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Research Article

Preparation and Performance of Capric-Myristic Acid Binary Eutectic Mixtures for Latent Heat Thermal Energy Storages

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The capric-myristic acid (CA-MA) binary eutectic mixture phase change material (PCM) was prepared for low-temperature latent heat thermal energy storage (LHTES). The thermal properties, thermal stability, and long-term cycling reliability of the PCMs were measured. Differential scanning calorimetry results showed that the CA-MA binary eutectic mixture at the mass ratio (72/28 wt%) indicated a high-performance PCM for its suitable phase change temperature (T_m: 18.21°C, T_f: 17.40°C) and high latent heat (H_m: 148.5 J/g, H_f: 134.0 J/g). Thermal gravimetric analysis results indicated that the CA-MA binary eutectic mixture had excellent thermal stability in its operating temperature range. The thermal cycling tests and Fourier transform infrared spectroscopy results revealed that the CA-MA binary eutectic mixture had good long-term cycling thermal chemical reliability. In summary, in terms of thermal property, thermal stability, and reliability, the prepared CA-MA PCM could be applied particularly for low-temperature LHTES systems and backfill materials of ground source heat pump systems.

1. Introduction

Thermal energy storage technologies have elicited increasing attention due to its broad application prospects in the fields of solar energy utilization, electric power peak-load shifting, industrial waste heat recovery, building heating, and air conditioning [1–5]. Latent heat thermal energy storage (LHTES) has elicited wide attention and application because of its larger storage energy density, less temperature change during energy storage, better stability, and higher safety compared with other methods [6, 7].

The core technology of LHTES is phase change material (PCM). Various inorganic, organic, and mixed PCMs, such as paraffin [8, 9], polyols [10, 11], inorganic salts [12], and fatty acids [13], have been studied in building energy conservation. Among these materials, fatty acid is one of the most relevant organic PCMs because of its several advantages, such as large latent heat, nontoxicity, suitable phase change temperature, zero corrosion, low degree of subcooling, no or minimal volume change, and good thermal stability [14, 15]. Furthermore, fatty acids can be extracted from oils, animal fat, and plants; thus, the raw materials are abundant and readily available [16, 17]. In addition, a low phase change temperature may be achieved by mixing two or more fatty acids on the basis of their eutectic effect [18–20]. Thus, the phase change temperature can be controlled by selecting an appropriate eutectic system formed by different fatty acids for several engineering applications.

Numerous recent studies have been conducted on the performance of fatty acids, such as on their thermal property, thermal stability, and long-term cycling reliability [21, 22]. Sarı et al. [23, 24] studied the thermal properties of many binary eutectic mixture systems, such as the mixtures of lauric-stearic, myristic-palmitic, palmitic-stearonic, lauric-myristic, lauric-palmitic, and myristic-stearic acids; the data measured by differential scanning calorimetry (DSC) showed that the melting temperatures of those binary systems were 37.00° C, 42.60°C, 52.30°C, 34.20°C, 35.20°C, and 44.10°C, respectively, and their phase change latent heat values were

182.70, 169.70, 181.70, 166.80, 166.30, and 182.40 J/g, respectively. Wen et al. [25] prepared a PCM of capric-lauric acid (CA-LA)/diatomite/EG composite and studied its thermal properties. Yuan et al. [26] and Wei et al. [27] prepared some ternary fatty acid composite PCMs and studied their thermal properties. These aforementioned research results prove that PCMs can be applied for solar energy utilization and building energy conservation and have a guiding effect on the development of eutectic mixture fatty acid.

Most of the aforementioned studies focus on single and binary or ternary eutectic fatty acids in the temperature range of 20°C–60°C, mainly used in building energy saving and solar energy utilization. However, as the backfill material in the ground source heat pump (GSHP) system, PCM with a phase change temperature of approximately 19°C has yet to be reported. GSHP is a heat pump technology that utilizes shallow geothermal energy. The heat transfer performance between the buried pipes and around the soil plays a decisive role in the operational stability and operating efficiency of the GSHP. The underground soil temperature is unchanged below 15 m. For example, the temperature is approximately 19°C in Shanghai, China [28]. However, after the GSHP system operates for a certain period of time, the soil temperature around the buried pipe will change, thereby reducing the system operating rate. Thus, the performance of the backfill materials around the buried pipe is particularly important.

