

Article

Analysis on the Improvement of Thermal Performance of Phase Change Material Ba (OH)₂·8H₂O

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Abstract: Benefitting from the characteristics of a high latent heat capacity and stable phase change behavior, phase change materials have widely received concerns in the field of thermodynamic management. Ba(OH)₂·8H₂O is an ideal phase change material (PCM) in the mid-to-low temperature range, but its large-scale application is still limited by severe supercooling during the nucleation process. In this paper, the experimental analysis and comparison are performed via an Edisonian approach, where Ba(OH)₂·8H₂O is adopted as an original substrate; BaCO₃, CaCl₂, NaCl, KH₂PO₄, and NaOH are selected as nucleating agents; and graphite is used as a heat-conducting agent. The results show that Ba(OH)₂·8H₂O containing 1.2% BaCO₃ and 0.2% graphite powder has the best performance. Compared with pure Ba(OH)₂·8H₂O, the supercooling degree is reduced to less than 1 °C, the phase change latent heat duration is extended, and the thermal conductivity is significantly improved. Therefore, this study not only provides a reference for the application of Ba(OH)₂·8H₂O, but can also be used as a guidance for other material modifications.

Keywords: phase change; Ba(OH)₂·8H₂O; nucleation; supercooling; latent heat



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1. Introduction

Nonrenewable fossil energy still plays an important role in most industrial fields around the world. However, fossil energy inevitably produces a large amount of greenhouse gas CO₂ and other pollutants, thus threatening human health and the global ecological environment [1–3]. In this consideration, mankind tends to explore renewable energy sources and improve the energy demand in the development of future societies. Phase change latent heat, as one of the effective methods of storage technology, can store a large amount of heat during the phase change process. This method mainly relies on the endothermic effect of the phase change material (referred to as PCM) to store the heat absorbed or released during the phase change process [4]. Phase change material (PCM) refers to a substance that converts its states as temperature evolution and provide latent heat. Thus, PCM is an effective means to improve energy efficiency and reduce energy consumption, which has become the focus of current research [5–8]. In the actual application of PCM, it is necessary to satisfy the requirements, e.g., suitable phase change temperatures, higher latent heat values, constant temperature, good economy, and high safety. Inorganic hydrated salt PCM is a commonly used material because of its high latent heat density and its desirable economic benefits. Nevertheless, the supercooling phenomenon of the inorganic hydrated salt leads to low latent heat recovery rates and insufficient material stability. This is a periodic change, and the major problem should be solved for the development of energy storage technology [9–11]. Supercooling, as a metastable state arising from the liquid–solid phase transition process, can provide the energy required for ion diffusion in the solution and interface, as well as crystal growth and crystal plane expansion [12,13]. In addition, the degree of the delay in the crystallization process is called the supercooling degree [14].

In terms of the supercooling degree, Lu et al. [15] have studied binary eutectic hydrated salt and analyzed the supercooling situation of two hydrated salts with different mass ratios. The results showed that when the mass ratio of two hydrated salts obtained 1:9, the lowest degree of supercooling was 8.1 °C. He et al. [16] used $\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$ and $\text{Ba}(\text{OH})_2$ as nucleating agents to improve the supercooling performance of calcium chloride hexahydrate, and the average supercooling degree was reduced to 1.07 °C. Yong et al. [17] explored the supercooling characteristics of $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$ phase change materials. After adding 5.3 wt% alumina as a nucleating agent, the temperature of supercooling was decreased to 1.4 °C. Although many researchers have made efforts to improve the supercooling performance through nucleating agents, the supercooling degree of modified PCM is still above 1 °C. Li et al. [18] analyzed the influence of the BaSO_4 , $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$, $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$, and $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$ nucleating agents on $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$. They found that the supercooling characteristics of the substrate was effectively improved, and an additional amount of 2% performed a more significant effect. Sheng et al. [19] showed that the phase transition temperature of $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$ was 78 °C. In the room temperature cooling open system, adding 3% or 5% borax and calcium chloride dihydrate to $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$, respectively, had a positive impact on reducing the supercooling degree of $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$. Based on the literature survey, it is known that $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$ is an important PCM, but its application in practical fields is limited by the supercooling issue. This article attempts to conduct further optimization on the basis of the above studies. The researched nucleating agent can not only reduce the degree of supercooling, but also increase the thermal conductivity so as to achieve a better ratio of adding as little as possible, thereby reducing the cost.

