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Preparation of polyvinyl chloride capsules for encapsulation of paraffin by coating multiple organic/inorganic layers

Yingbo Chen*, Linfei Zhao, Yang Shi

State Key Laboratory of Separation Membranes and Membrane Processes, School of Materials Science and Engineering, Tianjin Polytechnic University, Tianjin 300387, PR China

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ABSTRACT

Hysteresis of phase transition and leaking of phase change matters are main problems in encapsulation of phase change materials. In this paper, we aimed to solve the problems by coating multiple organic/inorganic layers on surface of capsules adsorbed with paraffin. The coating layers are formed through interfacial polymerization of piperazine and trimesoylchloride or polycondensation of tetraethyl orthosilicate, or self-polymerization of dopamine. Morphology and surface elemental analysis were observed by field emission scanning electron microscope and X-ray energy dispersive spectrometer, indicating formation of thin and dense coating layers. Thermal stability and phase change properties were characterized by thermal gravity analysis and differential scanning calorimetry. Phase change enthalpy and phase change temperatures of the coated capsules were similar whatever the coated layers were, suggesting little influence of coating layers on the phase change process. Tightness of encapsulation was evaluated by measuring weight loss percentage (WLP) of the capsules under repeated heating of the capsules at 70 °C. It was noticed that the capsules coated with three layers owned a WLP less than –5% withstanding for more than one day. Standing time and valid space were found to be important for tight encapsulation.

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1. Introduction

As a form of latent heat storage system, phase change materials (PCM) have attracted more and more attention for their various applications, such as thermal storage in buildings [1,2], heat and solar energy storage [2,3], the latent functional thermal fluids [3,4], thermoregulated fabrics [2,4], collection and storage of surplus heat [5] and some thermal management systems [6]. The application storing thermal energy in buildings can flatten the fluctuation of indoor temperature and shift building peak electric loads for cooling [7], which can reduce the energy consumption to reduce the use of fossil fuels and the CO₂ emissions [2,8]. Paraffin, as a traditional solid–liquid phase change material, was used as latent heat storage media, which showed many advantages, such as a phase change temperature dependent on its molecular weight, a high heat of fusion, a lower vapor pressure in the melt [9], an abundance of natural reserves, ideal phase change behavior and a relatively low cost to name a few [10]. Thus, paraffin is often used in applications such as thermoregulated building materials, thermoregulated fabrics and furniture [11]. However, it must be

well encapsulated into various shell materials to prevent it from leaking out when melted. A few examples of shell materials used for the encapsulation of paraffin include polyurethane [12], low-density polyethylene [13,14], melamine–formaldehyde resin [15], polymethyl methacrylate [16], diatomite [17], silica [18,19], carbon nanotube sponges [20] and so on. At present, paraffin is used to prepare microencapsulated PCM or form-stable PCM to store energy in buildings [2,21–24]. However, PCMs microencapsulated or dispersed in shell materials in micro or even nano size suffer from the problem of hysteresis of phase transition because the molecules of phase change materials were often restrained by microstructure of shell materials or had some interaction with shell materials such as hydrogen-bond interaction [25–27]. In application, because microencapsulated PCMs may degrade the mechanical properties of the building materials [28,29] and have high investment cost, macro-encapsulated PCMs are preferred and widely used in buildings as a latent heat storage system to flatten the fluctuation of indoor temperature and reduce the energy consumption [30–32]. Memon et al. [31] prepared macro encapsulated PCMs and confirmed that they have a function of flattening the fluctuation of indoor temperature and reducing the energy consumption while economic evaluation of macro encapsulated PCMs is satisfactory. Han et al. [26] and Li et al. [32] prepared

* Corresponding author.

E-mail address: bocy2009@hotmail.com (Y. Chen).

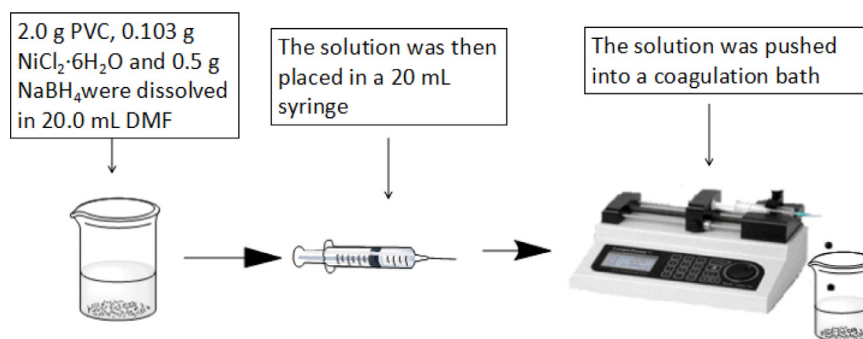


Fig. 1. The device diagram of fabricating PVC capsules.

macro-capsules containing microencapsulated PCMs, but the shell materials of the microcapsules limited the loading ratio of PCMs and the problem of hysteresis of phase transition was not solved.

