



Synthesis and characterization of microencapsulated myristic acid–palmitic acid eutectic mixture as phase change material for thermal energy storage



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HIGHLIGHTS

- Myristic acid–palmitic acid eutectic was microencapsulated with silica shell.
- Structure, morphology of microencapsulated phase change material were investigated.
- Thermal capacity, stability of microencapsulated phase change material were analyzed.
- Silica shell improved thermal stability of microencapsulated phase change material.

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ABSTRACT

In this work microencapsulation of myristic acid–palmitic acid (MA–PA) eutectic mixture with silica shell using sol–gel method has been attempted. The core phase change material (PCM) for thermal energy storage was myristic acid–palmitic acid eutectic mixture and the shell material to prevent the PCM core from leakage was silica prepared from methyl triethoxysilane (MTES). Thermal properties of the microcapsules were measured by differential scanning calorimeter (DSC). The morphology and particle size of the microcapsules were examined by scanning electronic microscope (SEM). Fourier transformation infrared spectrophotometer (FT–IR) and X–ray diffractometer (XRD) were used to investigate the chemical structure and crystalloid phase of the microcapsules respectively. The DSC results indicated that microencapsulated phase change material (MPCM) melts at 46.08 °C with a latent heat of 169.69 kJ kg^{−1} and solidifies at 44.35 °C with a latent heat of 159.59 kJ kg^{−1}. The thermal stability of the microcapsules was analyzed by a thermogravimeter (TGA). The results indicated that the MPCM has good thermal stability and is suitable for thermal energy storage application.

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1. Introduction

Energy conservation with continuous improvement in efficiency is the key to achieving humanity's goal of harvesting and consuming clean energy with minimal environmental pollution. Thermal energy storage is an important function in the field of energy conservation. It serves the purpose of smoothening fluctuations in gap between energy availability and demand for energy [1]. Thermal energy storage comes into play at different temperature ranges. At the high temperature side it is employed in electrical power generation, industrial waste heat recovery etc. At the lower temperature side it is employed in thermal comfort applications like heating and cooling of buildings, thermal comfort textile

etc. Using the latent heat capacity of a class of materials called phase change materials (PCM), to store thermal energy is a common technique due to their high density of energy storage capacity at the required operating temperature range. Phase change materials have certain undesirable properties like poor thermal conductivity and leakage upon melting. Microencapsulation enhances thermal and mechanical properties of phase change materials.

Microencapsulation of phase change materials has been achieved through both organic and inorganic shell materials. Currently organic shell materials are more common than inorganic shell materials. List of organic shell materials include gelatin with gum arabic as binding material [2,3] and polymers like PMMA [4–7], polystyrene [8], melamine–formaldehyde [9–11], urea–formaldehyde [12], polyurea [13], polyurethane [14], etc. There is also organic shell material which is co–polymers made up of combination of more than one organic polymer [15–19].

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Similarly, of the different class of organic phase change materials that are microencapsulated, *n*-alkane (paraffin) class materials are the most commonly used. Paraffin materials are chemically inert and they are insoluble in water which is favorable to microencapsulation process. Few examples of paraffin class of materials which have been microencapsulated in the past include *n*-octadecane [3,9,10,13,17,18], *n*-hexadecane [3,5,19], *n*-nonadecane [3], Rubitherm [4,16], *n*-heptadecane [6,8,19], *n*-eicosane [11], *n*-tetradecane [15,19], *n*-pentadecane [19] etc. Few examples of organic non-paraffin class of materials which have been microencapsulated in the past include xylitol [14], butyl stearate [20] and many fatty acids like capric acid [21], lauric acid [21], myristic acid [21], palmitic acid [22] and stearic acid [23] etc.

Currently there are many different techniques used for microencapsulation with organic shell materials. These include physical methods like spray-drying [2], complex coacervation [2,3] or chemical methods like suspension polymerization [4], emulsion polymerization [5–8], in-situ polymerization [9–12], interfacial polymerization [13] etc.

Organic eutectic mixtures have been microencapsulated in the past, but very rarely. One such rare example of microencapsulation of fatty acid eutectic is Sari et al. [24] in which they microencapsulated capric acid–stearic acid eutectic mixture with an organic shell material PMMA. Organic eutectic mixtures have advantages like sharp congruent melting process. They do not have issues like phase segregation and supercooling. Fatty acid eutectics have advantages like easy availability and favorable melting temperature range for low temperature thermal comfort applications. Therefore microencapsulation of fatty acid eutectic will be of great advantage to thermal energy storage.

Organic shell materials are usually toxic and flammable, and compared to inorganic polymers, they have poor heat transfer performance and thermal stability. Due to these disadvantages of organic shell materials, recently inorganic polymer materials such as silica [23,25–28], titanium dioxide [22,29] etc. have got more attention as shell materials. Most common microencapsulation technique for inorganic shell materials is the sol–gel process [22,23,25–28] which is a chemical method. However there are also few examples of using physical methods like spray-drying [29]. Microencapsulation of inorganic PCM is extremely rare as inorganic PCM are soluble in water. However there are few rare examples of microencapsulating inorganic PCM [30].