In terms of the thermal performance, Cui et al. [20] investigated the thermal performance and practicability of $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$ as a PCM. They designed different paraffin wax and $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$, and further conducted copper test tubes for phase analysis. The melting–solidification thermal cycle experiment indicated that the paraffin covering not only effectively inhibited the evaporation of crystal water, but also prevented the $\text{Ba}(\text{OH})_2$ reacting with CO_2 to form BaCO_3 , thus affecting its heat storage performance. Nevertheless, the analysis on the phenomenon of supercooling and thermal conductivity has not been made. Li et al. [21] adopted expanded perlite to improve the thermal conductivity of composite phase change materials.

Therefore, the composite PCM can compensate the shortcomings of a single material to a certain extent. Although there are many studies on the supercooling and thermal properties of phase change materials, few experimental studies have focused on the supercooling of $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$. In this paper, $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$ is used as the original base material of the composite material. Moreover, nucleating agents can provide crystallization sites to reduce the supercooling of the hydrated salt. Moreover, the nucleating agents are mostly selected from substances with a similar molecular structure to hydrated salt, or to substances with higher phase transition temperatures than that of raw materials. When the temperature drops to the freezing point of PCM, they can be employed to induce nucleation and crystallization of hydrated salt [22]. Five nucleating agents, i.e., BaCO_3 , CaCl_2 , NaCl , KH_2PO_4 , and NaOH , are selected here. Subsequently, the comparative analysis with pure $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$ is conducted to study the effects of several nucleating agents on the supercooling performance of the original substrate. After finding the best ratio, graphite is adopted as a thermal conductivity additive to improve the thermal conductivity of composite PCM, thus ensuring the stable performance of $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$ in practical applications.

2. Materials and Methods

2.1. Experimental Materials and Equipment

The materials used in this experiment are all analytical grades and provided by Fuchen (Tianjin, China) Chemical Reagent Co., Ltd. (as shown in Table 1).

The following equipment was used: Electric heating constant temperature water tank, DK-S600, made by Shanghai Shenxian Thermostat Equipment Co., Ltd. (Shanghai, China).

The specific temperature accuracy is $\pm 0.5\text{ }^{\circ}\text{C}$, temperature control range is $\text{RT} + 5\sim 99\text{ }^{\circ}\text{C}$; the T-type thermocouple is used for temperature measurement, manufactured by Shanghai Nanpu Instrument Co., Ltd. (Shanghai, China) and the accuracy is $\pm 0.5\text{ }^{\circ}\text{C}$, the temperature measurement range is $-200\sim 350\text{ }^{\circ}\text{C}$; the electronic mass balance FA2004, made by Shanghai Shangping Instrument Co., Ltd. (Shanghai, China) (accuracy $\pm 0.1\text{ mg}$), the maximum range is 200 g; Agilent data acquisition 34980A, American Agilent Technologies Co., Ltd. (Santa Clara, CA, USA) (accuracy $\pm 0.1\text{ }^{\circ}\text{C}$), range is 0~150 mm; the thermal conductivity test is performed by Hotdisk TPS 2500s, Sweden Hotdisk Co., Ltd. (Gothenburg, Sweden); the differential scanning calorimeter DSC 200F3 is used for the latent heat value test, Germany Netzsch Co., Ltd. (Selb, Germany), and its accuracy is $\pm 0.1\text{ }^{\circ}\text{C}$.

