

Research Article

Palmitic Acid-Stearic Acid/Expanded Graphite as Form-Stable Composite Phase-Change Material for Latent Heat Thermal Energy Storage

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The aim of this study is to prepare a novel form-stable phase-change material (PCM), palmitic acid-stearic acid/expanded graphite (PA-SA/EG) PCM, for latent heat thermal energy. A eutectic mixture of palmitic acid and stearic acid is incorporated with expanded graphite (EG). The thermal properties and microstructure of the novel PCMs were studied. The optimal mass ratio of PA-SA in composite PCM is approximately 92.8%. DSC results showed that the novel PCMs had suitable phase-change temperature (T_m : 55.18°C, T_f : 54.91°C) and high latent heat (H_m : 176.2 J/g, H_f : 175.6 J/g), and good thermal performance was maintained after 1500 times thermal cycling. The PA-SA binary eutectic mixture was uniformly distributed in the porous structure of EG, and the PCM did not leak even in molten state. EG had little effect on the thermal properties of the PA-SA binary eutectic mixture and remarkably improved the thermal conductivity of the composite PCMs. TGA test indicated that the composite PCM had nice thermostability in the range of operating temperature. Based on all these results, the PA-SA/EG composite PCM is a promising material for low-temperature thermal energy storage applications.

1. Introduction

Phase-change materials can store or release thermal energy during phase transition [1]. PCMs provide a higher stored energy density than sensible heat storage materials, and their temperature is almost unchanged during phase change [2]. Therefore, PCMs have been broadly used in building energy conservation [3, 4], photovoltaic solar energy [5], air-conditioning condenser heat recovery [6], solar energy storage [7, 8], cryogenic refrigeration [9], and indoor temperature control systems [10].

Fatty acids are widely used in PCMs because of their large heat storage capacities, low supercooling, nontoxic components, good heat and chemical stability, minimum volume changes during phase change, and no self-nucleation [11]. The eutectic mixture of binary or ternary aliphatic acids, which can be prepared by eutectic effect, can prevent disad-

vantages due to the fixed phase-transition temperature of in a single fatty acid and widen the phase-transition temperatures range of fatty acids [12, 13]. Hassan Nazir et al. [14] prepared the multiple fatty acid and determined the eutectic point of each eutectic solution according to the Schrader equation along with its thermophysical properties. These eutectic PCMs have a phase-transition temperatures range of 27–75°C and latent heat of 127–210 kJ/g, which are suitable for solar water heating, building energy saving systems, and photovoltaic solar heating applications. Kadir Tuncbilek et al. [15] prepared a eutectic solution of 69.0 wt.% lauric acid and 31.0 wt.% palmitic acid with a melting temperature of 35.2°C and latent heat of 166.3 J/g. Their results indicated that the LA-PA eutectic mixture is a promising storage material at low-temperature energy with nice heat performance.

However, single fatty acids and the eutectic solution of fatty acids have the disadvantage of low thermal conductivity,

which greatly reduces thermal transfer rate during the phase-change process [16]. For this problem, porous materials can be added to prepare form-stable PCMs. The strong adsorption of porous substrates can avoid the leakage of PCMs during the phase-change process [17, 18]. The main methods of preparing PCMs are microencapsulation [19], melt gel [20], physical adsorption, and press sintering [21]. Expanded graphite (EG) was widely used as an excellent porous substrate because of its high thermal conductivity, large pore volume, strong adsorption capacity, and low density [22].

Zhang et al. [23] prepared the CA-PA-SA/EG composite PCM for low-temperature heat storage. The mass ratio of CA:PA:SA is 79.3:14.7:6.0, and the CA-PA-SA mass content of CA-PA-SA/EG composites is 90%. The temperatures of melting and freezing were 21.33 and 19.01°C, respectively, and latent heats of melting and freezing of the composite PCM were 131.7 and 127.2 J/g, respectively. In Zhang et al.'s study, PCMs were uniformly distributed in the porous structure of EG and exhibited high thermal stability and thermal reliability. Yang et al. [24] prepared the MA-PA-SA/EG composite with an MA:PA:SA mass ratio of 52.2:29.4:18.4. The optimal mass ratio of the MA-PA-SA:EG was 13:1. The composite PCM had melting and freezing temperatures of 41.64°C and 42.99°C, respectively, and corresponding latent heat values of 153.5 and 151.4 J/g, respectively. Zhang et al. [25] prepared the PA-SA/EG composite PCM with a PA-SA:EG mass ratio of 13:1; the composite PCM phase-change temperatures were 53.89°C and 54.37°C, and the corresponding latent heat were 166.27 and 166.13 J/g, respectively.

