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Novel stearic acid/graphene core-shell composite microcapsule as a phase change material exhibiting high shape stability and performance

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ABSTRACT

A stearic acid (SA)/graphene composite microcapsule, having a core-shell structure, is prepared for the first time using a latex technology for use as a novel composite phase change material (PCM) for thermal energy management. Melted SA is dispersed in water using an anionic surfactant as a stabilizer and cooled to prepare a water-based latex containing negatively charged SA particles. A simple mixing of this latex with an aqueous dispersion of graphene, prepared with aid of a cationic surfactant, results in the self-assembly of graphene on a SA particle surface by an electrostatic interaction, allowing for the encapsulation of SA with graphene to form a core-shell composite microcapsule. The protective graphene shell gives an excellent shape stability to the composite during phase change and a substantial improvement in thermal stability of the active SA core. In addition, the core-shell having high thermal conductivity. This results in a high performance PCM with a large latent heat and an improved thermal conductivity.

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1. Introduction

The heat absorption or release as a latent heat accompanied with phase change provides a quick and facile thermal energy management mechanism [1–4]. Therefore, the use of phase change materials (PCMs) which have a high latent heat and a well-defined operating temperature to buffer a temperature variation through heat storage/release has been considered as one of the most effective ways to control heat dissipation. Inorganic compounds such as salt hydrates, metals, and alloys [5], or organic compounds such as paraffin waxes, fatty alcohols, and fatty acids are utilized as PCMs [6].

The heat storage/release accompanied with solid-solid, solidliquid, or liquid-gas phase change can be utilized for thermal energy management. Among them, solid-liquid PCM is the most useful for practical thermal energy control due to its high energy storage density and small volume change concomitant with the phase change [5,6]. However, the application potential of PCMs is limited by leakage at the liquid state, which results in the contamination of equipment as well as the loss of PCM [7]. Techniques such as impregnation, macro-encapsulation, microencapsulation and nano-encapsulation have been used to minimize PCM leakage. These techniques mostly utilize polymers as a container or a shell to inhibit leakage. They effectively increase the heat transfer area, reduce the contamination of periphery by PCMs and control the volume change accompanied with phase change [8–10]. However, the polymer wrapping of PCMs can cause other problems because polymer has low thermal conductivity, low thermal stability, poor mechanical stability, and can release toxic gases into environment at higher operating temperatures [11,12]. In addition, the weight fraction of active materials in polymerencapsulated PCMs is normally less than 80% because a thick polymer shell is necessary for effective encapsulation [7]. This low content of active materials results in low specific latent heat of the encapsulated PCM.

A high performance shape-stabilized composite PCMs can be fabricated with a porous rigid framework to impregnate PCMs. The composite keeps the solid state far above the melting temperature of the active PCM because the PCM is trapped in the framework of rigid structure which prevents the flowage of melted PCMs. For example, porous expanded graphite [13,14] or porous graphite oxide [15,16] holding paraffin waxes or stearic acid exhibits excellent shape stability, prevents leakage and stabilizes cycling performance. These composites also display a high heat storage/ release rate and an enhanced thermal conductivity. However, the application of these composites remains limited by the low content of active materials.

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Graphene, a single-atom-thick sheet of hexagonally arrayed *sp*²bonded carbon atoms, has attracted a great deal of attention because of its extraordinary electronic, electrochemical, thermal, and mechanical properties in addition to a large surface area and a high aspect ratio. Graphene has potential applications in a variety of areas such as in electronic and energy devices, catalysis, sensors, biomedicines, and composite materials [17–21]. Some reports have shown significant improvement in thermal conductivity of PCMs by incorporating a small amount of graphene [22,23]. The ultra-high aspect ratio and novel properties of graphene have motivated researchers to use it as functional shells in novel core–shell structures [24,25]. However, to the best of our knowledge, the use of graphene as shell to encapsulate PCMs has not been reported.

In this paper, we report a facile method to encapsulate stearic acid (SA) with graphene using a latex technology, forming a core-shell SA/graphene composite PCM, which has high weight fraction of the active core material due to thermally stable and ultra-thin graphene shell. Just a simple mixing of a water-based latex comprised of negatively charged SA particles with an aqueous dispersion of graphene coated with a cationic surfactant induces a self-assembly of graphene on SA particle surface by an electrostatic interaction, forming the core-shell composite microcapsules (Fig. 1). The compression molded composite exhibits excellent shape stability during phase change, enhanced thermal stability, high specific latent heat, stable cycling performance, and an improved thermal conductivity.

