



A simple sonochemical method for fabricating poly(methyl methacrylate)/stearic acid phase change energy storage nanocapsules



Guxia Wang^{a,b}, Weibing Xu^{a,*}, Qian Hou^b, Shengwei Guo^{b,*}

^a School of Chemistry & Chemical Engineering, Hefei University of Technology, Hefei 230009, China

^b School of Chemistry & Chemical Engineering, Beifang University of Nationalities, Yinchuan 750021, China

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ABSTRACT

In this study, stearic acid suitable for thermal energy storage applications was nanoencapsulated in a poly(methyl methacrylate) shell. The nanocapsules were prepared using a simple ultrasonically initiated in situ polymerization method. The morphology and particle size of the poly(methyl methacrylate)/stearic acid phase change energy storage nanocapsules (PMS-PCESNs) were analyzed using transmission electron microscopy, scanning electron microscopy, atomic force microscopy and dynamic light scattering. The latent heat storage capacities of stearic acid and the PMS-PCESNs were determined using differential scanning calorimetry. The chemical composition of the nanocapsules was characterized using Fourier transform infrared spectroscopy. All of the results show that the PMS-PCESNs were synthesized successfully and that the latent heat storage capacity and encapsulation efficiency were 155.6 J/g and 83.0%, respectively, and the diameter of each nanocapsule was 80–90 nm.

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1. Introduction

Phase change materials (PCMs) has attracted considerable attention and interest in recent years for applications such as thermal comfort in buildings, thermal protection, cooling, air-conditioning and solar heating systems. They are substances that melt and solidify at a nearly constant temperature and are capable of storing and releasing large amounts of energy when undergo phase changes [1–3]. A thermal energy transfer occurs when a substance changes from a solid to a solid, a solid to a liquid, a solid to a gas, or a liquid to a gas. We call this a change in state or phase [3]. Among PCMs, those that undergo solid to gas and liquid to gas phase changes have a higher latent heat. However, their application is limited by the emergence of gas during the phase change. Compared with solid to solid PCMs, solid to liquid PCMs have a relatively brighter future because of their higher latent heats. PCMs can be classified as organic or inorganic. In general, salt hydrates are common inorganic PCMs, and fatty acids and paraffin are examples of organic PCMs. PCMs come in many forms, including bulk materials, sheets, and particles dispersed in matrices.

In most cases, however, PCMs cannot be directly used in different applications because of several defects, including the necessity of using special devices or heat exchange surfaces that increase the

associated cost and thermal resistance between PCMs and the environment [4]. Furthermore, during the mixing process or the phase change from solid to liquid, PCMs tends to diffuse to the surface and gradually loosen [5].

Encapsulation is a good method for preventing leakage of melted PCMs, reducing their reactivity with the environment, increasing the heat transfer rate and controlling the change in volume when the phase change occurs [6]. Encapsulation is the process of coating a PCM with a continuous shell to produce a capsule that is a nanometer to a millimeter in size, known as a nano or micro capsule. Encapsulated PCMs are composed of two parts: a PCM core and a polymer container. These nano or micro capsules can be easily dispersed in a matrix or attached to a surface. The rate of heat exchange of encapsulated PCMs is high due to their large surface area to volume ratio. The encapsulation technique depends on the physical and chemical properties of the materials used. To date, there are several physical and chemical methods used for the production of nano or micro capsules [7–25]. However, the common problem with these methods is that they all require a complex or time-consuming preparation process.

As we all know, ultrasound is a type of mechanical vibration with a frequency between 2×10^4 and 10^7 Hz [26]. When such a wave is transmitted through a liquid medium, ultrasonic cavitation occurs, which generates a very extreme local environment, i.e., an extremely high local temperature and pressure and high heating and cooling rates [27,28]. A polymerization reaction can occur

* Corresponding authors.

E-mail addresses: weibingxu@hfut.edu.cn (W. Xu), feidehai@163.com (S. Guo).

without an initiator at room temperature in an ultrasonic field. Furthermore, ultrasonically initiated polymerization always occurs at a high reaction rate over a short time period.

Taking these issues into consideration, we tried to find a simple, efficient and environment-friendly technology. In this paper, a sonochemistry method was employed to prepare the poly(methyl methacrylate)/stearic acid phase change energy storage nanocapsules (PMS-PCESNs).

2. Materials and methods

2.1. Materials

Analytical grade methyl methacrylate (MMA) was purchased from Tianjin Beilian Chemical Co., Ltd., China. It was washed with 10% aqueous sodium hydroxide and water to remove the inhibitor and dried over anhydrous sodium sulfate, then, distilled under reduced pressure prior to use. The sealed purified sample was stored at 4 °C until required. Analytical grade stearic acid (SA), sodium dodecyl sulfate (SDS) and other common solvents were also purchased from Tianjin Beilian Chemical Co., Ltd., China. They were used as received.