In this study, a new type of palmitic acid-stearic acid/expanded graphite (PA-SA/EG) composite PCMs with suitable phase-change temperature and high phase-change latent heat for solar thermal storage system were prepared. The thermal performance and microstructure of the new composite PCMs were studied.

2. Experiments

2.1. Materials. Stearic acid (SA, analytically pure) and palmitic acid (PA, analytically pure) were purchased from Shanghai Shanpu Chemical Co., Ltd., China. Expandable graphite (350 meshes, expansion coefficient: 100 mL/g, Carbon content > 95%) was supplied by Qingdao Hengrunda Graphite Products Co., Ltd., China.

2.2. Preparation of Palmitic Acid-Stearic Acid Binary Eutectic Solution. Amounts of PA and SA were added to a beaker, melted in a 70°C centrifugal mixer with water separation, stirred for 1 hour until uniformly mixed, and then cooled to room temperature for solidification.

2.3. Preparation of the Composite PCMs. A certain amount of expandable graphite was preserved in a 50°C vacuum-drying oven for 18 hours and then placed in a preheated porcelain crucible. Subsequently, the crucible was placed into a 900°C muffle furnace for 40-second heating to obtain expanded graphite. A certain amount of dried expanded graphite was added to the PA-SA binary eutectic solution, added to a

beaker, and then heated in a thermostat at 80°C for 48 hours. After the PA-SA binary eutectic solution melted, the mixture was shaken every 2 hours for uniform mixing and full adsorption of the PA-SA binary mixture and EG. Then, the mixture was cooled to room temperature for the preparation of PA-SA/EG composite PCMs.

2.4. Characterization. The phase-change temperature and phase-change latent heat of the samples were determined using a differential scanning calorimetry (DSC, TA Q20, USA) calibrated with the Indium Standard in the temperature range of 20°C–80°C. The temperature increase of measurements had a 5°C/min heating rate under a constant stream of argon at a flow rate of 50 mL/min. The sample mass was about 7.5 mg. DSC measurements of the same samples were conducted three times, and the accuracy was $\pm 0.05^\circ\text{C}$ for phase-change temperatures and $\pm 0.1\%$ for latent heat. The thermal stability of the samples were analyzed by the thermogravimetric analysis (TGA) using a thermal analyzer (TA Q50, USA) in the temperature range of 25°C–500°C with 10°C/min heating rate under nitrogen gas atmosphere and accuracy of $\pm 0.2\%$. The samples were analyzed through the Fourier transform-infrared (FT-IR, Thermo Scientific Nicolet iS5, USA), and the FT-IR spectra were recorded on a KBr pellet at the frequency range of 4000 cm^{-1} to 400 cm^{-1} . The microscopic structures of the EG and PA-SA/EG composite PCMs were observed by using a scanning electronic microscope (SEM, Phenom LE, the Netherlands). Thermal conductivities of PA-SA/EG composite PCMs were measured at room temperature using Hot Disk Thermal Constants Analyzer (TPS2500, Hot Disk AB Company Sweden) based on the transient plane heat source technique, and the measurement accuracy of thermal conductivity is within $\pm 3\%$.

To investigate the effect of thermal cycling number on thermal properties, the PA-SA/EG composite PCMs 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 100, 500, and 1500. The changes in performance were measured by DSC.

3. Results and Discussion

3.1. Mass Ratio of Palmitic Acid and Stearic Acid. The thermophysical properties of PA and SA [26, 27] are shown in Table 1. The phase-change temperatures of PA and SA are higher than 60°C, whereas the phase-change temperature of PA or SA alone is extremely high for a solar thermal storage system. Therefore, the phase-change temperatures of the fatty acids can be reduced by preparing a eutectic solution. In this study, the PA-SA eutectic solution with suitable phase-change temperatures was prepared as PCMs for solar thermal storage systems. The melting temperatures (T_m) and latent heat values (H_m) of the different components of PA and SA can be calculated according to the Schrader Equation (1) [12], and the calculated values are shown in Table 2. The melting temperature was 54.21°C when the mass ratio of PA:SA was 72:28. The binary eutectic mixture of PA-SA was

TABLE 1: Thermal properties of PA and SA.

PCM	Chemical formula	Phase-change temperature, °C	Phase-change latent heat, J·g ⁻¹	Specific heat, kJ·(kg·°C) ⁻¹		Thermal conductivity, W·(m·K) ⁻¹ [26]
				Solid	Liquid	
PA	C ₁₆ H ₃₂ O ₂	64.03	185.4	1.9	2.8	0.162
SA	C ₁₈ H ₃₆ O ₂	70.71	203.0	1.6	2.2	0.172

TABLE 2: Thermal properties of PA-SA binary eutectic solution.

