



Synthesis and properties of microencapsulated octadecane with silica shell as shape-stabilized thermal energy storage materials



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ABSTRACT

In this work, shape-stabilized octadecane and silica microcapsules were prepared by using sol-gel method. In the microcapsules, the octadecane was used as phase change material (PCM) for thermal energy storage, and the silica prepared from methyl triethoxysilane (MTES) acted as shell material to prevent the octadecane from leakage. The thermal properties of the microcapsules were measured by differential scanning calorimeter (DSC). The microstructure and particle size of the microcapsules were surveyed by scanning electronic microscope (SEM). Fourier transformation infrared spectroscopy (FT-IR) and X-ray diffractometer (XRD) were used to investigate the chemical structure, crystalloid phase of the microcapsules. The DSC results showed that the satisfactory microencapsulated phase change material (MPCM) sample melts at 28.32 °C with a latent heat of 227.66 kJ/kg and solidifies at 26.22 °C with a latent heat of 226.26 kJ/kg. The thermal reliability of the microcapsules was analyzed by a thermogravimetric analyzer (TGA). The results indicated that the MPCMs have good thermal stability, which could be used as shape-stabilized phase change materials for thermal energy storage.

1. Introduction

With the rapid growth of world population and the development of economy, energy shortages have attracted increased attention. Finding efficient energy utilization solutions and new energy storage materials have become more and more important [1]. Latent heat thermal energy storage (LHTES) that uses phase change materials (PCMs) is a good choice for thermal energy storage because the PCMs have high storage density and can store energy in isothermal process [2–4]. Various PCMs have been studied for thermal energy storage in recent decades, such as paraffins [5,6], salt hydrates [7,8], fatty acids [9,10] and eutectic compound [11–13].

However, two disadvantages limit the application of the PCMs for thermal energy storage. One is the low thermal conductivity of most PCMs, the other is the leakage of the PCMs during the phase change process. Therefore the microencapsulation method is developed to encapsulate the PCMs with shell materials as shape-stabilized composites [14–17]. The shell materials of the microcapsules which have high melting point can prevent the PCMs from leakage during the phase change process and the large surface-to-volume ratio of the microcapsules can increase the heat transfer efficiency of the PCMs. According to the actual need, various methods are developed to prepare microcapsules, such as interfacial polymerization [18], in situ poly-

merization [19], spray drying [20], complex coacervation [21], suspension polymerization [22] and emulsion polymerization [23,24], which have been optimized by a series of experiments conducted by Sari et al. [25–28]. Moreover, the newly developed mini emulsion polymerization method [29,30] has attracted much attention. With the microencapsulation technique, the MPCM can be used in many applications. The MPCM can be used for solar thermal energy storage, textiles and buildings, and the MPCM dispersed fluid can be served as heat transfer fluid, generally to reduce energy consumption or help to shift energy elsewhere for the off-peak time.

Many materials have been used as shell materials. In previous studies, organic polymers are the most likely choices [31–33]. However, organic polymers are usually toxic and flammable, and have poor heat transfer performance and thermal stability. The disadvantages restrict the application of the microencapsulated phase change materials. Therefore, inorganic polymer materials such as silica, ALOOH, titanium dioxide and other inorganic polymers are developed as shell materials [34–36].

In this paper, synthesis and properties of microencapsulated octadecane with silica shell by using sol-gel method as shape-stabilized thermal energy storage materials are reported. The silica was prepared from methyl triethoxysilane (MTES). In the microcapsules, the octadecane was used as PCM for thermal energy storage

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while the silica acted as shell material to lower the flammability of the octadecane and prevent the leakage of the PCMs during the phase change process. The MPCMs have good thermal stability, and can be used as shape-stabilized phase change materials for thermal energy storage.

2. Experimental

2.1. Materials

Octadecane (Reagent grade, Sinopharm Chemical Reagent Co., Ltd.) was used as thermal storage material. Methyl triethoxysilane (Reagent grade, Tokyo Chemical Industry Co., Ltd.) was used to prepare silica. Anhydrous ethanol (Reagent grade, Sinopharm Chemical Reagent Co., Ltd) and distilled water were used as the solvent. Hydrochloric acid (Reagent grade, Nanjing Chemical Reagent Co., Ltd.) and ammonia solution (Reagent grade, Nanjing Chemical Reagent Co., Ltd.) were used to adjust pH value. Sodium dodecyl sulfate (SDS; Reagent grade, Shanghai Chemical Reagent Co., Ltd.) was used as the oil–water emulsifier.

2.2. Preparation of the octadecane oil–water emulsion

15 g of the octadecane and 1 g of the SDS were added into 100 mL of distilled water to form octadecane oil–water emulsion. The mixture was stirred by a magnetic stirrer at 45 °C for 60 min. The stirring speed was 800 r/min. Then the pH value of the mixture was adjusted to 9–10 by adding ammonia solution. Finally, the octadecane was evenly scattered into the distilled water to form a stable emulsion.