2.2. Apparatus

The equipment employed in this research was a VCX-750 ultrasonic processor (Sonics & Materials) with a frequency of 20 kHz. The titanium probe had a diameter of 13 mm. Ultrasonic irradiation was performed with the probe of the ultrasonic horn immersed directly in the reaction system. During the polymerization process, thermostated water was circulated to maintain a constant temperature, and the nitrogen purging rate was kept constant (Fig. 1).

2.3. The preparation of the PMS-PCESNs by ultrasonically initiated in situ polymerization

MMA (12.0 g) and SA (4.0 g) were mixed as the oil phase using a magnetic stirrer. The mixture was then added to an aqueous

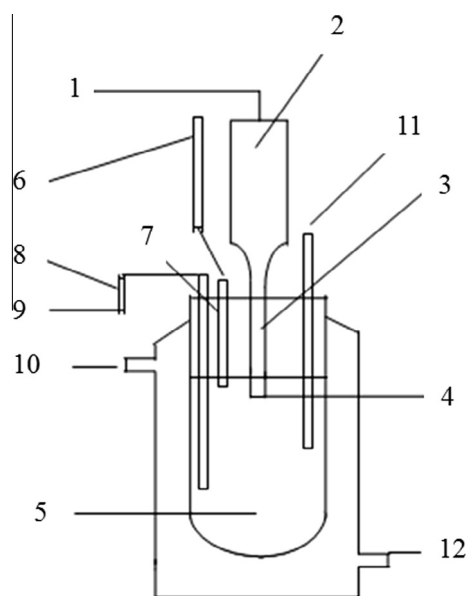


Fig. 1. A schematic diagram of the experimental set-up for ultrasonic irradiation: (1) ultrasonic generator; (2) transducer; (3) ultrasonic horn; (4) probe; (5) reaction vessel; (6) condenser; (7) N₂ outlet; (8) flowmeter; (9) N₂ inlet; (10) water outlet; (11) feed funnel; (12) water inlet.

solution of 1.0 wt% SDS (60 mL). The result was placed in the reaction vessel and deoxygenated by bubbling with nitrogen for 10 min. Then, the ultrasonic generator was switched on and the in situ polymerization was initiated by ultrasound irradiation. During the polymerization process, the nitrogen flow rate was kept constant at 50 mL/min, the cooling water was circulated to maintain a temperature of 20 °C, and the power output of the ultrasound was 750 W. The acoustic intensity generated at the tip of the horn and adsorbed by the solution was measured using an acoustimeter (YP0511F, Hangzhou Success Ultrasonic Equipment Co., Ltd., China) and found to be 12.8 W/cm². After a certain length of time, the ultrasonic irradiation was stopped, and the PMS-PCESNs were obtained. Part of the emulsion was poured into ice-cold methanol to coagulate and demulsify. Then, the precipitated material was filtered, washed, freeze-dried, and tested using FTIR and DSC. The remaining part of the emulsion was characterized using DLS, SEM, TEM and AFM directly.

2.4. Characterization

Dynamic light scattering (DLS) experiments were performed in 10-mm diameter round quartz precision light-scattering cells at an angle of 90° and temperature of 298 K. The apparatus employed was a BI-200SM spectrogoniometer (Brookhaven, USA) equipped with an Innova 304 Ar+ laser operating at a wavelength of 532 nm and a BI-9000AT digital correlator.

A Fourier transform infrared (FTIR) analysis was performed using a Nicolet 560 spectrometer with a scanning range of 500–4000 cm⁻¹.

The surface topography of the PMS-PCESNs was assessed using a scanning electron microscope (SEM, JSM-5900LV, Japan Electron Optics Laboratory). A very thin layer of gold was applied to the PMS-PCESNs using a sputtering unit (EMS 550, Electron Microscopy Sciences). The gold-coated PMS-PCESNs were placed in a microscope chamber and a strong vacuum was applied. The surface morphological features were determined using the microscope's 10 kV mode.

The morphologies and structures of the PMS-PCESNs were determined by transmission electron microscopy (TEM) using a JEM 100 CX microscope (Japan Electron Optics Laboratory). One drop of the suspension was diluted in water, placed on a 400-mesh carbon-coated copper grid and dried in air before observation.

Atomic force microscopy (AFM) testing was performed using a Nanoscope Multimode Explore (USA). For AFM imaging, two or three drops of the concentrated dispersion were placed on freshly cleaned mica plates and allowed to air dry. AFM images were obtained in the “tapping” mode. The maximum horizontal scanning range was 125 μm × 125 μm.

The thermal properties of stearic acid and the PMS-PCESNs, including the latent heat storage capacity and melting point, were obtained using differential scanning calorimetry (DSC, TA Instruments Q20). DSC measurements were performed with a heating and cooling rate of 10 °C/min. The samples were heated from 0 °C to 80 °C and cooled from 80 °C to 0 °C under a nitrogen atmosphere. From the DSC measurements, heating and cooling curves were obtained. By analyzing these curves, the thermal properties and heat storage capacities of stearic acid and the PMS-PCESNs were determined.

3. Results and discussion

3.1. Chemical analysis

The FTIR spectrum of the PMS-PCESNs prepared by ultrasonically initiated in situ polymerization is shown in Fig. 2. The peak

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