



Study on the synthesis and thermal properties of magnesium chloride hexahydrate–magnesium sulfate heptahydrate–activated carbon phase change heat storage materials

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Abstract

As the phase change heat storage materials, the hydrated salts have good application prospects. However, the supercooling and phase separation of hydrated salts are two key factors, which affect their thermal properties and thermal stability. Moreover, the thermochemical reaction of hydrated salts below 130 °C is very important for its phase-change heat storage performance. A new composite phase-change material (CPCM) with good heat storage performance and thermal stability composed of magnesium chloride hexahydrate (MCH), magnesium sulfate heptahydrate (MSH), and activated carbon (AC) was synthesized. The effects of AC on supercooling, phase separation, and phase transition temperature of CPCM were investigated. The results showed that phase separation and supercooling were completely eliminated in CPCM with 3.0 wt% AC, and the content of AC had a significant effect on thermal cycle stability, heat storage performance, and enthalpy balance. CPCM has good thermal cycle stability. CPCM with the content of 3.0 wt % AC possessed the phase transition temperature of 90.21 °C and the phase transition enthalpy of 156.14 kJ kg⁻¹, which is very suitable for household heat storage.

Keywords Composite phase-change material · Heat storage · Supercooling · Thermal cycle stability

1 Introduction

The accumulation of heat is one of the key technologies to realize the clean and efficient utilization of renewable energy. The storage and release of heat can solve the intermittent use of energy and improve its utilization efficiency. [1, 2]. At present, sensible heat storage, chemical heat storage, and latent heat storage are the three main heat storage technologies. Phase-change materials (PCM) have attracted much attention as the latent heat storage medium due to its advantages of high heat storage density, constant latent

temperature, and easy control. PCM can absorb heat (cold) from the environment and release heat (cold) to the environment, so as to control the ambient temperature [3]. Its thermal stability and heat storage capacity are the key factors in its application [4]. PCM are divided into inorganic PCM, organic PCM, or both. The inorganic PCM have the advantages of high latent heat, high thermal conductivity, and small volume change, but they have the disadvantages of corrosion, supercooling, and phase separation in the process of phase change [5]. Several salt experiments in a closed thermal cycle system showed that supercooling and phase separation were the key disadvantages of PCM [6–10]. It is worthy of attention to overcome the shortcomings of PCM to improve its comprehensive performance [11–15].

Bugaje [16] pointed out that phase-change time was an important parameter of phase-change heat storage system, because high thermal conductivity can ensure rapidly heat transfer speed. The Netherlands Energy Center (ECN) [17, 18] studied that the heat storage property of MgCl₂·6H₂O from room temperature to 130 °C was measured by XRD and TG/DSC, the results showed that the heat storage property of MgCl₂·6H₂O was greatly affected by the partial pressure of water vapor and dehydration temperature. Moreover,

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when the temperature was higher than 135 °C, the heat storage density of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ was seriously reduced due to the formation of HCl by hydrolysis reaction. Van Essen et al. [19] studied that the hydration and dehydration characteristics of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ as thermochemical heat storage materials by TG and XRD and proposed that $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ can be dehydrated below 150 °C to realize heat storage, but the release of heat from storage was difficult. The cycle stability and thermal conductivity of PCM can be improved by introducing carbonaceous materials, which was due to the high thermal conductivity and porosity of carbon materials [20–22]. Mauran S et al. [23] showed that the thermal conductivity of CaCl_2 with carbonaceous materials was 40 times higher than that of pure CaCl_2 , but they were easy to deliquesce [23, 24].

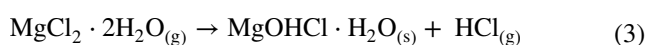
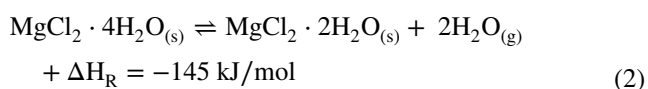
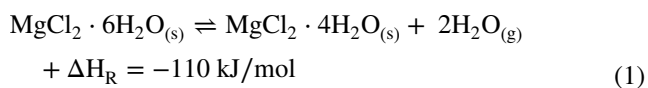
$\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$, as a phase change material, also has some inherent disadvantages, such as high undercooling, phase separation, corrosiveness, easy deliquescence, and unstable circulation. In this paper, $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ with stable thermal performance, high heat storage capacity, and common cation (Mg^{2+}) was introduced into $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$, and the MCH ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$)—MSH ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$)—AC composite phase change energy storage system was constructed by adding AC (activated carbon). The suppression effect of AC on MCH-MSH supercooling and phase separation was explored, and the heat storage capacity and heat storage stability of the system were studied.

