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Study on the Properties of Lauric Acid Paraffin Wax Volcanic Rock Shape-Stabilized Phase Change Materials

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Abstract. Taking lauric acid-paraffin wax (LA-PW) volcanic rock (VR) shapestabilized phase change materials with particle sizes of 1-3mm and 4-6mm as the research objects, analysis was made on the enthalpy, infrared spectrum, leakage characteristics, heat storage, heat release of shape-stabilized phase change materials. The results show that shape-stabilized phase change material has lower enthalpy with the increase in mass fraction of volcanic rock, but the material melting point and freezing point remain basically unchanged, and the shapestabilized phase change material does not produce new substances, displaying stable composition. It can be seen from the leakage characteristics that, under the same ratio, shape-stabilized phase change material with a volcanic rock particle size of 1-3 mm has greater thermal stability than that with a volcanic rock particle size of 4-6 mm.

Keywords. Volcanic rock, Lauric acid, Stearic acid, Shape-stabilized phase change material, Thermal properties

1. Introduction

Phase change materials (PCMs) tend to absorb or release heat while maintaining the temperature unchanged during the phase change process, which are used as temperature control materials in fields such as buildings, electronics, solar heat utilization. During the phase change process, the phase change material has leakage during the solid-liquid state transition [1-2]. Therefore, a suitable way should be chosen to stabilize the shape of the phase change material. At present, the encapsulation methods for phase change materials mainly include the following: melt blending method, microcapsule method and porous material adsorption method [3-5]. For example, Liu et al. successfully prepared paraffin/HDPE shape-stabilized composite phase change material by immersing paraffin wax into high-stability high-density polyethylene via hot-pressing melt blending method. With paraffin wax uniformly dispersed in HDPE, the composite material can be recycled and used stably for a long time, which reduces the problem of PCM leakage during melting [6]. Chen et al. used three polyethylene materials, HDPE, LDPE and LLDPE (linear low density polyethylene) as the support wrapping material of the phase change matrix, and selected paraffin as the phase change matrix to prepare

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a series of composite shape-stabilized phase change materials. The results show that, HDPE-supported PCM is more stable with minimal leakage [7]. Zhou Weibing et al. used lauric acid-stearic acid (LA-SA) as phase change material and expanded vermiculite as support material to prepare shape-stabilized composite PCM. The prepared composite PCM has good thermal stability and low leakage rate [68]. Shen Yongliang et al. used natural ceramsite as the support material and paraffin as the phase change material to prepare shape-stabilized composite PCM, which well overcome the leakage problem during the phase change process [9]. In addition, Guo Yong et al. used decanoic acid-myristic acid eutectic mixture as the core material to prepare phase change material microcapsules with thermoplastic resin as the wall material by solvent evaporation method. The results show that, by using polymethyl methacrylate (PMMA) as the wall material, the prepared microcapsules have good structure and good thermal stability [10]. Wei Jinghua et al. used the chemical precipitation synthesis method to prepare phase change microcapsule materials with palmitic acid as the core material and silica as the wall material. Studies on effect of hydrochloric acid concentration on properties of palmitic acid/silica phase change microcapsules show that the phase change microcapsules prepared when the hydrochloric acid concentration is 0.8 mol/L are spherical and have excellent latent heat properties [11]. In this paper, because the lauric acid-paraffin phase change material has a wide melting point range when mixed according to different mass fractions, and the volcanic rock has a honeycomb structure with good adsorption performance and low cost. Thus, the volcanic rock is used as the support material, and lauric acid-paraffin composite material is used as the phase change material to prepare shape-stabilized phase change materials with different volcanic rock particle size. Through diffusion-exudation circle method, infrared spectrum, cooling characteristic curve of material and thermal enthalpy (DSC) curve, the leakage rate, thermal properties, heat storage and release properties, and thermal stability of the composite PCM are analyzed and studied.

2. Preparation Process of LA-PW/VR Shape-Stabilized Composite Phase Change Material

2.1. Experimental Materials

- Lauric acid (LA): a saturated fatty acid with a melting point of 40°C~44°C.
- Paraffin wax (PW): alias crystal wax, melting point is 47°C~64°C, 58# paraffin wax is used herein.
- Volcanic rock: a granular solid with a honeycomb structure inside.

