

Preparation and thermal energy storage properties of D-Mannitol/expanded graphite composite phase change material

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ABSTRACT

A D-Mannitol/expanded graphite(EG) composite phase change material(PCM) was prepared for solar thermal energy storage or waste heat recovery applications performed at 180–240 °C. EG was produced by microwave irradiation performed. It was found that the EG prepared exhibited the maximum sorption capacity of 85 wt% for D-Mannitol. Scanning electron microscopy (SEM) images showed that D-Mannitol was uniformly dispersed in the micro holes of EG. Differential scanning calorimeter (DSC) analysis indicated that the phase change temperature of the composite PCM was close to that of D-Mannitol, and its latent heat was equivalent to the calculated value based on the mass fraction of D-Mannitol in the composite. With the assistance of hot disk analyzer, the thermal conductivities of the composite PCM first rise and then drop with increasing of its densities, and similar linear relationships between the maximum thermal conductivity and mass fraction of expanded graphite or between corresponding compress density and mass fraction of expanded graphite were obtained. Thermal energy storage performance of the composite PCM was tested in latent thermal energy storage (LTES) system.

The heat transfer in the composite PCM during the thermal energy storage process was enhanced through the thermal conductivity improvement, while the heat storage duration was affected by the mass fraction of EG and the apparent density of the composite PCM.

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1. Introduction

Latent thermal energy storage (LTES) using phase change material (PCM) as a storage unit is one of the most effective methods to obtain high energy efficiency and energy storage densities, which has already widely been applied in solar energy utilization, cooling of electronic devices, waste heat recovering and other fields. In the past decades, LTES systems have been extensively studied for high performance [1–3]. The PCMs with the properties such as high latent heat, chemical inertness, high thermal conductivity, and commercial availability will be taken as ideal thermal energy storage for desired applications.

In areas where solar thermal energy consumption focuses on high temperature, PCMs with high phase change temperature has come a main object of investigations in recent years. In

temperature range higher than 100 °C, a great interest is focused on inorganic eutectic salts as PCMs because of their lower investment costs and wide range of melting temperatures to apply different applications [4]. Binary and ternary eutectics are frequently applied for energy storage purpose in temperature interval of 100–300 °C [5–9], but these eutectic salts suffer a comparatively low latent heat (< 120 J/g), which leads to a reduction in energy storage capacity per volume of a LTES system. Besides that, the eutectic salts possess a poor thermal conductivities (< 1 W/m K), which will suppress their heat storage rates. Even more importantly, almost all salts have a corrosive action to ordinary metals at high temperatures. Thus, those drawbacks of salts will consequently obstacle their widespread applications. According to Kaizawa et al., Erythritol, Xylitol and D-Mannitol appears to be reliable PCMs for high-temperature LTES applications, due to large latent heat and high melting temperature [10]. Especially, D-Mannitol is a natural polyol having a high phase change temperature (> 160 °C) and large latent heat of melting (> 300 J/g) [11]. Its ability to retain heat effectively in phase change state affirms its capability for use as a PCM. Furthermore, D-Mannitol's

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relatively high density of 1.52 g/cm^3 allows a large quantity of *D*-Mannitol to be packed into a small volume in comparison with paraffin waxes. This, coupled with its high specific heat of melting allows for large amounts of energy to be absorbed for storage [10]. As regards the cost, *D*-Mannitol is quite cheap. Comprehensive above factors, *D*-Mannitol aroused our interest to be selected as a promising PCM for practical LTES application performed at 180–240 °C.

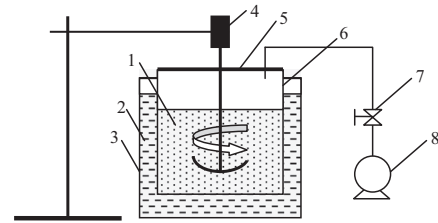
However, two problems must be resolved actually. One problem is that the slow rate of heat transfer is generally unacceptable, which is caused by the low thermal conductivities of PCMs [12,13]. A great amount of studies on have been carried out to enhance thermal conductivity of PCM. Techniques as addition of high-thermal-conductivity particles, insertion of fins, and impregnation of PCMs into highly conductive porous structures are attempted to be applied in LTES applications [14–17]. However, the configuration of fin tubes and addition of high-thermal-conductivity materials will bring significant weight and cost increase. Another problem is that it is very difficult to encapsulate PCMs in high-temperature applications (above 100 °C) [18,19]. Liquid PCM are usually easy to leak through crack generated by volumetric expansion upon melting. For this reason, a higher thickness of the capsule layer is required, but this leads to a decrease in the heat storage density of composite PCM. Porous materials with a high thermal conductivity are regarded as candidates for solving these two problems simultaneously. It is considered that porous materials can form a stable shape that avoids the leakage of liquid PCM because of the capillary and surface tension forces of a porous structure. Recently, expanded graphite (EG) has been adopted to enhance the heat transfer and prevent from liquid leakage upon melting in PCMs, due to its desirable properties of better stability, higher thermal conductivity, better compatibility with organic PCMs, and lower density as compared with metallic foams [19–24].