2 Experiment section

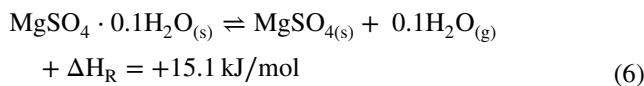
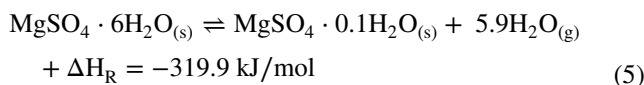
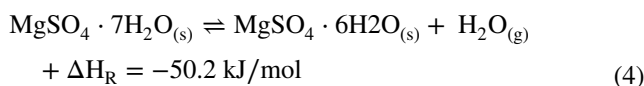
2.1 Materials

The materials used in the work were $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ (MCH), $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ (MSH), and activated carbon (AC), which were obtained, respectively, from Penglai Shuangshuang Chemical Co. LTD., China, Tianjin Beichen Founder Reagent Factory, China, and Henan Songshan Technology Co. LTD., China. The phase transition temperature and energy density $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ are 117 °C. The specific surface area of AC is 1529.8 m^2/g .

The dehydration/hydration reaction of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ in the system [25] is as follows:



The dehydration/hydration reaction of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ in the system [25] is as follows:



2.2 Sample preparation

About 10 g with different composite materials was prepared according to the proportion in Table 1, respectively. Firstly, the mixed salt-containing MSH and MCH was prepared according to the proportion in Table 1. The melting state of the mixture of MSH and MCH is shown in Fig. 1. The configuration process is to first mix the solid $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ and the solid powder AC uniformly in a proportional beaker, then heat the mixture to 120 °C and stir for 5 min to make the $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ liquid, then slowly add the powder of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$, again stir for 5 min to make the AC appear uniformly dispersed in the MCH-MSH system, and cool the MCH-MSH-AC system to room temperature to make it appear solid, as shown in Fig. 2.

The morphology of the phase change material in the phase change process will affect the phase change performance of the material. If phase separation occurs, it will affect the energy storage efficiency of the material, which is not conducive to practical applications. According to Fig. 1, it can be seen that the composite material of S0 did not exhibit phase separation after heating and melting, and S(0–1), S(0–2) and S(0–3) composites had phase separation and deposits at the bottom.

Table 1 System composition in the experiment

PCM	[m (MCH): m (MSH): m (AC)]/%
S0	95.00:5.00:0
S(0–1)	90.00:10.00:0
S(0–2)	85.00:15.00:0
S(0–3)	80.00:20.00:0
S1	94.05:4.95:1.00
S2	93.10:4.90:2.00
S3	92.15:4.85:3.00
S4	91.20:4.80:4.00

It can be seen from Fig. 2a that the mixture of MCH and MSH presents a transparent layered structure. Figure 2c–e shown that there was still a little population stratification when AC content was low, as shown in Fig. 2b, but the AC was evenly distributed in the MCH-MSH with the increase in AC content, which indicated that AC can be well integrated with MCH-MSH at the appropriate AC content.

2.3 Test method

The schematic diagram of the experimental process is shown as Fig. 3. The beaker containing MCH-MSH-AC system was moved to the self-designed incubator together with electrical heater (100 W). The thermocouple was placed in the middle of the solution and connected to a data acquisition unit (34970A BenchLink Data Logger), and the data acquisition unit was connected to a computer. The electrical heater heats

