



Microencapsulated *n*-alkane eutectics in polystyrene for solar thermal applications



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ABSTRACT

Some *n*-alkanes (*n*-tetracosane: C24, *n*-eicosane: C20, *n*-nonadecane: C19, *n*-octadecane: C18, and *n*-heptadecane: C17) and their eutectic mixture have been encapsulated in polystyrene (PS) as phase change materials (PCMs) in order to benefit from solar energy. The eutectic melting points and combination ratios of C17-C18, C20-C17, C20-C19, and C20-C24 eutectic mixtures (EMs) were found out theoretically and proven experimentally before the encapsulation processes. Microencapsulated eutectic mixtures (MEEMs) were structurally proven using a Fourier Transform Infrared (FTIR) Spectroscopy Analysis and morphologically investigated using a Scanning Electron Microscopy (SEM), a Polarized Optical Microscopy (POM), and a Particle Size Distribution (PSD) analyzer. Differential Scanning Calorimetry (DSC) analysis was primarily considered for characterization. MEEMs had reversible and mostly isothermal phase change temperatures between 21.0 and 35.9 °C and enthalpy between 61.2 and 146.1 J/g. These values were intentionally suitable for solar thermal applications. Thermogravimetry Analysis (TGA) was performed to reveal thermal stability limits of the MEEMs. Accelerated thermal cycling experiments were performed to investigate stability of MEEMs.

1. Introduction

Thermal energy storage has a wide range of applications due to potential materials working at different temperatures (Sergio et al., 2015; Zhang et al., 2016). Materials store and release energy upon temperature changes as sensible heat or latent heat. Latent heat thermal energy storage (LHTES) is very popular because of intensive storage density and isothermal operation (Khudhair and Farid, 2004; Xu et al., 2015). Phase change materials (PCMs) have been widely investigated for building (Pasupathy et al., 2008; Baetens et al., 2010; Cabeza et al., 2011; Barreneche et al., 2013; Bouadila et al., 2014; Mirahmad et al., 2016), thermal comfort in textiles (Khan and Pervez, 2015; Tong and Tong, 2015; Liang et al., 2016; Alay et al., 2017), preservation of biological materials (Yeo et al., 2001; Cheng et al., 2011; Gin and Farid, 2010), protection of electronics from overcooling (Ling et al., 2014), among others. Paraffins with isothermal and reversible solid-liquid phase change are widely preferred among all PCMs (Waqas and Din, 2013; Sharma et al., 2015; Karaipekli and Sari, 2016). They have low vapor pressure, considerable thermal durability, and inertness. However they have some handicaps caused by liquid handling problem and they should be encapsulated to prevent seepage during usage (Tang

et al., 2014). *n*-alkanes are encapsulated in macro (Li et al., 2012), micro (Yu et al., 2014; He et al., 2014; Sarier et al., 2015; Qiu et al., 2015; Sari et al., 2016), or even nano sizes (Tang et al., 2013; Barlak et al., 2015; Sari et al., 2015; Konuklu et al., 2015a,b; Mohammadi et al., 2016). The micro or nano encapsulation of PCMs caused some property developments like free of liquid handling problem due to decreased interference to close environment, resistance to volume change, improvement in heat conduction due to extended surface area, non permeability, and improved stability (Konuklu et al., 2015a,b; Giro-Paloma et al., 2016; Figueiredo et al., 2016; Zhao et al., 2016; Haurie et al., 2016) in general. The encapsulated PCMs have generally been incorporated to fibers, fabrics, coatings, and foams (Liang et al., 2016; Alkan et al., 2015; Siddiqui and Sun, 2015). Besides they have also been used in some special applications in insulation panels and buildings (Borreguero et al., 2010; Su et al., 2012; Jin et al., 2013) and electronic equipments (Rokni et al., 2015). Microencapsulated PCMs (MEPCMs) are prepared by various methods (Yang et al., 2013; Malekipirbazari et al., 2014; Simón et al., 2014; Morita et al., 2015; Yin et al., 2015; Pan et al., 2015; Latibari et al., 2015). Synthesis by means of emulsification is the most widely used among all because of simplicity, cheapness, technical feasibility, eco-friendly, and reproducible

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(Shirin-Abadi et al., 2011; Alkan et al., 2011).

Polymeric shells are generally prepared with formaldehyde and isocyanide ingredients in order to be resistant to outdoor conditions (Palanikkumaran et al., 2009; Mohaddes et al., 2014) inspite that there were remnant toxic residuals after the synthesis. There are some other polymer based encapsulants (Zhang and Wang, 2009; Bayés-García et al., 2010; Alay et al., 2011; Qiu et al., 2012, 2013; Sari et al., 2015). Polystyrene (PS) is a cost effective, transparent and lightweight thermoplastic compared to other inorganic and organic microcapsule shell materials. It is used for mechanical and thermal protection at large amounts. It is well known that the monomer of PS, styrene is cheap and easily available in the market. PS is cheap and therefore a cost effective precursor of new materials.

Synthesis of *n*-alkane core MEPCMs is an up to date issue because of the advantages of *n*-alkanes like isothermal working and phase change variability according to the molecular weight. However, *n*-alkanes are sold in a wide range of costs and some are very expensive. In spite of temperature variability, there are still no possible pure materials for any of desired working temperatures. Therefore, alternative means should be developed for economy and for working temperatures possibility. Eutectic mixtures (EMs) of *n*-alkanes could make it possible to reach more cost effective blends than pure compounds with desirable phase transition temperatures and satisfactory enthalpy. For this reason, *n*-heptadecane (C17), *n*-octadecane (C18), *n*-nonadecane (C19), *n*-eicosane (C20), and *n*-tetracosane (C24) were blended at eutectic combinations to serve as new PCMs. Then the produced eutectics were encapsulated in PS shell for the first time in this work. Microencapsulated eutectic mixtures (MEEMs) produced in this work are called as PS/(C17-C18), PS/(C20-C17), PS/(C20-C19), and PS/(C20-C24).

