



The preparation of the hydrotalcite-based composite phase change material



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HIGHLIGHTS

- Hydrotalcite that had single crystal phase and high crystallinity were prepared.
- Composite PCM was prepared with organic HT and paraffin via liquid intercalation.
- The thermal conductivity of the composite PCM is 17.0% higher than that of paraffin.
- The composite PCM has a good heat storage and heat release performance.

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ABSTRACT

Mg–Al–CO₃-Hydrotalcite (HT) was prepared via hydrothermal synthesis method in this paper. Then the HT was organically modified. The composite PCM was prepared with the organic HT and paraffin via the liquid intercalation. The interlayer groups of HT were investigated using a Fourier Transform Infrared Spectroscopy (FTIR) before and after modification. The microscopic structure of the organic HT/paraffin composite PCM was characterized using a Scanning Electron Microscope (SEM). The thermal properties were measured using a Differential Scanning Calorimeter (DSC) and a thermal constant analyzer. The results showed that the HT prepared via the hydrothermal synthesis had single crystal phase and high crystallinity. The layer spacing of the organic HT was expanded. The intercalation rate of paraffin was 25.9 wt% in the organic HT. The phase transition temperature and the phase change enthalpy of the organic HT/paraffin composite PCM were 26.2 °C and 29.8 J/g, respectively.

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1. Introduction

Phase change materials (PCMs) act as energy storages because the energy can be stored during the phase change process. Therefore, PCMs have been widely used in the storage and thermal fields [1–3]. When PCMs are used in the buildings, the building energy consumption can be reduced. Paraffin is a most common solid–liquid PCM [4–6]. The solid–liquid PCM is accompanied by the appearance of liquid phase in the storage and release process, which causes the leakage leak in its practical application.

The leakage of PCM after thermal cycling will affect the thermal storage properties of PCM [7,8]. To solve the leakage issue, many encapsulation technologies, which can divided into encapsulation and composite technology, were developed [9–11]. Karkri et al. [12] pointed that one possible method for suppressing leaching is

based on the microencapsulation of wax within a polymeric shell. Encapsulation of PCM is a kind of technology that PCM is covered by film forming material (capsule wall) to become tiny particles. Chai et al. [13] designed and synthesized a sort of bifunctional PCMs-based microcapsules with an n-eicosane core and a crystalline titanium dioxide shell. The microcapsules showed good performance in energy storage and sterilization. Fang et al. [14] synthesized the polystyrene(PS)/n-dotriacontane (Dot) composite nanoencapsulated phase change material (NEPCM) for thermal energy storage by a miniemulsion polymerization method. The composite material has a high latent heat capacity and good thermal stability. Li et al. [15] prevented the leakage of paraffin by mixing hydrophobic nano-powder modified PX25, cement, and water to produce thermal energy storage composites. The mechanism of preventing PCM leakage is that hydrophobic nano-powders can absorb most exuded paraffin. Memon et al. [16] prepared the macro-encapsulated paraffin-LWA by incorporation of paraffin into porous LWAs through vacuum impregnation and developed structural–functional integrated NWAC with macro-encapsulated

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paraffin–LWA. Xu and Li [17] prepared paraffin/diatomite composite PCM. The results showed that paraffin can be well incorporated in diatomite pores.

The layered materials have great surface effect and interface effect, which can prevent the solid–liquid PCM from releasing out of the interlayer and thus solve the leakage problem. Meanwhile, the thermal conductivity of the layered materials is higher than that of paraffin. The layered materials can improve the thermal conductivity of paraffin. Hydrotalcite (HT) is a kind of anionic layered compounds. It consists of laminates with positive charges and interlayer anions which are orderly stacked. Because of the special structure and properties, HT have great application prospect in the field of catalysis, adsorption, photochemical and electrochemical [18–20]. However, the layer spacing of the HT is small because of the high interlayer charge density which results in the strong interlayer electrostatic interaction. So, it is difficult for organic molecules to enter the interlayer of the HT. In order to expand the interlayer spacing of the HT and improve the inorganic–organic compatibility, organic anion surfactants are usually used to modify the HT.

Zhong [21] prepared modified hydrotalcite with sodium dodecylbenzenesulfonate (SDBS) and itaconic acid by the ion-exchange method. The results show that SDBS and itaconic acid can enter the layer of hydrotalcite and the layer spacing is greatly improved. Wang [22] intercalated dodecylsulfonate (DSO) and dodecylbenzenesulfonate (DBS) into the Mg–Al layered double hydroxides (LDHs) using the co-precipitation method. The surface properties of DSO–LDHs and DBS–LDHs were modified from hydrophilic to hydrophobic characteristics. Gao [23] made the organic modification of nano LDH panicles with benzoic acid by means of intercalation. It was found that the modified nano LDH particles could dispersed efficiently whether in polar ethylene vinyl acetate or non-polar polyethylene.

