



Preparation and characterization of polyurea microencapsulated phase change material by interfacial polycondensation method



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ABSTRACT

Phase change materials (PCMs) for heat energy storage have received an extensive attention in recent years. For heat energy storage application, a new type of polyurea (PU) microencapsulated phase change materials (MicroPCMs) were prepared by interfacial polycondensation method with isophorone diisocyanate (IPDI) and ethylene diamine (EDA) as shell monomers and paraffin as core material. The influences of monomer mass ratio, emulsifier type, emulsifier dosage and emulsifying stirring speed on MicroPCMs were investigated systematically. The morphology, chemical composition and particle size distribution of the MicroPCMs were characterized by using scanning electron microscope (SEM), fourier transform infrared (FT-IR) spectrum and laser particle size analyzer respectively. The results show that the MicroPCMs prepared under the optimal conditions have spherical shape and an average diameter of 2.42 μm . The results of differential scanning calorimeter (DSC) analysis show that the phase latent heat of the MicroPCMs is 92.5 J/g with 44.5% core content. The results of anti-osmosis test confirm that the MicroPCMs with the core/shell ratio of 75/25 have good compactness and stability. The microencapsulation technology developed is expected to be used in air conditioning, heating and other fields.

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1. Introduction

Microencapsulation is a widely accepted technique, and phase change materials (PCMs) have been used in energy storage field [1]. During the phase change process, PCMs can absorb, store and release the latent heat at their melting points with the change of ambient temperature [2]. Although very high latent heat can be obtained, the bulk PCMs are not easy to handle in practical applications [3]. To overcome their inherent shortcomings of these solid–liquid PCMs, such as volume expansion, liquid migration and supercooling, the technology of microencapsulation of PCMs (MicroPCMs) has been developed and studied intensively [4]. MicroPCMs have a core–shell structured to protect specific functional materials from or to release them into an outer phase for a long period of time [5,6]. MicroPCMs can handle PCMs as core material with many advantages: increasing thermal conductivity and heat transfer area, improving durability and preventing phase separation and supercooling phenomena [7,8]. Further, MicroPCMs can increase the thermal mass of building and clothing without increasing their real mass which makes them suitable for textiles and building applications [9,10].

Many methods have been developed for microencapsulation including interfacial polymerization [11,12], in situ polymerization [13,14], complex coagulation [15,16] and suspension polymerization [17,18].

The interfacial polycondensation has been widely concerned since it has several unique advantages, such as high reaction speed, mild reaction course and low penetrability [19]. The selection of shell material is also crucial to facilitate applications of MicroPCMs because their chemical and thermal stability, release properties and compatibility are all decided by the shell structure, while shell structure is governed by the monomers and the preparation technique [20]. Presently, polyurea (PU) [21], melamine–formaldehyde (MF) resin [22], urea–formaldehyde (UF) resin [23], polystyrene (PS) [24] and poly(methyl methacrylate) (PMMA) [25] are usually selected as shell materials of MicroPCMs.

Polyurea is generally synthesized by isocyanate and amino compounds as reactive monomers [26]. The selection of reactive monomers as shell material is very important for interfacial polymerization of MicroPCMs. Toluene-2,4-diisocyanate (TDI) with good reactivity and rigidity has been widely used for preparing microcapsules by interfacial polymerization [27]. However, polymerization reaction of TDI easily happens when water is in the initial emulsion due to TDI's high reactivity, and the quick reaction results in the partial or whole coalescence of the emulsion droplets and the stability reduction of the emulsion [28]. Moreover, NCO group connected on benzene ring of TDI has the certain rigidity, which causes easily the shell of MicroPCMs to rupture at certain pressure. Thus, the brittleness of this material limits the practical applications of the MicroPCMs [29]. In addition, TDI with high toxicity and strong irritant also results in environmental issues and health problems. Therefore, the use of appropriate reactive monomers as shell material is

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very important. Whereas the isocyanate group connected by alicyclic in isophorone diisocyanate (IPDI) show more flexibility, lower activity and lower toxicity compared with TDI, IPDI is considered as a promising material for the shell in preparation of MicroPCMs. Furthermore, MicroPCMs used for different products have diverse requirements on properties, and it is necessary to develop various types of MicroPCMs for different purposes.

In this study, a new type of MicroPCMs is synthesized by interfacial polymerization, and the properties of the products, such as morphology, size distribution, chemical structure, thermal properties and anti-permeability, are also investigated experimentally. The originality lies in the use of oil phase monomer IPDI, which provides superior properties for MicroPCMs.

2. Experimental

2.1. Materials

Isophorone diisocyanate (IPDI) with a purity of 98 wt.% and ethylene diamine (EDA, 99 wt.%) used as shell-forming monomers were supplied by Chengdu Xiya Chemical Reagent Company (Chengdu, China). Paraffin (RT21) as a core material was purchased from Ruhr energy science and technology Co., Ltd. (Hangzhou, China). Cyclohexane (CHX) as an assistant reagent was obtained from Tianjin Damao Chemical Reagent Factory (Tianjin, China). Sodium dodecylbenzenesulfonate (SDBS), polyethylene glycol sorbitan monostearate (Tween60) and polyoxyethylene(10) nonyl phenyl ether (OP-10) as the emulsifiers were also supplied by Tianjin Damao Chemical Reagent Factory (Tianjin, China). Petroleum ether, anhydrous ethanol and acetone as extracting solvent were obtained from Chengdu Aike Chemical Reagent Factory (Chengdu, China).

