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Microencapsulation of caprylic acid with different wall materials as phase change material for thermal energy storage

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ABSTRACT

In this study, caprylic acid (octanoic acid) suitable for thermal energy storage applications was microencapsulated with different wall materials, including urea-formaldehyde resin, melamine-formaldehyde resin, urea+melamine-formaldehyde resin. Microcapsules were prepared using coacervation method. Hardening process of microencapsulated phase change material (PCM) was done with formaldehyde. The morphology and particle sizes of microencapsulated PCM were analyzed by scanning electron microscopy, (SEM). The latent heat storage capacities of caprylic acid and microencapsulated caprylic acid were determined with differential scanning calorimetry (DSC). The chemical characterization of microcapsules was determined by Fourier transformed infrared (FTIR) spectroscopy. It is concluded that urea-formaldehyde resin was the best capsule wall material for caprylic acid. Based on all results, it can be considered that the microcapsules were synthesized successfully and that, the phase change enthalpies of melting and freezing were about 93.9 J/g and 106.1 J/g, respectively, the particle diameter was 200 nm–1.5 μm.

1. Introduction

Many heating and cooling applications [1,2] benefit from using thermal energy storage in phase change materials (PCMs). What make PCMs very favorable is their isothermal nature and high volumetric capacity. Phase change materials (PCMs) are "Latent" heat storage materials. They use chemical bonds to store and release the heat. The thermal energy transfer occurs when a material changes from solid to liquid, or liquid to solid. This is called a change in state, or phase [3]. Phase change materials (PCMs) are divided into two groups as inorganic and organic. Salt hydrates and clathrate hydrates can be given as examples of inorganic PCM. Paraffin and fatty acids can be shown as example of organic PCM. Fatty acids have more advantageous features compared to many PCMs in use. These are congruent melting, chemical stability and non-toxicity [4,5]. More important features are volume change being less that other PCM during phase change and low melting point. Additionally another advantage of fatty acid is that it is obtained from vegetable and animal oils that provide a continuous supply [6].

Microencapsulation technique has been used in chemical industry for different types of products (fertilizers, pesticides, pharmaceuticals, carbonless paper, scented products, detergents, etc.) since 1930s. The purpose of using microencapsulation in these products is likely to be to achieve controlled release, to protect sensitive materials from their environment or to make active materials easier and/or safer to handle. The ultimate aim for PCM microencapsulation is not only to make PCMs easier and safer to handle but also to reduce reactivity and to improve thermal properties by increasing the heat transfer area [7].

The selection of the capsule wall material for the microencapsulation of PCM plays an important role in regulating the properties of the microcapsules such as morphologies, heat capacities and thermal stabilities [8]. Many methods are developed for microencapsulation including coacervation [9–13], interfacial polymerization [14–16], in situ polymerization [17,18] and suspension-like polymerization [8,19–21].

In different studies, microPCMs containing *n*-octadecane [22–24], *n*-tetradecane [25], *n*-pentadecane [26], paraffin wax [26], *n*-hexadecane [27] were synthesized. Garcia et al. [28] successfully prepared microcapsules of Rubitherm (RT27) with different shell materials using complex coacervation method. Sterilized Gelatine/Arabic Gum and Agar–Agar/Arabic Gum were used as coating materials. Oui et al. [8] microencapsulated *n*-octadecane with different methylmethacrylate-based copolymer shells as phase change materials for thermal energy storage. In recent times some researches have shown interest in fatty acid esters and microencapsulation of fatty acid esters. For example; polyurea microcapsules containing butyl stearate materials were prepared using interfacial polycondensation [29]. Galactitol hexa

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stearate and galactitol hexa palmitate were prepared as novel solidliquid PCM by means of esterification reaction of the galactitol with palmitic acid and stearic acid [30].

As seen from the literature survey above, generally microencapsulation process focused on paraffin microcapsules, but there is little study in the literature on microencapsulation of fatty acids. Because fatty acids have functional group, controlling their microencapsulation process is harder than paraffin and its derivatives. Ozonur et al. prepared coco fatty acid mixture microcapsules by simple and complex coacervation methods for thermal storage. The aim of this study was to make natural coco fatty acid mixtures easier to handle as a potential PCM for thermal energy storage applications to conserve energy in building sector [7].

Caprylic acid is a promising fatty acid that may be used in various fields (building, textile, agriculture, food transportation, etc.) due to its melting point (15–17 °C) and high latent heat storage capacity (158 J/g). Caprylic acid is generally in liquid phase at room temperature and its application becomes difficult under these conditions. Microcapsules ensure the liquid materials to be used as solids. Therefore, no publication seems to be in the literature on this topic. In this study, caprylic acid microcapsules were prepared by using simple coacervation method. The obtained microcapsules were morphologically, chemically and thermally characterized.