In this study, the capric-myristic acid (CA-MA) binary eutectic mixture PCM was prepared for LHTES and as backfill materials around the buried pipe of a GSHP system. The thermal properties and thermal cycling reliability of the materials were tested via DSC, and the thermal decomposition stability was investigated via thermal gravimetric analysis (TGA). In addition, Fourier transform infrared (FTIR) spectroscopy was used to investigate whether the chemical composition of the PCMs changed before and after preparation and determine the possible reason that caused the change of the thermal properties of the materials with the increase in the thermal cycling number.

2. Experiments

2.1. Materials. Capric acid (CA, ≥98.5% purity) and myristic acid (MA, ≥98% purity) were purchased from Shanghai Zhunyun Chemical Co. Ltd.

2.2. Preparation of CA-MA PCMs. The solid CA and MA were weighed separately at different weight ratios from 0 wt% to 100 wt%, and the sample weight errors were controlled within 0.1 mg. Then, the CA and MA were mixed in a beaker. Then, the beaker was stored in a vacuum drying oven at a constant temperature of 80°C for 2 h. After completely melting, the fatty acid mixtures were stirred for 30 min at 60°C and 500 r/min in a magnetic stirrer. The beaker of the molten liquid fatty acid mixtures was then placed in an ultrasonic water bath; the temperature was controlled at 60°C to ensure that the fatty acids were constantly in the melting state. Furthermore, the time of ultrasonic vibration was approximately 2 min to ensure that the two types of fatty acids were sufficiently mixed to form binary eutectic mixtures. With these methods, a set of CA-MA binary eutectic mixtures was prepared.

2.3. Characterization. The phase change temperature (melting temperature (T_m) and freezing temperature (T_f)) and phase change latent heat (melting latent heat (H_m) and freezing latent heat (H_f)) of the samples were determined by DSC (TA Q20, USA) calibrated with indium standard in the temperature range of 0°C–80°C. The temperature increasing rate of DSC measurements had a 5°C/min heating rate. DSC measurements of the same samples were conducted three times, and the accuracy was ±0.1% for phase change temperatures and ±4% for latent heat.

To investigate the effect of the thermal cycling number on thermal properties, the CA-MA eutectic mixtures were heated from solid to liquid state and then cooled from liquid to solid state by a heating controller. The above thermal cycling process was conducted continuously until the values were 500, 1000, and 2000. The changes in the performance of the mixtures were measured by DSC and FT-IR.

The thermal stability of the CA-MA PCM was analyzed by TGA (TA Q50, USA) in the temperature range of 20°C–450°C with a 10°C/min heating rate under nitrogen gas atmosphere and an accuracy of ±0.2%.

The samples of CA, MA, and CA-MA eutectic mixtures were analyzed by FTIR (Thermo Scientific Nicolet iS5, USA). The uncycled and cycled samples were measured by FTIR to explore the reason of the variation of thermal properties of the CA-MA mixtures after thermal cycling.

3. Results and Discussion

3.1. Thermal Properties of CA and MA Used. The CA-MA PCM was prepared by mixing CA and MA at different mass ratios. The phase change temperatures and latent heat of the CA and MA in some references [29–32] are listed in Table 1. The DSC curves of the CA and MA used in this study are shown in Figure 1. The phase change temperatures and latent heat from the curves are shown in Table 1. From the table, the melting points of CA and MA used are 31.17°C and 52.68°C, respectively, and the latent heat values of fusion are 169.4 J/g and 188.6 J/g, respectively. These results suggest that CA and MA can be used as raw materials to produce the CA-MA PCM with suitable phase change temperatures and large latent heat.

3.2. Mass Ratio of CA-MA PCM. In a binary system, if the two solid components are completely immiscible and can form a eutectic system, then the phase system will become a eutectic binary system [33]. In a solid-liquid two-phase equilibrium system of a two-component system whose liquids are completely mutually soluble while the solids are completely immiscible, a temperature lower than the phase change temperature of the two pure components will be observed. At this temperature, the solid mixtures, named the lowest eutectic, which has the same composition as the liquid phase, will precipitate, and its temperature is the lowest eutectic temperature or the lowest eutectic point; similarly, the mechanical mixture of the two solids can also be melted together at the minimum eutectic temperature [34].