Table 1. Experimental material parameters.

Type	Name	Chemical Formula	Purity Content
Nucleating agent	Barium carbonate	BaCO_3	$\geq 99.0\%$
	Calcium chloride	CaCl_2	$\geq 96.0\%$
	Sodium chloride	NaCl	$\geq 99.5\%$
	Potassium Dihydrogen Phosphate	KH_2PO_4	$\geq 99.5\%$
	Sodium hydroxide	NaOH	$\geq 96.0\%$
Thermal conductive agent	Graphite	—	$\geq 98.0\%$

2.2. Experimental Method

$\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$ has the advantages of a high latent heat value during the phase change process, low phase change temperature, high efficiency, and low economic cost. Due to these benefits, this experiment uses $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$ as the original base material. What is more, the nucleating agents can provide additional crystallization sites to reduce the supercooling of hydrated salt. Five kinds of nucleating agents are selected, BaCO_3 , CaCl_2 , NaCl , KH_2PO_4 , and NaOH , and graphite is considered as a thermal conductive agent for the experimental analysis. At present, the use of nucleating agents is one of the most effective and economical methods for reducing the supercooling of the hydrated salt phase change materials. However, while the search for nucleating agents has a certain theoretical basis, most of them need to rely on the “Edisonian approach”, that is, using intuition to find the best nucleating agent through multiple experiments with a large number of materials. In this study, the Edisonian approach is taken to screen the nucleating agent. The preparation method is as follows:

Firstly, an electronic balance was used to weigh 6 parts of 50 g $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$ so to put them into 6 identical 50 mL beakers. Then, weighing 5 parts of 0.5 g (1%) of BaCO_3 , CaCl_2 , NaCl , KH_2PO_4 , and NaOH as nucleating agents and mixing them with the previously weighed 5 parts of $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$, followed by a mechanical stirring with the glass rod. In order to ensure the accuracy of the measured data and to prevent the loss of crystal water during the heating process, the beaker lid was covered with a small round hole in the middle and a T-type thermocouple was inserted to monitor the temperature evolution at the center of sample to ensure the accuracy of data. Then, the sample was put it into an electric thermostatic water tank and heated to $90\text{ }^{\circ}\text{C}$ again. After the sample was completely melted, it was removed, and a cooling test was performed at room temperature. Following the same steps as illustrated above, five different types of nucleating agents were weighed as 1.0 g (2%) and 1.5 g (3%) and analyzed through heating and cooling tests. According to the test results, the best ratio of the nucleating agents could be determined. Then, the refinement experiment was performed again to select the best nucleating agents from the different mass fractions. Based on the optimal ratio of the nucleating agent, graphite powder as the heat-conducting filler was added to the composite $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$ phase change material. Besides, different amounts of graphite powder were considered and mixed with the above composite, and the step cooling curve test was conducted. Apart

from that, the DSC and thermal conductivity of as-prepared composite PCM was measured again after multiple cycles to obtain its thermophysical properties.

2.3. Phase Change Material Experiment Platform Construction

Figure 1 shows the system platform designed for this experiment. The main experimental equipment is as follows:

