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Synthesis, characterization and thermal properties of nanoencapsulated phase change materials via sol–gel method

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ABSTRACT

This study focuses on preparing PCM (phase change material) nanocapsules which contain PA (palmitic acid) as core and SiO_2 as shell materials. For the first time encapsulation of phase change materials is synthesized in nano scale via the sol–gel method by changing the value of pH in the range of 11–12. The morphology and the mean size of three samples are compared and the influences of different pH values on the particle size studied. This investigation reveals that the encapsulation ratio of PA is increased from 83.25 to 89.55 percent by increasing the pH value in the range of 11–12. The nanoencapsulated PCMs are arranged uniformly and spherically with mean diameter sizes 183.7, 466.4 and 722.5 nm for pH values of 11, 11.5 and 12, respectively. A thermal cycling test is done by 2500 melting/freezing cycles to determine thermal reliability and chemical stability of the nanoencapsulated PCMs. The thermal conductivity of the encapsulated PA is significantly improved compared to pure PA. As a result, the prepared PA/SiO_2 nanocapsules are appropriate PCMs for slurry thermal energy storage applications because of their acceptable thermal properties, good thermal reliability, chemical stability, uniform morphology and thermal conductivities.

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1. Introduction

TES (thermal energy storage) is one of the most important energy technologies today, and plays an effective role in the use of thermal energy applications [1,2]. PCMs (phase change materials) are attractive materials for thermal energy storage due to their large latent heat and their characteristic of constant temperature in the course of absorbing or releasing energy [3]. They can be used as energy storage material in TES applications like solar energy collectors and industrial waste heat recovery [4,5].

There is a wide variety of PCMs available which melt and solidify at a broad range of temperatures and are utilized in many applications. They are categorized into two main groups: organic and inorganic materials. One of the most widely used organic low temperature PCMs is paraffin wax as it is cheap but has only moderate thermal conductivity values. Salts are higher temperature materials with higher volumetric energy density and thermal conductivity. Other organic materials which have been studied in the last several decades are fatty acids such as palmitic acid and stearic acid [6–8]. The palmitic acid has been introduced as one of the most important PCMs because of its desirable thermal and heat transfer characteristics. But these kinds of PCMs are not easy to be used directly in practical solar thermal applications because of their low thermal conductivity, flammability and instability. Encapsulation of PCMs is a practical solution for these kinds of problems [9].

Microencapsulated PCMs are formed from PCM as core and a material as shell to retain the shape and prevent PCM leakage during the phase change process [10]. The PCM microcapsules or nanocapsules have some advantages, such as increasing the heat transfer area and controlling the volume changes of storage material during the period of phase change [11,12]. Most attention in preparing micro/nanocapsules was on using organic materials as shell, but the use of inorganic shell materials has gained growing interest recently [13]; silica materials, especially, have been employed as carriers in controlled drug release. Encapsulating with an inorganic shell can also reduce the reactivity of the PCM with the outside environment [14]. Silica materials are nontoxic [15]; in addition, they have high surface area and tunable pore sizes. Their structures are stable and their surface properties well-defined. Silica spheres have high storage capacity and desirable thermal

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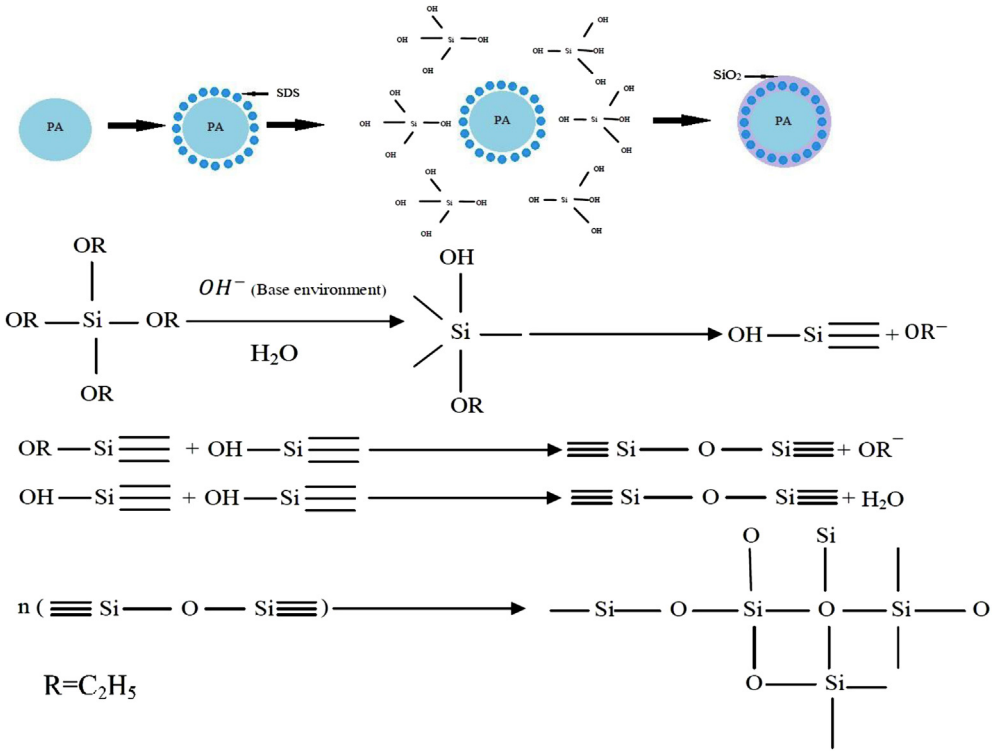