PA-SA, wt.%	T _m , °C	H _m , J·g ⁻¹
0.0-100.0	69.1	201.8
60.0-40.0	51.2-54.2	183.7
62.0-38.0	51.8-53.5	182.9
63.5-36.5	52.1-52.8	182.3
64.2-35.8	52.3-52.3	181.7
64.9-35.1	52.5-51.7	181.4
65.6-34.4	52.8-51.1	181.1
85.0-15.0	56.7-45.9	183.1
100.0-0.0	58.9	189.6

prepared according to the calculated results, and the mass ratio of the eutectic mixture was PA : SA = 72 : 28. A recent work [28] shows that the Schrader equation is not sufficiently accurate to determine the exact eutectic of binary fatty acids. There are various reasons such as the purity of a single fatty acid supplied, measure methods, and experimental error. Therefore, more experiments are required to determine the actual mass ratios and thermal properties of the eutectic mixtures.

$$\begin{cases} T_m = \left[\frac{1}{T_i} - \frac{R \ln X_i}{H_i} \right]^{-1} \\ H_m = T_m \sum_{i=1}^n \frac{X_i H_i}{T_i} \end{cases} \quad (1)$$

where T_m is the melting temperature of the eutectic solution (in K); T_i is the melting temperature of the i th component (in K); H_i is the latent heat of phase change of the i th component (in J/mol); H_m is the latent heat of phase change of the eutectic solution (in J/mol); X_i is the molar content of the i th component (in %), and $\sum X_i = 1$; and R is the gas constant, which is 8.315 J/(mol·K).

3.2. Optimal Mass Ratio of Components in the Composite PCMs. EG has excellent adsorption capacity. Theoretically, heat stored in the PCMs increases with the amount of fatty acids. However, in fact, when the PCMs exceed the maximum adsorption limit of EG, excess composite PCMs may leak after being melted into liquid and affect the properties of the composites. Therefore, the optimal mass ratio of the PA-SA eutectic mixture in the composite PCMs should be determined.

In this study, PA-SA/EG composites with different PA-SA contents (91%–94%) were prepared. The prepared PA-SA/EG composites (0.50 g) were uniformly dispersed in a test area with 30 mm diameter at the center of the filter paper. The sample was then heated at 80°C for 1 hour in a vacuum-drying oven and then taken out. After the samples were brushed off from the filter paper, the seepage degree for the eutectic mixture fatty acids of the PCMs at the test area was determined. The photographs of PA-SA/EG composite PCMs before (a) and after (b) the thermal treatment and the filter paper (c) with samples brushed off after thermal treatment are shown in Figure 1. The sample with 94% PA-SA content leaked obviously, whereas the sample with 92% PA-SA did not; hence, the biggest mass ratio of PA-SA was intended to be 92%–93%. Accordingly, we prepared samples with 92%–93% PA-SA content (shown in Figure 1(a)). Figure 1(b) indicates the samples status after heat treatment. When the PA-SA content was 93%, after the samples on the filter paper was brushed off, a few eutectic mixture fatty acid leaks were confusingly observed on the filter paper (shown in Figure 1(c)). The molten PCMs did not leak until 92.8%. Therefore, the maximum absorption mass ratio of PA-SA content in the composite PCMs should be approximately 92.8%. The mass ratio of PA-SA content in the PA-SA/EG composite PCMs used in subsequent studies was 92.8%.

3.3. Thermal Properties of the Composite PCMs. The thermal properties of PA-SA and PA-SA/EG were measured by DSC. The results are shown in Table 3 and Figure 2. The curves were similar, and only one endothermic peak and one exothermic peak in the melting and solidification processes were observed. The comparison of thermal properties of the PA-SA and PA-SA/EG composite PCM showed that EG had no remarkable influence on the phase-change temperature of the composite PCM. The melting temperature of the composite PCM were 55.18°C and freezing was 54.91°C, and the corresponding latent heat values were 176.2 and 175.6 J/g, severally. The latent heat of the phase change of composites after adding EG has a slight decrease because EG acted only as adsorption and support in PCM. Hence, the latent heat of the phase change of PA-SA/EG PCMs per unit mass was reduced, and only PA-SA absorbed and released heat during melting and freezing [29]. The latent heat capacity of the composite PCM was proportional to its PA-SA content.

