



# Salt hydrate/expanded vermiculite composite as a form-stable phase change material for building energy storage

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## ABSTRACT

An investigation on a new type of shape-stabilized phase change material (PCM) prepared by impregnating eutectic salt hydrate ( $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ - $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$  with 1:1 in mass ratio) into expanded vermiculite (EV) is carried out in this study. The maximum mass percentage of  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ - $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$  eutectic within the composite is determined as 60%. The chemical composition of EV is measured by X-Ray Fluorescence Spectrometer (XRF). The specific surface area of EV and raw vermiculite (RV) is characterized by Brunauer-Aemmett-Teller (BET) method. The results from Scanning electron microscope (SEM), Fourier transform infrared spectroscopy (FT-IR) and X-ray diffraction (XRD) tests reveal that eutectic salt hydrate fully filled in the multilayer structure of EV and had good compatibility among the components of the composites. The phase change temperature and the melting enthalpy of the composites are 23.98 °C and 110.3 J/g respectively, which are measured by differential scanning calorimetry (DSC). Good thermal and structural reliability are also demonstrated by thermal gravimetric analysis (TG) techniques and thermal cycling tests. Moreover, the low thermal conductivity with 0.192 W/(m K) tested by the Hot Disk via transient plane source method certifies that the composite is very applicable for insulation. The composite PCM is then tested in a reduced-scale test chamber outfitted with four thin PCM panels as insulated wallboards. The indoor temperature variations of the PCM room is compared with another identical chamber without PCM layer for evaluating the thermal regulating performance. The results show that the composite has the function of maintaining the indoor temperature at a comfortable range for a longer period and distinctly influenced the time delay. All analysis results indicate that the low-cost salt hydrate/EV composite can exert a great deal of influence on insulation in building energy conservation.

## 1. Introduction

Driving by expanding population, growing economic and demanding quality of life, the worldwide energy consumption rates are predicted to grow unceasingly [1]. The increasing energy consumption will lead to adverse impacts on global environment due to the substantial use of fossil fuel and emission of greenhouse gas. Therefore, the effective conservation of energy is a significant issue nowadays. It cannot be neglected that building sector accounts for 40% of the overall global energy consumption and it is expected to increase by 28% within 2035. Design of thermal insulated construction envelops with high heat capacity, consequently, tend to be a crucial topic for energy saving at present [2–5]. Latent heat thermal storage using phase change

materials (PCMs) is widely regarded as one of the most effective technologies to reduce indoor heating and cooling loads from variable outside environment, and therefore overcomes the mismatch between availability and supply of energy. Building envelops integrated with PCMs can not only slow down the indoor temperature fluctuations but also improve human comfort, thanks to their prominent characteristics of providing high storage density, adsorbing and releasing as well as reusing heat within negligible temperature variation [6,7]. Several methods are generally utilized to combine PCMs with building envelops such as inserting PCMs into multi-layer wallboards, fabricating PCMs hollow bricks and impregnating PCMs into construction materials like concrete, cement, gypsum and etc [8–10]. Nevertheless, leakage problem of PCMs often occurs to the PCM-based building envelops after

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PCMs enduring solid-liquid phase transitions and the consequent poor reliability is a major concern for the practical application of PCMs in buildings. To deal with this problem, great attentions have been paid to preparing composite PCMs through microencapsulating PCMs into microcosmic shell or impregnating PCMs into porous supporting materials. It is expected that with assistance of materials with microcellular structures, the liquid PCMs can be confined in micro-scale regions and therefore stay form-stable. Although numerous investigations have proved the effectiveness of this approach, it should be noted that microencapsulated PCMs often require complex preparation procedures and are not cost-effective, which makes them difficult to be put into mass production. [11,12]. Alternatively, impregnating PCMs into porous supporting materials to prepare shape-stabilized PCMs is a predominantly attracted method due to their inherent merits of simple manufacturing procedure, low cost and undemanding shapes for application, which has large potential for development [13].

Commonly, to prepare a composite PCM with desirable properties, the selections of PCMs and porous supporting materials are both of great significance. As for PCMs, paraffin, fatty acids and salt hydrate are underlying candidates for construction use. However, paraffin and fatty acids always suffer from flammability and pungent odour, which would make it difficult to achieve human comfort. By contrast, salt hydrate PCMs which possess outstanding features of low cost, inflammability and higher storage capacity exhibit great potentials in building energy conservation fields [14,15].  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$  (moderate melting temperature of  $32.34^\circ\text{C}$ ) is a kind of promising salt hydrates with large heat fusion, wide sources and low cost. These favorable characteristics endow  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$  with high value for building use. However, the defects of supercooling and phase separation of  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$  limit its practical application. Currently, several researches are focusing on solving these shortcomings. Shape-stabilized phase change material was prepared by integrating  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$  with modified  $\text{SiO}_2$  by Wu [16]. Confined in the silica matrix, the composite PCM had no phase separation problem. Polyvinylpyrrolidone (PVP) was then coated on the surface to improve thermal cycling performance. However, corresponding latent heat was relatively low. Wu [17] also impregnated  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}/\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$  eutectics into expanded graphite (EG) to prepare composite PCM and further coated by paraffin. Even though there were no phenomena of leakage and phase separation problems, the thermal conductivity of this composite was found to be  $3.65\text{ W}/(\text{m K})$ , which would give rise to bad thermal insulation outcomes for buildings. Based on the researches above, it can be deduced that if  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$  is combined with low-conductivity materials with porous structure, phase separation and leakage problems may effectively be restricted. The fabricated composite PCM would also possess both good thermal insulation property and large heat storage capacity, which could be widely applied to building envelopes. Moreover, Liu [18] prepared a kind of eutectic hydrated salt (melting temperature of  $31.5^\circ\text{C}$ ) which was constituted with  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$  and  $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$  (melting temperature of  $38.42^\circ\text{C}$ ). This demonstrated the eutectic mixtures of salt hydrate can reduce the melting temperature of single components and then provide  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$  with suitable temperature for utilizing in building envelopes. As a result,  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$  can be considered as a kind of excellent PCMs as building materials.