Table 1: Thermal properties of pure CA, pure MA, and CA-MA binary eutectic mixture.

PCM	Thermal properties				References
	T_m (°C)	H_m (J/g)	T_f (°C)	H_f (J/g)	
CA	31.53	165.21	32.05	168.43	Sarı et al. [29]
MA	53.51	192.68	53.24	195.36	
CA	27.69	164.7	32.06	163.5	Fu et al. [30]
MA	50.78	203.7	55.17	201.0	
CA	31.5	155.5	—	—	Gao and Qian [31]
MA	51.6	204.5	—	—	
CA	32.14	156.04	32.53	154.24	Karaipekli et al. [32]
MA	53.86	192.58	53.74	190.11	
CA	31.17	169.4	31.69	170.3	This study
MA	52.68	188.6	51.63	193.1	
CA-MA eutectic mixture	18.21	148.5	17.40	134.0	This study

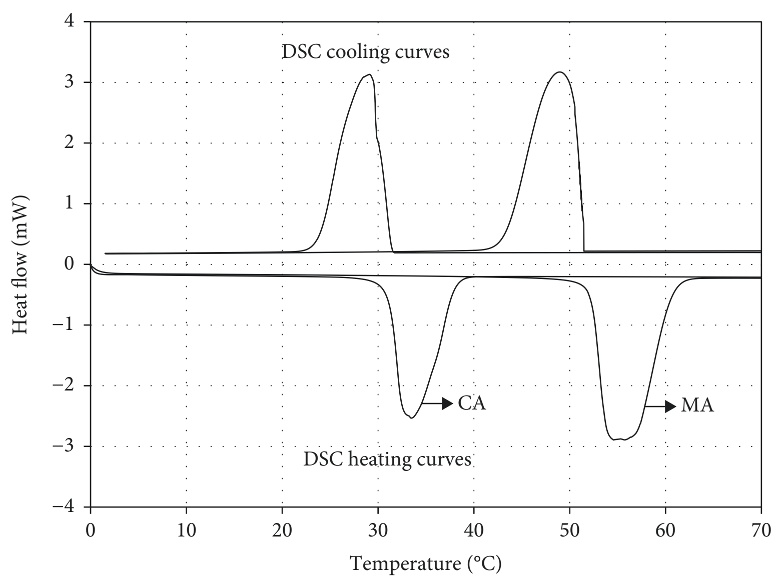

Figure 1: DSC curves of pure CA and pure MA.

Thus, the binary eutectic mixture fatty acids have a lower phase change point than any of the fatty acids. Zhang et al. [35] derived the relationship of melting temperature, latent heat, and content of components A and B and eutectic mixtures based on the second law of thermodynamics and phase equilibrium theory, shown as follows:

$$\begin{cases} -\dfrac{H_A(T_m - T_A)}{T_A} + RT_m \ln\,(1 - X_A) + G_{A,ex} = 0, \\ -\dfrac{H_B(T_m - T_B)}{T_B} + RT_m \ln\,(1 - X_B) + G_{B,ex} = 0, \end{cases} \quad (1)$$

where T_m is the phase change temperature of the eutectic mixtures (K); T_A and T_B are the melting temperatures of constituents A and B, respectively (K); H_A and H_B are the latent heat of constituents A and B, respectively (J/mol); X_A and X_B are the mole percent ratio of constituents A and B of the eutectic mixture, respectively, (%) and $\sum(X_A + X_B) = 1$; R is the gas constant (8.315 J/(mol·K)); and $G_{A,ex}$ and $G_{B,ex}$ are the excess free enthalpy of constituents A and B, respectively.