3.4. Infrared Spectral Analysis of the Composite PCMs. The interactions between the components were characterized by determining the chemical miscibility between PA-SA and EG by FT-IR. The FT-IR spectra of PA-SA and PA-SA/EG

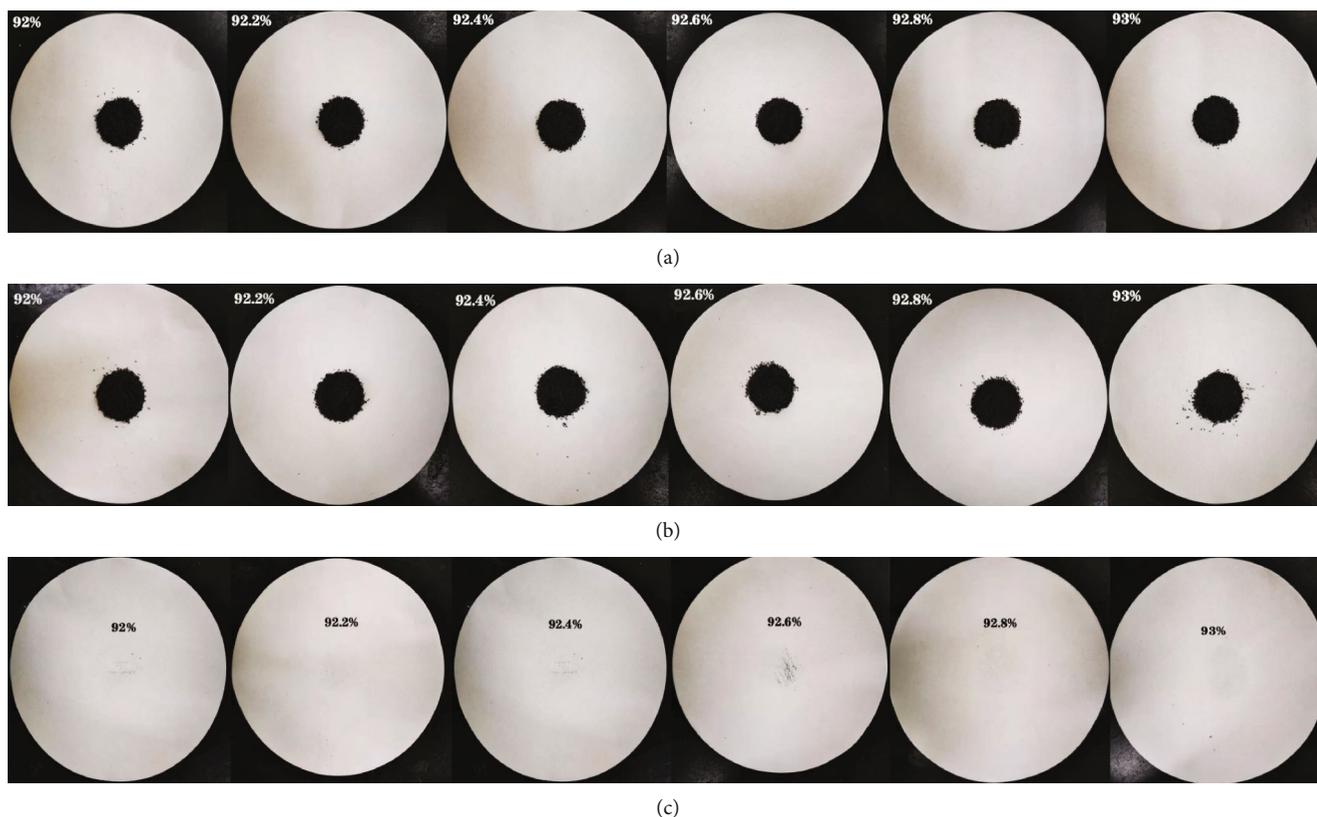


FIGURE 1: Photos of PA-SA/EG before and after thermal treatment. (a) Samples before thermal treatment. (b) Samples after thermal treatment. (c) Filter paper with samples brushed off after thermal treatment.

TABLE 3: Thermal properties of PA-SA and PA-SA/EG.