2.2. Preparation of LA-PW/VR

Step 1: The LA-PW binary hybrid material was prepared by water bath heating method. Each group of materials has a total weight of 200g. LA and PW were initially mixed physically according to the ratio of 35% (70g) lauric acid and 65% (130g) paraffin. After the physical mixing, the material was heated to 80°C by the water bath heating method and stirred uniformly for 20 min to melt the LA and PW. After further mixing, the LA-PW binary hybrid material was poured out and cooled before grinding.

Step 2: Put the volcanic rock in the crucible and smash it, use a sieve to screen the volcanic rock of different particle sizes (1~3mm, 4~6mm), and then put the volcanic rock into the electrothermal blowing dry box with a temperature of 40°C for 8h drying. After drying, take it out and let it cool down.

Step 3: Weigh LA-PW with different mass ratios and VR with different particle sizes using an electronic balance and put them into a labeled beaker. The beaker was placed in an electric vacuum drying oven with a pressure of 0.08 MPa and a temperature of 80°C for 24h vacuum adsorption. Afterwards, take it out and stir evenly and let it cool. After cooling, tableting was performed by weighing and placing 4~5g material into a powder tableting machine, and a cylindrical sample was obtained when the pressure was 5 MPa.

3. Research on the Properties of Shape-Stabilized Phase Change Materials

3.1. LA-PW/VR Enthalpy Analysis

The DSC test of LA-PW/VR was carried out using a thermal comprehensive analyzer. 10 mg LA-PW/VR with a volcanic rock particle size of 1-3 mm and 4-6 mm and lauric acid-paraffin mass fraction of 35%, 40%, and 50%, respectively was selected to test and analyze the Tm peak and enthalpy of LA-PW/VR. The DSC curves of LA-PW/VR are shown in Figures 1 and 2. The Tm peak and enthalpy statistics are shown in Tables 1 and 2.



Figure 1. DSC curve of size of 1-3mm.

Figure 2. DSC curve of v size of 4-6mm.

Table 1	. Enthalpy	data record	d table of v	olcanic roc	k with a	particle siz	e of 1-3mm
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Parameter	Particle siz	ze 1-3mm	Particle size 4-6mm		
Material sample	Tm peak (°C)	$\Delta H (J/g)$	Tm peak (°C)	$\Delta H (J/g)$	
35%M+65% VR	50.18	30.71	51.38	61.54	
40%M+60% VR	51.41	84.36	51.64	71.48	
50%M+50% VR	52.48	100.42	51.73	100.01	

From Figure 1, it can be seen that the composite phase change material has a double peak where LA and PW overlap. It can be seen from Table 1 that the T_m peak of the volcanic LA-PW/VR composite phase change material is in the range of 50°C1-52°C, indicating that the mass fraction of volcanic rock has little effect on the melting point of the composite phase change material. In addition, it can be seen from the statistical results that the enthalpy ΔH of the phase change material decreases gradually with the increase in mass fraction of volcanic rock in the composite PCM.

3.2. LA-PW/VR Leakage Rate Study

The leakage rate of the composite phase change material LA-PW/VR was tested by the diffusion-exudation circle method, and the thermal stability of the shape-stabilized phase change material was studied. First, draw a circle with a radius of 1.5cm on the paper before the test, then draw four concentric circles with radius successively increased by 0.5cm, and then adjust the heating platform temperature to 50°C and let it stabilize. Secondly, place the prepared circular sample at the center of the concentric circle, then place it on the heating platform, and start timing at the same time. Record the time required for the experimental sample to diffuse to the edge of the 1st-4th circles, respectively. The test sample is shown in Figures 3, 4. After the experiment, the samples were removed, and the recorded experimental data are shown in Table 2.





Figure 3. DSC curve of size of 1-3mm.

Figure 4. Leakage rate experiment graph.