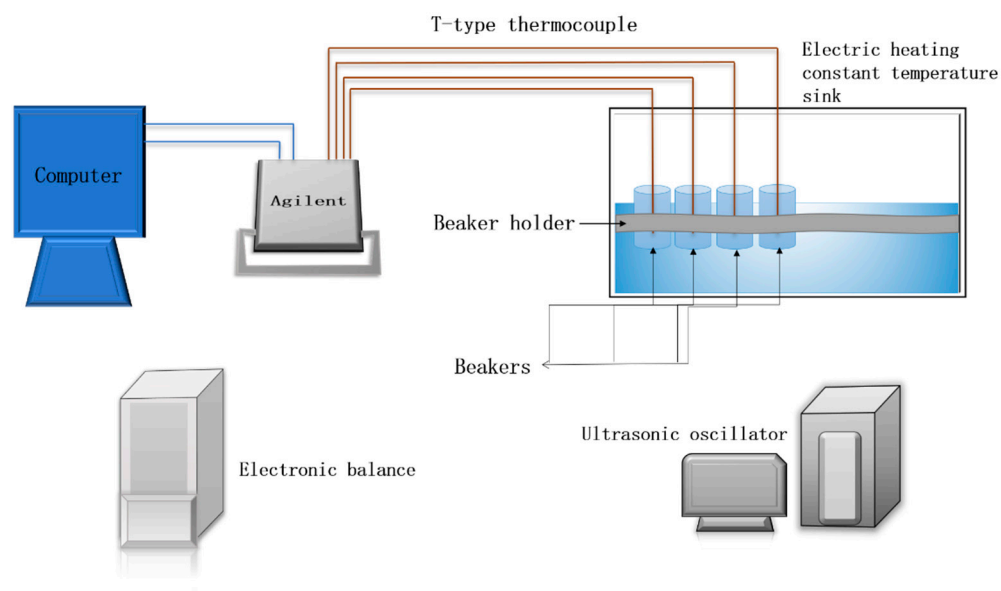


Figure 1. Experimental platform system.

3. Experimental Results and Discussions

3.1. Test Results and Analysis of Substrate Supercooling

In general, the heat storage performance of the phase change heat storage materials is mainly determined by factors such as the supercooling degree and the latent heat value. Moreover, the thermophysical parameters of $\text{Ba(OH)}_2 \cdot 8\text{H}_2\text{O}$ exhibit slight variation to some extent, which is caused by the difference of manufacturer and product specifications. Hence, in this experiment, the supercooling degree of $\text{Ba(OH)}_2 \cdot 8\text{H}_2\text{O}$ material is tested according to the temperature measurement test bench, thus providing accurate parameters for the subsequent data analysis. The measured results of pure $\text{Ba(OH)}_2 \cdot 8\text{H}_2\text{O}$ are shown in Figure 2.

The experiment of $\text{Ba(OH)}_2 \cdot 8\text{H}_2\text{O}$ is shown in Figure 2, and it can be concluded that the phase transition temperature of $\text{Ba(OH)}_2 \cdot 8\text{H}_2\text{O}$ is about 78.1°C . During the exothermic process from liquid to solid, the PCM begins to crystallize and release latent heat when temperature drops to around 72°C , the degree of supercooling is about 6.1°C , and the duration of the latent heat of phase change is 2265 s. Notably, the duration of the phase change will affect the latent heat performance of the PCM in the latent heat stage.