PCM	Phase-change temperature, °C	Melting Peak change temperature, °C	Phase-change latent heat, J·g ⁻¹	Phase-change temperature, °C	Freezing Peak change temperature, °C	Phase-change latent heat, J·g ⁻¹
PA-SA	54.81	66.62	187.0	54.06	43.42	179.7
PA-SA/EG	55.18	63.11	176.2	54.91	48.86	175.6

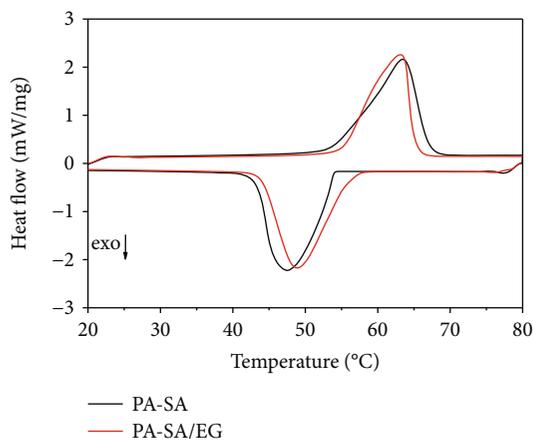


FIGURE 2: DSC graphs of PA-SA and PA-SA/EG.

are shown in Figure 3. The FT-IR spectra of PA-SA showed that the peaks at 2916 and 2849 cm^{-1} represent the stretching vibrations of $-\text{CH}_3$ and $-\text{CH}_2$ groups, respectively. The absorption band of O-H stretching vibration is in the range of 3000–2750 cm^{-1} and usually overlays with the absorption band of the C-H stretching vibration of fatty acids. The peak at 1701 cm^{-1} is the characteristic absorption peak of C=O stretching vibration. The peak at 1470 cm^{-1} is the $-\text{CH}_2$ bending peak, and 1294 cm^{-1} represents the C-H and C-C bending. The peaks at 935 and 720 cm^{-1} correspond to rocking vibration and bending, which are characteristics of the PA-SA aliphatic chain.

The FT-IR spectra of the PA-SA/EG composite PCM showed that the characterized absorption peaks at 2916, 2849, 1701, 1470, 1294, 935, and 720 cm^{-1} also existed, and no obvious new absorption peaks appeared. These findings indicated that PA-SA and EG in the composite PCMs were physically mixed and had no chemical action.

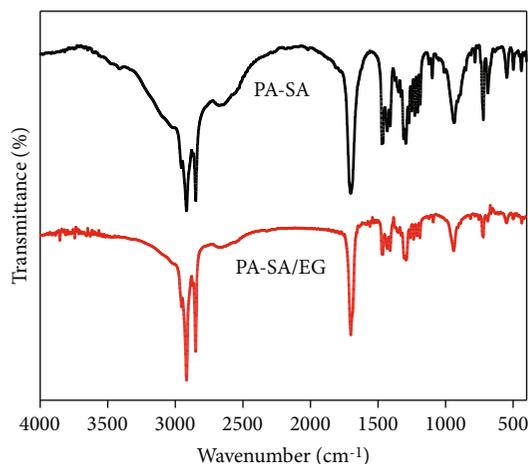


FIGURE 3: FT-IR spectra of PA-SA and PA-SA/EG.

3.5. Microstructure of the Composite PCMs. The microstructures of the EG and PA-SA/EG composites were observed through SEM, and the results are shown in Figure 4. The SEM image of EG (Figure 4(a)) shows that its pores were mainly micron in size and had wormlike porous structures. Hence, EG had a larger specific surface area and higher surface-active edges in the form of scales. This structure tightened the connection between EGs and promoted the adhesion of molten PA-SA. Figure 4(b) shows that PA-SA was uniformly dispersed and adhered to the pore network structure of EG. The capillary action and surface tension in the mesh structure between the PA-SA and EG prevented the leakage of PA-SA in the molten state [30]. The appearance of the PA-SA/EG composite material was not changed in macroscopic view during the phase change. This result indicated that the multivoid structure of EG provided a certain mechanical strength for the composite PCM. In the above observation, PA-SA in the molten state had good adhesion and compatibility with the pore network structure of EG during the phase change of composites, and PA-SA was uniformly distributed in the EG and did not leak.

3.6. Improvement of the Heat Performance of the Composite PCM. Single fatty acids and eutectic mixtures of multi-fatty acids have poor thermal conductivity. Therefore, adding multihollow substrates is necessary to improving their thermal conductivity. EG has high thermal conductivity; hence, the thermal conductivity of PA-SA/EG composite PCMs was greatly improved because of EG addition. In this work, the thermal conductivity of PA-SA/EG composite PCM was measured using the Hot Disk Thermal Constants Analyzer based on the transient plane heat source technique, and the improvement in the thermal conductivity of PA-SA/EG composite PCM was further verified by heat storage and discharge experiments.

