Energy Conversion and Management 69 (2013) 116–121

Contents lists available at SciVerse ScienceDirect

Energy Conversion and Management

journal homepage: www.elsevier.com/locate/enconman

The effects of compounding conditions on the properties of fatty acids eutectic mixtures as phase change materials

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ARTICLE INFO

Article history:
Received 10 November 2012
Accepted 31 January 2013
Available online 6 March 2013

Keywords:
Phase change material
Stearic acid
Myristic acid
Eutectic mixture
Heating operation
Ultrasonic vibrating operation

ABSTRACT

This work was focused on investigating the effects of compounding conditions on the properties of fatty acids eutectic mixtures as phase change materials (PCMs), and the binary eutectic mixtures of stearic acid (SA) and myristic acid (MA) were selected as representative. The melting points of SA–MA mixtures with varying combination proportions were tested to determine eutectic ratio. Adopting heating-ultrasonic method, the SA–MA mixtures with eutectic ratio were blended under different conditions to obtain a series of eutectic mixtures, and then their thermal properties and chemical structures were analyzed by differential scanning calorimetry (DSC) and Fourier transform infrared (FT-IR), respectively. The results showed that the eutectic mass ratio of SA to MA was 36–64%; within certain time range, more heating time can result in lower melting point, but not the longer, the better; when the heating temperature increased, the melting point of eutectic mixture had a little irregular change, but the melting time of solid mixture obviously reduced, so, heating temperature should be considered synthetically to save energy; ultrasonic vibrating process hardly had influences on the properties of eutectic mixtures, and can be canceled; the preparation of fatty acids eutectic mixture did not have any damages to the chemical structures of fatty acids.

1. Introduction

With the rapid development of urbanization and the aggravation of energy shortage in China, building energy conservation is becoming more and more important. Traditional insulation materials were and are being used in thicker or multiple layers as the thermal-protective coating of building, but adversely affecting building details, net-to-gross floor area and load bearing constructions [1]. Phase change materials (PCMs) can make up for these defects of traditional insulation materials. The main advantage of PCMs is to store heat energy in a latent form, resulting in greater heat storage capacity per unit volume than that of conventional building materials [2–9]. For example, when the ambient temperature rises, solid PCMs can melt into liquid, absorbing heat; as the ambient temperature drops again, liquid PCMs will turn to the solid state, releasing the absorbed heat. Therefore, PCMs can effectively decrease the indoor temperature fluctuations, reducing energy consumption and the cost of Heating, Ventilation and Air Conditioning system [10–12].

Fatty acids, as PCMs for the energy storage, have attracted more and more research interests in recent years, due to small volume variation during the phase change, high fusion latent heat, good chemical stability, good thermal stability, nonflammability, low corrosion, non-toxicity and low cost [13–15]. Nevertheless, most unitary fatty acids cannot be used directly to regulate room temperature, because of their higher melting temperatures. Therefore, according to the ideal solution model, many researchers have prepared binary or ternary fatty acids eutectic mixtures with low melting temperatures by blending two or three kinds of fatty acids [16–30].

In general, heating-ultrasonic method was used to compound the eutectic mixtures of fatty acids. But, for different research groups, there were great differences in practical compounding conditions (temperature and time), or even some groups did not provide detailed compounding conditions. In order to prepare the binary fatty acids of capric–lauric acid (CA–LA), capric–palmitic acid (CA–PA), capric–stearic acid (CA–SA), LA–PA, LA–SA and PA–SA, Li et al. [18] put the samples in a water bath with constant temperature of 70 °C for complete melt, and then placed them in an ultrasonic cleaner for dispersion. Focused on the preparation of CA–LA, CA–SA, CA–myristic acid (MA) and LA–MA eutectics, Wang and Meng [19] kept two fatty acids in the drying oven at 80 °C for 2 h, and then vibrated them in an ultrasonic cleaner at 50 °C for 2 min. Gao and Qian [20] melted CA and MA at 90–100 °C, maintained them at 51 °C for 30 min, and vibrated them in an ultrasonic cleaner for 2 min. Sarı et al. [21–27] just melted the selected fatty

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0196-8904/$ - see front matter © 2013 Elsevier Ltd. All rights reserved.
http://dx.doi.org/10.1016/j.enconman.2013.01.026

acids (CA–PA, LA–MA, LA–PA, LA–SA, MA–PA, MA–SA and PA–SA) and stirred them for about 10 min to ensure the homogeneity of the mixture. The compounding conditions in the research work of Jiao et al. [28], Yao et al. [29] and Li et al. [30] were not presented in detail.

Obviously, the production cost of binary or ternary fatty acids as PCMs would be influenced directly by the practical compounding conditions. Higher compounding temperature and longer compounding time inevitably consume more energy, leading to the increase of cost. Hence, it is very significant to investigate the effects of compounding conditions on the properties of fatty acids eutectic mixtures, however, few researches on this field have been found in the existing literature.