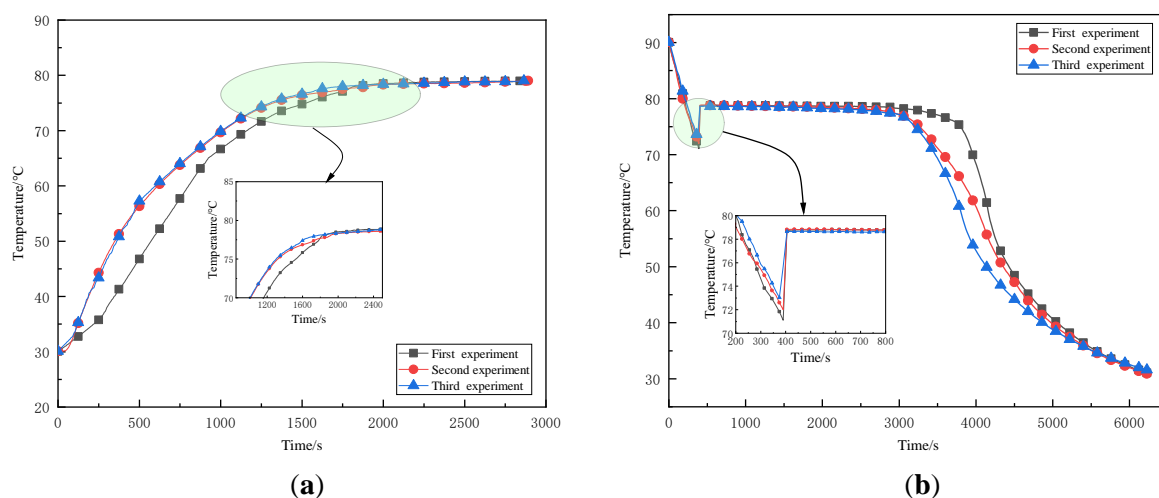


Figure 2. (a) Base material $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$ heat curve; (b) base material $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$ exotherm curve.

3.2. Analysis of the Influence of Different Types of Nucleating Agents on Supercooling Degree of $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$

In order to determine the optimal ratio among different types of nucleating agents, we performed a series of tests for different types of nucleating agents. When 0.5 g (1%), 1.0 g (2%), and 1.5 g (3%) are added, the corresponding result is shown in the Figure 3 below:

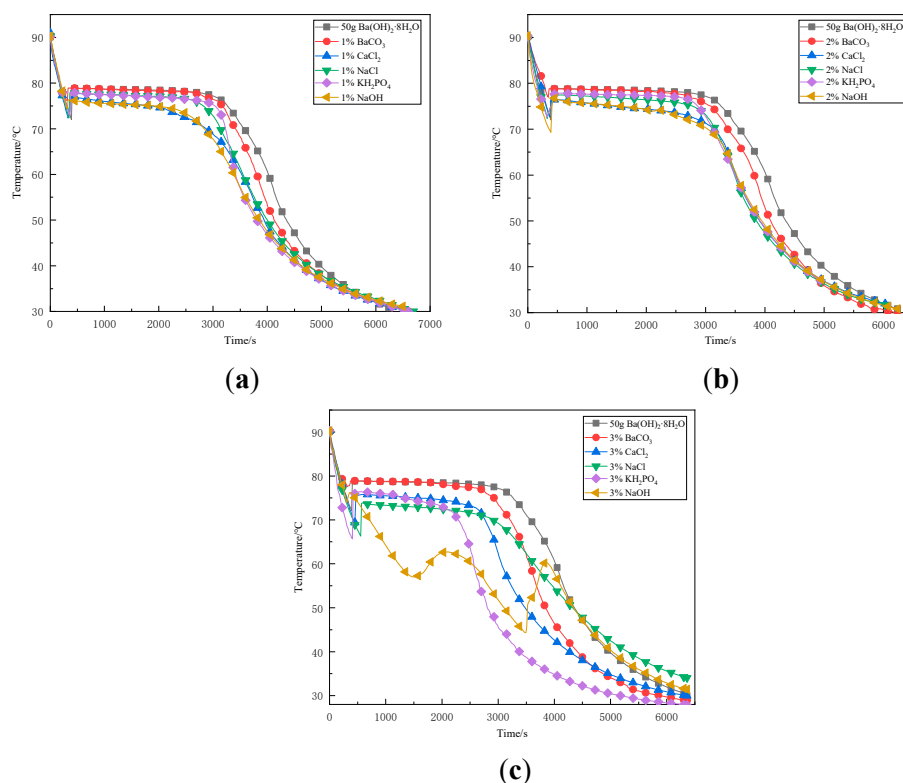
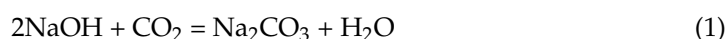


Figure 3. (a) The addition amount of nucleating agent is 1%; (b) the addition amount of nucleating agent is 2%; (c) the addition amount of nucleating agent is 3%.

