

Polystyrene-based caprylic acid microencapsulation for thermal energy storage



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ABSTRACT

In this study, caprylic (octanoic) acid microcapsules were synthesized with polystyrene shell material using the emulsion polymerization method. The influence of the type and concentration of the cross-linking agent on the phase-change properties of the microcapsules was examined. The structure and properties of the microcapsules have been characterized by scanning electron microscopy (SEM), differential scanning calorimetry (DSC), and thermal gravimetric analysis (TGA). A second main contribution of this work is to investigate whether we could synthesize microcapsules with the same thermal properties during serial production. The effects of serial production on microencapsulated caprylic acid (microPCMs) have been investigated by thermal methods. The results show that reproducibility is an important parameter in the microencapsulation process. It was determined that when the synthesis amount is increased, we obtained lower efficiency in the microencapsulation of caprylic acid.

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1. Introduction

Fatty acids are phase-change materials for latent heat thermal energy storage applications [1]. Caprylic acid is one of the most promising fatty acids because of its melting point (15–17 °C) and high latent heat storage capacity (158 J/g). These properties allow their use in many thermal energy storage (TES) applications such as buildings, textiles, agriculture, food transportation, etc. [2]. It can be said that thermal energy storage has been recognized as one of the most significant technologies for the utilization of renewable energy sources and conserving energy [3]. Phase-change materials (PCMs) are substances that melt and solidify at a nearly constant temperature. Large amounts of energy can be stored and released during the phase change [4]. Materials used as PCMs in TES applications should have a large latent heat, suitable melting temperature, melt congruently with minimum subcooling and be chemically stable, low in cost, non-toxic and non-corrosive. The use of PCMs that are in the liquid state at room conditions is difficult in thermal energy storage applications. Since PCMs repeatedly change phase from solid to liquid during the heat storage process, they need to be kept in some kind of containers or matrices [5]. Enclosing the liquid materials in microcapsules allows

them to be treated as solids. Furthermore, with microencapsulation the core materials can be protected from the effects of the environment. Recently, microencapsulation of PCMs has been widely studied for TES, for example *n*-dodecanol microcapsules were fabricated with an acrylic polymer shell using suspension-like polymerization and photo-induced polymerization methods [6]. Also, *n*-pentadecane was encapsulated into methyl methacrylate (MMA) shells by Taguchi et al. [7] using the suspension polymerization method. Li et al. [8] encapsulated *n*-octadecane with different copolymer shells by suspension polymerization. In another study, a series of microencapsulated phase-change materials (micro-PCMs) with acrylate-based polymer shells and binary core materials were prepared using suspension-like polymerization method [9]. Konuklu et al. synthesized a series of four nanocapsules containing *n*-alkanes (C_nH_{2n+2}), namely tetradecane, pentadecane, hexadecane, and heptadecane, in poly(styrene-*co*-ethylacrylate) shells using an emulsion copolymerization method [10].

In our previous work [2,11,12] we microencapsulated caprylic acid with different shell materials. We started with the microencapsulation of caprylic acid with urea formaldehyde resin, melamine formaldehyde resin, and melamine-urea-formaldehyde resin [2]. Secondly, microcapsules containing caprylic acid and polyethylacrylate shells for thermal energy storage applications were prepared using an emulsion polymerization technique. Ethylene glycol dimethacrylate was used as a crosslinking agent [11]. Thirdly, caprylic acid/chitosan-gelatine microcapsules were

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prepared according to the complex coacervation method and were cross-linked by glutaraldehyde. The influences of the glutaraldehyde mass ratio upon encapsulation, as well as both the physical and thermal properties of the resulting microcapsules, the properties of microencapsulated phase-change materials (microPCMs) were analyzed [12].

Using an appropriate shell material is very important for the application of microcapsules in latent heat storage applications [11]. For example melamine–formaldehyde, urea–formaldehyde and melamine-urea-formaldehyde (MUF) shells are very popular shell materials for the microencapsulation of PCM process. But these shell materials can include formaldehyde residue, which can lead to health and environmental problems [13]. In order to increase the application areas of the microcapsule, shell materials that do not contain any formaldehyde residue can be preferred. Recently, Polystyrene (PS) shell material is also used as a popular shell material in microPCM manufacturing [14]. In the last few years there has been increasing interest in the micro-nanoencapsulation of PCMs with PS and styrene/copolymer shell materials [10,13,15–20]. Among the advantages of PS shell materials are: they are clear, inexpensive, non-toxic and are easy to handle/process. Because of these advantages PS is now used in many applications such as electrical/electronic, automotive and industrial films [21]. Also, since PS has thermal insulating properties; it can be used in insulation applications [22].