SA and MA are common fatty acids, and are often used as raw materials to prepare fatty acids eutectic mixtures. In this study, using heating-ultrasonic method, binary eutectic mixtures of SA and MA, as representative of fatty acids eutectic mixtures, were prepared under different conditions, and the thermal properties and chemical structures of these binary eutectic mixtures were tested. Based on the test results, the effects of heating temperature, heating time, ultrasonic vibrating temperature and time on the properties of SA–MA eutectic mixture were investigated, and the corresponding influence mechanisms were discussed. In addition, the research conclusions about SA–MA binary eutectic mixture were promoted into general rules about fatty acids eutectic mixtures. This research would make up for the vacancy of existing research, and provide some theoretical basis and application reference for the future works about compounding binary or polynary eutectic mixtures of fatty acids as PCMs.

2. Materials and methods

2.1. Materials

Stearic acid (SA, 98.0% pure) was purchased from Sinopharm Company (Beijing, China). Myristic acid (MA, 98.0% pure) was purchased from Kermel Company (Tianjin, China). And the basic properties of SA and MA are listed in Table 1.

2.2. Methods

2.2.1. Preparation of fatty acid mixtures

Heating-ultrasonic method was adopted to prepare fatty acid mixtures. SA and MA were weighed according to certain proportions, and were heated in the water bath at designated temperatures for designated times to obtain liquid mixture. The liquid mixture was put in an ultrasound cleaner (KQ3200E, Kun Shan Ultrasonic Instruments Co., Ltd.) at certain temperatures for certain times, and then was cooled to room temperature.

2.2.2. Characterization

Differential scanning calorimetry (DSC 822^e, Mettler) was used to test the thermal properties of fatty acid mixtures. The heating rate was 5 °C/min in a constant stream of nitrogen. The melting temperature was obtained by drawing a line at the point of maximum slope of the leading edge of the DSC peak and extrapolating the base line on the same side as the leading edge of the peak. The latent heat of fusion was calculated as the area under the peak by numerical integration.

Fourier transform infrared (FT-IR) spectrophotometer (Avatar370) was employed to study the chemical structures of fatty acid mixtures. FT-IR spectra were taken on a KBr disk at a frequency range of 4000–400 cm^{-1}.

3. Results and discussion

3.1. The determination of the eutectic mass ratio of SA and MA

A series of the binary systems of SA and MA in different combination proportions were prepared. The solid samples, which were weighed within ±0.2 mg, were melted and stirred at 85 °C for 45 min, and then the liquid mixtures were vibrated for 2 min. After cooling to room temperature, in order to determine the eutectic ratio, melting temperatures of these binary mixtures were measured by DSC, and the results are shown as Fig. 1. It can be seen that the melting point of SA was reduced by the addition of MA; meanwhile, the melting point of MA was decreased by the introduction of SA; however, both fatty acids melted simultaneously at 44.72 °C, where the mass ratio of SA to MA was 36–64% known as the eutectic mass ratio, which was accord with the data of Sarı [21].

3.2. The effect of heating time on the thermal properties of the eutectic mixture

Five samples weighed according to the eutectic proportion of SA and MA were melted at 70 °C, and maintained for 5 min, 15 min, 25 min, 35 min and 45 min, respectively, without ultrasonic vibrating. The thermal properties of eutectic mixtures were measured by DSC as shown in Fig. 2. It can be seen that their peak shape and position changed little with the variation of heating time, except for the eutectic mixture with heating time of 5 min. There were two peak values in the DSC curve of the eutectic mixture heated for 5 min, meaning that 5 min was too short to mix SA and MA completely. In addition, Fig. 3 shows that when heating time increased from 5 min to 45 min, the melting point of eutectic mixtures decreased from 45.59 °C to 44.24 °C, and the rate of decrease before 15 min was faster than that after 15 min which was very slow. This indicated that more heating time would lead to more fully blending, and in order to obtain uniform mixture, the heating time should not be less than 15 min. Therefore, for preparing fatty acids eutectic mixtures, when the heating temperature was enough, it was not the longer heating time, the more efficient mixing, and too long heating time (2 h for example) just wasted energy. The best time need to be determined roughly by simple experiments.

3.3. The effect of heating temperature on the thermal properties of the eutectic mixture

Because SA began to slowly volatilize as the ambient temperature was higher than 90 °C, while the solid mixture of SA and MA was very difficulty to melt as the ambient temperature was below 60 °C. The heating temperatures were selected as from 60 °C to 85 °C.

Six mixtures of SA and MA with eutectic mass ratio were melted at 60 °C, 65 °C, 70 °C, 75 °C 80 °C and 85 °C, respectively, and then

Table 1
The basic properties of SA and MA.

Sample	Molecular formula	Molecular weight	Melting point (°C)	Latent heat of fusion (J/g)
Stearic acid (SA)	$C_{18}H_{36}O_2$	284.47	65.57	246.6
Myristic acid (MA)	$C_{14}H_{28}O_2$	228.38	50.27	229.9