For fatty acids, $G_{A,ex} = G_{B,ex} = 0$ is an excellent approximation. Accordingly, equation (1) can be rewritten as

$$\begin{cases} T_m = \left[\dfrac{1}{T_A} - \dfrac{R \ln X_A}{H_A}\right]^{-1}, \\ T_m = \left[\dfrac{1}{T_B} - \dfrac{R \ln X_B}{H_B}\right]^{-1}. \end{cases} \quad (2)$$

The relevant parameters of CA and MA in Table 1 are replaced in equation (2), and equation (3) can be obtained.

$$\begin{cases} \ln X_{CA} = \dfrac{11.53 - 3509.6}{T_m}, \\ \ln X_{CA} = \dfrac{15.90 - 5179.9}{T_m}. \end{cases} \quad (3)$$

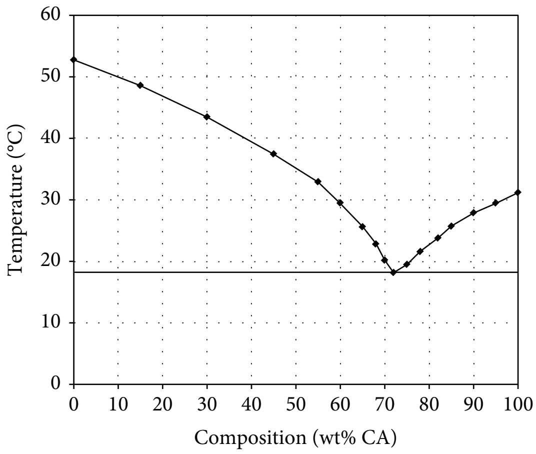

Figure 2: Effects of composition mass ratios (wt%) on the melting temperatures of CA-MA binary eutectic mixtures.

The theoretical phase diagram of the CA-MA eutectic mixtures can be drawn, and the corresponding theoretical ratios and phase change temperatures can be determined by equation (3).

However, experiments have proven that some errors occur if equation (3) is adopted because the purity of a single fatty acid supplied may affect the mass ratio and the phase change temperature. Thus, the actual mass ratios of the eutectic mixtures must be experimentally determined.

The effects of the composition mass ratios on the melting temperatures of the CA-MA binary eutectic mixtures are shown in Figure 2, and the data are taken from our experiments. The melting temperature of the CA-MA binary eutectic mixtures decreases with the increase in the mass ratio of CA, and the temperature range narrows when the mass ratio approaches the eutectic mixing ratio. At the eutectic mixing ratio, the lowest melting temperature is obtained. Then, the melting temperature of the CA-MA mixture increases again with the CA mass ratio. As shown in Figure 2, the lowest melting temperature is 18.21°C at the mass ratio (72% CA/28% MA w/w). The melting temperature of the CA-MA PCM is lower than that of any of the pure fatty acids.

3.3. Thermal Property of CA-MA PCM. The DSC curve of the prepared CA-MA PCM is shown in Figure 3, and the thermal performance parameters are shown in Table 1. The melting and freezing temperatures are 18.21°C and 17.40°C, respectively, and the melting and freezing latent heat values are 148.5 J/g and 134.0 J/g, respectively. The indoor comfortable temperature range is 16°C–25°C; thus, the eutectic mixture can be used for building LHTES to reduce the load of heating, ventilation, and air conditioning systems in winter and summer. The soil temperature around the buried pipe of the GSHP is approximately 19°C. Therefore, the eutectic mixture can be used as a backfill material of the geothermal heat exchanger to enhance the heat transfer effect and improve the operating efficiency of the GSHP system.

The phase change temperatures and latent heat of the CA-MA mixtures in some references [29–32] are listed in Table 2; the values slightly differ from the data in Table 1. Two causes for the different results are probable. One is that the single fatty acids supplied contain a certain amount of impurities, and the other is experimental error [36].

3.4. FTIR Analysis of CA-MA PCM. FTIR spectroscopy was conducted to ascertain the chemical structure of the CA-MA PCM. The FTIR spectra of the single fatty acid CA and MA and the CA-MA mixture are shown in Figure 4.