From the experimental results illustrated in Figure 3a, it can be seen that the addition of BaCO_3 , CaCl_2 , NaCl , KH_2PO_4 , and NaOH to $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$ all takes a positive part in modifying the supercooling to some extent. When the addition amount of BaCO_3 is 1%, it

can be found from the curve that the degree of supercooling is as low as 76.1 °C, which is 4.1 °C lower than pure Ba(OH)₂·8H₂O. When the addition of CaCl₂, NaCl, KH₂PO₄, and NaOH reaches 1%, the degree of the supercooling of the curve is reduced to 72.4 °C, 72.5 °C, 72.9 °C, and 74.4 °C, respectively, and the enhancing effect is not obvious compared with pure Ba(OH)₂·8H₂O. The reason is attributed to the relatively small amount of addition and the different effects of various materials' supercooling degree. In other words, it is related to the amount of addition. In order to further verify the relationship with the amount of the nucleating agent added, the amount of the nucleating agent is continuously increased (as shown in Figure 3b). When the content of the nucleating agent is improved to 2%, the supercooling degree of BaCO₃ decreases to 77.3 °C. It is worth noting that the addition of 2% NaOH is less effective. The explanation is that NaOH chemically reacts with air more easily. Once it is exposed to a small amount of CO₂, the following reaction will occur:



and when exposing with excess CO₂, the following reaction will occur:



In order to ensure the accuracy of the data, the behavior of the composite PCM with 3% nucleating agent was tested (as shown in Figure 3c). Owing to the existence of BaCO₃, the degree of the supercooling is reduced to 77.5 °C, which is further improved compared to the addition of 1% or 2%. However, the duration time of the phase change is significantly shortened. On the other hand, after incorporating CaCl₂, NaCl, KH₂PO₄, and NaOH, the supercooling degree is gradually increased in the opposite direction. In addition, the profile of NaOH also fluctuates greatly, which verifies that NaOH reacts very easily with air and is not suitable as a nucleating agent for Ba(OH)₂·8H₂O.

By comparing the above three sets of experimental data, it can be seen that the addition of an amount of BaCO₃ in the nucleating agent is similar to that of pure Ba(OH)₂·8H₂O and the exothermic curve is relatively similar. In this case, the latent heat performance does not significantly decrease, while the effect becomes obvious when the addition amount is 1% and 2%. With the gradual expansion of the addition amounts, the degree of supercooling is gradually reduced, but the duration time of the phase change is significantly shortened. Therefore, the phase change behavior and the latent heat duration of the composite PCM is also varied with the addition of the nucleating agent. The experimental results show that BaCO₃ has the best effect among the five nucleating agents, and the optimal addition amount is 1% and 2%.

3.3. Adding Different Mass Fractions of Nucleating Agents in the Supercooling Test

Figure 4 shows the exothermic profiles of Ba(OH)₂·8H₂O with different addition amounts of BaCO₃ from 1% to 2%. When the addition of BaCO₃ is 1.2%, the supercooling degree reduced to about 77.1 °C and the latent heat release time during the process is 2310 s. Compared with the original substrate, the phase change performance of the composite PCM is effectively improved. When the addition amount of BaCO₃ is 1.4%, the supercooling degree of the curve is reduced to about 76.4 °C, and the latent heat release time is 2205 s. When the addition amount of BaCO₃ is 1.6%, the supercooling degree of the curve is reduced to about 76.8 °C, and the latent heat release time is 2145 s. When the addition amount of BaCO₃ is 1.8%, the supercooling degree of the curve is reduced to about 77.2 °C, and the latent heat release time is 1920 s, which is 345 s shorter than that of pure Ba(OH)₂·8H₂O. It can be seen from the data that when the addition amount is between 1.4% to 1.8%, the supercooling degree of the Ba(OH)₂·8H₂O composite PCM gradually decreases, but the latent heat duration of the phase change is the opposite. Therefore, considering the trade-off between the nucleation effect and the latent heat capacity of the composite PCM, the optimal mass fraction of BaCO₃ is regarded 1.2% in the original base

material of pure $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$ due to its best effect. In this consideration, the degree of supercooling is as low as possible, with a value less than 1°C .

