



Microencapsulated capric–stearic acid with silica shell as a novel phase change material for thermal energy storage



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HIGHLIGHTS

- Microencapsulated C–S with silica shell as phase change material for thermal energy storage.
- Agitation rate plays a crucial role in determining the PSD of C–S/SiO₂ MEPCM.
- The phase change temperature and latent heat are measured to be 21.4 °C and 91.48 J/g.
- MEPCM shows good thermal reliability with respect to 1100 times thermal cycling.
- MEPCM has great potential in the application of thermal energy storage.

ARTICLE INFO

Article history:

Received 19 February 2014

Accepted 21 May 2014

Available online 29 May 2014

Keywords:

Fatty acid

Silica

MEPCM

Thermal reliability

Thermal energy storage

ABSTRACT

Microencapsulated phase change materials (MEPCM) with polymeric shells have shortcomings including noxious gas releasing, flammability, poor thermal stability, low mechanical strength and heat conductivity. To overcome these disadvantages, capric–stearic eutectic (C–S) was microencapsulated in silica shell as a novel phase change material for thermal energy storage. The fabrication process was carried out without use of any emulsifiers, which would significantly deteriorate the latent heat storage capacity within conventional method. Such C–S/silica MEPCM possesses typical core–shell structured spherical morphology and the agitation rate plays a crucial role in determining the particle size distribution. DSC analysis shows that the melting and freezing temperatures of MEPCM are 21.4 °C and 22.2 °C, and the latent heats of melting and freezing are 91.48 J/g and 90.52 J/g, respectively. Besides, the thermal stability of PCM is greatly enhanced after embedded in silica shell. Above all, MEPCM reveals good thermal reliability after subjected to 1100 repeated melting/freezing cycling. Based on all results, the C–S/silica MEPCM has great potential in the application of thermo-regulated textiles, air-condition buildings, heat-transfer fluids and so on.

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1. Introduction

Sustainable and renewable energy has aroused great concern as energy crisis and environmental problem is increasingly prominent [1–5]. Phase change materials (PCMs) can absorb/release large amounts of heat in solid/liquid phase transition (reversible) [6–10]. Therefore, thermal energy storage (TES) technology using PCMs is found to be an effective solution as it not only reduces the

mismatch between energy supply and demand in time and space [11,12], but also it has a wide range of application, such as solar energy storage, industrial waste heat recovery, intelligent air-condition buildings, temperature adaptable greenhouses, aviation, electric appliance, thermo-regulated textiles and so on [13,14].

Among the PCMs investigated, fatty acids have attracted the most intensive attention for their suitable price, relatively large latent heat and good thermal reliability after a large quantity of thermal cycling [2,15]. Particularly, their binary or ternary eutectics are more competitive PCMs, which lie in the possibility to adjust their composition in accordance with desired temperature for human body comfortable [16,17]. In order to overcome the shortcomings of bulk PCMs, such as leakage [18], low thermal conductivity [10,19], and high volume changes [20],

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microencapsulated phase change materials (MEPCM) have been developed recently [21]. MEPCM have superiorities over solid–liquid PCMs as follows: increasing heat transfer area, reducing PCMs reactivity toward the outside environment, and permitting the core materials to withstand frequent volume changes during the phase transition [13].

To date, extensive studies have been carried out to microencapsulate PCMs within different polymeric shells, such as melamine–formaldehyde (MF) resin [22], urea–formaldehyde (UF) resin [23], and polystyrene [24]. Unfortunately, there are still some great disadvantages of using polymeric shell in practical application. For instance, polymers are apt to degrade and release noxious gas continuously, which causes environmental and health problems. Moreover, polymeric shells are restricted in some applications for their flammability [25], poor thermal stability [26], low mechanical strength and heat conductivity [27]. Most of all, various kinds of emulsifiers, used within conventional method to synthesize MEPCM, usually occupy as high as 10–30 wt% of MEPCM [28]. This will significantly sacrifice the latent heat storage capacity, and deteriorate the overall performance of MEPCM [29]. Therefore, it is meaningful for the thermal energy storage to introduce a high performance shell yet efficient method to fabricate MEPCM exempted from using emulsifiers.

Silica is nonflammable, and possesses higher mechanical strength, thermal conductivity and better chemical resistance compared with polymers. Moreover, silica has been widely used to encapsulate organic and inorganic active substances due to its non-toxicity and biocompatibility [30,31]. Therefore, silica is a promising shell material to prepare MEPCM. Despite many studies on MEPCM, a research on the facile preparation of fatty acid eutectic/silica MEPCM without any emulsifier is still limited. In this work, silica microcapsules containing capric–stearic acid eutectic (C–S) as a novel MEPCM were prepared by self-templating method [32]. The chemical structure, particle size distribution, thermal stability, thermal properties and long-term reliability of the synthesized MEPCM for the potential application such as thermo-regulated textiles, air-condition buildings, and heat-transfer fluids are investigated.