The heat storage and discharge rate of PCM were obtained by measuring the time required for the temperature change of the center temperature of PCM during the melting and freezing processes to reach the setting temperature. The setting temperatures of the melting and freezing processes

were 80°C and 15°C, respectively. The test results are shown in Figure 5. The melting and freezing curves of the PCM are shown in Figures 5(a) and 5(b), respectively. The time required for heating PA-SA and PA-SA/EG from 15°C to 80°C was 77 and 50 min, respectively, and the time required for PA-SA/EG to complete heat storage was 28% shorter than that of PA-SA. The cooling time of PA-SA and PA-SA/EG from 80°C to 15°C was 123 and 71 min, respectively, and the heat release time of PA-SA/EG was 42% shorter than that of PA-SA. All the results showed that the porous structure of EG allowed the uniform distribution of PA-SA and greatly enhanced the thermal conductivity, heat storage, and heat release rate of the PA-SA/EG composite PCM.

PA-SA/EG composite PCM cylinders with smooth and compressed surfaces and with different bulk densities were prepared by dry pressing, as shown in Figure 6. Their effect on thermal conductivity was investigated. The block parameters of composite PCM cylinders are shown in Table 4. Figure 7 shows the thermal conductivity test data of the cylindrical samples with different bulk densities. Thermal conductivity increased considerably with the increase in bulk density. The bulk densities of the samples were 571.04, 625.95, 745.71, 776.63, and 858.06 kg/m³, and their heat conductivities were 1.648, 1.878, 2.191, 2.459, and 2.556 W·(m·K)⁻¹, respectively. Data from the fitting formula in Figure 7 show that the thermal conductivity (in Y) of the composite PCM was nearly linear with bulk density (in X). The fitting formula is $y = -0.17128 + 0.00323x$ ($R^2 = 0.95982$). In conclusion, impregnating PCM with porous structure materials, such as EG, can greatly improve the thermal conductivity of PCM.

3.7. Thermostability and Thermoreliability of the Composite PCM. The thermal stability of PA-SA and PA-SA/EG composite PCM was measured through TGA, and the TGA curves are shown in Figure 8. The samples were heated from room temperature to 80°C in a nitrogen atmosphere and stored at this temperature for 60 min for the maintenance of a constant weight loss rate. The initial 1 wt.% weight loss temperature (T_{ini}), the epitaxial starting temperature (T_{onst}), the temperature of the maximum mass loss rate (T_{max}), and the temperature with 99% fatty acids weight loss ($T_{99\%}$) are presented in Table 5. It can be found from Table 5 that T_{ini} , T_{onst} , T_{max} , and $T_{99\%}$ of the PA-SA/EG composite PCM were all higher than those of the PA-SA. The mass losses are mainly caused by the volatilization of PA-SA. The initial 1 wt.% weight loss temperature of PA-SA/EG composite PCMs was approximately 173.6°C. The mass decreased sharply with the increase in temperature. The weight loss percentage peak was reached at 265.1°C and was volatilized almost completely at a temperature of approximately 304.7°C. Therefore, PA-SA/EG composite PCMs have good thermostability in low-temperature applications below 100°C.

It is important to have a good thermoreliability for the composite PCMs. Thermal reliability refers to whether the thermal storage performance of composite PCMs changes after repeated heat storage and release. The thermal properties of excellent composite PCMs should not change or only

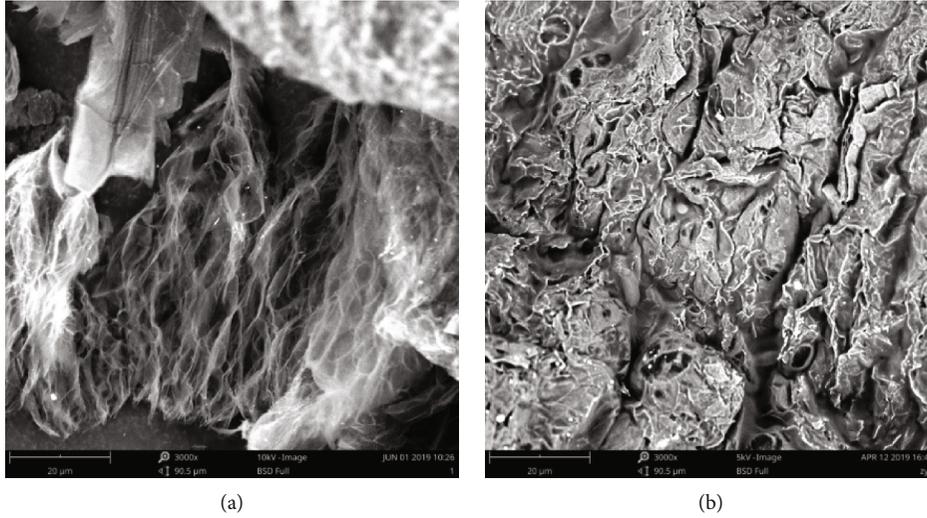


FIGURE 4: Microstructure of (a) EG and (b) PA-SA/EG.

