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Study on preparation and thermal properties of sodium nitrate/silica composite as shape-stabilized phase change material

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ABSTRACT

A sodium nitrate ($NaNO_3$)/silica (SiO_2) composite was prepared as a shape-stabilized phase change material by a sol–gel procedure. In this composite, $NaNO_3$ acted as the phase change material and SiO_2 was used as the supporting material. The maximal weight percentage of $NaNO_3$ in the composite was determined to be 60 wt.%. The chemical composition, morphology, structure and thermal properties were investigated by Fourier transform infrared spectroscopy (FT-IR), scanning electron microscope (SEM), thermal gravimetric analysis (TGA), differential scanning calorimeter (DSC) and Laser thermal conductivity meter. The DSC results indicated that the enthalpies of melting and freezing of the $NaNO_3/SiO_2$ (60 wt.% $NaNO_3$) composite were 108 kJ/kg and 110 kJ/kg, and the corresponding temperatures of the phase transition were 302 °C and 300 °C, respectively. In the temperature range of lower than 500 °C the phase change enthalpy of the composite was increased with the increase of the roasting temperature.

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1. Introduction

Phase change material (PCM), which is a type of functional material, can absorb, store and release latent heat of material during solid–solid or solid–liquid phase change process. Due to its high thermal storage density and small temperature variation during phase transition, the latent heat energy storage employing the PCM is one of the most effective ways of thermal energy storage [1–4]. The PCMs have been applied in many fields, such as solar energy storage, building energy saving, temperature control textiles, waste heat recovery and storage from industrial process [5–8].

So far, many PCMs such as hydrated salts [9], inorganic salts [10–12], paraffins [13], fatty acids [14], polyesters [15] and polyethers [16] have been investigated for thermal energy storage at different temperature range. Compared with other PCMs, inorganic salts usually exhibit high thermal stability and are advantageous for the application at high temperatures (>200 °C) [17–19]. However, a major issue in using inorganic salts by itself for thermal energy storage is that inorganic salts needs encapsulation to prevent leakage of melted inorganic salts during the phase change process, which results in extra thermal resistance and increased cost. Shape-stabilized PCMs, which are composed of working substance and supporting material, can remain in solid state and avoid the leakage of melted working substance even when the working substance changes from solid to liquid, making the practical application more convenient. Salt-ceramics composites are a type of shape-stabilized PCMs using inorganic salts as working substance. In the field of salt-ceramics, there are usually two preparation methods which are the infiltration methods and mixing method. In the infiltration method, the salt melts in an electric furnace and infiltrates into the porous ceramics, and the composite is formed when the furnace cools to room temperature. In the mixing method, a mixture of ceramics powder, salts, and an appropriate amount of an additive is pressed to form a specific shape and sintered at high temperature. There have been some reports on the salt-ceramics [20–23]. These works were mainly focused on the preparation of new materials by changing the phase change substance, carrier and preparation methods, as well as the further characterization of the composites in terms of thermal properties. In the inorganic salts, sodium nitrate ($NaNO_3$) is regarded as a promising inorganic salt PCM due to its high heat of fusion, suitable melting temperature range and good stability [24–26]. Rysiakiewicz-Pasek et al. [27] prepared $NaNO_3$/porous glasses composite by infiltrating melted $NaNO_3$ into porous glass with different average pore diameters and studied the phase transition properties of $NaNO_3$ in the composite. However, this preparation process was carried out for long time at high temperature, and it

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was not convenient for application. Lopez et al. [10] prepared $NaNO_3$––KNO_3/graphite composite for thermal energy storage by means of physical mixing of the as-received salt powders and expanded graphite particles and followed by auni-axial compression (50–100 bar). Although shape-stabilized $NaNO_3$––KNO_3/graphite PCMs with good thermal properties were prepared, one drawback of the preparation method was the usage of high pressure, which resulted in high energy consumption. Therefore, it is important to develop new methods of preparing the shape-stabilized PCMs with $NaNO_3$ as working substance at room temperature and atmospheric pressure.

In this work, a novel shape-stabilized PCM, $NaNO_3/SiO_2$ composite was prepared under ambient conditions using Na_2SiO_3 as the silicon source. The difference of our work from the previous publications was that we prepared the PCMs by a new method which was sol–gel procedure. The maximal weight percentage of $NaNO_3$ dispersed in the PCM composites without any leakage of the melted $NaNO_3$ was determined. The composition, structure and thermal properties of the composite were characterized by Fourier transform infrared spectrophotometer (FTIR), scanning electronic microscope (SEM), thermal gravimetric analysis (TG) and differential scanning calorimeter (DSC). The effect of the roasting temperature on the phase change enthalpy was also studied. The results demonstrate that this composite has good properties and will be useful for the thermal energy storage at high temperature.

2. Experimental

2.1. Chemicals and materials

Sodium metasilicate nonahydrate ($Na_2SiO_3{\cdot}9H_2O$, A.R.) and sodium nitrate ($NaNO_3$, 98%, AR) were purchased from Beijing Modern Eastern Fine Chemicals (Beijing, China). Deionized water was prepared in our laboratory.

Cation exchange resin of sodium form (732) was provided by Beijing Modern Eastern Fine Chemicals (Beijing, China). It was transformed to be the hydrogen form after treatment with sulfuric acid aqueous solution.