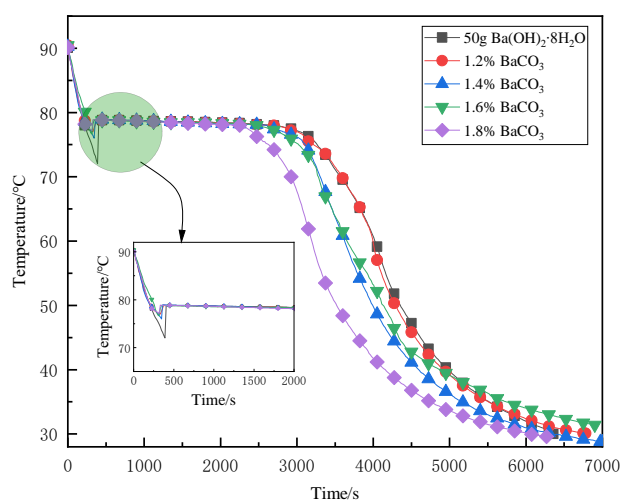


Figure 4. Exothermic curve diagram of different mass fraction BaCO_3 .

3.4. Add Graphite Powder with Different Mass Fractions for Testing

Figure 5 shows the results for the composite phase change heat storage material sample (BaCO_3 added at 1.2%) incorporating different qualities of graphite powder. The experiments show that as there is an increase of graphite powder, the phase change heat release time and the phase change temperature of the composite phase change heat storage materials are gradually decreased. In the graphite addition range, the phase transition temperature and the phase transition platform of the graphite powder with the addition amounts of 0.1% and 0.2% in the latent heat exothermic stage are close to as those without the graphite powder. Besides, the temperature decrease rate in the sensible heat stage is faster than that of the $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$ sample without the graphite powder. When the amount of the graphite powder is more than 0.2%, the temperature decrease rate is relatively fast, the phase transition temperature is greatly reduced, and the phase transition duration is significantly shortened. When the addition of the graphite powder increases, the thermal conductivity of the sample correspondingly improves, and the heat transfer rate also grows accordingly. Through the DSC test and Hotdisk test, the results are shown in Figure 6 below.

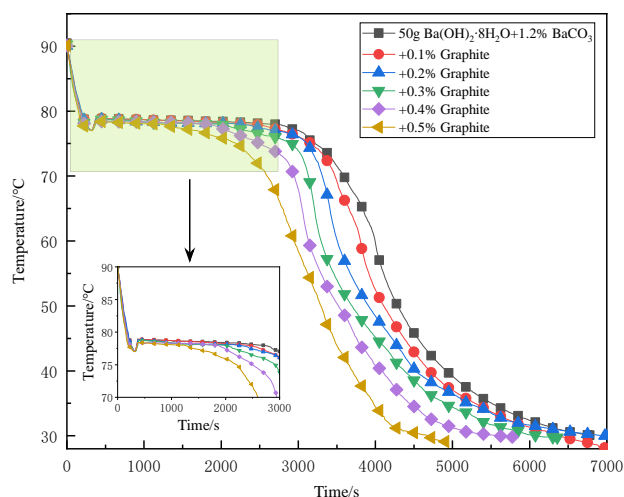


Figure 5. Supercooling curve diagram of graphite powder with different mass fractions.