The infrared spectrum curves of CA and MA in Figure 4 show a strong peak at 1696.66 cm^{-1} (CA) and 1700.45 cm^{-1} (MA), which is the absorption of C=O stretching vibration in the hydroxyl group; this absorption peak is highly typical for fatty acids. Fatty acids usually exist in the form of bimolecular associations due to the presence of hydrogen bonds, and its O–H stretching vibration at 3100–2500 cm^{-1} usually overlaps with the C–H bond stretching vibration peak of the aliphatic group. In this characteristic band, the stretching vibration peak corresponds to $-CH_3$ at 2916.16 cm^{-1} (CA) and 2914.45 cm^{-1} (MA), which correspond to $-CH_2-$ at 2844.53 cm^{-1} (CA) and 2847.70 cm^{-1} (MA).

The infrared spectrum curve of the CA-MA in Figure 4 shows that 1708.79 cm^{-1} is the characteristic peak of C=O of the CA-MA eutectic mixtures, thereby indicating that fatty acid molecules still exist in the form of a dimer in the binary eutectic CA-MA. Furthermore, the absorption peak of the –OH group is in the range of 3050–2800 cm^{-1}. The infrared spectrum of the binary eutectic mixture is similar to the pure fatty acids; thus, its molecular structure has not changed and the phase change heat storage properties and chemical properties of the fatty acids are maintained.

3.5. Thermal Stability of CA-MA PCM. Thermal stability refers to the resistance of PCMs to high temperatures. Thus, whether a significant mass loss occurs in the temperature range in which the PCM was used can be determined. Generally, PCMs, especially organic PCMs, often undergo significant mass loss because of oxidation, decomposition, and volatilization reactions when subjected to high-temperature tests. Therefore, the temperature range must be controlled when using those materials. The thermal stability of fatty acid PCMs is commonly analyzed via TGA.

As shown by the thermal gravimetric (TG) and DTG curves of the CA-MA PCM in Figure 5, its initial mass loss temperature is approximately 110°C, which indicates that the CA-MA eutectic mixture begins to evaporate slowly, the epitaxial starting temperature is 181.84°C, and the temperature range of 120°C–230°C is the main mass loss area. The mass loss is caused by the volatilization of the samples. The temperature of the maximum mass loss rate is 217.61°C. At 243°C, the weight loss of the samples is nearly 98.5%, and the samples have almost completely evaporated; the remaining components are impurities in the system. On the basis of the analysis, the CA-MA eutectic mixture has high thermal stability below 100°C; however, when the working temperature rises above this temperature, the CA-MA eutectic mixture easily decomposes and volatilizes, thereby resulting in high mass loss. The thermal energy storage performance and the service life of the materials will be greatly affected if mass loss occurs. Therefore, the CA-MA PCM cannot

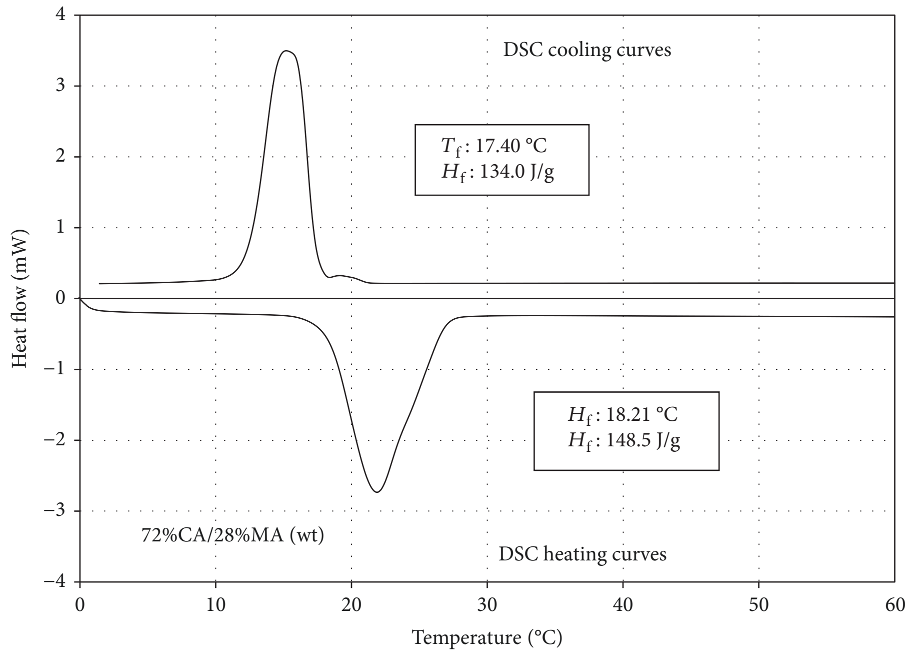

Figure 3: DSC curve of the CA-MA binary eutectic mixture.