With regarded to the supporting materials for preparing shape-stabilized PCMs within construction field, most of the clay mineral materials such as kaolin [19], diatomite [20–22], sepiolite [23], montmorillonite [24], expanded perlite [14,25,26], vermiculite [27–29] and fly ash [30] with porous structures and considerable surface area are prospective candidates. The interactions of surface tension, capillary force and hydrogen bonds between salt hydrates and the supporting materials can effectively restrict leakage and phase separation of PCMs during solid-liquid transition. Different methods can be utilized for preparing shape-stabilized PCMs including vacuum impregnation and direct adsorption. Vermiculite is a kind of multi-layered

silicate rocks with excellent specific surface area on account of its apparent volume expansion after high-temperature calcining. The calcining processing leads to release of interlayer water for several times and finally the vermiculite evolves into expanded vermiculite (EV) with highly porous structure. EV is a traditional insulation material with properties of wide sources, low conductivity, fire and sound resistance as well as lightweight, which have been always used as components for roof and wallboards [31–33]. Several studies have been carried out about shape-stabilized PCMs based on EV. Wen [34] prepared two novel composite PCMs by impregnating fatty acid eutectics into EV and expanded perlite (EP). It was found that EV could adsorb more liquid fatty acid in the same situation. Zhang [25] etc. prepared a composite by incorporating eutectic fatty acid into EV. In order to increase thermal conductivity, 5 wt% Cu power was added. Xu [35] etc. developed lightweight cement-based composites by adopting paraffin/EV composite PCM as aggregate. The good thermal resistance performance endows the composite with large potential in reducing indoor temperature. The fabricated composite was then made as aggregate for developing light-weight cement. However, the latent heat is relatively low. Similar researches also carried out by Chung [36], Guan [37] and Karaipekli [38]. The obtained results above not only provide powerful proof that EV is an excellent supporting material for adsorbing organic PCM, but also indicate that it is very appropriate for insulating due to the low conductivity. It is prospective to develop novel composite PCMs comprised by inorganic salt hydrates and EV, which would have merits of inflammable, large latent, excellent insulation performance along with low cost [39]. Up to now, few works have been reported about salt hydrate/EV composite PCMs yet.

In this study, a novel EV-based composite with  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ - $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$  binary eutectic salt hydrate as PCM for building insulation was prepared by vacuum impregnation.  $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$  (melting temperature of  $32.46^\circ\text{C}$ ) was used to adjust phase transition temperature of  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$  due to its similar properties with  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ . Borax is used as nucleating agent to reduce supercooling degree of the system. Physical and chemical properties of the composite PCM were investigated. Moreover, the thermal and insulating performance of the composite PCM panels were evaluated in two test chambers as well.

## 2. Materials and experimental methods

### 2.1. Materials

Sodium sulfate decahydrate ( $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ , analytical grade) and sodium carbonate decahydrate ( $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ , analytical grade) were purchased from Kemiou Chemical Reagent Co., Ltd (Tianjin, China) and used as phase change materials. Borax ( $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ , analytical grade), which is always considered as excellent nucleating agents for  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ , was supplied by Aladdin Reagent Co., Ltd (Shanghai, China). Raw vermiculite (RV) samples were obtained from Dongbai Mineral Co., Ltd (Hebei, China). Chemical composition of RV samples determined by X-ray fluorescence spectrometer (XRF, JSX-3201Z). The RV sample consists of 43.82%  $\text{SiO}_2$ , 20.25%  $\text{Al}_2\text{O}_3$  and other metal oxide. For the sake of getting better adsorption and thermal insulation performance, RV was calcined at  $800^\circ\text{C}$  for 1 h to obtain expanded vermiculite (EV). This calcination temperature enables RV to expand as much as 8–30 times of its original volume [35,37,40]. Polystyrene box was purchased from Xinan Polystyrene box Co., Ltd (Shandong, China). Polyvinyl chloride board was provided by Meiyi Plastic Industry (Dongguan, China). Insulation cotton was purchased from Chenye crystal fiber Co., Ltd (Zhejiang, China).

### 2.2. Preparation of binary eutectic hydrated salt

The  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ - $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$  binary eutectics were fabricated by replacing  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$  with 20, 30, 40 and 50 wt%

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