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Novel Na₂SO₄@SiO₂ phase change material with core-shell structures for high temperature thermal storage



Solar Energy Material

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ABSTRACT

Microencapsulated composite material using Na₂SO₄ as core and SiO₂ as shell for high temperature thermal energy storage is prepared. The effects of silica mass percentages within the Na₂SO₄@SiO₂ PCM composites on thermal conductivity, thermal stability, melting temperature, and latent heat are investigated. No new phases are formed during the encapsulation process. The spherical silica nanoparticles with diameters at around 300 nm are well decorated on the surface of Na₂SO₄. The PCM composite with 5.4% silica addition is determined as the optimal sample due to its excellent comprehensive properties. The inhibition of liquid leakage during melting can be effectively realized. The thermal conductivity of Na₂SO₄@SiO₂ PCM under high temperatures (600–800 °C) can be increased to 0.59 W/(m K), 0.62 W/(m K), and 0.87 W/(m K), respectively. The initial and peak melting temperatures of the Na₂SO₄@SiO₂ are tested at 885.20 °C and 887.91 °C, respectively. The latent heat is determined as 110.6 J/(g K), and the total heat storage density including latent and sensible heat with a temperature span of 100 °C is 390.6 J/g, and the mass loss is observed < 1% under 1000 °C. In addition, the latent heat loss percentage after 50 thermal cycle tests is $\leq 4.3\%$.

1. Introduction

With the increase of energy consumption, the shortage of fossil fuels has become a big challenge for human society. Concentrated solar power (CSP) technology uses focused sunlight to provide heat at higher temperatures for electricity generation or supply energy to other energy-intensive industries, which has shown great potential in renewable energy sector [1]. The core issue of CSP technology is the utilization of thermal energy storage (TES) system to continuously generate electricity in nighttime when there is no sunlight. Phase change material (PCM) possesses high energy storage density due to the phase change process and it has been widely used in the TES system even though not limited to CSP plants. However, previous researchers have been mostly focused on the low temperature phase change materials, including salt hydrates, fatty acids and paraffin waxes, etc [2-6]. To the best of our knowledge, fewer papers on the high temperature phase change materials (melting temperature > 300 °C) have been published due to the corrosion issue, low thermal conductivity and low thermal stability at high temperatures [7].

The high temperature PCMs can be mainly classified into two categories: eutectic salt and alloys compounds. Compared to eutectic salts, the alloys compounds based PCMs generally possess lower melting temperatures, more complicated fabrication processes and most of all a higher cost [8–10]. The eutectic salts based high temperature PCM show advantages of more cost-effective, higher melting temperature, and larger heat of fusion [11]. Jiang [12] et al. developed a eutectic Na₂CO₃-NaCl salt which had a melting temperature at 637 °C with a heat of fusion of 283.3 J/g and the thermo-physical properties varied marginally after 50 and 100 thermal cycle tests. Thomas [13] et al. proposed a high temperature of 306 °C with a reported melting enthalpy of about 178 J/g. Philip [14] et al. pointed out that the chloride salts may be promising PCMs due to their excellent thermal stability and lower cost compared to nitrates.

The eutectic salts based high temperature PCMs have been mainly faced four challenges: corrosion to the containers, liquid leakage, low thermal conductivity, and poor thermal stability under high temperature. Many researchers have paid attention in solving these issues of salt based PCMs. Zheng [15] et al. used the stainless steel 304 as the shell and eutectic NaNO₃ and NaCl-MgCl₂ as the core materials to develop core-shell PCM composites. Huang [16] et al. reported LiNO₃/KCl-expanded graphite composite PCM, which utilized graphite to increase

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the thermal conductivity of the salt PCM. Guo [17] et al. presented a NaNO₃/SiO₂ composite PCM using the sol-gel technique. The enthalpies of melting and freezing of the composite were reported at 108 J/g and 110 J/g, with the corresponding phase change temperatures at 302 °C and 300 °C, respectively. Zhou [18] et al. proposed a method using silica ceramic composite as the porous matrix for preparing the paraffin/silica ceramic composite. Results showed that the composite remained solid above the melting temperature of paraffin even the mass fraction of paraffin reached to 75%.

In recent years, some researchers have focused on PCMs working under even higher temperature, within which Na₂SO₄ salt has attracted much attention for its melting temperature above 850 °C, good thermal stability and relatively weak corrosion to the container. Wu [19] et al. developed a new type of high temperature PCM consisting of SiC ceramic foam and Na₂SO₄ salt. Results showed that no chemical reaction occurred between Na₂SO₄ and SiC matrix. However, its latent heat decreased to 30.03 J/g, and the heat storage density was about 161 J/g in the temperature range of 800–900 °C. Liu [1] et al. prepared a Na₂SO₄ based high temperature PCM using the impregnation method. Mullite was used as the porous matrix, while Na₂SO₄ was loaded with different volumetric ratios, and the measured latent heat was 105.33 J/ g.