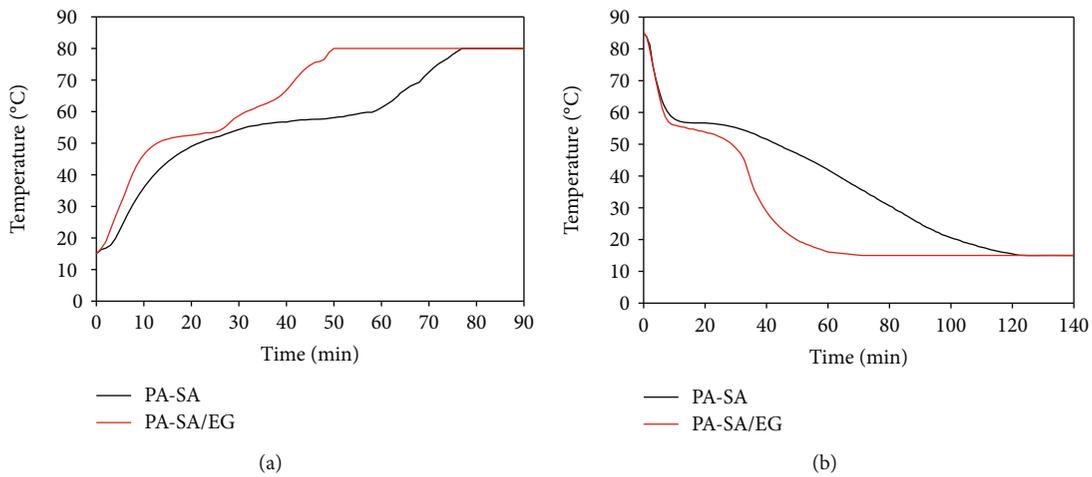


FIGURE 5: Melting (a) and freezing (b) graphs of PA-SA and PA-SA/EG.

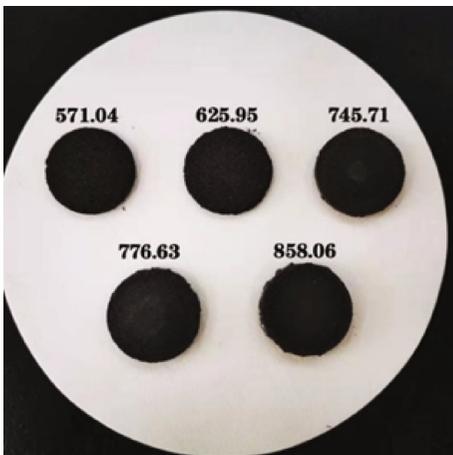


FIGURE 6: PA-SA/EG cylinder block with different bulk densities (density/kg·m³).

have little change after long-term use. The thermal reliability of PCMs can be tested through accelerated thermal cycling experiments. Two thermal properties, phase-change temperature and phase-change latent heat, of the PA-SA/EG composite PCMs were investigated after 200, 500, and 1500 thermal cycles. The comparison of the DSC curves of PA-SA/EG composite PCM after 200, 500, and 1500 thermal cycles are shown in Figure 9, and the specific parameters are shown in Table 6. Figure 9 shows that DSC curves before and after multiple cycles are very similar. Table 6 shows that the phase-change temperatures of PA-SA/EG composite PCM after 200, 500, and 1500 thermal cycles varied by 0.59, 0.71, and 1.02°C, separately, and the phase-change latent heat decreased by 0.28%, 1.14%, and 3.46%, respectively. These results showed that the phase-change temperature and latent heat of PA-SA/EG composite PCM have little change after several thermal cycles; therefore, the composite PCM have good thermal reliability.

TABLE 4: Parameters of PA-SA/EG cylinder block with different bulk densities.

No.	Mass, g	Thickness, mm	Diameter, mm	Density, kg·m ⁻³	Thermal conductivity, W·(m·K) ⁻¹
1	1.3323	3.31	29.97	571.04	1.65
2	1.6817	3.76	30.17	625.95	1.88
3	2.3725	4.51	29.98	745.71	2.14
4	2.7454	4.97	30.10	776.63	2.46
5	3.1546	5.22	29.95	858.06	2.56

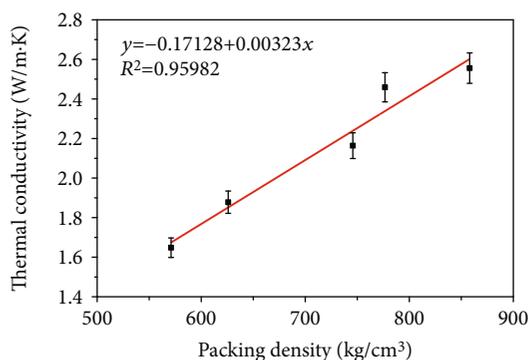


FIGURE 7: PA-SA/EG thermoconductivity variations with the packing density bulk density.