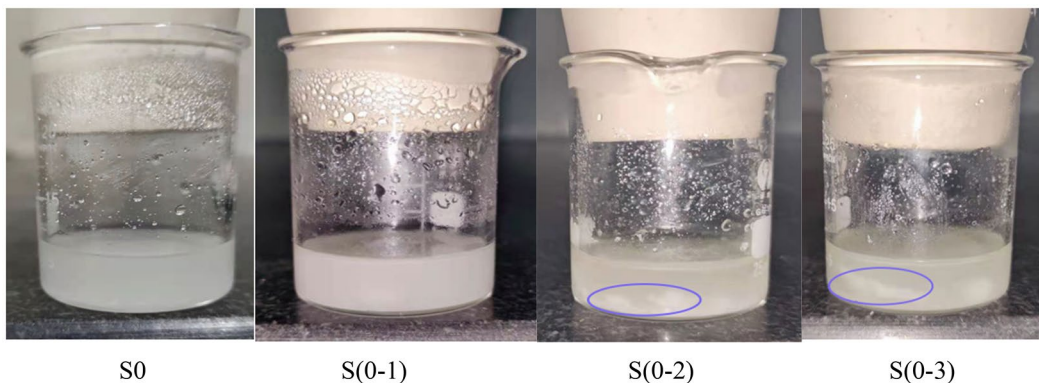


Fig. 1 The picture of S0, S(0–1), S(0–2), and S(0–3) after heating and melting

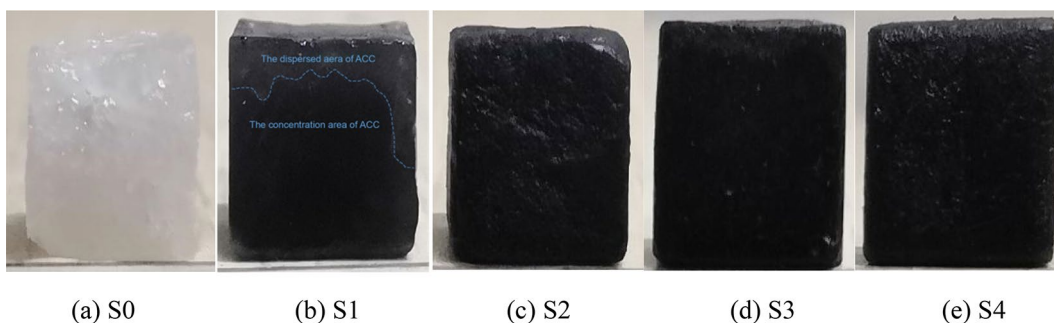


Fig. 2 The picture of samples with different contents of AC

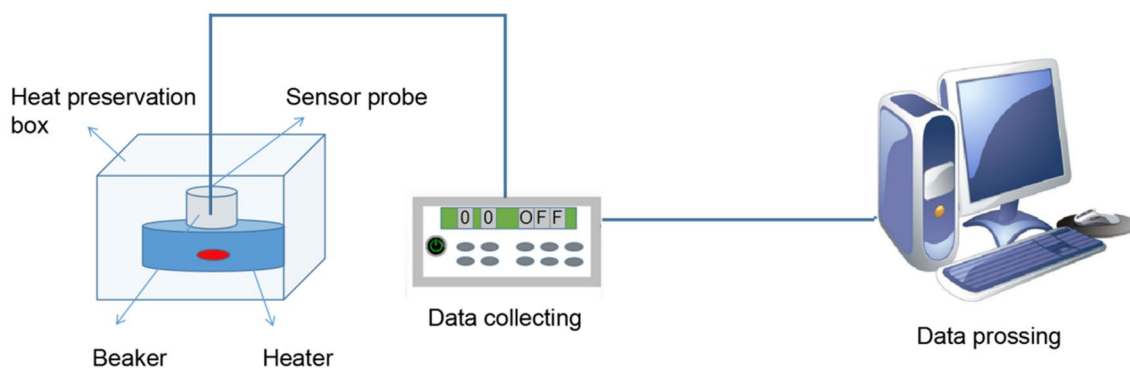


Fig. 3 Schematic diagram of the experimental process

the MCH-MSH-AC system to 120 °C making it melt completely, and the raised temperature data were measured and stored synchronously to the computer. The electrical heater stops heating and MCH-MSH-AC system cools naturally, and the computer records and stores cooling data synchronously. This cycle process was repeated many times for confirming MCH-MSH-AC system stability and data accuracy. The characteristics of temperature change and stability of MCH-MSH-AC system were analyzed during endothermic and exothermic processes.

The reaction enthalpy and mass changes of 20 mg MCH-MSH-AC system were analyzed by using TGA/DSC during hydration and dehydration process. Cyclic stability of MCH-MSH-AC system was investigated by measuring the reaction enthalpy of sample during multiple dehydration/hydration cycles. The temperature-rising gradient for the sample dehydration was programmed to 140 °C at 5 K/min and then naturally cools down. The phase analysis of MCH-MSH-AC system after heat cycles was carried out by D/MAX2500PC X-ray diffractometer (XRD) in continuous scan mode with speed of 10°/min, scanning range of 10° ~ 100°, acceleration voltage of 40 kV, and current of 100 mA. Microstructural characterization was performed using scanning electron microscopy (SEM) to investigate distribution of MCH-MSH and AC.

3 Results and discussion

3.1 Effect of AC on thermal properties of MCH-MSH-AC system

Figure 4 illustrated the heating DSC curves of five kinds of composite MCH-MSH-AC systems with different AC contents. According to Fig. 4, the phase-change enthalpy of composite MCH-MSH-AC systems of S0, S1, S2, S3, and S4 was 230.89, 217.06, 205.25, 186.45, and 174.15 kJ kg⁻¹, respectively. However, the test results were quite different from those reported in the literature. Firstly, the purity of the sample and the test conditions were different. Secondly, the inorganic salt dissolves in the crystal water removed by the hydrated salt and produces the heat of dissolution, and the concentration difference thermal effect produced in the dissolution process [25]. Thirdly, the evaporation of crystal water, the evaporation heat of crystal water was 2257 kJ kg⁻¹. The three phenomena will affect the test results. The addition of activated carbon will reduce the phase-change enthalpy of the composite material, but the material still maintains good heat storage performance.

And their starting phase-change temperature was 102.51, 96.54, 98.97, 95.45 and 89.27 °C, ending temperatures-112.65, 110.77, 104.94, 102.45, 99.37 °C, respectively.