Although *D*-Mannitol has been mentioned previously and EG is widely used to enhance heat transfer process and encapsulate PCMs, few studies about the utilizations of *D*-Mannitol in LTES applications and the thermal conductivity improvement of *D*-Mannitol with the help of EG has been reported. Hence, the development and study on a new *D*-Mannitol/EG composite PCM is valuable for the promotion of solar thermal energy utilization and waste heat recovery. In the present work, a composite PCM with *D*-Mannitol as PCM and EG as encapsulation heat transfer promoter was prepared. The thermal properties of the composites were studied by measuring the melting temperature, latent heat of phase change and thermal conductivity. Meanwhile, its thermal energy storage performance with EG mass fraction and compressing density in a LTES unit were experimentally tested and compared.

2. Experimental

2.1. Preparation of composite PCM

In the experiments, analytically pure *D*-Mannitol ($\text{C}_6\text{H}_{14}\text{O}_6$) with a melting point of 166–169 °C was selected as the PCM, which was made of the Shanghai Bio Science & Technology CO. Ltd. Expanded graphite was used to serve as a support in the composite process. Expanded graphite was made by the expandable graphite with average particle size of 500 μm and expansion rate of 300 ml/g. The expandable graphite was supplied by the Qingdao Hengsheng Graphite Co. Ltd. After being dried in a vacuum oven at 120 °C for 2 h, the expandable graphite of about 10 g was put into a microwave oven (800 W, Midea, China) for 20 s every time in order to obtain the expanded graphite. A *D*-Mannitol/Expanded graphite composite PCM was produced by absorbing the liquid *D*-



1- Mixture of *D*-Mannitol and expanded graphite 2-Conduction oil 3-Stainless steel tank
4-Mixer 5-Seal cover 6-Glass container 7- Valve 8-Vacuum pump

Fig. 1. Schematic diagram of the composite process.

Mannitol into the expanded graphite. Fig. 1 shows a schematic diagram of the composite process. First, solid *D*-Mannitol and expanded graphite were placed in a glass container with a seal cover. Then, the glass container was put into the stainless steel tank containing conduction oil which was heated to about 220 °C. Next, the vacuum pump started to remove the air from the glass container and the mixer also started to stir the mixture for about 2 h. Finally, the vacuum pump was turned off and the composite PCM was removed from the glass container and then it was allowed to cool down until the PCM was solidified. For each EG composite PCM, based on the sorption curve as described in our earlier investigation [25], the maximum sorption capacity was determined by the experimental method. In this study, the mass percentage of *D*-Mannitol in the composite PCM was used to express the sorption capacity. The maximum sorption capacity of *D*-Mannitol can reach 85% for the expanded graphite sample prepared. The composite PCMs with different mass percentage of *D*-Mannitol were chosen as tested PCM samples.

2.2. Characterizations of PCM

In this study, a scanning electron microscope (SEM, Quanta, FEI, Hotland) was used to observe the micro structures of the expanded graphite and the composite PCM. The melting and heat storage behaviors of the pure *D*-Mannitol and composite PCM were studied by a differential scanning calorimeter (DSC, DSCQ20, TA Instruments Inc., USA) under the protection of nitrogen. The data were collected at a scan rate of 5 °C/min from 120 °C to 210 °C. The quantity of each sample in the calorimeter sample cells made of aluminum is in the range from 6 to 10 mg. The latent heat and melting temperature for PCM were obtained by DSC. The thermal conductivities of PCM with a type 8563 probe acting as both heat source and sensor was measured by Hot disk thermal constant analyzer (Hot Disk TPS2500, Hot Disk Inc., Sweden). A transient plane source method, in which two disk-like samples for the test materials with a similar thickness were required to be placed in contact with the probe and heated at constant power for a setting scanning time, was selected for these measurements.

2.3. Testing device of thermal energy storage performance

The thermal energy storage characteristics of *D*-Mannitol and composite PCM in a LTES system were investigated, respectively. The schematic diagram of the experimental system shown in Fig. 2 mainly consisted of an automatic temperature test chamber and a LTES unit. The temperature of test chamber was automatically controlled by a temperature control system with an accuracy of ± 1 °C. The tested PCM samples which were produced by steel shell mould and tablet press (DY-40, Tianjing Keqi Co. Ltd, China) could be packaged completely by the cylindrical alloy aluminum container with inner diameter of 50mm, outer diameter of 48mm and height of 100 mm. The K-type thermocouple was set in the center of each cylindrical PCM sample to investigate the

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