2.3. Preparation of the MPCMs

Different mass ratios of the MTES, anhydrous ethanol and distilled water were mixed together in a beaker, the values of the mass ratios are shown in Table 1. Then hydrochloric acid was added into the mixture to adjust the pH value to 2–3. The mixture was stirred constantly at 45 °C for 20 min. The stirring speed was kept at 400 r/min. In this process, the hydrolysis reaction of the MTES occurred, producing the methyl silicate sol solution as microencapsulation precursor. The Scheme 1 of the reaction is as follows:

Then the stirring speed of the octadecane oil–water emulsion was reduced to 300 r/min, while stirring temperature remained unchanged. The methyl silicate sol solution was added to the octadecane oil–water emulsion drop by drop and then the emulsion was kept stirring for 2 h. In this process, the silica shell was formed on the surface of the octadecane droplets by the condensation reactions of the methyl silicate. The shell formation process of the silica and the Schemes 2 and 3 of the reaction are as follows:

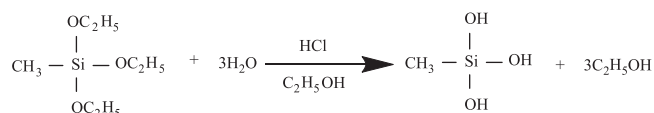
Finally, the microcapsules were collected by filtering, and dried at 45 °C for 24 h in vacuum oven. Three kinds of microcapsules were acquired, and were named MPCM1, MPCM2 and MPCM3.

2.4. Characterization of the MPCMs

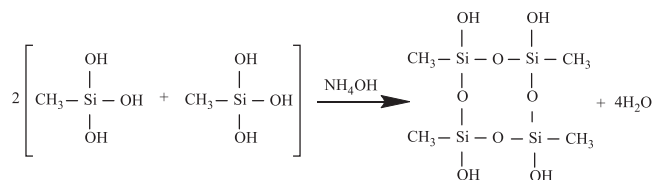
The chemical structure analysis of the MPCMs was performed using

Table 1
Composition of the octadecane emulsion and MTES solution.

Samples	Octadecane emulsion	MTES solution.
MPCM1	15 g octadecane+1 g SDS+100 mL distilled water	15 g MTES+15 g ethanol +25 mL distilled water
MPCM2		20 g MTES+20 g ethanol +30 mL distilled water
MPCM3		25 g MTES+25 g ethanol 40 mL distilled water



Scheme 1. The hydrolysis reaction of the MTES.



Scheme 2. The condensation reactions of the methyl silicate.



Scheme 3. The shell formation process of the silica.

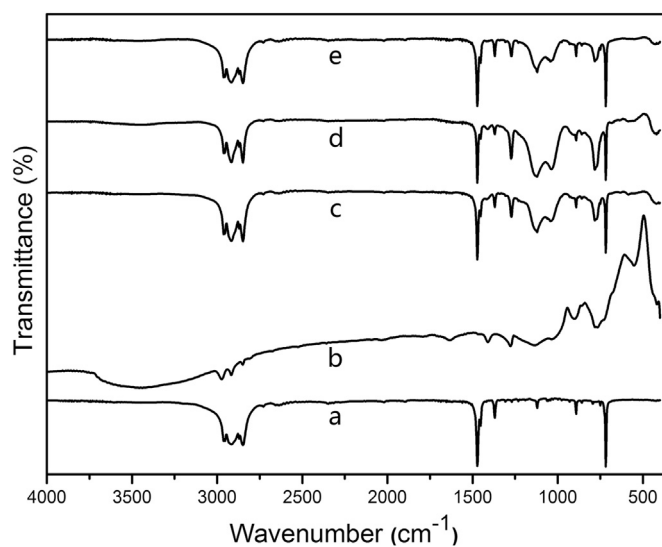


Fig. 1. FT-IR spectra of the (a) octadecane, (b) silica, (c) MPCM1, (d) MPCM2 and (e) MPCM3.

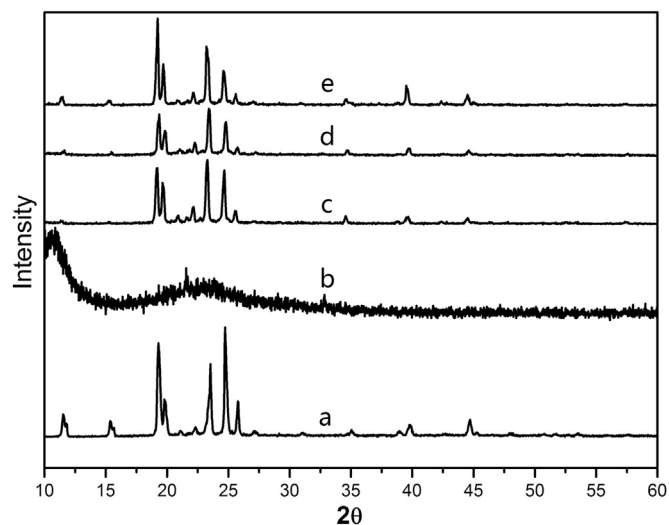


Fig. 2. XRD patterns of the (a) octadecane, (b) silica, (c) MPCM1, (d) MPCM2 and (e) MPCM3.

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