However, as far as we know, there is no report in the literature about the microencapsulation of caprylic acid within a polystyrene shell. As shown in Fig. 1, polystyrene is a polymer which is produced from styrene monomer by polymerization. The aim of this work was to develop caprylic acid microcapsules suitable for thermal energy storage applications by using polystyrene shells. Secondly, the influence of the type and amount of polymer crosslinkers was tested. The fabrication of caprylic acid/polystyrene microcapsules under the same conditions was repeated approximately 50 times in order to produce approximately 1 kg of microcapsules. At the end of production all the microcapsules were mixed and the thermal properties were analyzed.

The rest of the paper is organized as follows: Section 2 describes the experimental process. Section 3 shows the test results, and Section 4 presents the conclusions.

2. Experimental

2.1. Material

Caprylic acid (Merck, Germany, melting point; 15–17 °C) was used as the core material. The characteristics of caprylic acid are shown in Table 1 [2]. Styrene (> 99%, Sigma Aldrich Company, USA) was used as the monomer for the shell material; ethylene glycol dimethacrylate (EGDMA), C₁₀H₁₄O₄, (Merck, Germany) and allyl methacrylate (AMA), C₇H₁₀O₂, were used as the crosslinking agents. Triton X-100 (Merck, Germany) was used as emulsifier. The initiator, which was ammonium peroxodisulphate (Merck, Germany), and other analytical reagents such as *tert*-butylhydroperoxide (70%, Merck, Germany), Triton X-100 (Merck, Germany), iron

Table 1
Characteristics of caprylic acid [2].

Molecular formula	C ₈ H ₁₆ O ₂
Molar mass	144,21 g/mol
Appearance	Clear, viscous liquid
Color	Light yellow
Density	0,91 g/mL(25 °C)
Melting point	15–17 °C

(II) sulphate 7-hydrate (FeSO₄ · 7H₂O) (Panreac, Spain) and sodium thiosulphate (Na₂S₂O₃) (Merck, Germany) were used without further purification. The chemical structures of the EGDMA and AMA are shown in Table 2.

2.2. Method of microencapsulation of caprylic acid in polystyrene shell

All chemicals were used as received from commercial sources without further purification. Polystyrene (PS) shells were prepared by the emulsion microencapsulation technique. Firstly; in a 2-necked flask equipped with a magnetic stir bar and condenser, the 'A' solution (water+caprylic acid+Triton X100), which was prepared by stirring for 30 min at 40 °C, was added. Then the 'B' mixture (styrene+EDGMA+FeSO₄ · 7H₂O solution+Ammonium peroxodisulphate) was added to 'A' and stirring was continued for 60 min at 1000 rpm. Then the 'C' solution (Na₂S₂O₃+*tert*-butylhydroperoxide) was added to the emulsion and heated at 95 °C with stirring for five hours. Then the solution with its polymer microcapsule suspension was cooled to room temperature and washed with water five times. Subsequently the solid microcapsules were filtered off and dried. Table 3 shows the caprylic acid/polystyrene microcapsule (microPCMs) synthesis recipe.

For PCMs with high volatility, especially those that are in the liquid state at room temperature, the desired yield has often not been achieved in the microencapsulation process. Therefore, as it shown in Fig. 2, a condenser was used in the experimental setup. The refluxing condenser connected to the balloon neck was provided to retain volatile PCMs in the emulsion.

2.3. Analysis of microcapsules

The morphology of the microcapsules was characterized by using a scanning electron microscope (SEM, JEOL JSM-6400). All samples were coated with a layer of gold prior to the observation. The melting/crystallization temperatures and melting/crystallization enthalpies of the caprylic acid and caprylic acid/polystyrene capsules were evaluated with a differential scanning calorimetry (DSC) instrument (PerkinElmer, Waltham, MA, USA) at a rate of 5 °C/min in the range of –20 to 50 °C under nitrogen atmosphere. DSC analysis shows the melting temperature (T_m) and solidification temperature (T_s) of the sample and the enthalpy value for each process (melting and solidification, H_m and H_s respectively) which is equivalent to the area under the curve [14]. During DSC analysis, about 5 mg of sample was used. The determination of the phase change material content in the microPCMs followed from the formula:

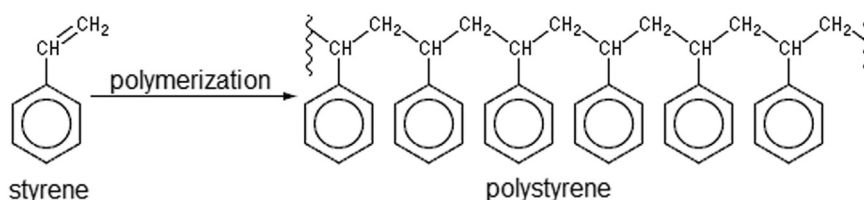


Fig. 1. The reaction scheme of polystyrene.

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