



Experimental investigations of Alum/expanded graphite composite phase change material for thermal energy storage and its compatibility with metals



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ABSTRACT

A novel composite phase change material (CPCM) were prepared with Aluminum potassium sulfate dodecahydrate (Alum, $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$) as PCM and expanded graphite (EG) as nucleating agent and porous matrix for thermal conductivity enhancing. The detailed thermo-physical properties and thermal energy storage performance were studied. The DSC revealed that the melting temperature and latent heat of fusion of Alum/EG CPCM were 87.92°C and 214.9J/g , respectively. The thermal conductivity of Alum/EG CPCM was improved from $0.497\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ to $5.875\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ due to the addition of EG, which can also be confirmed by the infrared thermal imager during charging/discharging process. The Alum/EG CPCM exhibited prominent chemical stability and thermal reliability before and after 500 thermal cycle tests. Furthermore, the corrosion of three metals and one metal alloy were studied and the gravimetric analysis and the results of element composition of four samples exhibited that the brass was the suitable materials for the container for long-term used while stainless steel 304L and aluminum were severely corroded. The results demonstrated that Alum/EG CPCM was a prospective candidate for thermal energy storage and accelerated the research on the Alum/EG heat storage system.

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1. Introduction

With the improvement of the human's living standard, the requirement of the fossil fuel was on the up and up. The immediate problem generated was that the emission of carbon dioxide would be increased. Thereby, the global warming would be ulterior aggravated. An effective approach to deal with this problem was energy conversion and improving the efficient of the energy employment [1]. A large number of researchers and scholars studied the thermal energy storage aimed to save the energy. Thermal energy storage was divided into three types: sensible heat storage, latent heat storage and chemical reaction heat storage. Chemical reaction energy storage underwent exothermic/endo-thermic processes through the endothermic and exothermic reactions of compounds in reversible chemical reactions. With the disadvantages of complicated technology, high cost and low overall efficiency, this technique was difficult to put into practical

applications. Conversely, with the advantage of large energy storage density and isothermal characteristic during exothermic/ endothermic, latent heat storage was applied in various domains, such as solar energy storage, industrial exhaust heat recovery [2–4].

Phase change materials (PCMs) attracted much attention on enhancing thermal energy utilization and reducing environment pollution efficiently [5]. The PCMs are commonly divided into two types: organic and inorganic phase change material. As for organic PCM, such as fatty acids, paraffin et al., have attracted considerable interest in thermal energy storage [6–8]. However, the organic PCMs have the disadvantages of inflammable, relatively low heat storage capacity etc, which hindered the extensive application. On the other hand, the inorganic PCM [9,10], with the strengths of constant phase change temperature, nonflammable and high heat storage capacity, was a potential candidate for heat storage. According to the phase change temperature, inorganic PCM contained of hydrated salt (such as $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ [11,12], $\text{CH}_3\text{COONa} \cdot 3\text{H}_2\text{O}$ [13,14], Glauber's salt [15]) and anhydrous salt (for example, NaNO_3 [16–18], KNO_3 [19,20], KCl [21]). Hydrated salts [22] play an important role in low and middle temperature heat storage,

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especially industrial exhaust heat recycle and solar energy storage. Herein, aluminium potassium sulfate dodecahydrate (Alum), with appropriate phase change temperature of 80 °C–100 °C, high melting enthalpy of more than 240 J/g, was rarely researched by the researchers, which was worthy of further investigation.

Most of the PCMs suffered from leakage due to the low viscosity while melting to liquid state. Hence, it is necessary to package the PCM with other structures. Encapsulating the PCM and compositing with other porous structure were two useful ways to solve the above weakness. Whereby, complicated preparation techniques, long preparation period, low utilization ratio of raw material and low latent heat of the products are the most noteworthy drawbacks of the encapsulation the PCM, which hindered the extensive applications. Svetlana Ushak et al. [23] packaged $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ and bischofite with acrylic as shell polymer and chloroform as solvent via a fluidized bed method. The melting thermal properties of the microencapsules were 104.6 °C and 95 J/g for bischofite, and 95.3 °C and 118.3 J/g for $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$. $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ /expanded perlite composite was obtained with melting temperature and latent heat of 27.38 °C and 87.44 J/g, respectively [24]. Moreover, $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ - $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}/\text{SiO}_2$ composite with various mass ratio of $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ was prepared and the latent heat of the composite was 88.13 J/g with no leakage appeared during the phase change [25]. All techniques mentioned above can restrain the leakage of liquid PCM. However, most of the PCM suffered from low thermal conductivity during application. Therefore, an effective encapsulation method should be chosen to package the Alum with thermal conductivity improvement and without leakage.

