

# Preparation and properties of myristic–palmitic–stearic acid/expanded graphite composites as phase change materials for energy storage

Xiaojiao Yang, Yanping Yuan<sup>\*</sup>, Nan Zhang, Xiaoling Cao, Cheng Liu

*School of Mechanical Engineering, Southwest Jiaotong University, 610031 Chengdu, PR China*

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## Abstract

Based on theoretical calculation, myristic acid–palmitic acid–stearic acid ternary eutectic mixture (MA–PA–SA) with a mass ratio of MA:PA:SA = 52.2:29.4:18.4 was prepared firstly. Then, the MA–PA–SA/expanded graphite (EG) composite phase change material (PCM) with an optimum mass ratio of MA–PA–SA: EG = 13:1 was fabricated. The prepared MA–PA–SA and MA–AP–SA composite PCM are characterized by the scanning electron microscope (SEM), Fourier transformation infrared spectroscopy (FT-IR), differential scanning calorimetry (DSC) and thermogravimetry analyzer (TG). The SEM and FT-IR results showed that the MA–PA–SA was uniformly adsorbed into the network porous structure of EG. The DSC results indicated that the melting and freezing temperatures and latent heats of MA–PA–SA/EG composite PCM were 41.64 °C and 42.99 °C, and 153.5 J/g and 151.4 J/g respectively. TG analysis test revealed that the prepared MA–PA–SA/EG composite PCM has a high thermal stability in working temperature range. Thermal cycling test result showed the melting and freezing temperatures and latent heats of the prepared composite PCM changed by 0.28 °C and 0.48 °C, and –1.63% and –1.32% respectively after 1000 thermal cycles. The thermal conductivity of MA–PA–SA/EG composite PCM was improved by the high thermal conductivity of the EG. All results indicated that the prepared MA–PA–SA/EG composite PCM has proper phase change temperature, high latent heat and thermal conductivity, and good thermal reliability and stability for thermal energy storage in solar heating, waste heating recovery systems and other potential applications.

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**Keywords:** Fatty acids; Ternary eutectic mixture; Composite phase change material; Thermal properties

## 1. Introduction

Latent heat thermal energy storage (LHTES) by using phase change materials (PCMs) is considered to be the most potential way to solve the energy shortage problem. Thermal energy can be stored/released during the melting/freezing process of PCMs. The storage of thermal energy can realize the control of environmental temperature and match the energy supply and demand in time and space (Nallusamy et al., 2007; Karaiepli and Sari,

2009; Wang et al., 2012). The application of PCMs in LHTES are now under the spotlight in many fields such as low temperature refrigeration, heat of condensation recovery, building energy saving engineering and solar energy thermal storage (Diaconu et al., 2010; Mondal, 2008; Gu et al., 2004; Kenisarin and Mahkamov, 2007; Sharma et al., 2002; Neeper, 2000; Khudhair and Farid, 2004).

At present, inorganic salt hydrates, paraffin waxes and fatty acids have been widely studied as PCMs for LHTES applications (Ting et al., 1997; Xu and Li, 2013; Sari, 2003; Rozanna et al., 2004). Among the investigated PCMs, fatty acids were considered as potential PCMs because of the

<sup>\*</sup> Corresponding author. Tel.: +86 13880871068; fax: +86 28 87634937.  
E-mail address: [ypyan@home.swjtu.edu.cn](mailto:ypyan@home.swjtu.edu.cn) (Y. Yuan).

advantages of low supercooling, high heat capacity, good thermal and chemical stability, small volume change, self-nucleating behavior, non-toxic, etc. (Yuan et al., 2014). Moreover, fatty acid esters and binary and ternary eutectic mixtures of fatty acids with different phase change temperatures can be obtained, and they expand the application of fatty acids as PCMs (Yuan et al., 2011). Capric acid–palmitic acid eutectic mixture was used as PCM to prepare the phase change gypsum wallboard as novel phase change wallboard for latent heat thermal energy storage (Karaïpekli and Sari, 2007). Capric acid, lauric acid, myristic acid and stearic acid were selected to prepare binary fatty acid eutectic for the sake of decreasing phase change temperature. form-stable PCMs of PMMA as supporting material and binary fatty acid eutectic as the heating–absorbing materials were prepared for building energy conservation (Wang and Meng, 2010). Based on the calculated mixing proportions of the six binary eutectic mixtures of CA, LA, PA and SA, Li et al. (2011a), via phase diagram thermodynamic method, prepared such six mixtures which were verified later via the absorption curve and DSC curve. The six binary eutectic mixtures showed a phase change temperature range of 21.1–55.0 °C. Cai et al. (2012) prepared five binary eutectic mixtures of LA–MA, LA–PA, MA–PA, MA–SA and PA–SA whose melting and temperatures ranged from 33.27 °C to 53.69 °C; and freezing temperatures ranged from 33.71 °C to 53.45 °C. Afterwards, Cai et al. synthesized stable-form fatty acid eutectics/PET composite PCMs with the five eutectic mixtures and polyethylene terephthalate (PET) by means of the electrospinning technique. Zhang et al. (2013), on the basis of theoretical calculation, prepared a LA–MA–PA ternary eutectic mixture with the phase change temperature of 31.41 °C and latent heat of 145.8 J/g. And LA–MA–PA/EG composite PCM for low temperature thermal storage was fabricated.

