



A polymer-coated calcium chloride hexahydrate/expanded graphite composite phase change material with enhanced thermal reliability and good applicability

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ARTICLE INFO

Article history:

Received 13 November 2017
Received in revised form
18 December 2017
Accepted 21 December 2017
Available online 27 December 2017

Keywords:

Phase change material
Hydrated salt
Expanded graphite
Polymer-coating
Thermal reliability

ABSTRACT

A novel polymer-coated composite PCM was prepared by mixing the $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}/\text{EG}$ composite particles with a photo-curing polymer, followed by irradiating the mixture under UV light to make the polymer cured. It is shown that the surfaces of the composite particles have been sealed by the polymer. And the melting and freezing enthalpy values of the obtained polymer-coated $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}/\text{EG}$ composite PCM are as high as 113.2 and 114.1 J g^{-1} , respectively, and its melting point is quite close to that of $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$. After experiencing 500 heating-cooling cycles, polymer-coated $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}/\text{EG}$ exhibits the decreases only by 1.68% and 0.52% in the enthalpies of melting and freezing, much less than 25.54% and 30.55% for $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}/\text{EG}$, respectively, revealing much enhanced thermal reliability. The applicability of the uncoated and polymer coated $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}/\text{EG}$ composite PCMs was investigated by directly mixing them with gypsum powder and water, followed by forming into plasterboards. It is found that, the plasterboard containing polymer coated $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}/\text{EG}$ not only exhibits superior thermal performance for delaying temperature rise over the one with $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}/\text{EG}$ but also possesses much better thermal reliability. These good characteristics render the polymer-coated $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}/\text{EG}$ composite PCM with great potentials in building energy conversation.

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1. Introduction

Thermal energy storage is one of the key technologies for increasing thermal energy utilization efficiency, thus playing an important role in relieving the energy crisis [1,2]. Among various thermal energy storage technologies, latent heat storage (LHS) based on phase change materials (PCMs) is the most important one, which has been widely applied in many fields, including heating and cooling buildings, waste heat recovery, off-peak power utilization, heat pump systems, thermal management, etc. [3–5]. It is undoubted that high-performance and low-cost PCMs are essential for developing high-efficiency latent heat storage systems [6].

PCMs can be broadly classified into two types: organic and inorganic. Although organic PCMs (e.g. paraffin waxes, fatty acids,

fatty alcohols) have the advantages of high phase change enthalpies as well as nearly no supercooling and phase separation, they suffer from relatively high cost, low thermal conductivity and flammability [3]. Comparatively, inorganic PCMs, especially hydrated salts, possess good characteristics of high latent heat, extensive raw sources, low cost and incombustibility, making them very promising PCMs [7]. However, the wide applications of hydrated salts are greatly hampered by the shortcomings inherent in them, which include phase segregation, supercooling, and causticity. Besides, the tendency of losing water of crystallization makes hydrated salts always need to be sealed in containers when used, thereby decreasing their applicability. Consequently, hydrated salts have been long considered as a difficult topic in PCM fields. In the past few decades, the researchers on hydrated salts have been mainly focused on minimizing supercooling and eliminating phase segregation, by the use of nucleating agents and thickening agents [8–10] or the addition of extra water [11–13]. However, these efforts made no improvements on their causticity and the liquid leakage problem after hydrated salts changed their phases from solid to liquid. Apparently, it is of great significance to develop

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novel hydrated salt-based PCMs that not only do not suffer from all the aforementioned drawbacks but also possess good applicability.

Developing form-stable composites, e.g. by grafting a PCM on the nanosheets of graphene oxide [14] or microencapsulating PCMs [15], is an effective route for solving the liquid leakage problem for solid-liquid PCMs along with modulating their thermal conductivity [16]. Among those various routes for preparing composite PCMs with good form-stability, adsorbing PCMs into porous carriers is the simplest one, which just involves physical process. Since the pioneering work by Zhang et al. [17] in 2006, expanded graphite (EG) has been verified to be an excellent supporting material for preparing composite PCMs, due to its large pore volume and high thermal conductivity. And various organic PCMs, such as paraffin, fatty acids, alcohols, etc., have been combined with EG [18–20], owing to the good compatibility between these organics and EG. It has been found that the obtained EG-based composites exhibit good form stability and enhanced thermal conductivity at a cost of losing latent heat by only around 10%, as compared with the corresponding PCMs [21–23]. For the inorganic PCMs, although several types of research have been reported on the EG-based composites containing the molten salts with phase change temperatures of more than 150 °C [24–27], only a little work has been done on the ones containing the hydrated salts, such as $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ and $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$ [28] or $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ and $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$ [29]. It is noticeable that, among various hydrated salts, $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ is a very important one, owing to its extensive raw sources, low cost along with its suitable phase change temperature for use in building energy conversation [4,30]. Duan et al. [31] prepared a $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}/\text{EG}$ composite PCM and found that its supercooling and phase segregation were overcome by the combination with EG. However, they didn't report the thermal reliability of this composite. Besides, in order to apply the $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}/\text{EG}$ composite PCM into buildings, Ye et al. [32] encapsulated this composite using PVC sheets, followed by compacting to obtain a PCM panel. Consequently, one can expect that, if the surfaces of the $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}/\text{EG}$ composite particles can be sealed off, the obtained PCM will not only avoid all the drawbacks inherent in hydrated salts but also can be mixed with building materials directly, thereby showing improved thermal reliability and applicability.