Table 2: Thermal properties of CA-MA mixtures in this study and other references.

PCM	Phase change temperature (°C)	Phase change latent heat (J/g)	References
CA/MA mixture (75.0/25.0 wt%)	22.17	153.19	Sarı et al. [29]
CA/MA mixture (76.0/24.0 wt%)	23.64	147.70	Fu et al. [30]
CA/MA mixture (78.0/22.0 wt%)	19.65	149.02	Gao and Qian [31]
CA/MA mixture (73.0/27.0 wt%)	21.70	168.37	Karaipekli et al. [32]
CA/MA mixture (72.0/28.0 wt%)	18.21	148.50	This study

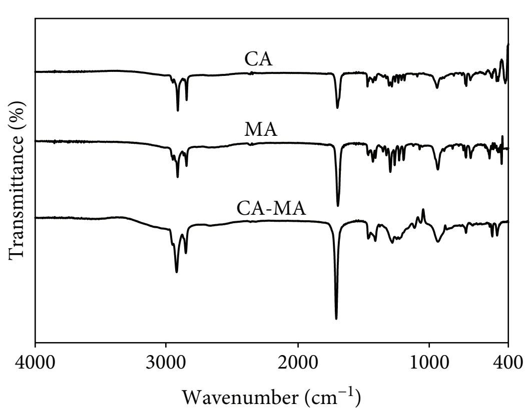

Figure 4: FTIR spectra of CA, MA, and CA-MA.

be used in medium–high-temperature phase change thermal energy storage, although it can be applied for low-temperature phase change thermal energy storage, such as a building energy conservation system and backfill material around the buried pipe of the GSHP system.

3.6. Thermal Reliability of CA-MA PCM. The thermal cycle reliability of PCMs refers to whether the thermal energy storage performance decays after repeated storage/discharge processes. The reliability is an important parameter to measure the service life of PCMs. The thermal cycle reliability of PCMs is often tested by accelerated thermal cycling to study the changes of two important thermodynamic parameters, phase change temperature, and latent heat, before and after thermal cycling. The DSC curves of the CA-MA PCMs after 500, 1000, and 2000 thermal cycles are shown in Figure 6.

The changes in melting and freezing temperatures of the CA-MA PCM with the thermal cycling number are shown in Figure 7. After 500, 1000, and 2000 thermal cycles, the T_m values change to 0.07°C, 1.21°C, and 0.46°C, respectively, and the T_f values change to −0.75°C, −0.52°C, and− 0.87°C, respectively. As the thermal cycling number increases, the variations of the T_m and T_f values are irregular and the change values are extremely small, such that the influence on the phase change energy storage system is negligible. Therefore, on the basis of the changes in the melting and freezing temperatures, the CA-MA PCM has high thermal cycle reliability.

As the thermal cycling number increases, the variations of the melting and freezing latent heat values of the CA-MA PCM are shown in Figure 8. After 500, 1000, and 2000 thermal cycles, the melting latent heat (H_m) values of the