In spite of many desirable properties of fatty acids, the disadvantage of low thermal conductivity limits their application. To overcome the problem, various modified methods to enhance the thermal conductivity of PCMs have been investigated. The main methods are concentrated in inserting fins, adding particles with high thermal conductivity and incorporating porous structure materials (Agyenim et al., 2009; Zhang et al., 2006; Wang et al., 2010; Li, 2013; Karaïpekli et al., 2007). Expanded graphite is a kind of light carbon material featuring porous structure and a high thermal conductivity. It is always used as the supporting material to increase the thermal conductivity of PCMs without much reduction in the latent heat energy storage capacity (Sari and Karaïpekli, 2009; Zhao et al., 2011).

This research is focused on a novel potential PCM for thermal energy storage in solar heating, waste heating recovery systems. Myristic–palmitic–stearic acid ternary eutectic mixture (MA–PA–SA) with heating and freezing temperature of 41.72 °C and 42.38 °C was prepared. Then, MA–PA–SA/EG composite PCM used for thermal energy storage in solar heating, waste heating recovery systems

was prepared. The microstructure and thermal properties of MA–PA–SA/EG composite PCM were characterized by scanning electron microscope (SEM), Fourier transformation infrared spectroscopy (FT-IR) and differential scanning calorimetry (DSC). Afterwards, the thermal reliability and stability and thermal conductivity of the composite PCM were also investigated.

## 2. Experimental

### 2.1. Materials

Myristic acid (MA, 98% pure), Palmitic acid (PA, AR) and Stearic acid (SA, 98% pure) were obtained from Aladdin Industrial Corporation, Shanghai China. Expandable graphite (80 meshes, expansion coefficient: 200 mL/g, Carbon content: 99%) was supplied by Jinrilai Electronic Materials Factory, Qingdao, China.

### 2.2. Preparation of MA–PA–SA ternary eutectic mixture

A certain amount of MA, PA and SA were weighted and put in a 100 ml beaker, sealed by a piece of plastic wrap, and then heated in a thermostatic water bath at 80 °C. When the three fatty acids melted completely, they were stirred in the magnetic stirrer at 400 rpm/min for 30 min to make sure a homogeneous mixing and then cooled down to the room temperature. Afterwards, the mass ratio, phase change temperature and latent heat of the mixture were measured by DSC.

### 2.3. Preparation of MA–PA–SA/EG composites

Expanded graphite (EG) was prepared by microwave treatment using a microwave oven at a microwave irradiation power 700 W for 30 s. A series of MA–PA–SA/EG composite PCMs were prepared by using the obtained MA–PA–SA as the PCM and EG as the supporting material. EG was put into ten 50 ml beakers with 0.2 g for each beaker, then MA–PA–SA weighing differently was placed on EG in the ten beakers respectively. The beakers were sealed with the plastic wrap and put in the oven for 24 h at 65 °C, during the process of which, the sample in each beaker was stirred every 8 h to make sure a homogenous absorption of EG to MA–PA–SA. Afterwards, the samples were cooled down to the room temperature.

### 2.4. Characterization

The melting temperatures and latent heats of PA, SA, MA–PA–SA eutectic mixture and MA–PA–SA/EG composite PCM were obtained by using a differential scanning calorimeter (DSC, TA Q20 USA) at 5 °C/min under a constant stream of argon at a flow rate of 50 ml/min. DSC instrument was calibrated with indium as a standard reference material, and the accuracy of enthalpy measurement was  $\pm 4\%$ . The morphology and microstructure of the EG

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