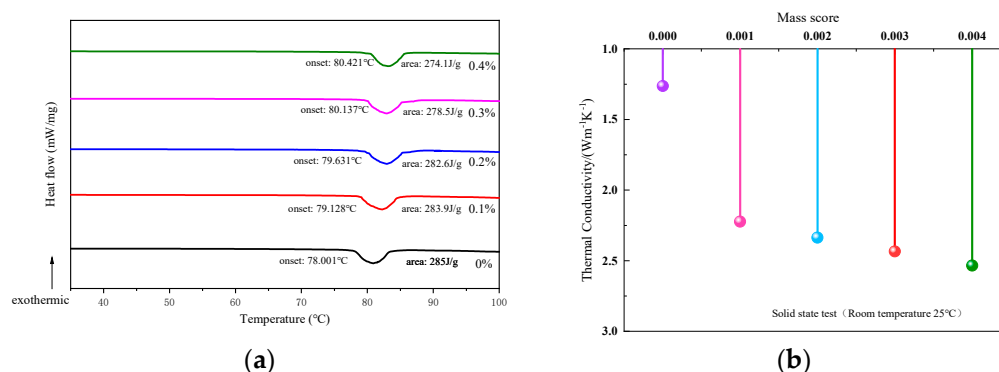


Figure 6. (a) DSC test chart; (b) Hotdisk test chart.

From the results of Figure 6a, it can be seen that with the increasing amount of thermally conductive graphite, the phase transition temperature change of the composite PCM is closer to that without the graphite, and the fluctuation is small. For the variation of the latent heat value, with the addition amount of thermally conductive graphite, the latent heat value shows a downward trend. Especially when the addition amount is greater than 0.2%, the rate of decrease is faster. Due to the addition of thermally conductive graphite, the solidification nucleation of $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$ composite phase change heat storage material and the heat exchange with environment are accelerated, thereby affecting the latent heat storage and the heat release performance of the material. Therefore, from the DSC test results, the graphite addition at 0.1% and 0.2% exhibits better effects in the test range. From Figure 6b, it is observed that the thermal conductivity of the composite PCM increases rapidly as the addition of graphite powder also increases. When the amount of the graphite powder gradually increases, its thermal conductivity will gradually expand. Therefore, it can be seen that adding graphite is an effective method to improve the thermal conductivity. Due to the above experimental DSC test, when the graphite addition amount is 0.1% and 0.2%, the latent heat value is closer to the latent heat value of adding no graphite, and the phase transition temperature fluctuation is small. What is more, the heat conduction coefficient at 0.2% graphite addition is higher than that at 0.1% graphite addition, which can enhance the heat transfer efficiency of the substrate. Through the above analysis and comprehensive consideration, the effect is better when the addition amount of the graphite is 0.2%.

4. Conclusions

In this paper, the supercooling and thermal properties of phase change materials are studied by using $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$ as the original basis material. In addition, the effects of the different nucleating agents and thermal conductivity agents are investigated through experimental comparison, and the following conclusions are drawn:

(1) In order to improve the supercooling performance of the original base material $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$, the Edisonian approach is adopted to compare the different nucleating agents. The experimental results show that BaCO_3 acting as the nucleating agent can greatly reduce the supercooling degree with an addition amount of 1.2%. When it is lowered to less than 1 °C, the latent heat duration of phase change is longer than the pure $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$.

(2) Graphite powder is selected as the thermal conductive agent for the composite PCM, and tests are performed to explore effect of graphite under different mass fractions. The temperature, DSC, and Hotdisk tests are conducted to measure the step cooling curve, the latent heat value, and the thermal conductivity, respectively, thus obtaining the best heat conducting property. According to experimental analysis, with the aim of improving the heat storage efficiency of PCM during the latent heat stage and to strengthen the heat transfer effect of the base material, the optimal addition amount of graphite powder is 0.2%, with little effect on the latent heat value.

Therefore, based on the comprehensive considerations of the supercooling degree, the phase change latent heat value, and the latent heat duration, it is concluded that adding 1.2% BaCO₃ + 0.2% graphite powder to the Ba(OH)₂·8H₂O phase change material has the best effect. In this case, the supercooling degree of the PCM can be reduced to less than 1 °C, and its thermal conductivity is also effectively improved. This work provides a reference for the application of Ba(OH)₂·8H₂O, and can also be used for the study of other material properties.

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