In this work, a novel core-shell structured Na₂SO₄/SiO₂ PCM composite is prepared using the so-gel technique. Na₂SO₄ is used as the high temperature PCM, while the silica is encapsulated on the surface of the Na₂SO₄ particles. The effects of silica mass percentages (0%, 2.7%, 3.8%, 5.4%, 13.4% and 26.8%) within the Na₂SO₄@SiO₂ composite on the thermal conductivity, thermal stability, melting temperature, and latent heat of the composite are investigated. The silica particles encapsulated on the surface of Na₂SO₄ increase the thermal conductivity of the PCM composite, as well as preventing liquid leakage of the melted Na₂SO₄. The thermophysical properties vary marginally after 50 cycling tests. One appealing application of this microencapsulated PCM is that it may be mixed and move with the heat transfer fluid (HTF), another salt with lower melting temperature, to form a slurry flow for creating a new TES system for CSP plants, and thus highly increases the adoption of latent heat thermal storage in the CSP sector.

2. Experimental section

2.1. Materials and synthesis methods

Sodium sulfate (Na₂SO₄), tetraethyl orthosilicate (TEOS), concentrated ammonium hydroxide (NH₄OH), ethanol (EtOH), deionized water (H2O) were purchased from Sigma-Aldrich Corporation. All of them were of analytical reagent grade and no further purification was carried out. In a typical synthesis route, 40 mL EtOH was firstly mixed with 10 mL water and this mixture was stirred for 10 min under room temperature. 1 mL concentrated NH₄OH solution was then added into the mixture, further stirred for 10 min. The pH value of the solution was about 13, which would promote the sol-gel reaction for the formation of core-shell structures. Different amounts of Na₂SO₄ powders (10 g, 7 g, 5 g, 2 g and 1 g) were then added into the mixture, and the mixture was further stirred for 30 min 1 mL TEOS was then dropped into the Na₂SO₄ solution, therefore the silica mass percentages in the $Na_2SO_4@SiO_2$ PCM can be calculated as 2.7%, 3.8%, 5.4%, 13.4% and 26.8%, respectively. The blank sample with no TEOS addition is also prepared as reference. The composite solution was stirred at room temperature for 12h to induce the sol-gel reaction, followed by water wash, filtration and vacuum drying at 80 °C for 24 h. The core-shell structured Na₂SO₄@SiO₂ PCM composites with different silica ratios were then derived for further characterization and evaluation.

2.2. Characterizations

The X-ray diffraction (XRD) patterns were recorded using a Rigaku

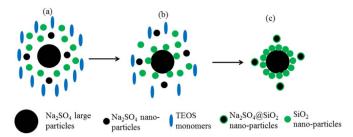


Fig. 1. Schematic diagram of the SiO_2 shell growth mechanism on the Na_2SO_4 core.

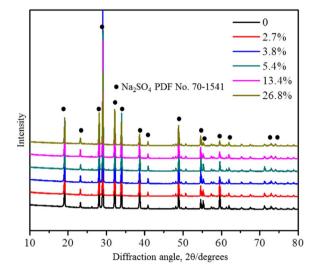


Fig. 2. XRD patterns of Na_2SO_4 and $Na_2SO_4@SiO_2$ PCM composites with different silica mass percentages.

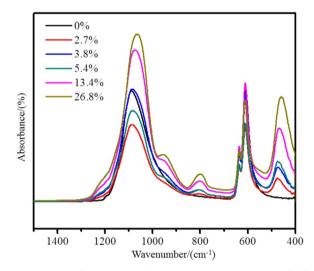


Fig. 3. FTIR spectra of pure Na₂SO₄ and Na₂SO₄@SiO₂ PCM composites with different silica mass percentages.

Smartlab X-ray diffraction analysis with CuK α 1 radiation ($\lambda = 0.15406$ nm). The X-ray tube was operated at 40 kV and 40 mA. The particle size distribution of pure Na₂SO₄ powder and Na₂SO₄@SiO₂ PCM composite were performed on the Microtrac S3500 laser scattering particle analyzer. Thermal gravimetric analysis (TGA) and differential scanning calorimetry analysis (DSC) were performed by SDT Q600 Thermogravimetric analyzer under flowing nitrogen of 100 mL/min at a heating rate of 10 °C/min to 800 °C. Scanning electron microscopy (SEM) was conducted using a FEI Quanta FEG 450 Scanning Electron Microscope. Transmission electron microscope (TEM) and selected area electron diffraction (SAED) images of the PCMs were taken using FEI

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