Fig. 1. Schematic formation of nanoencapsulation of silica shell and palmitic acid as core.

conductivity [15]. There are many methods for encapsulating PCMs, like in situ polymerization [16,17] spray drying, complex conservation [18,19], interfacial polymerization [20], miniemulsion polymerization [21,22] and the sol–gel method [23].

However, in some fields, especially in latent functionally thermal fluids, microencapsulated PCMs are not appropriate for repeated cycling, because the large size particles of the PCM microcapsules increase the fluid's viscosity; thus it is obligatory to develop PCM nanocapsules with smaller particle sizes as compared with microcapsules [1].

In recent years, several kinds of nanoencapsulated PCMs were synthesized by different methods. Nanocapsules of n-octacosane with PMMA (poly (methyl methacrylate)) shells were investigated by Sari et al. [10]. The nanocapsules had energy storage and release capacity of 86.4–88.5 kJ/kg during their phase change. Kwon also prepared nanocapsules of n-octacosane [24] the mean size of which was less than 100 nm. Nanocapsules of n-hexadecane with the shell of poly (alkyl methacrylate) were fabricated by Black et al. [25] while Alay et al. synthesized PMMA/n-hexadecane nanocapsules as a fiber additive in textile application [26].

Two main studies have been done on encapsulating PCMs with silica shell: Zhang et al. prepared microcapsules of n-octadecane as core and silica as shell with mean diameter size 7–16 μm via sol–gel method in acidic environment [15], and Fang et al. investigated the thermal properties of microencapsulated paraffin and silica core/shells with mean diameter size 8–15 μm through the sol–gel method with surfactant with a pH value in the range of 2–3 [23].

However, the mechanism of formation of inorganic silica shell at the O/W interface is still not clear; also, no work has been done on nanoencapsulation of palmitic acid by SiO_2. This study, therefore, has focused on synthesizing nanocapsules of palmitic acid with silica shell via the sol–gel method and studying the thermal characterization and the influence of different pH values on the particle sizes.

2. Experiment

2.1. Materials

The following materials (purchased from Fisher Scientific Inc.) were used in this experiment: palmitic acid ($C_{16}H_{32}O_2$) (PA) as latent heat storage PCM (99%), sodium dodecyl sulfate ($NaC_{12}H_{25}SO_4$) (SDS) as surfactant, tetraethoxysilane ($SiC_8H_{20}O_4$) (TEOS) (98%) as the precursor, absolute ethanol (C_2H_6O) (99.9%) as the solvent and ammonia solution (NH_4OH) as the activator.

2.2. Method

2.2.1. Preparation of palmitic acid emulsion

0.4 g SDS was dissolved in 200 ml distilled water at 70 °C (more than the melting point temperature of PA); 20 g PA was then added into this solution which was stirred continuously with a magnetic stirrer at the rate of 1000 rpm for 2 h.

2.2.2. Preparation of silica nanoparticles

20 ml TEOS and 180 ml absolute ethanol were mixed with 54 ml distilled water and the pH of the solvent was controlled by adding ammonium hydroxide. The mixture was stirred with a magnetic stirrer at the rate of 500 rpm for 30 min. The pH of the solvent was controlled at 11(S1), 11.5(S2) and 12(S3) in three different beakers. When the hydrolysis reaction of the silica was completed, the resultant sol solution was used as the encapsulation precursor.

2.2.3. Preparation of nanocapsules

The sol solution was added drop wise into the PA emulsion which was stirred at the rate of 500 rpm for 4 h. The temperature of the emulsion was controlled at 70 °C. Then the emulsion was cooled to room temperature and washed with distilled water and centrifuged. Formed white powder was collected and dried at 50 °C for

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