



## Sodium nitrate – Diatomite composite materials for thermal energy storage



Guizhi Xu<sup>a</sup>, Guanghui Leng<sup>b,c,\*</sup>, Cenyu Yang<sup>a</sup>, Yue Qin<sup>d</sup>, Yuting Wu<sup>e</sup>, Haisheng Chen<sup>f</sup>, Lin Cong<sup>b</sup>, Yulong Ding<sup>b</sup>

<sup>a</sup> Global Energy Interconnection Research Institute, China State Grid, Beijing, China

<sup>b</sup> Birmingham Centre of Energy Storage, University of Birmingham, Birmingham, UK

<sup>c</sup> Institute of Process Engineering, Chinese Academy of Sciences, Beijing, China

<sup>d</sup> Civil Aviation University of China, Tianjin, China

<sup>e</sup> Beijing University of Technology, Beijing, China

<sup>f</sup> Institute of Engineering Thermophysics, Chinese Academy of Sciences, Beijing, China

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### ABSTRACT

The work reported in paper concerns the use of diatomite to form-stabilise sodium nitrate, a phase change material (PCM) for medium temperature thermal energy storage applications. The composite was found to be able to retain up to 70% of the nitrate salt. X-ray diffraction (XRD) analyses suggested an excellent chemical compatibility between diatomite and the salt. Scanning electron microscope (SEM) analyses demonstrated an even distribution of the salt within the diatomite structure. Differential scanning calorimetry (DSC) measurements showed that melting temperature of the material was approximately 307.8 °C with a latent heat of 115.79 kJ/kg. Mechanical characterization of the composite material showed a compressive strength of the composite materials as high as 22.17 MPa. The composite materials were found to have a fairly low thermal conductivity of ~0.5 W/m K and an addition of graphite could give a substantial thermal conductivity enhancement (~6-fold with an additional of 10 wt% graphite).

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### 1. Introduction

Due to potential applications in peak shaving of electrical power grids, solar thermal power generation, energy-efficient buildings and industry waste heat recovery, significant attention has been paid to high performance phase change based composite thermal energy storage (TES) materials over the past few decades (Xu et al., 2015; Marliacy et al., 2000; Kang et al., 2015; Rogeta et al., 2013; Galione et al., 2015; López-Sabirón et al., 2014). Form-stable composite phase change thermal storage materials have been attracting great attention, as they possess properties which contribute to enhanced heat transfer performance and reduced high temperature corrosion effect. Also their good mechanical properties make them applicable in a variety of applications. When phase change occurs, the shape of the composite material remains because the PCMs whose phase state changes are enclosed in the shape stabilize material. As phase change materials (PCMs) store

heat in both sensible and latent forms and a significant part of the stored heat is due to the latent heat, PCM based TES materials offer a high energy density. Industrial uptake of the technology, however, has been hindered by a number of challenges including materials, processes and economical aspects. There are numerous PCMs that can be used for making composite materials. Examples include paraffin waxes (Cai et al., 2015), fatty acids (Zeyu et al., 2014) and hydrated salts (Kenisarin and Mahkamov, 2016) with low phase change temperatures, and nitrates, carbonates, chlorides, sulphates and their combinations with medium to high phase change temperatures (Zalba et al., 2003). There also exists a range of shape stabilization materials including inorganic metal oxides, metals, large molecular polymers and expanded graphite (Nomura et al., 2015; Alkan et al., 2009; Zhong et al., 2010). Normally, large specific surface area, good adsorption material such as vermiculite, diatomite, and expanded graphite are used as shape stabilization materials for the preparation of composite thermal storage material to impregnate the liquid phase change material. Xu et al. (2015) used vacuum impregnation method for impregnating the melted paraffin liquid into the expanded vermiculite. The mass fraction of adsorption paraffin is 37.5% in the composite

\* Corresponding author at: Birmingham Centre of Energy Storage, University of Birmingham, Birmingham, UK (G. Leng).

E-mail address: [a280794125@163.com](mailto:a280794125@163.com) (G. Leng).

thermal storage material, and the latent heat of the composite material is around 77.6 J/g. Li et al. (2015) used impregnation method to prepare Paraffin/nanosilica form-stable PCM composites which were then mixed with fibers, and compressed as vacuum insulation panels (VIP). The results indicate that the insulating effect of the VIP was better than the normal organic foam insulations. The thermal mass of VIP was improved, and it saves construction costs without installing PCM and insulation separately. In particular, the PCM will be contained in the backbone material. Karaman et al. (2011) used vacuum impregnation method and diatomite to shape stabilize polyethylene glycol, which has a phase change temperature of 27.7 °C and a latent heat of 87.09 J/g. They found that the composite materials could hold about 50 wt% of the PCM with a good chemical and physical stability in cyclic heating-cooling experiments. Nomura et al. (2009) studied diatomite-erythritol composites using a vacuum impregnation method. Erythritol has a high liquid-solid phase change latent heat of 294.4 J/g. They observed that liquid erythritol fully populated within pores of diatomite and the pore size had an effect on the melting point; the smaller the pore size in the diatomite the lower the melting point of the PCM. They also found that the latent heat of the composite material could be as high as 83% that of pure erythritol. However, a considerable loss of the phase change material was observed during thermal cyclic tests with 25% mass loss after four cycles between the room temperature and 150 °C.