2. Experimental

2.1. Materials

Tetraethoxysilane (TEOS, AR), stearic acid (SA, AR), capric acid (CA, AR), hydrochloric acid (37%), anhydrous ethanol (AR), ammonia hydroxide (25–28%) were all purchased from Sino-pharm Chemical Reagent Co. Ltd. Deionized water was self-made in our laboratory. The thermal properties of SA and CA are listed in Table 1.

2.2. Preparation of C–S eutectic

As can be seen that both the melting points of CA and SA are higher than the human body comfortable temperature (15–25 °C). Hence, C–S is prepared to decrease the phase change temperature to a suitable value. The blending process of CA and SA is regarded as

Table 1
Thermal properties of CA and SA.

Sample	Melting		Freezing	
	Melting point T_m (°C)	Latent heat ΔH_m (J/g)	Freezing point T_f (°C)	Latent heat ΔH_f (J/g)
CA	31.5	165.7	30.6	163.5
SA	68.7	221.1	66.4	219.2

an ideal solution model and the eutectic ratio can be calculated by Schroder's equation: $\ln x_A = \Delta H_{m,A} (1/T_{m,A} - 1/T)$ [16,33]. Where $\Delta H_{m,A}$ and $T_{m,A}$ are the latent heat and melting point of component A, R is the gas constant, T and x_A are melting point of eutectic and the molar fraction of component A, respectively. By calculation, the mass fraction of CA to SA at eutectic point is 86%–14%. Then, samples are weighed according to the calculated proportion, and put in the water bath at a constant temperature of 80 °C until they melt completely. The mixed melt is stirred for 30 min with a magnetic stirring apparatus at 200 rpm and then cooled to room temperature.

2.3. Modification of silica precursor

TEOS is evolved as silica precursor in this work. TEOS and ethanol are equivalently mixed. The pH of mixture is adjusted to ~2.5 by adding a few drops of hydrochloric acid. Then, the solution is stirred at a rate of 600 rpm for 6 h at room temperature with a magnetic stirrer. The clear solution is evaporated by a rotary vacuum evaporator at 50 °C for 1 h to remove the ethanol and by product. Such obtained viscous liquid is partially hydrolyzed TEOS, which contains silanol and ethyl group. As a result, the modified silica precursor is turned into amphiphilic as well as higher reaction activity in weak basic solution.

2.4. Preparation of C–S/silica MEPCM

The fabrication process of MEPCM is as followed: in a three-neck flask, 5 g C–S eutectic and 50 ml distilled water is added. Then, the sample flask is immersed in a constant temperature water bath, and 12 ml modified TEOS is added dropwise. Continuous high-speed stirring is followed to impulse the oil–water mixture to disperse into uniform emulsion. Subsequently, few drops diluted ammonia hydroxide (~1%) is added to adjust pH of the emulsion to ~10, and keeps stirring for 10 min. The resultant suspension is left for 1 h without stirring. Finally, the C–S/silica microcapsules are collected by centrifugation, washed with water and dried in vacuum oven at 60 °C overnight.

2.5. Characterization

The chemical structure analysis of samples is carried out on a FTIR spectrophotometer (Thermo Nicolet Nexus). FTIR spectra are

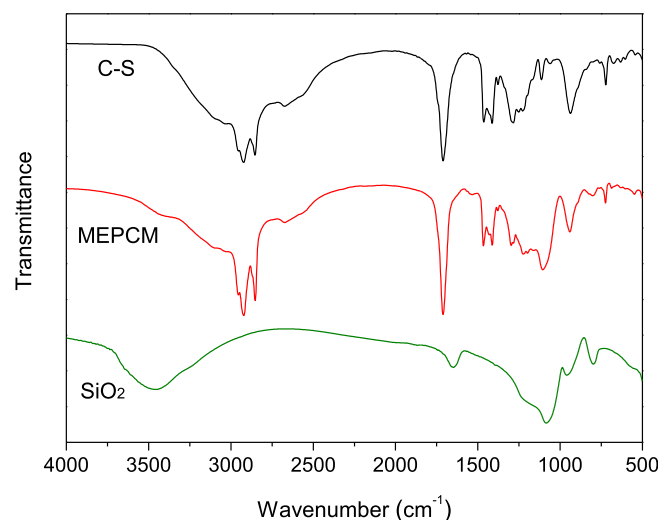


Fig. 1. FTIR spectra of C–S, SiO₂ and MEPCM.

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