2. Experimental

2.1. Materials

Expandable graphite (ES350 F5, average particle size $\sim 280 \,\mu\text{m}$, Qingdao Kropfmuehl Graphite Co., Ltd., China) was grinded to a smaller size of around 50 μ m and used for graphene preparation. Stearic acid (SA) (above 98.5%, Aldrich), hexadecyltrimethylammonium bromide (CTAB) (above 99%, Aldrich), sodium dodecyl sulfate (SDS) (above 99%, Aldrich), and acetone (Aldrich) were used as received.

2.2. Preparation of graphene

Graphite oxide was prepared by the chemical oxidation of the grinded expandable graphite using the Brodie method, as described



Fig. 1. Schematic illustration of the SA/graphene composite fabrication (a), and photographs of the SA latex, the aqueous dispersions of graphene and SA/graphene composite (b).

in previous papers [26–29]. Thermally reduced graphene was prepared by rapid heating of the dry graphite oxide at 1100 $^{\circ}$ C for 1 min under N₂ atmosphere.

2.3. Preparation of composites

The SA/graphene core-shell composite microcapsule was prepared using a latex mixing method (Fig. 1). In a typical experiment, a pre-determined amount of SDS was dissolved in distilled water (100 g). SA (15 g) was then added to the above SDS solution at room temperature. The temperature of the mixture was then raised to 90 °C. allowing SA to melt, followed by agitation at 800 rpm for 3 h to form a liquid SA dispersion in water stabilized by SDS. The dispersion was then cooled down to room temperature to form a water-based latex containing solid SA particles coated with SDS. Two SA latexes were prepared with different amounts of SDS, in which the sample SL1 is a SA latex prepared with one part SDS per one hundred parts SA (1 phr SDS); the sample SL2 is the latex prepared with 2 phr SDS. A pre-determined amount of graphene was dispersed in water (300 times the mass of graphene) using CTAB (with a mass identical to graphene) as a dispersing agent by sonication for 1 h. The graphene dispersion was then added to the above SA latex, followed by agitation for 1 h, filtration, washing thoroughly with distilled water, and drying in a vacuum at 50 °C for 1 day to obtain the composite. A series of composites was prepared using the two SA latexes above and with various graphene loadings. For example, the sample SL1G-1 is the composite prepared using the latex sample SL1 with one part graphene per one hundred parts SA (1 phr graphene); the sample SL2G-1 is the composite prepared using the latex sample SL2 with 1 phr graphene. For comparison, a composite was prepared using a conventional solution mixing method with acetone as a liquid medium both for dispersing graphene and for dissolving SA. In this case, a pre-determined amount of graphene was dispersed in acetone (100 g) by sonication for 1 h. SA (10 g) was dissolved in acetone (250 g) followed by the addition of the graphene dispersion, then mixed in an opened beaker on a hot plate set at 150 °C to remove acetone until the mixture became viscous. It was then dried in an oven at 60 °C for one day followed by further drying in a vacuum at 60 °C for an additional day to obtain a composite. The composite was prepared with various graphene loadings. For example, the sample SSG-1 is the composite prepared by solution mixing method with one part graphene per one hundred parts SA (1 phr graphene). SA and all composites were then compression molded at room temperature under 100 MPa for 5 min to form sheets to conduct measurements.

Another composite was also prepared by the latex mixing method using the same SA latex (SL1) but with expanded graphite dispersed in water by CTAB for self-assembly. The expanded graphite was prepared by the expansion of the expandable graphite at 1100 °C for 1 min under N₂ atmosphere. The obtained expanded graphite was dispersed in water by sonication for 6 h using CTAB as surfactant with the same amount as that used for dispersing graphene. The dispersion was then allowed to stand for 1 h in order for the unexfoliated graphite and the large and thick graphite flakes to settle down. The supernatant part containing thin graphite sheets stably dispersed in water was taken by decantation for use in composite preparation.

2.4. Characterization

X-ray diffraction (XRD) patterns were acquired with an X-ray diffractometer (Ultima IV, Rigaku) using Cu K α radiation (λ = 0.1542 nm) as the X-ray source. The morphology of the samples was observed using a field emission scanning electron microscope (FE-SEM, JSM-6500F, Jeol). Particle size analysis was performed

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