.

2.2. Preparation of GO with different oxidation degrees

GO was prepared from pristine flake graphite powder based on modified Hummers Method [35] and was exfoliated to lamellar structure in distilled water by ultrasonication. 2 g graphite and 1 g sodium nitrate were mixed into 46 mL concentrated sulfuric acid in an ice bath and stirred for 30 min. Then 6 g potassium permanganate was slowly added into the mixed liquid and agitated for another 2 h. Next, the suspended liquid was moved to 35 °C oil bath and oxidated for a certain time. 92 mL distilled water was added and the temperature was subsequently increased to 98 °C and kept for 15 min. In the post-processing stage, 15 mL H₂O₂ (30%) was added until no bubbles created. The reaction was terminated by 140 mL distilled water and washed by 8*250 mL water until the supernate was neutral. Finally, the resultant brown liquid was exfoliated by ultrasonication for 50 min. The oxidation time was 30, 60, 90 min respectively and the corresponding samples were denoted as GO-1, GO-2, GO-3.

2.3. Preparation of MEPCM modified by GO

The preparation of MEPCM was divided into three parts: 1) Preparation of wall prepolymer: 10 g melamine and 21.5 g formaldehyde were added and stirred under heating until the liquid became clear. Then a certain amount of GO water dispersion was mixed, and the PH was adjusted to 8–9, followed by agitating at 70 °C for another 3 h; 2) Preparation of core pre-emulsion: 25 g dodecanol, 30 g water and 2.5 g SMA1000HNa were mixed and vigorously stirred for 1 h, then the emulsion was transferred to a PH of 3–4 with citric acid and heated to 70 °C; 3) Preparation of MEPCM: The wall prepolymer was added to the emulsion with a dropping rate of 3 s per drop, and subsequently reacted for 3 h.

The reaction production was washed with 50 mL 50% alcohol-water and 50 mL distilled water to remove residual dodecanol and ions for 5 times, and dried in a vacuum oven at 50 °C for 48 h.

2.4. Characterization

The morphology and thickness of GO were measured by atomic force microscope (AFM, MFP-3D-S, USA) in tapping mode with

vibration frequency of 300 kHz and force constant of 40 N/m. GO water dispersion (0.01 mg/mL) was added on the mica plate and dried at 50 °C for 30 min. Particle sizes were recorded on Particle Analyser (Malvern ZEN5600, UK). The Raman spectra were carried out by Raman system (Renishaw, England) with the laser wavelength of 532 nm. The scanning range was 0–3000 cm⁻¹. The chemistry composition of GO was measured by Element Analyser (Vario EL III, Elementar Analysensysteme GmbH, Germany). Scanning electron microscope (SEM, FEI Nova NanoSEM 430, Holland) and transmission electron microscope (TEM, TECNAI) were used to observe the morphology of MEPCMs. SEM analysis does not require special sample preparation, while TEM analysis needs to drop MEPCM alcohol dispersion (0.01 mg/mL) onto 300-mesh copper grid and then dry. The latent heat was analyzed by DSC (200 F3, NETZSCH, Germany). 5 mg MEPCM sample was sealed in an aluminum pan for DSC analysis at the heating/cooling rate of 10 °C/min under nitrogen atmosphere, and the testing temperature ranged from -30 °C to 60 °C. The thermal conductivity was obtained by Hot Disk (TPS2500, Sweden). 10 g MEPCM sample was added into a cylindrical mold and compacted, and then tested at room temperature. Thermal cycling test was made by alternately kept the MEPCM samples at -30 °C or 50 °C for 30 min, and the process was repeated by 100 times.

3. Results and discussion

3.1. Structure and element composition of GO

Raman spectra of GO samples are shown in Fig. 1, the D band at around 1350 cm⁻¹ is connected with the defect concentration and disorder caused by oxidation process, while the G band at about 1580 cm⁻¹ is associated with ordered structure of aromatic matrix. The Raman result suggests that the structure is destructed by oxidation, which is in accord with the previous research [36]. The oxygen content of GO detected by element analysis greatly reflects degree of oxidation. The elementary composition of GO is shown in Fig. 2. As is respected that, with oxidation time prolonging, oxygen content significantly increases and hydrogen content almost keeps unchanged, while carbon content obviously decreases, which implies that the C/O ratio reduces and the degree of oxidation increases.

3.2. Size and morphology of GO

The GO samples with various degrees of oxidation have excellent water dispersion and are brownish yellow in color. AFM shows that GO samples with various oxidation degrees possess good morphology of lamellar structure and all have uniform heights of 1.0–1.2 nm, which is the characteristic of complete exfoliation and monolayer (Fig. 3).

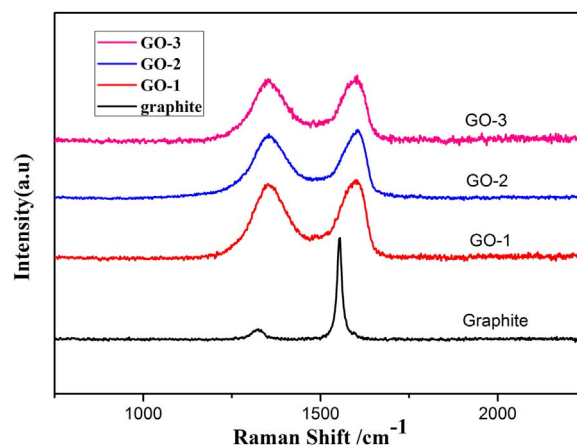


Fig. 1. Raman Spectra of graphite and graphene oxide.

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