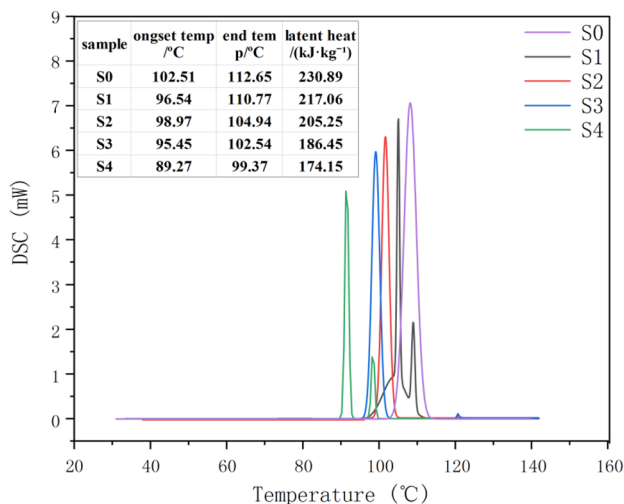


Fig. 4 DSC curve of the MCH-MSH systems with different contents of AC

The results show that AC reduced the initial phase-change temperature of the system to below 100 °C, although it reduced its enthalpy, MCH-MSH-AC systems still have high enthalpy. The properties of MCH-MSH-AC systems with phase transition temperatures below 100 °C and high enthalpy make them suitable for solar heat storage of water cycle in households.

Thermal stability is an evaluation index of the ability of PCM to adapt to temperature change in application [26]. The composite PCMs with good properties should have good thermal stability within their operating temperature range. Thermogravimetric analysis (TGA) is used to test the thermal stability of MCH-MSH-AC composite PCMs between room temperature and 140 °C. Figure 5 shows the progressive dehydration process of MCH-MSH-AC composite PCMs with different AC contents before and after circulation. According to the weightlessness curve before circulation in Fig. 5, the initial dehydration temperature of MCH-MSH-AC composite PCMs is around 80 °C. The mass loss between 80 and 120 °C is related to the loss of water molecules. The weight loss rates of samples S0, S1, S2, S3 and S4 are 6.39%, 4.92%, 4.58%, 4.22%, and 3.98%, respectively, and the most part of the heat storage process completes at less than 120 °C. The results show that AC/MCH-MSH composite PCMs have good thermal stability.

Figure 6 showed the effect of AC content on the undercooling and phase-change temperature of the MCH-MSH-AC system under the same exothermic environment. The sample of S0 without AC had a large undercooling degree, the addition of AC had a significant effect on the exothermic time, the exothermic platform, and the undercooling degree of the systems, but the effect was little on the maximum

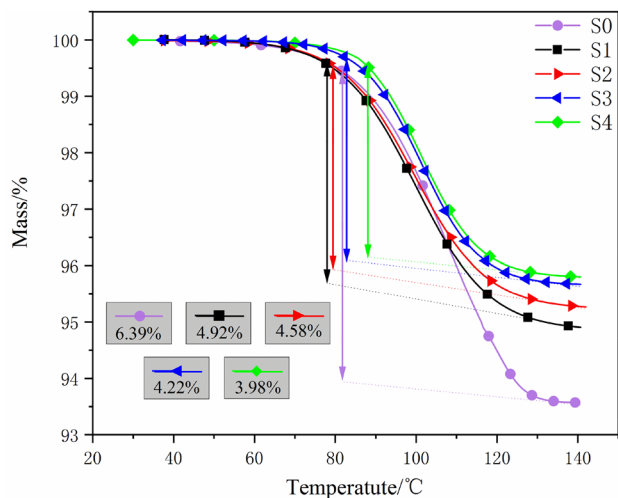


Fig. 5 TG curve of the MCH-MSH systems with different contents of AC

phase-change temperature that MCH-MSH system can reach. The presence of undercooling would absorb the heat released in the phase change process by itself leading to a drooping endothermic peak on the curve of the exothermic platform (arrow mark as shown in Fig. 6), which will reduce released heat rate to the environment. From the figure, the sample of S1 with 1.0 wt% AC has still a large degree of undercooling although it is less than that of sample S0. With the increase in AC addition, the undercooling degree of the composite system was gradually decreased, there was still a small undercooling degree in the sample of S2 (2.0wt%AC), but the undercooling was completely eliminated in the samples of S3 (3.0wt%AC) and S4 (4.0wt%AC), which imply good thermal stability. However, the samples of S4 showed shorter heat release time and greater slope of the heat release platform than that of sample S3. Because activated carbon has a high thermal conductivity, which accelerates the rate of heat release to the environment, reduces the absorption of heat by the system itself, thereby reducing the degree of subcooling. The other is because the inorganic salt hydrate phase change material is in the liquid state when a little inorganic salt is dissolved in its own combined water, the system is in an ion-dispersed state, and the surface of the activated carbon can be combined with the ions in the phase change material, which helps to improve the wettability between the activated carbon and the phase change material. According to the theory of heterogeneous nucleation of crystals, the specific surface area of activated carbon is large, which provides a larger non-uniform nucleation interface and more nucleation points for the recrystallization of phase change materials, which can promote the non-uniform nucleation of the system and reduce the nucleation barrier, and then reduce the undercooling.