Composite membranes which are suitable for fuel cell applications are developed by introducing LDHs in the polymeric structure. In particular, LDHs which introduced into the polymeric matrix produces increase the polymer tortuosity that reduces the permeation of the fuel and improve the mechanical properties of polymers for high temperature operation [24]. The nanocomposite based on low density polyethylene (LDPE) and Mg–Al based layered double hydroxide (Mg–Al LDH) show the excellent flame retardant performance and thermal stability [25].

However, the current research mainly focuses on the application of HT in the polymers. Few researches are conducted about the application of HT in the energy storage materials. In this paper, Mg–Al–CO₃–HT was prepared via hydrothermal synthesis in this paper. HT was organic modified by sodium dodecylbenzenesulfonate (SDBS). The organic HT/paraffin composite PCMs were prepared with the liquid intercalation method. The thermal conductivity of the organic HT/paraffin composite PCMs was studied.

2. Experimental

2.1. Materials

Aluminum nitrate nonahydrate (Al(NO₃)₃·9H₂O), Magnesium nitrate hexahydrate (Mg(NO₃)₂·6H₂O), Sodium carbonate anhydrous (NaCO₃), Sodium hydroxide (NaOH) and Ethylene glycol C₂H₆O₂ were purchased from ChengDu Kelong Chemical Reagent Co., Ltd., China. Sodium dodecylbenzenesulfonate (SDBS) were supplied by Sinopharm Chemical Reagent Co., Ltd., China. All the chemical reagents above are analytical reagents. Paraffin was purchased from Shijiazhuang Caldecott Phase Change Materials Co., Ltd., China. The melting temperature of the paraffin is 28 °C and

the density of the paraffin is 0.9 g/cm³. The thermal conductivity of paraffin is 0.182 W/m K.

2.2. Experimental method

2.2.1. Synthesis of hydrotalcite

- (1) 7.68 g Mg(NO₃)₂·6H₂O and 3.75 g Al(NO₃)₃·9H₂O were dissolved in 30 ml deionized water, and the solution was stirred until uniform to obtain solution A.
- (2) 3.20 g NaOH and 0.53 g Na₂CO₃ were dissolved in 30 ml deionized water, and the solution was stirred until uniform to obtain solution B.
- (3) Solution A and solution B were mixed rapidly. 10 ml (volume fraction 15%) glycol was added into the solution and stirred until uniform.
- (4) The pH value of the solution was adjusted to 12 with saturated NaOH solution and then poured into a hydrothermal reaction kettle. The solution was reacted at 120 °C for 16 h.
- (5) The cooled solution was filtrated in vacuum. The product was washed to neutral.
- (6) The reaction product was dried in the vacuum at 80 °C. The product was ground to obtain the Mg–Al–CO₃–HT.

2.2.2. Organic modification of HT

- (1) 1 g HT was dissolved in 80 ml glycol and stirred at 1500 rpm until uniform to obtain HT suspension.
- (2) 1 g SDBS was dissolved in 20 ml ultra pure water and then poured into the HT suspension. The mixture was stirred at 1000 rpm until uniform.
- (3) The pH value of the mixture was adjusted to 4.7 with 0.1 mol/L HNO₃ solution and then reacted at 100 °C for 2 h.
- (4) The reacted solution was cooled and filtered. The produce was washed. This process was repeated for four times.
- (5) The reaction product was dried in the vacuum at 80 °C and then ground to obtain HT-SDBS.

2.2.3. The preparation of HT-SDBS/paraffin composite PCMs

- (1) 4 g HT-SDBS was dissolved in ethanol and stirred at 1500 rpm until uniform to obtain organic HT suspension.
- (2) 1.4 g paraffin was dissolved in ethanol and then poured into the HT-SDBS suspension. The mixture was stirred for at 1000 rpm for 15 min.
- (3) The mixture was heated to 75 °C and then stirred at 400 rpm and 75 °C. Ethanol was recycled by condensation in vacuum until evaporated.
- (4) The product was dried in vacuum at 80 °C and then ground to obtain HT-SDBS composite PCMs, which was labeled as HT-SBDS/paraffin.

The schematic diagram of the compounding device was shown in Fig. 1.

2.3. Characterization and testing

FTIR spectrometer (Thermo Scientific Nicolet iS10, NETZSCH, Germany) was used to test the functional group-modified OHT, the scanning range is 400–4000 cm⁻¹. The surface morphology of the composite PCM was examined with field emission SEM (Sirion 200, FEI Company, Netherlands). The accelerating voltage was 20 kV. The changes of HT Layer spacing were characterized before and after modifying and Intercalating by XRD (D/max-2500, Rigaku Company, Japan). The thermal conductivities of OHT/paraffin composite PCM was tested by a thermal constant

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