2.2. Preparation of MicroPCMs

Microencapsulated paraffin with polyurea shell was prepared by interfacial polycondensation method. Fig. 1 is the process of preparing the MicroPCMs. Typical procedure used to prepare MicroPCMs is as follows: the oil solution was prepared by adding 2.2 g IPDI, 5.7 g paraffin and 6 ml CHX in a round-bottom flask. The mixture was stirred for several minutes. Then the oil solution was dropped into 70 ml emulsifier aqueous solution to form a stable oil-in-water (O/W) emulsion at room temperature. The emulsion was stirred for 10 min at a certain stirring rate by using QSL high-speed disperse-machine (Shanghai Hengci Ltd., Shanghai, China). The aqueous soluble monomers (EDA) were dissolved in 15 ml distilled water, and then EDA aqueous solution was slowly dropped into the paraffin emulsion to start interfacial polycondensation with the stirring speed of 400 rpm. After all of the EDA aqueous solution was added, the suspension was heated to 60 °C and maintained at this temperature for 4 h with a continuous stir. Subsequently, the suspension was cooled down to the room temperature. Finally, the resultant MicroPCMs were filtered and washed twice with distilled water and dried in a vacuum oven for 24 h.

2.3. Characterization

The chemical structures of MicroPCMs and paraffin were determined by a Fourier transform infrared (FT-IR) spectrometer (Nicolet 560, USA). The samples were ground and mixed with KBr to make pellets. Spectra were collected by averaging 32 scans at a resolution of 4 cm⁻¹ in the wave number range of 400–4000 cm⁻¹.

The surface morphologies were observed with SEM (FEI QUANTA-450, America) at an accelerated voltage of 30 kV with 5000× magnification.

The mean diameter and particle distribution of MicroPCMs were determined with a laser particle size analyzer (Malvern Zetasizer 3000HS, Malvern, UK).

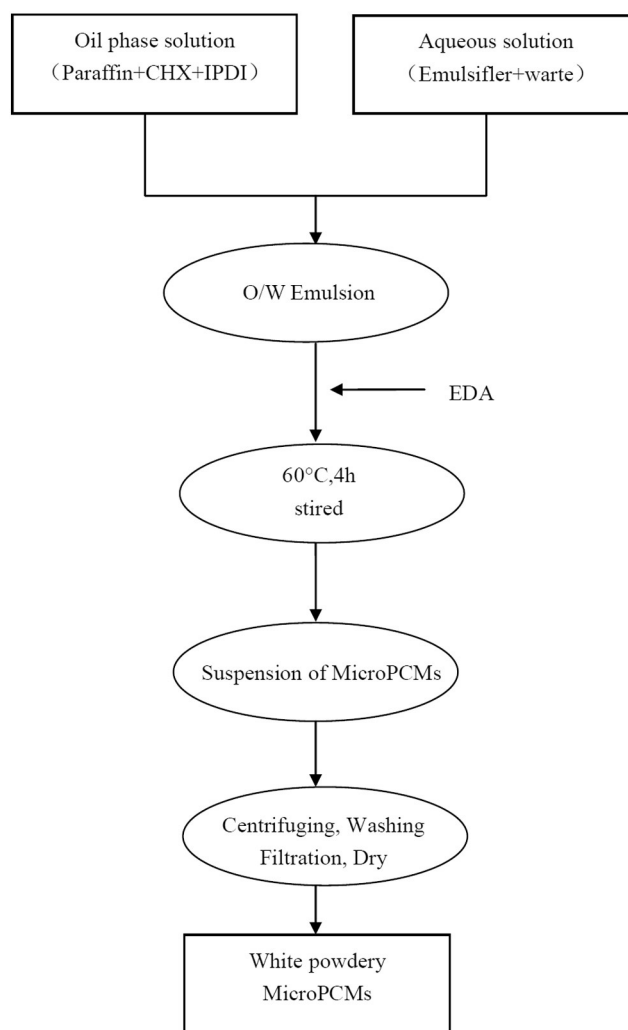


Fig. 1. Process of preparing MicroPCMs.

The thermal storage properties of MicroPCMs were measured with differential scanning calorimetry (CDR-34P, China) at a rate of 3 °C/min among the range of –20–50 °C under nitrogen atmosphere. Anti-osmosis of MicroPCMs was performed with spectrophotometer (721G-100, China). According to the phase change latent heat of the test, the core material content was calculated by the formula follows:

$$\text{Paraffin content} = \frac{\Delta H_{\text{MicroPCMs}}}{\Delta H_{\text{Paraffin}}} \times 100\%.$$

Here, $\Delta H_{\text{MicroPCMs}}$ and $\Delta H_{\text{Paraffin}}$ are the phase change latent heat of MicroPCMs and paraffin (208 J/g) respectively.

Anti-osmosis tests were conducted. The dried MicroPCMs were dispersed in distilled water [30]. The dispersion was filtered and 5 ml filtrate was taken to detect the absorbance (A) at 600 nm in a room temperature range. Absorbance of fully released solution is A_1 and the absorbance of blank solution is A_0 . The release rate of MicroPCMs core materials is $(A - A_0)/(A_1 - A_0)$. The taken filtrate was poured back into the original container after the test, and duplicated the test every hour.

3. Results and discussions

3.1. Dry weight of MicroPCMs prepared with different mass ratio of monomers

In the process of preparing MicroPCMs, core material, phase change paraffin, had no effect on synthesizing the polyurea microcapsule shell,

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