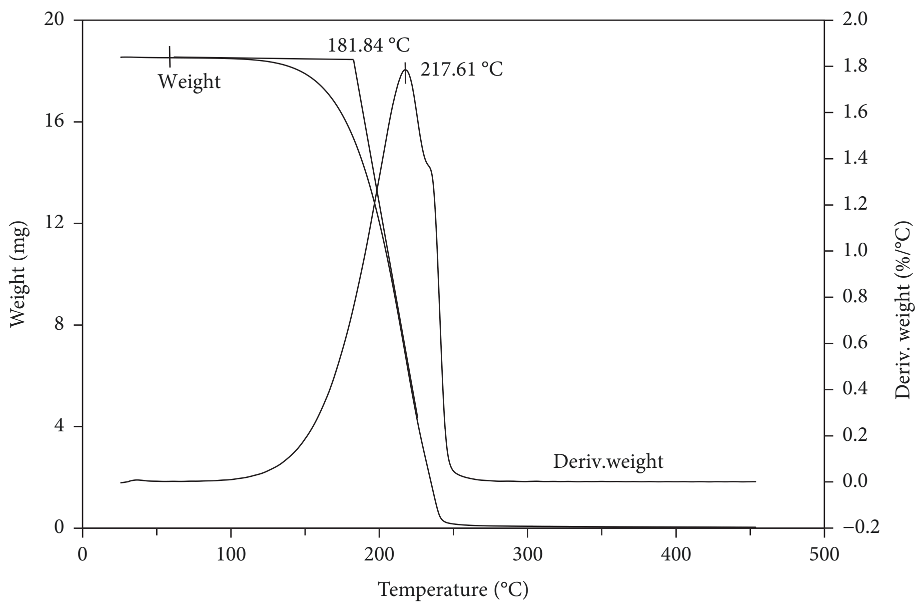

Figure 5: TG and DTG curves of CA-MA PCM.

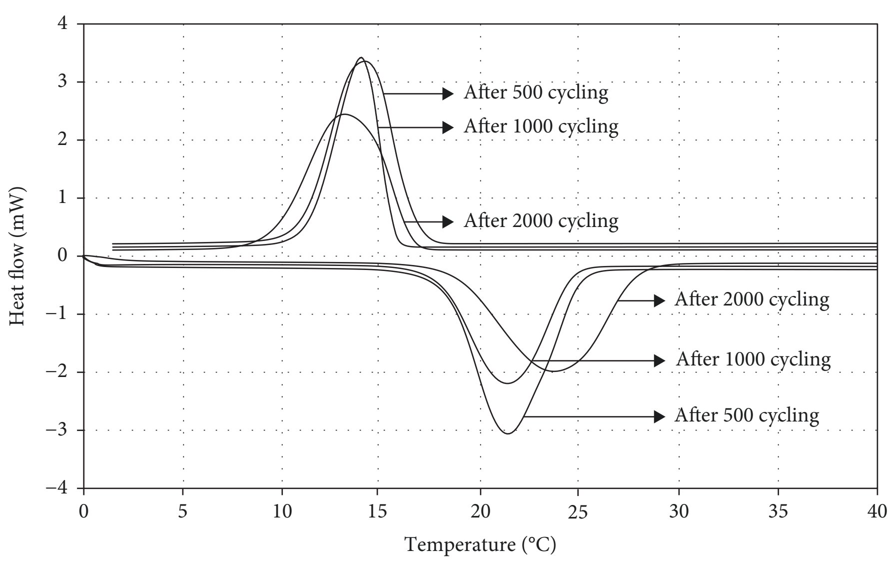

Figure 6: DSC curves of CA-MA PCM after 500, 1000, and 2000 thermal cycles.

mixtures changed by −3.2%, 1.15%, and− 12.1%, respectively, and the freezing latent heat (H_f) values changed by 3.7%, 0.5%, and− 4.4%, respectively. As the number of thermal cycling increases, the variations of H_m and H_f values are irregular. When the number of thermal cycling increases from 500 to 4000, the values of H_m and H_f vary between −12.1% and 3.7%, respectively. These results are acceptable for PCMs applied to LHTES and GSHP systems.

As shown in Figures 7 and 8, small variations of the thermal properties of the PCMs occur before and after thermal cycling, which are caused by the following two factors: one is that the single fatty acids supplied contain a certain amount of impurities, and the other is that the PCMs may undergo chemical degradation [36]. The uncycled and cycled (after 2000 times) samples of CA/MA eutectic mixtures were

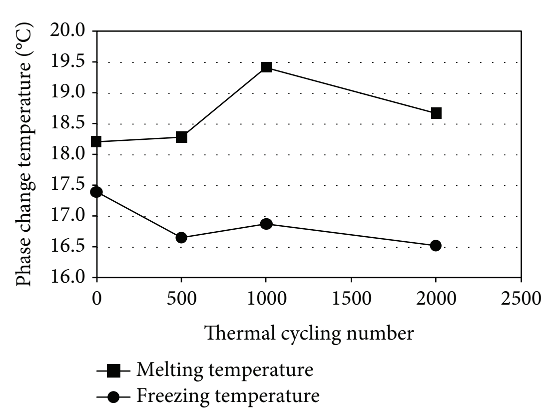

Figure 7: Variations of the phase change temperatures of the CA-MA PCM with the thermal cycling number.