Pincemin and Py (2008) used cold compressed method to prepare eutectic NaNO<sub>3</sub>/KNO<sub>3</sub> salt based graphite composites for heat storage at a medium temperature of 200–300 °C. They found changes in the enthalpy of the material due to structural change of graphite after 200 charge-discharge cycles. Qin et al. (2015) used cold compressed and sintering method to prepare the Na<sub>2</sub>SO<sub>4</sub>/diatomite composite materials. The composite material containing 55% salt gives an optimal formulation and has an energy density over 360 kJ/kg in range of 700–900 °C.

This work concerns sodium nitrate based composite PCM materials. Such a PCM has a phase change temperature of approximately 308 °C and hence is suitable for medium temperature applications. Diatomite is used for shape stabilization. The reasons for the use of diatomite lie in low raw material costs, high specific surface area, great absorbability, and good thermal and chemical stability even at elevated temperatures (Roberts, 1999; Nakajiman and Harrell, 2002; Khraishem et al., 2004). In this work, the samples were prepared with cold compress method, and then sintered at high temperature. This method can effectively improve the thermal conductivity of the material. Furthermore, the phase change material encapsulated within the material does not leak and the samples which can be formed into any shape that we need has good mechanical properties, and are easy to be installed. Available literature on the use of diatomite for shape stabilizing medium temperature PCMs seems rather lacking, which have many potential applications including solar thermal power generation, peak shaving of electrical power grids, decentralized energy systems, and unsteady industrial waste heat recovery. This forms the main motivation of the work reported in this paper. The work involves formulation and fabrication of sodium nitrate based diatomite composite materials, characterization of their thermal and physical properties, and the measurements of their chemical and physical stabilities.

## 2. Experimental

This section gives the details of the raw materials used for formulating diatomite based composite, procedures for fabricating the composites, and experimental methods for characterizing the materials.

### 2.1. Raw materials

Both the sodium nitrate and diatomite were in a solid form at the ambient temperature. The sodium nitrate was analytic grade and purchased from China Xilong Chemicals Inc., whereas the diatomite was provided by China Shengzhou Diatomic Manufacturing Inc. Table 1 shows chemical compositions of the diatomite obtained with an X-Ray fluorescence analyser (AXIOS-MAX X, PANalytical B.V.). One can see that silica (82.6%) and alumina (8.2%) are two main components of diatomite and the rest 15 minor components account for less than 10%. The diatomite has a disc-like shape with many nanoscale pores; see late for scanning electron microscopic images and further discussion. BET measurements using an Autosorb-1 device (Quantachrome, USA) showed that the diatomite had a specific surface area of 66.29 m<sup>2</sup>/g. In order to enhance thermal conductivity, analytical grade graphite purchased from China Xilong Chemicals Inc. was used in the formulation.

### 2.2. Fabrication of diatomite-sodium nitrate composites

The fabrication process involved weighing the particulate materials, followed by grinding separately at the ambient temperature to a size that can go through a 120-mesh sieve, then thorough mixing and tableting to give disc-like green pellets. To ensure sufficient mechanical strength of the green pellets for subsequent drying and sintering processes, a small amount of 1 wt% starch solution was added during mixing. The tableting was done at a pressure of 10 MPa and the resulting green pellets had a mass of 50 g and were 50 mm in diameter. The green pellets were dried at 120 °C for 2 h before sintering at 370 °C for an hour in an electrical oven. Different formulations were studied using different mass ratios of diatomite to sodium nitrate. Table 2 shows the compositions of the formulations without graphite. When 10 wt% graphite is added, the cost of this composite thermal storage material was estimated at \$ 800/ton which is cheaper than the paraffin and metal thermal storage material. It is more cost-effective than molten salts. This type of thermal storage material can be directly used in large-scale thermal storage systems. Because this material was made form-stabilise. The gaseous heat transfer fluids can transfer heat by flowing through the loose packed pellet piles while the liquid flow (such as water, oil) cannot, because of the material is dissolved in this liquid.

### 2.3. Measurement and analyses

A micro-hardness instrument (MX-6, Shenzhen Shuanhua Instruments, China) was used to measure the hardness of the composite thermal energy storage materials, which relates to materials strength. The hardness measurements were done at a load of 2 N at the ambient temperature. The ATSM C39 standard was followed in measurements. Chemical composition was determined by using an X-Ray fluorescence spectrometer (PANalytical B.V., Netherlands) as mentioned before, whereas chemical compatibility was investigated with a PANalytical X-Ray Diffractometer (XRD). A JSM-7001F SEM (JEOL, Japan) was used to observe the material surface

**Table 1**  
Chemical compositions of the diatomite.

Diatomite	SiO <sub>2</sub> (wt%)	Al <sub>2</sub> O <sub>3</sub> (wt%)	Fe <sub>2</sub> O <sub>3</sub> (wt%)	MgO (wt%)	CaO (wt%)	Pb (mg/Kg)	As (mg/Kg)
X	≥85	<5	≤1.5	<0.4	<0.5	≤4	≤5

X is the percentage of chemical compositions. Standard uncertainties u is u(x) = 0.01.

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