In previous work [33], we prepared PVC hollow macro-capsules encapsulated paraffin and coated one layer with silica, as the size of the pores of silica ceramics is small enough to keep the melted paraffin in pores even over the melting temperature of the paraffin. The composite has a high phase change enthalpy and no hysteresis of phase transition since paraffin was encapsulated in macroscopic scale. We found that encapsulation of paraffin with the silica material can prevent it from leaking out when melted and enhance thermal transfer and phase change properties of the PCMs. But the leaking problem was not solved completely and thermal transfer and phase change properties of the PCMs can be enhanced more by coating more silica.

In this paper, PVC macro-capsules are prepared to encapsulated paraffin directly by coating multiple layers of polyamide, silica or polydopamine (PDA). The composite we prepared not only solves the problems of paraffin leaking and hysteresis of phase transition, but also has a high phase change enthalpy. The composition and morphology of the composite capsules were investigated in detailed by X-ray energy dispersive spectrometer and field emission scanning electron microscope. Phase change enthalpy and thermal stability of the capsules were recorded using differential scanning calorimeter and thermal gravity analysis. Sealing of the capsules were studied by differential weight method. Thermal cycling test was performed to determine thermal reliability of the encapsulated capsules.

2. Experimental

2.1. Materials

N,N-dimethylformamide (DMF, 99.5%), tetraethyl orthosilicate (TEOS, 98.0%) and pure ethanol were purchased from Kermel Chemical Reagent Co., Ltd. (Tianjin, China). Nickel chloride 6-hydrate ($\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$, 98.0%), sodium borohydride (NaBH_4 , 98.0%) piperazine (PIP, 99.0%) and tris (hydroxymethyl) aminomethane (Tris, 99.5%) were obtained from Tianjin Guangfu Fine Chemical Research Institute. Sodium hydroxide (96.0%) and ammonia solution ($\text{NH}_3 \cdot \text{H}_2\text{O}$, 25.0%) were purchased from Tianjin Fengchuan Chemical Reagent Technologies Co., Ltd. Dopamine hydrochloride (DA, 98.0%) and trimesoylchloride (TMC, 99.0%) were obtained from Aladdin Industrial Corporation (Shanghai, China). Paraffin was supplied by Shanghai Yi Yang Instrument Co., Ltd. All reagents were applied as received without further purification and all the aqueous solutions were prepared with distilled water.

2.2. Fabrication of PVC capsules and adsorption of paraffin

PVC capsules were prepared according to previous report [33], briefly, 2.0 g PVC, 0.103 g $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ (catalyst for NaBH_4 hydrolyz-

ing) and 0.5 g NaBH_4 (foaming agent) were dissolved in 20.0 mL DMF to obtain a homogeneous solution. The solution was then placed in a 20 mL syringe with a needle (inner diameter of 1.0 mm). The solution was pushed by a syringe pump (Fig. 1) into a coagulation bath (distilled water) with stirring at a constant speed. After formation of capsule, it was taken out from the coagulation and air dried.

Adsorption of paraffin was carried out in vacuum. The dried capsules were immersed in melted paraffin at 70 °C in a flask. The flask was put in a vacuum oven at the same temperature under pressure of -0.08 MPa. After being filled with paraffin, the capsules were taken out from paraffin by removing the paraffin on the outer surface using a tissue paper. In order to form the first coating layer on the surface of the capsules, 20 wt% TEOS (used to prepare silica at the next step) or 1 wt% TMC (used to prepare PA at the next step) were added in melted paraffin.

2.3. Encapsulation of capsules

Many researchers have prepared phase change composites using silica as a shell [18,19,37], proving that silica can effectively hold phase change materials and get it in a facile condition. Besides, polyamide has a compact structure, and can be prepared easily. So, we choose them as the layering materials respectively.

The first SiO_2 layer was formed by condensation of TEOS (Fig. 2) in ammonia solution (8 mL distilled water, 28 mL ethanol and 4 mL $\text{NH}_3 \cdot \text{H}_2\text{O}$ preheated in a water bath at 60 °C). Capsules (containing paraffin and TEOS) were added to the solution with stirring until the condensation reaction of TEOS was complete. The capsules were taken out, washed and dried to obtain the capsules with coating SiO_2 layer. The first PA layer was formed by interfacial polymerization of PIP and TMC (Fig. 2). Capsules (containing paraffin and TMC) were added to 2 wt% PIP aqueous solution with stirring until the polymerization of TMC and PIP completed.

To further improve the seal of the capsules, the secondary SiO_2 layer was synthesized out of the first SiO_2 layer or PA layer. In order to enhance the combination between two layers, a polydopamine (PDA) layer which was used as a surface-adherent film onto a wide range of inorganic and organic materials [34] was coated after the formation of the first layer.

The PDA layer was formed by adding the capsules in a solution (10 mM Tris buffer solution, pH was adjusted to 8.5 with hydrochloric acid, followed by addition of 3 mg/mL dopamine hydrochloride) with slightly shocking until the self-polymerization of dopamine completed (Fig. 3). The coated capsules were washed with 10 mM Tris buffer solution at the same pH and dried to obtain capsules with coating SiO_2 /PDA (or PA/PDA) layers.

The outer SiO_2 layer was formed by adding the PDA coated capsules in TEOS ammonia solution (Fig. 3) (8 mL distilled water, 28 mL ethanol, 4 mL $\text{NH}_3 \cdot \text{H}_2\text{O}$ and 1 g TEOS preheated in a water bath at 60 °C) with stirring until the condensation reaction of TEOS

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