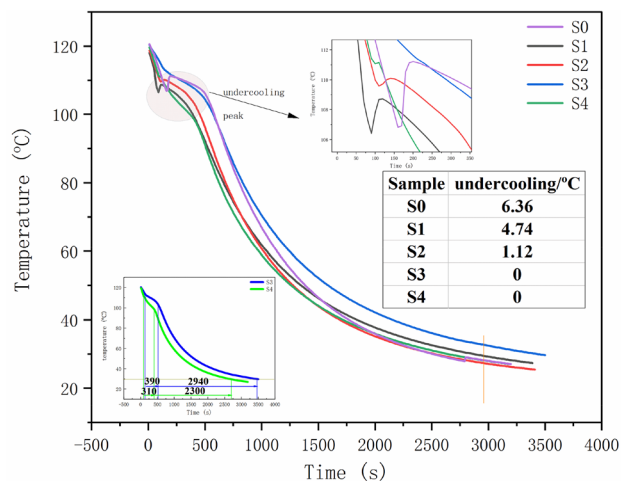


Fig. 6 Undercooling of MCH-MSH-AC systems with different contents of AC

All above indicated that the addition of AC can effectively inhibit the undercooling of the composite system and improve the thermal stability of the MCH-MSH-AC heat storage system, but excessive AC makes negative affect for MCH-MSH-AC system. The illustration in bottom left of Fig. 6 showed the phase-change temperature and phase-change time of the S3 and S4 system. It can be seen that the phase-change time of S3 system was longer than that of S4 system, and its cooling time is also longer from maximum temperature to room temperature than that of S4 system, which is still 32.6°C when the S4 system was room temperature (25°C).

3.2 Thermal stability of MCH-MSH-AC system

Because AC had good thermal conductivity making dehydration of hydrated rate salts promotion [26], which lead to decline the phase change temperature of MCH-MSH-AC systems, and the pores of AC can absorb water molecules, which prevent water molecules escaping from the systems and make the systems show very good stability after many thermal cyclic, as showed in Fig. 7. It can be observed from Fig. 7 that the cyclic stabilities of the samples were related to the content of AC. After 20 cycles of storage/release heat, the weight loss rate of sample S3 is only 13.12%, and that of S4 is 12.9%, but that of samples S1 and S2 increase greatly with the number of cycles resulted from the loss of water molecules. It indicates that the samples with low content of AC cannot provide enough space to store the water of salt hydrate dehydration, and the dehydrated water was gradually lost because of phase separation and water evaporation in the heat cycles. This result also indicates that appropriate addition of AC can significantly improve the thermal

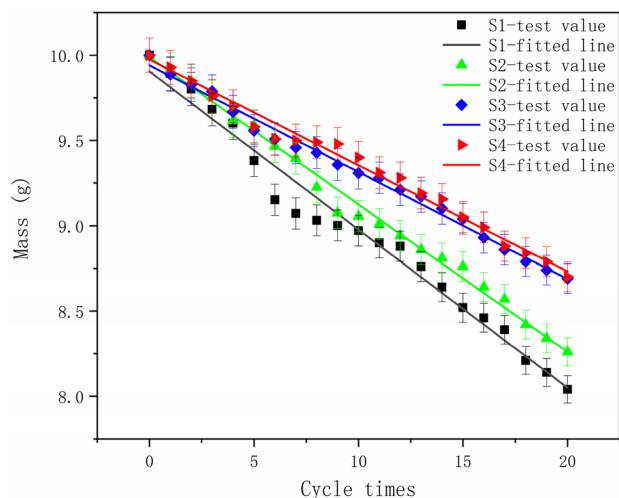


Fig. 7 Mass losses of the MCH-MSH-AC

stability of MCH-MSH-AC system, although it reduces the heat storage capacity of the system.

The initial temperature and enthalpy of phase change of S3 during endothermic process were measured in several cycles, as shown in Fig. 8. It is found that the initial temperature of phase change decreases slightly with the increase in cycle frequency, which finally stabilizes at about 90.21°C after 20 cycles, and the enthalpy stabilizes finally at about 165.14 kJ kg⁻¹ after 20 cycles, which decreases only 7.4% compared to the initial values. This is because that the MCH-MSH-AC system becomes homogenized gradually in the process of repeated cycling, and the dehydration and hydration reactions reach stable equilibrium state.

Figure 9a, b showed the micromorphologies of pure AC and MCH-MSH-AC system with 3.0 wt% AC. It can be observed from Fig. 9a that the AC presents porous structures and its surface is irregular and uneven, which are conducive to the adsorption of PCM in the voids [21]. From Fig. 9b, AC was dispersed in MCH-MSH system and adhered to MCH-MSH surfaces, which will allow dehydrated water molecules to be adsorbed in voids of AC because of the effect of capillary pore and surface tension, preventing water molecules escaping and phase separation in heating process, but the water molecules can be expelled from the pores and hydration reaction takes place during exothermic process. Therefore, the MCH-MSH-AC system displayed good cyclic stability and prolong its service life (Fig. 9).