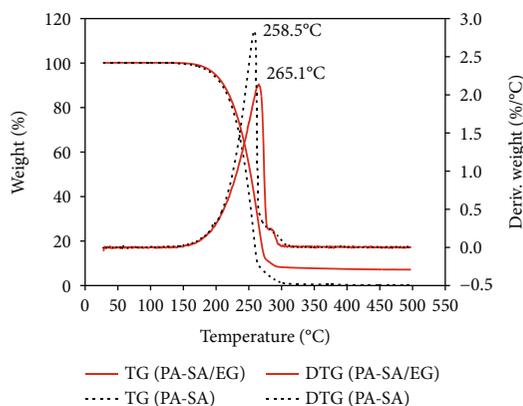


FIGURE 8: Thermogravimetric graphs of PA-SA and PA-SA/EG.

TABLE 5: TGA Characteristic temperatures of the PA-SA and PA-SA/EG PCMs.

Samples	T_{ini} , °C	T_{es} , °C	T_{max} , °C	$T_{-99\%}$, °C
PA-SA	165.7	228.2	258.5	297.6
PA-SA/EG	173.6	238.2	265.1	304.7

4. Conclusions

The form-stable PA-SA/EG composite PCM with an optimum absorption ratio (92.8% PA-SA) was prepared as a novel latent heat thermal energy storage material. The

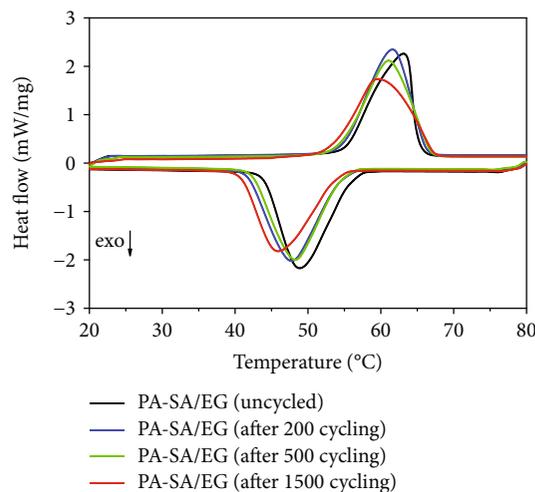


FIGURE 9: DSC graphs of PA-SA/EG composite PCMs before and after heating cycle.

PA-SA eutectic mixture as PCM is confined in the porous EG without melted PCM seepage from the composites. The SEM and FT-IR results indicate that the PA-SA was uniformly distributed in the porous structure of EG and the PCM did not leak even in molten state; PA-SA binary eutectic mixture and EG only have physical interaction without chemical reaction. By using the DSC analysis method, the temperatures of melting and freezing were measured as 55.18 and 54.91°C, respectively, and latent heats of melting and freezing of the composite PCM were and 176.2 J/g and 175.6 J/g, separately. The thermal conductivities of the composite PCMs were much higher than purity PA-SA and linearly increased with the packing density. The addition of EG into PA-SA resulted in a remarkable improvement in the thermal conductivity, which was verified by comparing the melting/freezing times of PA-SA with those of the PA-SA/EG composite PCM. The TGA test indicated that the composite PCM had nice thermostability in the range of operating temperature. The results of thermal cycling test showed that the PA-SA/EG composite PCM has good thermal reliability in terms of thermal properties after 1500 times thermal cycling. Based on all results, it was also concluded that PA-SA/EG especially as a novel form-stable composite PCM can be widespread used in building energy efficiency, solar energy storage, air-conditioning system, utilization of waste heat, and other low-temperature thermal energy storage systems.

TABLE 6: Thermal properties of PA-SA/EG composite PCM before and after thermal cycling.

Number of thermal cycling	Melting		Freezing	
	Temperature, °C	Latent heat, J·g ⁻¹	Temperature, °C	Latent heat, J·g ⁻¹
0	55.18	176.2	54.91	175.6
200	54.59	175.7	54.24	174.8
500	54.47	174.2	54.19	172.1
1500	54.16	170.1	54.08	168.7

Data Availability

The data availability of the manuscript can be found at <https://figshare.com/s/081609e2474cf360a211>.

Conflicts of Interest

The authors declare that there are no conflicts of interest.

Acknowledgments

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