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# Micro/nano-encapsulated *n*-heptadecane with polystyrene shell for latent heat thermal energy storage

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ABSTRACT

This study is focused on the preparation, characterization and determination of latent heat thermal energy storage (LHTES) properties of polystyrene (PS)/*n*-heptadecane micro/nano-capsules as a novel encapsulated phase change material (EPCM). The micro/nano-EPCM was synthesized via emulsion polymerization method and characterized chemically using Fourier transform infrared (FTIR) spectroscopy. The surface morphology and particle size of the micro/nano-capsules were investigated by particle size distribution (PSD) analysis, polarized optical microscopy (POM) and scanning electron microscopy (SEM) techniques. From differential scanning calorimetry (DSC) analysis, the melting temperature and latent heat of the prepared EPCM were measured as 21.48 °C and 136.89 J/g, respectively. The results of thermogravimetric (TG) analysis showed that the fabricated micro/nano-EPCM had good thermal durability. Thermal reliability, chemical stability, thermal conductivity and phase change reversibility of the micro/nano-EPCM were also studied. All of the results revealed that the fabricated PS/*n*-heptadecane micro/nano-capsules had promising LHTES potential especially for passive solar thermal regulation of textile, building, food storage container, medical and electronic materials. © 2014 Elsevier B.V. All rights reserved.

## 1. Introduction

Phase change materials (PCMs) are very alluring materials for solar passive LHTES applications because of their capabilities to store high latent heat per unit volume via phase change at a nearly invariable temperature [1]. PCMs are classified basically into two groups as organic and inorganic materials [2]. Organic PCMs are basically known as paraffin and non-paraffin. Paraffins, i.e, *n*-alkanes are chemically inert and reliable, non-corrosive, nontoxic and commercially accessible at rational cost. These kinds of PCMs have also no phase segregation, low vapor pressure, little or no sub-cooling and good thermal durability properties, and high latent heats of fusion and proper solid-liquid phase change temperatures for many LHTES applications [3-7]. However, they have some drawbacks such as low thermal conductivity, flammability and high volume change during phase change [8-10]. Moreover, inorganic PCMs have large high latent heat capacity and good thermal conductivity, non-flammable and low-cost properties in comparison with organic PCMs. On the other hand, they are corrosive and show unstable phase change behaviors and high sub-cooling degrees, which

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can appreciably affect their LHTES characteristics. Therefore, these problems regarding organic and inorganic PCMs can be overcome substantially by their encapsulation into a capsule matrix in macro-[11–13], micro- [14–16] and even nano-sizes [17,18]. The encapsulated PCM (EPCM) is described as a very small bicomponent system consisting of a core material (PCM), and an outer shell or capsule wall material. Besides, the use of EPCM helps dramatically to solve the problems mentioned above; it provides some advantages ordered as [19–23]: (i) lessening the reactivity of PCM with its close environment, (ii) expanding the heat transfer area, (iii) preventing the leakage problem of PCM during its phase changing from solid to liquid, and (iv) resisting against the volume change of PCM. Therefore, in recent decades, the utility of EPCMs have drawn attention in the preparation of thermal regulating fabric [24–29], foam [30,31], building [32–35] and electronic materials [36]. Recent investigations have been generally focused on the development of a cheap and technically feasible process to be used in the fabrication of organic EPCMs. The most common methods employed to produce organic EPCMs are complex coacervation, interface, in-situ and emulsion (or miniemulsion) polymerization methods [37]. Among these, miniemulsion polymerization is a simple, cheap, stable and ecofriendly method for the preparation of EPCMs with appropriate particle sizes [38].

In recent years, several works have been realized about the encapsulated paraffin wax or *n*-alkanes as EPCMs using various polymer shells such as melamine–formaldehyde resin [39,40], urea–formaldehyde resin [41,42], polyurea [43–45], and acrylicbased polymers [46–50]. Among these, especially melamine– formaldehyde and urea–formaldehyde resins-based EPCMs may contain irrepressible residue formaldehyde and isocyanides after shell forming reaction, which have negative effects on the environment and health. It is also hard to find useful ways to eradicate the wastes that are dissociative and can be incessantly released from the products.

Polystyrene (PS) is one of the aromatic polymers made from monomer styrene in the chemical industry. It is very inexpensive and therefore commonly used in several trading applications [51,52]. Polystyrene foams are also suitable for the insulation of building concrete materials and panel systems. It has also reasonably good mechanical and protective properties against the exterior environment. All of these advantages have facilitated it to be considered as shell material in the fabrication of new EPCMs [53-56]. Therefore, the utilization scale of PS can be extended by its usage in different passive solar LHTES systems. n-Heptadecane known as a hydrocarbon with linear chain has a desirable melting temperature of about 21 °C and relatively high latent heat of fusion (about 216 J/g). By taking into account the literature survey, it can be noted that there is no study about the preparation and thermal characterization of micro/nano-encapsulated *n*-heptadecane using PS shell. With this purpose, the present work was focused on the synthesis of PS/n-heptadecane micro/ nano-capsules as novel EPCM using emulsion polymerization method and chemical characterization by FT-IR spectroscopy technique. The morphology and particle size of the fabricated EPCM were investigated using SEM, POM and PSD analyses. The LHTES properties, thermal reliability and thermal durability were determined using DSC and TG techniques. In addition, the chemical stability,

thermal conductivity and phase change reversibility properties of the prepared EPCM were studied.

# 2. Experimental

# 2.1. Materials

The chemicals used in the synthesis of micro/nano-capsules include monomer styrene (Sigma-Aldrich Company), *n*-heptadecane (Merck Company), divinylbenzene (Merck Company), Triton X100 (Merck Company), and ferrous sulfate heptahydrate and ammonium persulphate (Sigma-Aldrich Company). The monomer styrene was washed three times with an aqueous solution of 10 wt% NaOH prior to use. The other chemicals were used without further purification.

### 2.2. Preparation of the PS/n-heptadecane micro/nano-capsules

PS/*n*-heptadecane micro/nano-capsules were synthesized using the experimental set-ups shown in Fig. 1(a and b). For the preparation of PS/*n*-heptadecane(2:1) micro/nano-capsules, the initial weight ratio of styrene monomer/*n*-heptadecane was selected as about 67%/33% (w/w). According to this ratio, 6.7 g monomer and 2 g divinylbenzene as cross linking agents were mixed with 40 mL deionized water in the three-necked glass reactor (Fig. 1a). 3.3 g *n*-heptadecane in melted state was added to the reactor. This mixture was homogenized using a homogenizer at 12,000 rpm for 15 min. Then, 3 g Triton X-100 surfactant was added to the mixture and the solution was re-homogenized at 50 °C for 60 min at the same homogenization rate. In the next step, 1 mL initiator (freshly prepared by solving ferrous sulfate heptahydrate (0.15 g) and ammonium persulphate (0.1 g) into 100 mL deionized water) was



Fig. 1. Photograph images of experimental apparatus used in the synthesis processes and the obtained final product.

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