In order to further verify and analyze the foregoing research conclusions, the element surface distribution analysis was performed on the 3% AC content system. The results are shown in Fig. 10. The parts with relatively high Mg element content also have higher contents of C, O, and

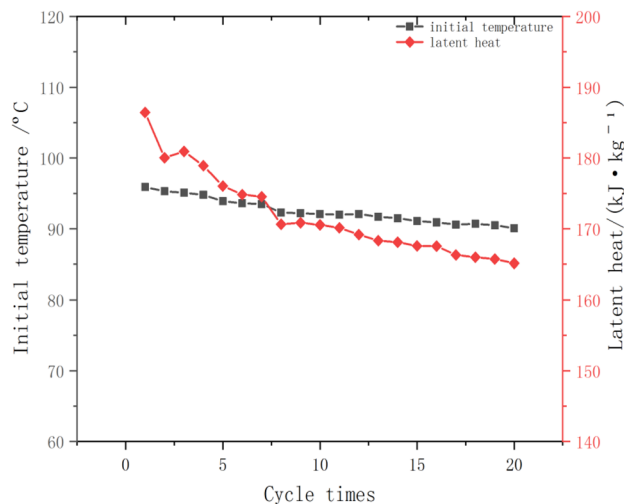


Fig. 8 Initial phase-change temperature and enthalpy of sample S3 in 20 cycles

S elements. It has the effect of dispersing and strengthening the composite material system.

The transmission electron microscope (TEM) was further used to observe the microstructure of the 3% AC composite material in the experiment, as shown in Fig. 11. It can be seen from Fig. 11a that graphitized carbon black appears in the material and adheres to the surface of the phase change material, which is consistent with the phenomenon described in Fig. 9. Figure 11b shows that the composite material is a polycrystalline material with no regular geometric shape and a fixed melting point.

Figure 12 showed the exothermic stability curve for 20 cycles of the sample S3. It indicated from the phase-change range that the MCH-MSH-AC system of the sample S3 has basically the same initial and ending phase-change temperature, which is about 111.62°C and 104.51°C. Compared with the heating process (as shown in Fig. 4), the high temperature point of phase change was somewhat higher, but the low temperature point is the same during the cooling process, which implied rapid heat storage in heating process and larger exothermic range in heat release process. The cycle curves tend to coincide with increase in cycles, which indicated that the system not only maintained good thermal stability, but also the stability becomes better with the increase in the number of cycles (Fig. 12).

Figure 13 shows the infrared spectrum of the 3% AC composite after modification cycle. There are strong absorption peaks at 3389 cm⁻¹ and 3240 cm⁻¹, which are attributed to the stretching vibration peaks of the -OH bond; there are sharper absorption peaks at 2955 cm⁻¹ and 2854 cm⁻¹, and the analysis may be liquid. The sum frequency of variable-angle vibration and rocking vibration of water molecules at 1634 cm⁻¹

Fig. 9 SEM of AC and MCH-MSH-AC system after 10 cycles

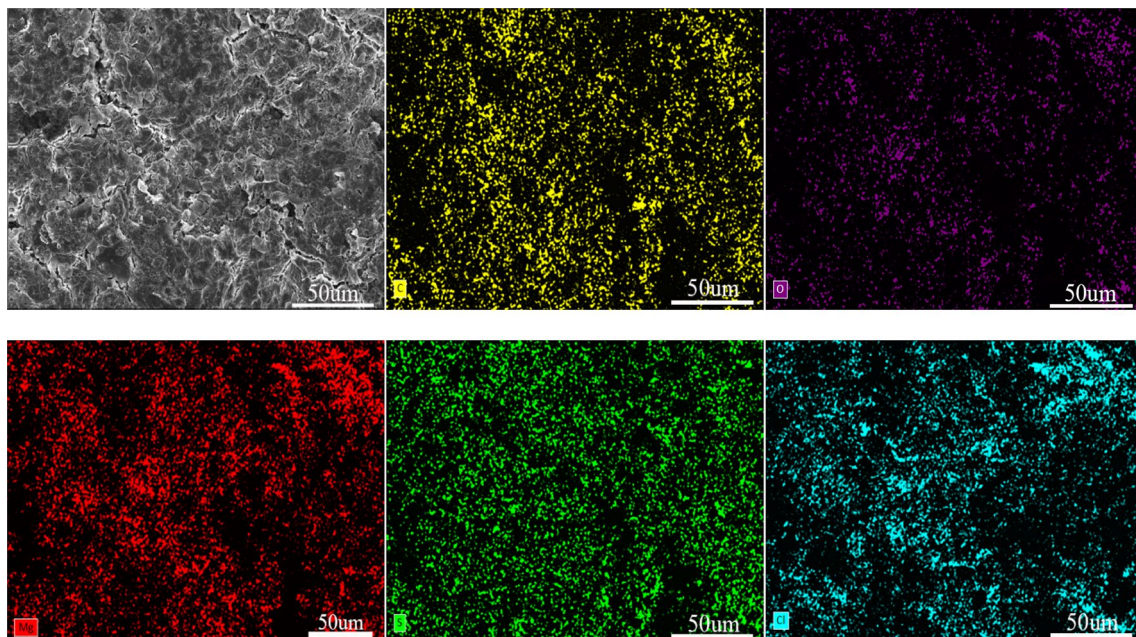
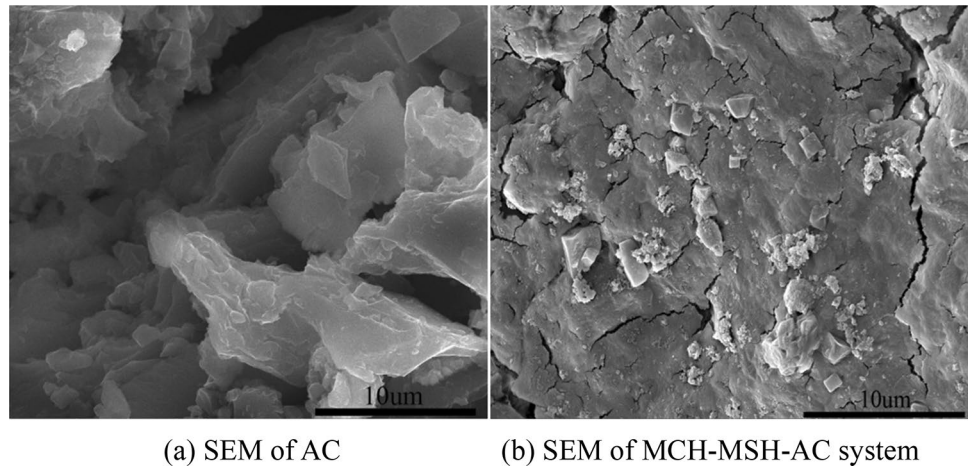