2.2. Methods

The procedure for the preparation of $NaNO_3/SiO_2$ composite consisted of two steps: determination of SiO_2 content and preparation of composite.

Determination of SiO_2 content: Na_2SiO_3 solution (0.5 mol/L) was passed through the cation exchange resin of hydrogen form until the pH of the eluent reached 7.0–8.0, so that Na^+ in the solution was replaced by H^+, and the silicic acid solution was formed. Then 10 mL of silicic acid solution was gelatinized at 50 °C, and then dried in the oven at 100 °C for 24 h to prepare the pure SiO_2 xerogel. The dried SiO_2 xerogel was weighed and used as a standard, and the ratio of SiO_2 and $NaNO_3$ can be controlled by adding $NaNO_3$ with different weights.

Preparation of composite: the silicic acid solution (pH 7–8) was stirred vigorously for 10 min after adding $NaNO_3$. Then the mixture was gelatinized at 25 °C in a water bath for 24 h. The gel formed was dried in the oven at 100 °C for 48 h to produce a $NaNO_3/SiO_2$ composite. To improve the heat storage performance of composite, the prepared $NaNO_3/SiO_2$ composite powder was heated up to 500 °C with heating rate of 10 °C/min and roasted for 5 h in the oven.

The composites with different content of $NaNO_3$ were prepared, and there was leakage of $NaNO_3$ from the surface of the composite after roasting when the mass percentage of $NaNO_3$ in the composites was higher than 60 wt.%. So the maximum mass percentage of $NaNO_3$ dispersed into the PCM composites was determined to be 60 wt.%.

2.3. Characterization

The composition of the prepared composite was determined by Fourier transform infrared spectroscopy (FT-IR, Tensor27, Bruker, Germany) in the wavenumber range of 400–4000 cm^{-1} at room temperature. The morphology and structure were observed using a scanning electron microscope (SEM; JSM7401, Shimadzu, Kyoto, Japan) at a 30 kV accelerating voltage. The thermal stabilities were investigated by thermal gravimetric analysis (TG, STA409PC, Netzsch, Germany) at a heating rate of 10 °C /min in the air from 30 °C to 1000 °C. The phase change properties of $NaNO_3$ and $NaNO_3/SiO_2$ composite were characterized using a differential scanning calorimeter (DSC; STA409PC, Selb, Netzsch, Germany) in the nitrogen atmosphere with a heating rate of 10 °C /min from 30 °C to 350 °C. The heat transfer properties of composite were investigated by Laser thermal conductivity meter (LFA447 Netzsch, Germany).

3. Results and discussion

3.1. Chemical composition of the composite

The FTIR spectra of SiO_2, $NaNO_3$ and the $NaNO_3/SiO_2$ composite are shown in Fig. 1. In the FTIR spectrum of SiO_2, there were peaks at 1100 cm^{-1}, 793 cm^{-1} and 471 cm^{-1}, which were caused by the Si—O—Si antisymmetric stretching vibration, the symmetric stretching and bending vibrations, respectively [28]. The peak at 3465 cm^{-1} represented the stretching vibration of the OH groups, which was contributed by unreacted Si—OH groups during the gelation. For $NaNO_3$, the peaks at 1390 cm^{-1} and 2431 cm^{-1} were caused by the N—O stretching vibration and the peak at 833 cm^{-1} was caused by the N—O symmetric stretching vibration [29]. In the FTIR spectrum of the $NaNO_3/SiO_2$ composite, the characteristic peaks at 1100 cm^{-1}, 793 cm^{-1}, 471 cm^{-1}, 3465 cm^{-1}, 1390 cm^{-1}, 2431 cm^{-1} and 833 cm^{-1} were found without significant new peaks. This result indicated that the prepared $NaNO_3/SiO_2$ composite using sol–gel method was a physical hybrid, and $NaNO_3$ and SiO_2 did not occur chemical reaction during the preparation.

3.2. Morphology and structure of the composite

The prepared samples of the roasted $NaNO_3/SiO_2$ composite were pressed to form circular shapes at 6 MPa for 2 min, and were then tested in repeated thermal cycles between 150 °C and 350 °C

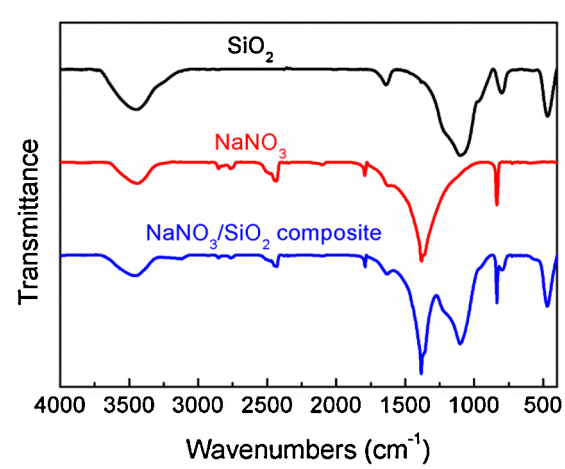

Fig. 1. FTIR spectra of SiO_2, $NaNO_3$ and $NaNO_3/SiO_2$ composite.

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