In the current work, a novel polymer-coated $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}/\text{EG}$ composite PCM was prepared by mixing the $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}/\text{EG}$ composite with a photo-curing polymer, followed by irradiating under UV light to make the polymer cured. The morphology, microstructure and thermal properties of the obtained polymer coated $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}/\text{EG}$ composite PCM were characterized and compared to those of the uncoated one. Furthermore, the applicability of the uncoated and polymer coated $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}/\text{EG}$ composite PCMs was investigated by mixing them with gypsum powder and water, followed by forming into plasterboards. And the thermal performance and thermal reliability of the plasterboards containing the uncoated and polymer coated $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}/\text{EG}$ composite PCMs were evaluated by placing them into a test room, respectively. It is found that the thermal reliability of the polymer coated $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}/\text{EG}$ composite is much better than that of the uncoated one, and the plasterboard containing the polymer coated $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}/\text{EG}$ composite exhibits superior thermal performance and reliability over the one with the uncoated $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}/\text{EG}$ composite.

2. Experimental section

2.1. Preparation of polymer-coated $\text{EG}/\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ composite

The UV curable resin system used to coat the $\text{EG}/\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ composite PCM particles is composed of trimethylolpropane triacrylate, modified polyurethane acrylic polymers and 2-hydroxy-2-

methyl-1-phenyl-1-propanone. The procedures for preparing the $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ sample and the $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}/\text{EG}$ composite are described in Supplementary Information. In a typical synthesis, 3 g of trimethylolpropane triacrylate and 1 g of the modified polyurethane acrylic polymer were mixed with 0.2 g of 2-hydroxy-2-methyl-1-phenyl-1-propanone used as the sensitizer. And then, the obtained resin was added to the $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}/\text{EG}$ composite powder under continuous stirring. After being mixed thoroughly, the obtained mixture was irradiated by UV light to make the resin cured to form a cross-linked polymer (shown in Figure S1). The obtained sample was the polymer-coated $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}/\text{EG}$ composite PCM.

2.2. Characterizations and measurements

The characterizations of structure, morphology, thermal properties and thermal reliability of the PCMs are described in Supplementary Information. To test the supercooling degree of the composite PCMs, an experimental system was set up for recording the change in temperature with time during heating and cooling, as shown in Fig. 1a. The set-up mainly consisted of a thermostatic silicone oil bath box and a unit of thermal energy storage (TES). The temperature of the silicone oil bath was automatically controlled and measured with the help of a temperature control system with an accuracy of ± 0.01 °C. The TES unit consisted of two identical cylindrical glass containers with an inner diameter of 25 mm, a wall thickness of 1 mm and a length of 200 mm. Two K-type thermocouples were placed into two identical cylindrical glass containers to monitor the temperature variations of each sample within a measurement error of ± 0.1 °C. Locations of the thermocouples are also presented in Fig. 1. The two test points were placed at the same depth, 20 mm away from the bottom of the container. Initially, two-thirds of the TES unit volume was filled with each composite PCM at 5 °C, and the remaining space was left for accommodating the thermal expanding of the PCM. During the heat storage process, the thermostatic silicone oil bath box was kept at a constant temperature of 55 °C. A data logger (Agilent34970A, USA) was used to collect the temperature data.

The evaluation of applicability and thermal performance of the polymer-coated and uncoated $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}/\text{EG}$ composite PCMs was conducted as follows. The two composite PCMs were added into a mixture of gypsum powder and water whose mass ratio was 2:1, respectively, and their mass fractions were set at 3 wt%. After being mixed thoroughly, the obtained mixtures were pouring into a 10 cm \times 10 cm square mold to obtain two kinds of thermal energy storage plasterboards. For comparison, the plasterboard without PCM was also fabricated. Moreover, an experimental apparatus for testing the thermal performance of the as-fabricated plasterboards is illustrated in Fig. 1b, which consists of a light source, a test room and a data collection system. The ordinary plasterboards and the ones with the polymer-coated and uncoated $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}/\text{EG}$ composite PCMs were placed at one wall of the test room, respectively, which wall faced to a light source for simulating the solar irradiation. And the heat storage and release of the plasterboards were carried out when the light was turned on and off. A 1000 W solar simulator (SOLAREEDGE3700, Perfectlight, China) was used as the irradiation source, and the light intensity at the wall was measured to be 100 mW/cm^2 by using an irradiatometer (ST-80C, Photoelectric Instrument Factory of Beijing Normal University, China). The data collecting system consisted of a thermoelectric couple, a data collector and a computer. The thermocouple was positioned at the center of each plasterboard for monitoring their temperature variations, which linked to the data collector. Furthermore, the three kinds of plasterboards were subject to experience 500 heating-cooling cycles by heating them to 50 °C and keeping for 1 h

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