Fig. 10 Morphology and elemental surface distribution AC 3% SEM composite phase change materials

is the absorption peak of the stretching vibration of the Cl–O bond; the peaks appearing at 1460 cm^{-1} and 1384 cm^{-1} are caused by the stretching vibration of the S–O bond.

3.3 XRD analysis

Figure 14 shows that phase of the sample S0 without AC and S3 with 3.0wt% AC was analyzed after 10 cycles. The grain size of AC is calculated to be $15.6\text{ }\mu\text{m}$. It is found that the

phase types of the samples S0 and S3 are basically the same, but the contents are greatly different. The AC addition promotes the phase change of the system to $\text{Mg}_4\text{Cl}(\text{OH})_7\cdot 6\text{H}_2\text{O}$, making $\text{Mg}_4\text{Cl}(\text{OH})_7\cdot 6\text{H}_2\text{O}$ as the main phase of the system, which has excellent performance of buffering, reactivity, adsorbability, and stable heat absorption, and exothermic performance[27]. This may also be one of the reasons why the system tends to be stable after multiple cycles (Fig. 14).

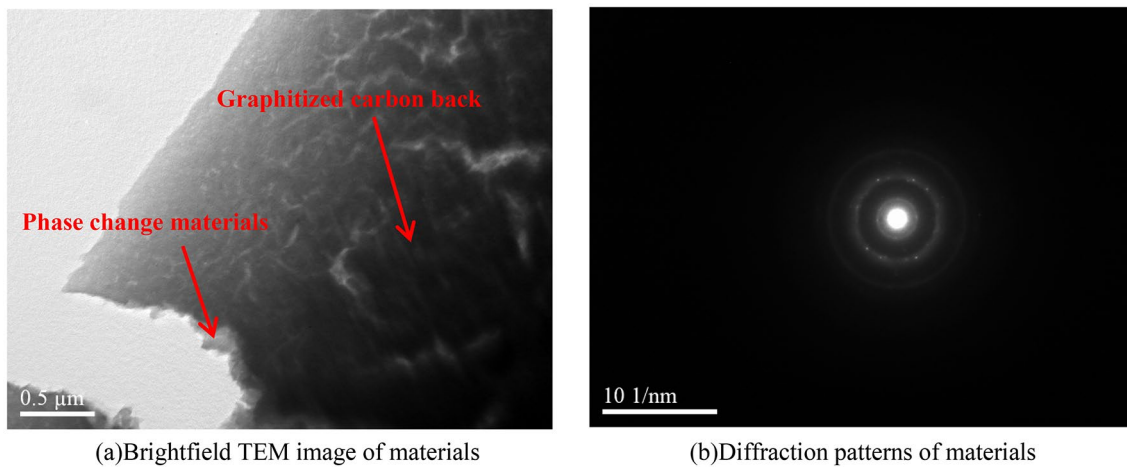


Fig. 11 3%AC composite transmission electron microscope(TEM) image

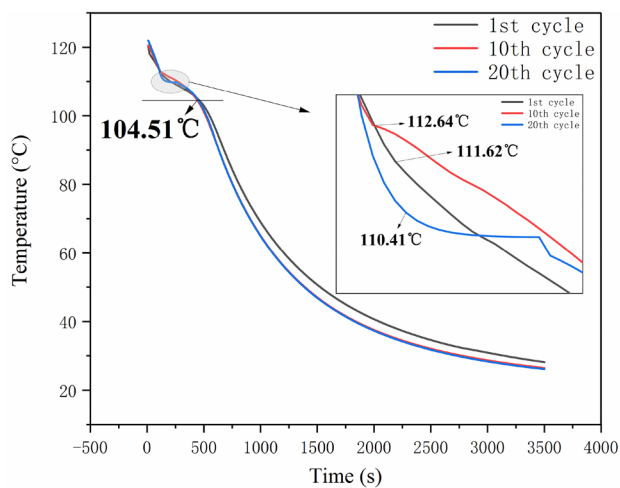


Fig. 12 Thermal stability curve of composite material

4 Conclusions

MCH-MSH-AC system was prepared by adding AC, and the effects of AC on the system's undercooling degree, phase-change temperature, heat storage capacity, and cycling stability were studied by measuring the

temperature change curve, DSC, and thermal cycle curves. The microstructure and phase composition of the system were analyzed by SEM and XRD. It is concluded that:

- 1) More than 3.0 wt% AC can effectively eliminate undercooling degree and reduce the phase-change temperature of the MCH-MSH-AC system, but reduce its heat storage capacity. The starting and ending phase-change temperature of MCH-MSH-3.0 wt% AC system was 95.45°C and 102.54 °C in the heating process, respectively, and its initial phase-change enthalpy reaches 186.45 kJ kg⁻¹ and stable enthalpy is 165.14 kJ kg⁻¹, which was suitable for solar heat storage of water cycle in households.
- 2) The system possessed good thermal stability, which reaches gradually stable phase-change temperature and stable phase-change enthalpy with the increase in the number of cycles. The weight loss rate of MCH-MSH-3.0 wt% AC system is only 13.1% and the enthalpy decreases only 7.4% after 20 cycles compared to the initial values, and the values do not change after that.
- 3) AC is dispersed in MCH-MSH system and adhered to MCH-MSH surface, and Mg₄Cl(OH)₇·6H₂O is the main phase of MCH-MSH-3.0wt% AC system after 20 cycles.

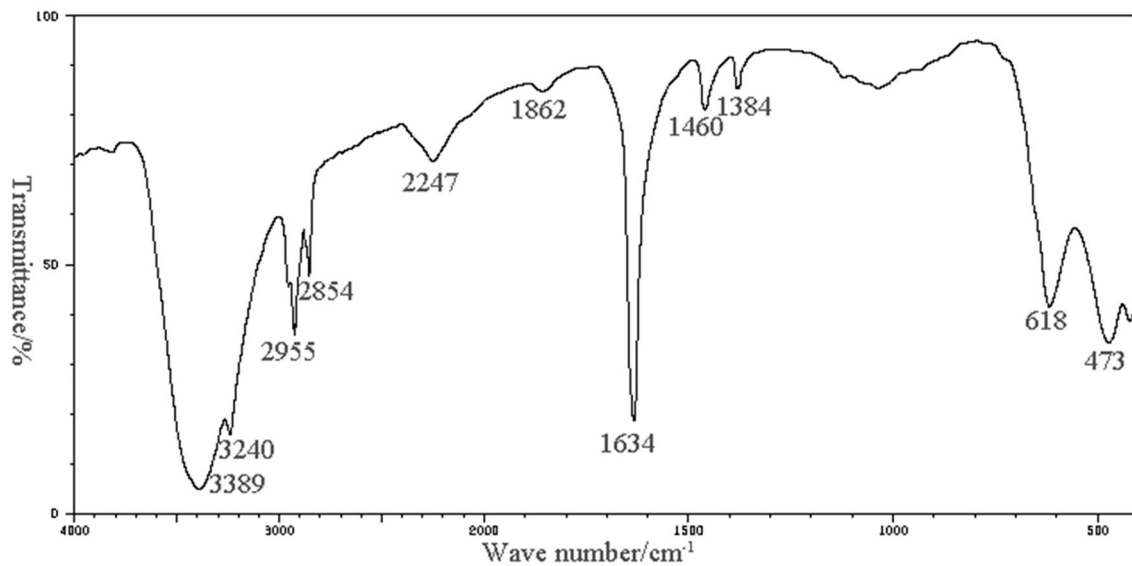


Fig. 13 The infrared spectrum of the 3% AC composite after cycle

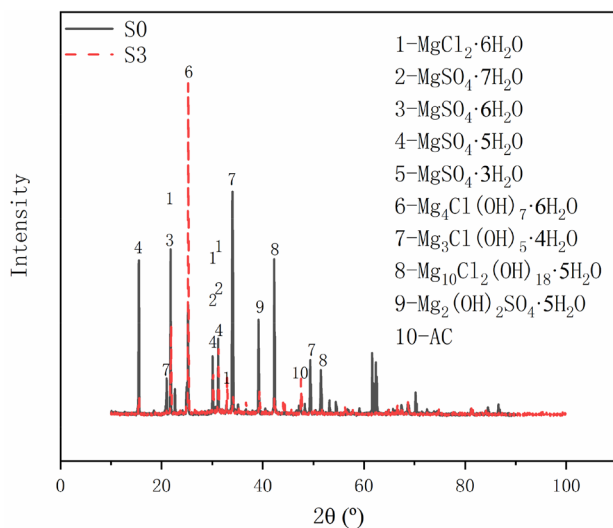


Fig. 14 XRD patterns of composite materials of S0 system and S3 system after 20 cycle

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Declarations

Conflict of interest The authors declared that there is no conflict of interest.

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