Parameter	Particle s	size 1-3mm	Particle size 4-6mm		
Material sample	1st lap (min:s)	3rd lap (min:s)	1st lap (min:s)	3rd lap (min:s)	
90%M+10%VR	01:04	02:57	01.46	04:52	
80%M+20% VR	02:09	06:15	01:41	05:13	
70%M+30% VR	01:51	07:38	01:59	05:12	
60%M+40% VR	03:35	09:06	03:32	05:55	
50%M+50% VR	04:46	11:29	02:24	09:07	
40%M+60% VR	07:36	20:01	03:51	22:30	

Table 2. Experimental data of sample leakage rate.

The experimental results show that under the same mass fraction, volcanic rock with a particle size of 1-3 mm generally has better thermal stability than volcanic rock with a particle size of 4-6 mm. Secondly, regardless of particle size 1-3mm or 4-6mm, the greater the mass ratio of volcanic rock, the better the thermal stability. Therefore, volcanic rock has a certain bearing capacity against the LA-PW binary hybrid material, which can increase the flow of LA-PW low eutectic mixture, reduce the leakage rate of the composite phase change material, and enhance the thermal stability of the phase change material.

3.3. LA-PW/VR Fourier Transform Infrared Spectroscopy

LA-PW/VR with the LA-PW mass ratio of 35%, 40% and 50% was selected to analyze its Fourier transform infrared spectrum, with results shown in Figure 5.



Seen from the Fourier transform infrared spectrum, after the LA-PW binary hybrid material is mixed with VR, phase change material with LA-PW mass fraction at 35%M, 40%M, 50%M, 100%M exhibit obvious characteristic peaks. The highest peaks are all between (2800-3000) cm-1, indicating that while preparing LA-PW/VR composites by adding VR to the binary hybrid material LA-PW, no new substances are generated and the physicochemical properties of the materials are unchanged.

3.4. LA-PW/VR Heat Storage and Heat Release Characteristics Study

The heat storage characteristics and heat release characteristics of LA-PW/VR were experimentally studied by water bath method. The LA-PW/VR composite material with LA-PW mass fraction of 90%, 55%, 50% and volcanic rock particle size of 1-3mm was selected as the test sample. A 50ml glass Erlenmeyer flask was used to hold the LA-PW/VR phase change material. The temperature sensors were buried in LA-PW/VR and water, respectively, and the endothermic temperature and exothermic temperature of LA-PW/VR and water were acquired by USB-2000 data acquisition system. When testing the heat storage characteristics of the shape-stabilized material, the LA-PW/VR composite phase change material was heated to 80°C, and the water temperature was kept 0-8°C higher than the temperature of the composite phase change material during the heating process. When testing exothermic characteristics of the shape-stabilized material, the melted composite phase change material released heat in a water bath environment, and the temperature of the composite phase change material was kept 0-8°C higher than the water temperature during the test. The measured heat storage and heat release characteristics of the LA-PW/VR hybrid material are shown in Figures 6 and 7.







Figure 7. Solidification curve of LA-PW/VR.

The figure shows that during the melting process of the composite phase change material, the melting point is in the range of $51^{\circ}C-53^{\circ}C$, and during the solidification process, the freezing point is in the range of $50^{\circ}C-52^{\circ}C$, indicating that the melting point and freezing point temperature of the composite phase change material are approximately equal. In addition, the mass fraction of volcanic rock in the composite phase change material creates little effect on the melting point and freezing point of the composite phase change material.

4. Conclusion

Through the preparation of LA-PW/VR with different particle sizes of volcanic rock and the test of thermophysical properties, the results show that:

- The Tm peak of LA-PW/VR composite phase change material with different mass fractions of volcanic rock is in the range of 50°C1-52°C, indicating that mass fraction of volcanic rock has little effect on the melting point of the composite phase change material. The enthalpy △H of the phase change material decreases gradually with the increase in the mass fraction of volcanic rock in the composite PCM.
- 2. Under the same mass fraction, volcanic rock with a particle size of 1-3 mm has better thermal stability than volcanic rock with a particle size of 4-6 mm, and the larger the mass fraction of the volcanic rock, the better the thermal stability.
- 3. In the prepared volcanic rock shape-stabilized composite phase change material, the physicochemical properties of the material LA-PW and volcanic rock were not changed, and no new substances were produced.
- 4. The cooling characteristics experiments of the composite phase change material show that adding PE in different mass ratios to the binary hybrid material LA-PW creates little effect on the melting point and freezing point characteristics of the material.

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