Expanded graphite (EG) is a universal material which has the advantages of light-weight, bulky surface area, great thermal conductivity and small complicated permittivity [26,27]. Yuping Wu et al. [28] developed a novel CPCM with Sodium sulfate decahydrate-sodium phosphate dibasic dodecahydrate eutectic hydrated salt as PCM and EG as porous filler by impregnation method and further coated with paraffin wax. The thermal conductivity of the coated composite was up to $3.643 \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$ and the thermal properties of the coated composite after 100 melting and freezing cycles was almost constant with that before. Thereinto, EG displayed excellent thermal stability performance. Zhijun Duan et al. [29] synthesized $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}/\text{EG}$ CPCM with OP-10 as surfactant via vacuum impregnation method. The thermal conductivity of the composite was as high as $8.796 \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$ which was 14 times higher than the pure hydrated salt. Hye Kyoung Shin et al. [30] obtained sodium acetate trihydrate (SAT)/EG CPCM using carboxymethyl cellulose (CMC) to preventing the phase segregation of SAT and the aggregation of EG with simple blending method. The thermal conductivity of the CPCM was improved to $18.5 \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$ by adding 2.5 wt% EG and 5 wt% CMC and the phase change latent heat was 202.2 J/g comparing to 250.3 J/g for pure SAT. There were only five thermal cycles done to measure the phase change characteristic which was not enough to explain the thermal stability of the CPCM. Huang Z et al. [31] prepared LiNO_3 -KCl/EG CPCM by physical mixing method for solar energy storage with the melting temperature and enthalpy of 165.55 °C and 178.10 J/g, respectively. The thermal conductivity of the CPCM had remarkable improvement. Liu J et al. [32] obtained MgCl_2 -KCl/EG CPCM with a novel process with the melting temperature and enthalpy of 424.14 °C and 161.37 J/g, respectively. The CPCM possessed excellent thermal reliability, better uniformity and smaller volume expansion. Herein, a new method was employed to synthesize form-stable phase change composites with high enthalpy by the aqueous solution physical adsorption method.

Compatibility with container materials played a crucial role in thermal energy storage system for latent heat storage. The corrosion characteristics for salts to metals and metal alloys were studied

during the recent decades. The corrosion tests between nine kinds of low temperature PCM and metals and polymers were carried out by Eduard Oró et al. [33]. Polymers did not show evident changes in weight or physical appearance of the samples while the metals were corroded in different degree. Dheep G R et al. [34] measured the corrosion of glutaric acid with aluminum, copper and stainless steel. The results show that the stainless-steel specimen has shown very low corrosion rate in contact with glutaric acid compared to aluminum and copper specimens which was recommended as container material. Besides, there are few literature learning about the compatibility of CPCM with container material, especially for hydrate salt composite. Huang Z et al. [35] tested the corrosion characteristics of LiNO_3/KCl -EG CPCM to stainless steel, carbon steel, copper and brass. The stainless steel and carbon steel had good corrosion resistance to the LiNO_3/KCl -EG CPCM whereas brass was not a suitable choice as container material for this CPCM. From the above, it is necessary to test the compatibility with container materials for new kind of PCM or CPCM.

The objective of this study was to develop the Alum/EG CPCM with a novel method, the aqueous solution physical adsorption method, which was suitable for solar energy latent heat storage system. Thereinto, the irregularly worm-like porous structure of EG acted as high thermal conductivity filler [36]. The thermal stability was studied by TG analysis. Moreover, the heat storage performances of Alum and Alum/EG CPCM were investigated and the melting characteristics were also revealed. Furthermore, the compatibility between the CPCM and copper, brass, 304L stainless steel and aluminum was tested. In the present work, as one of inorganic hydrated salts Alum, the porous materials with high PCM load, high shape stability, good thermal conductivity and low cost are the optimal choices for thermal energy storage. The results of compatibility would give help to design and construct the CPCM heat storage system.

2. Experimental details

2.1. Materials

Expandable graphite powders (mesh 50, expandable volume: 300 ml/g) were purchased from Qingdao Graphite Co. Ltd., China. The Alum (98%) was supplied by Tianjin Damao reagent Co. Ltd, China. The chemicals were employed as received without further purification.

2.2. Preparation of Alum/EG CPCM

EG was gotten from expandable graphite powders through microwave treatment at a power of 700W for 40s [37]. The porous support EG was treated to remove the tiny granules with the help of 600 mesh sieves before the blending experiment. The Alum was milled into powder and put into a beaker with the corresponding water to obtain c. The mixers of alum and water were heated at 85 °C with magnetic stirring at 400 rpm for 2h to obtain homogeneous solution. EG were weighed by an analytical balance with precision of 0.1 mg and added to the Alum saturated solution. The CPCMs were stirred and dispersed through ultrasonic cleaner (KQ2200DE, Kunshan) at 80 °C for 3h. Afterwards, the Alum/EG CPCM was gotten via suction filtration by the Buchner funnel and then drying the mixer at 30 °C for 24h with stirring every 30min to avoid the agglomeration of the resultant product. Ultimately, Alum/EG CPCM containing 90 wt% Alum was successfully prepared in this study.

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