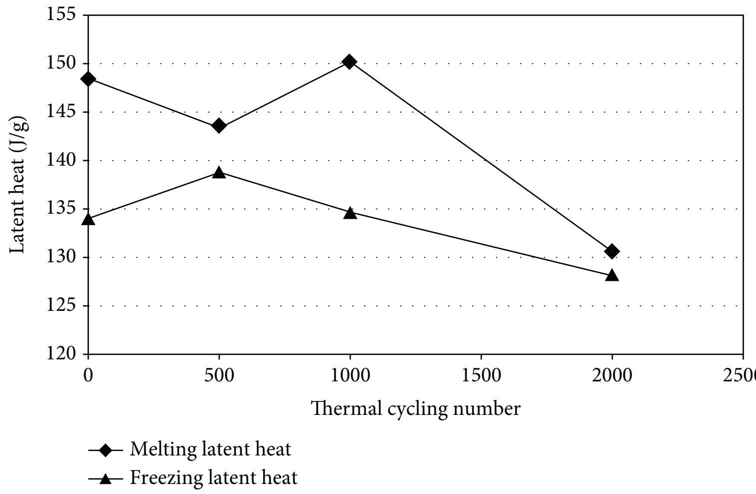

FIGURE 8: Changes in the latent heat of CA-MA PCM with the thermal cycling number.

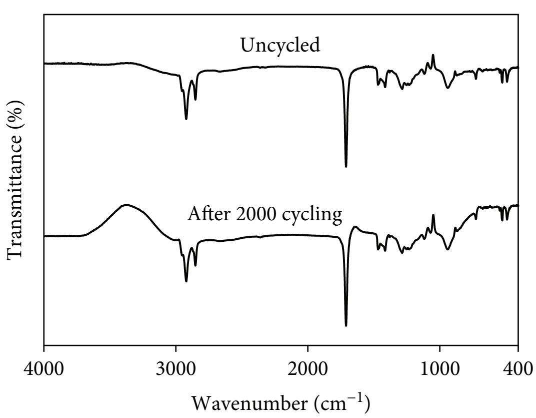

FIGURE 9: FTIR spectra of the uncycled and cycled CA-MA PCMs.

measured by FTIR for the second factor, and the infrared spectrum is shown in Figure 9. The figure shows that the peaks of the two spectra are in the same frequency band and match each other. Thus, the mixtures do not undergo any chemical degradation after thermal cycling. Thus, as the thermal cycling number increases, the thermal properties of the materials change only because the supplied single fatty acids contain a certain amount of impurities.

4. Conclusion

The CA-MA binary eutectic mixture PCM was prepared. The thermal properties, thermal stability, and long-term cycling reliability of the binary eutectic mixtures were studied.

(1) The DSC results show that the CA-MA PCM is highly suitable for LHTES and as backfill materials around the buried pipe for GSHP systems because of the phase change temperatures (T_m: 18.21°C, T_f: 17.40°C) and latent heat (H_m: 148.5 J/g, H_f: 134.0 J/g)

(2) The TGA results show that the CA-MA PCM has excellent thermal stability below 100°C. Thermal cycling tests show that the CA-MA PCM has good long-term cycling thermal reliability because of the small variations of phase change temperatures and latent heat with thermal cycling number

(3) The FTIR results indicate that the molecular structure of the CA-MA binary eutectic mixture has not changed; the phase change heat storage performance and chemical properties of fatty acid are maintained. In addition, the CA-MA PCM does not undergo any chemical degradation after thermal cycling, and its thermal property changes only because the single fatty acids supplied contain a certain amount of impurities

The CA-MA PCM has promising application prospects in LHTES and as backfill materials around the buried pipe of GSHP systems due to its good performance.

Data Availability

The data availability of the manuscript can be found at https://figshare.com/s/5d017c6ae301c9bdffd9.

Conflicts of Interest

The authors declare that there are no conflicts of interest.

Acknowledgments

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