Microencapsulated PCM has applications in both passive and active thermal energy storage systems. Their main applications include usage in passive thermal energy storage systems of buildings, textile, automobile interior, medical products etc. They are also used in microencapsulated phase change material slurry for use as heat transfer fluid in active systems [31]. Thermal inertia of the buildings can be increased by incorporating phase change materials. Increased thermal inertia of the buildings results in reduced temperature fluctuations inside buildings when the temperature outside fluctuates during the day and night times. MPCM can be embedded into flooring, drywalls, concrete, ceilings, panels, gypsum boards, insulation panels, wallboards etc. MPCM has an edge over direct impregnation of phase change material, because porous nature of building materials like mortar can lead to leakage issues when the temperature is above the melting point of PCM. Phase change materials like fatty acids are chemically affected by the alkalinity of some concretes due to presence of $\text{Ca}(\text{OH})_2$. Similarly thermo-regulating textiles also use MPCM to improve human thermal comfort in extreme cold weathers [32]. MPCM can also be embedded into polyurethane composite foams used for insulation purpose in automobile interiors and medical storage boxes etc [33]. In HVAC systems for building thermal comfort applications, typical heat transfer fluids like water has a melting point of 0 °C. This is very low to be able to utilize its latent heat for thermal stor-

age. At the human thermal comfort temperature range where most thermal comfort applications operate, water only has sensitive heat capacity which is very low compared to its latent heat. MPCM enhances the heat capacity of water at the required operating temperature and improves heat transfer coefficient. Microencapsulated PCM slurry can serve the purpose of both heat transfer medium [34] and thermal energy storage medium. The direct use of phase change material in PCM slurries can result in clogging of heat transfer ducts due to solidification and agglomeration of PCM at low temperatures. Microencapsulation resolves this problem by avoiding direct contact between different PCM droplets.

In this work sol–gel method is used with myristic acid–palmitic acid eutectic mixture as core phase change material (PCM) and silica prepared from methyl triethoxysilane (MTES) as shell material. Fatty acid eutectics provide us with greater flexibility in adjusting the operating temperature through combination of different pure components. They also have sharp melting feature with almost no supercooling. They are economical and easily available. However currently there still exists a knowledge gap with respect to microencapsulation process of fatty acid eutectic mixtures. Unlike paraffin, fatty acids are chemically reactive due to their acidic nature which can affect the microencapsulation process. Moreover, eutectic PCM microencapsulation has an additional stage of eutectic preparation process which has been covered extensively in this work. This work also generates new FT-IR and XRD characterization data for myristic acid–palmitic acid eutectic mixture. Microencapsulation of fatty acid eutectic with inorganic shell material has never been attempted in the past. In the past works, inorganic silica shell has been synthesized for various PCM materials with compounds like tetraethoxysilane (TEOS) and sodium silicate. In this work a new compound, methyl triethoxysilane (MTES) was used for generating silica shell.

2. Synthesis and characterization

2.1. Materials

Eutectic mixture of myristic acid ($\text{C}_{14}\text{H}_{28}\text{O}_2$, tetradecanoic acid, analytical reagent, Sinopharm Chemical Reagent Co., Ltd.) and palmitic acid ($\text{C}_{16}\text{H}_{32}\text{O}_2$, hexadecanoic acid, analytical reagent, Sinopharm Chemical Reagent Co., Ltd.) was used as thermal storage material. Methyl triethoxysilane (Reagent grade, Tokyo Chemical Industry Co., Ltd.) was used to prepare silica. Anhydrous ethanol (Reagent grade, Sinopharm Chemical Reagent Co., Ltd) and distilled water were used as the solvent. Hydrochloric acid (Reagent grade, Nanjing Chemical Reagent Co., Ltd.) was used to adjust pH value. Sodium dodecyl sulfate (SDS; Reagent grade, Shanghai Chemical Reagent Co., Ltd.) was used as the oil–water emulsifier.

2.2. Preparation of the MA–PA eutectic mixture

Myristic acid (MA) and palmitic acid (PA) were blended together to form eutectic mixture. Following equation was used for theoretically estimating the eutectic point [35].

$$T = \frac{1}{\left(\frac{1}{T_A} - \frac{R \ln x_A}{\Delta H_A}\right)} \quad (1)$$

where T , T_A , x_A , ΔH_A and R represent melting temperature of eutectic mixture, melting temperature of component A, molar fraction of component A, enthalpy of component A and gas constant respectively.

Based on Eq. (1), the melting temperature of the MA–PA eutectic was calculated for different MA–PA mass ratios. Results are shown in Fig. 1. For ease of calculation, the molar fraction has been

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