2. Experimental

2.1. Materials

C17, C18, C19, C20, and C24 were purchased from Merck Company and used as received. Triton X100 (Sigma Aldrich), ammonium persulfate (Sigma Aldrich) and ferrous sulfate heptahydrate (Sigma Aldrich) were used as received. Styrene, divinylbenzene (DVB) were purchased from Sigma Aldrich company and NaOH solution (10 wt/v%) were used for inhibitor purification.

2.2. Preparation of EMs

Eutectic composition ratios and phase change temperatures were calculated according to the Schröder Equation (Kauranen et al., 1991) for prepared EMs. The eutectic points of C17-C18, C20-C17, C20-C19, and C20-C24 blends were experimentally determined by using DSC measurements on mixtures at various composition ratios. Single melting and crystallization temperatures in DSC curves were accepted as an evidence of eutectic point and ratio.

2.3. Synthesis of the MEEMs

The MEPCMs have been prepared according to the literature (Sari et al., 2010; Alkan et al., 2011). The method is called as miniemulsion process which is a slightly modified version of emulsion polymerization. The monomer was previously mixed with core materials and homogeneously distributed in water phase using a suitable surfactant and then the system was maintained at polymerization temperature for several hours. The yield of encapsulation was considerably higher than suspension method. The MEEM with 2/1 polystyrene/eutectic *n*-alkane shell/core ratio was given as an example for the preparation. In this example, firstly, 10 g styrene and 2 g divinylbenzene monomers were mixed vigorously with 0.01 M 50 mL NaOH in water solution in a separatory funnel. The organic phase was decanted to the reactor filled

with 40 mL deionised water directly. Secondly, 5 g of eutectic *n*-alkane mixture (2/1) and surface active material were added to the reactor and heated to 50 °C. The mixture was homogenised with 10,000 rpm mixing for 60 min. As a third, initiators (1 mL solution of FeSO₄ and NH₄S₂O₇ in water) were added and the system was maintained as mixing at 500 rpm and 70 °C for 6 h. At the end of the reaction, MEEM was cooled to 25 °C, filtered off through a filter paper and then washed for 3 times with distilled water.

The MEEMs were prepared with 3/1, 2/1, 1/1, and 1/2 shell/core ratios. The various shell/core ratios were studied because the core content of the MEPCMs is important for thermal storage capacity and shell/core ratio is reversibly proportional to mechanical property. The real encapsulation ratios were calculated according to the phase change enthalpy of the MEEMs over those of EMs determined by Differential Scanning Calorimetry (DSC) analysis.

2.4. Characterization of MEEMs

The shell formation of the MEEMs through emulsion polymerization was investigated on KBr disks using a Jasco 430 model FT-IR spectroscopy instrument. Leica model Polarised Optical Microscopy (POM) and Leo 440 model Scanning Electron Microscopy (SEM) instruments were used for morphology investigation. The particle size and uniformity of the MEEMs were determined using a Malvern Mastersizer 2000 model particle size analyzer instrument. Particle size analyses of MEEMs were suspended by Triton X-100 prior to analysis. Thermal properties of the MEEMs were determined by using a Perkin Elmer Jade model DSC instrument with a heating-cooling ramp of 3 °C/min. Further analyses to prove life time (reliability) of the MEEMs was done by FT-IR spectroscopy and DSC before and after accelerated thermal cyclings (heating/cooling operations of 5000 times). The accelerated heating/cooling was carried in a BIOER TC-25/H model thermal cyler. The thermal stability of the MEEMs was carried out using a Perkin Elmer TGA7 model Thermogravimetric Analyzer (TGA) instrument. A 10 °C/min heating ramp was applied to any of the samples in nitrogen atmosphere. Thermal conductivity of the MEEMs was determined using a Decagon KD2 model thermal property analyzer.

3. Results and discussion

3.1. Evaluation of DSC thermograms of the EMs

One important selection criteria of a PCM is its application range which depends on the type of the LHTES system. Suitable working temperature ranges of textile and building applications are 18–35 °C and 19–26 °C, respectively. Therefore eutectic compositions working in these ranges were chosen (the eutectic composition ratios were firstly theoretically calculated and verified experimentally by considering the DSC thermograms of the components at different combination ratios). Fig. 1 represented DSC thermograms of C17-C18, C20-C17, C20-C19, and C20-C24 mixtures in which eutectic compositions of each material had only one phase transition. In the thermograms the small peaks at the left of the melting curves were due to solid phase transition of *n*-alkanes. The small former peaks were due to solid–solid phase change of the C20-C19 and C20-C24 mixtures as the latter large ones were assigned to the solid–liquid phase change of the eutectic mixture. C17-C18, C20-C17, C20-C19, and C20-C24 eutectic mixtures were formed with the composition ratios of 17/83, 20/80, 20/80, and 90/10 respectively with solid–liquid phase change temperatures of 25.6, 21.3, 31.9, and 35.8 °C, respectively.

DSC heating/cooling curves of C17-C18, C20-C17, C20-C19, and C20-C24 EMs were shown all together in Fig. 2 for comparison. The eutectic composition ratios and the LHTES properties of pure *n*-alkanes and their eutectic mixtures were also tabulated in Table 1. As seen from the Table, the latent heat temperatures and capacities of C17-C18, C20-C17, C20-C19, and C20-C24 eutectic mixtures were in the range of

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