Table 1
Characteristics of caprylic acid.

Molecular formula	$C_8H_{16}O_2$
Molar mass	144.21 g/mol
Appearance	Clear, viscous liquid
Colour	Light yellow
Density	0.91 g/ml (25 °C)
Melting point	15–17 °C

2. Experimental

2.1. Material

Caprylic acid (Merck, Germany, melting point; 15–17 °C) core material, 10% urea (Tekkim, Turkey, Germany), 10% melamine (Sigma- Aldrich, USA), 37% formaldehyde (Merck Germany), gum Arabic (not pure, commercial product), and gelatin powder (not pure, commercial product) were used as wall materials in the preparation of microcapsules. 10% NaOH and 10% acetic acid solutions were used to control the pH during polymerization. Three different surfactants, Tween 40 (Merck, Germany), Tween 80 (Merck, Germany) and TritonX-100 (Merck, Germany), were used as emulsifier (E) in this study. The characteristics of Caprylic acid are shown in Table 1.

2.2. Process of microencapsulation PCMs

Caprylic acid was used as the core material of microcapsules. In the preparation of microencapsulated PCMs, simple and complex coacervation techniques were used. Urea-formaldehyde (UF), melamine-formaldehyde (MF), urea+melamine-formaldehyde (UMF) couples were tested in simple coacervation technique and gelatin–gum Arabic (GG) couple was tested in complex coacervation technique to encapsulate the core material. Coacervation technique was prepared by taking the Ozonur et al. (2006) publication into consideration [7]. Before encapsulation core material, caprylic acid was dispersed into a first monomer of shell solution with emulsifier. After 30 min the second monomer of shell solution was added to the prepared dispersion. The mixture was emulsified 2–3 h mechanically at a stirring rate 1000 rpm. The next steps were addition of acid solution for changing the pH, and addition of water for dilution of the emulsion solution. Finally, the prepared microcapsules were stabilized by formaldehyde which is used as crosslinking agent. The used method is schematized in Fig. 1.

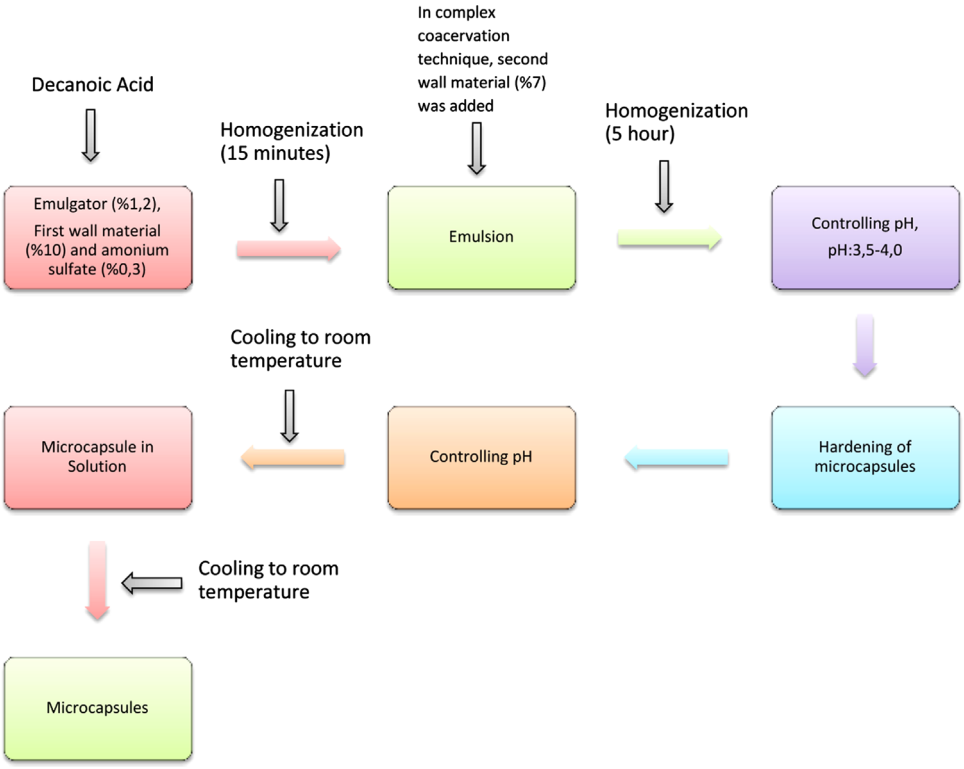

Fig. 1. Schematic formation of the microCA with coacervation method.