



# Preparation and thermal properties of stearic acid/titanium dioxide composites as shape-stabilized phase change materials for building thermal energy storage

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## ABSTRACT

In this study, stearic acid (SA)/titanium dioxide (TiO<sub>2</sub>) composites with different mass ratios were prepared by mixing titania powder with stearic acid–water emulsion. In the composites, the SA performed as phase change material for thermal energy storage, and TiO<sub>2</sub> was used as supporting material. The thermal properties of the composites, such as phase change temperature and phase change latent heat, were measured by differential scanning calorimetry (DSC). Fourier transformation infrared spectroscopy (FT-IR) analyses indicated that there was no chemical interaction during the preparation process. X-ray diffractometer (XRD) and scanning electronic microscope (SEM) were used to survey crystalloid phase and microstructure of the SA/TiO<sub>2</sub> composites. Besides, the thermal reliability of the composites was investigated by a thermogravimetric analyzer (TGA). The satisfactory SA/TiO<sub>2</sub> composite with 33% mass ratio of the SA melts at 53.84 °C with a latent heat of 47.82 kJ/kg and solidifies at 53.31 °C with a latent heat of 46.60 kJ/kg. Due to its non-inflammability, nontoxicity and good thermal stability, the composite can be used as shape-stabilized phase change materials for building thermal energy storage.

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## 1. Introduction

In recent decades, the increasing energy consumption has been a big challenge for the development of human society. Therefore, the investigations of new energy resources and efficient ways of energy storage and recovery have attracted much attention all over the world [1]. Latent heat thermal energy storage (LHTES) is one of the most efficient ways to store thermal energy. In the LHTES system, thermal energy is stored in phase change materials (PCMs) during a melting process while it is recovered during a freezing process. It is due to the fact that the PCMs have great endothermic and exothermic capacity during the phase change process [2]. The PCMs can be applied in various fields according to their phase change temperatures, such as air-conditioning systems [3,4], building energy conservation [5–8], solar heating systems [9–13].

Currently, various PCMs such as inorganic salt hydrates [14], *n*-alkanes [15], fatty acids [16,17] have been studied for LHTES application. Among all these PCMs, fatty acids have advantages in proper melting temperature range, lower vapor pressure, small

volume change, etc. [18]. However, two disadvantages are also obvious when PCMs are used in thermal energy storage applications. One is that the low thermal conductivity of most PCMs restricts the transmission of heat during phase change process and the other is the liquid migration during solid–liquid melting process. Therefore, microencapsulation and combining PCMs with other supporting materials as shape-stabilized composites are developed [19,20]. Many organic and inorganic materials are chosen as supporting materials so far, such as opal [21], styrene–butadiene–styrene copolymer [22], expanded graphite [23], molecular sieve [24] and calcium carbonate [25]. The inorganic materials are superior to organic ones in buildings because most of the latter are flammable and toxic.

The previous researches are commonly using organic polymers as supporting materials [26,27]. However, the application of PCMs with polymeric supporting materials is usually restricted due to their toxicity, flammability, low heat conductivity and poor thermal stability [28]. So some inorganic supporting materials such as SiO<sub>2</sub> [29] are used in composite PCMs recently, but the preparation and properties of composite PCMs with titanium dioxide shells is little reported up to now.

In recent decades, many efforts have been done to develop PCM in buildings [30,31]. The aim of this work is to analyze the thermal

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**Table 1**  
The compositions of the SA/TiO<sub>2</sub> composites.

Samples	Compositions
CPCM1	40 g SA + 500 g deionized water + 200 g TiO <sub>2</sub>
CPCM2	40 g SA + 500 g deionized water + 160 g TiO <sub>2</sub>
CPCM3	40 g SA + 500 g deionized water + 120 g TiO <sub>2</sub>
CPCM4	40 g SA + 500 g deionized water + 80 g TiO <sub>2</sub>
CPCM5	40 g SA + 500 g deionized water + 40 g TiO <sub>2</sub>

properties of stearic acid/titanium dioxide composites as shape-stabilized phase change materials for building thermal energy storage.

In this paper, the preparation and thermal properties of the SA/TiO<sub>2</sub> composites as shape-stabilized phase change materials for building thermal energy storage are presented. In the composites, the SA is used as phase change material for thermal energy storage, and the TiO<sub>2</sub> acts as supporting material. The satisfactory composite with 33% mass ratio of the SA melts at 53.84 °C with a latent heat of 47.82 kJ/kg and solidifies at 53.31 °C with a latent heat of 46.60 kJ/kg. The composites have a good thermal stability, and can be used as shape-stabilized phase change materials for building thermal energy storage.

## 2. Experiments

### 2.1. Materials

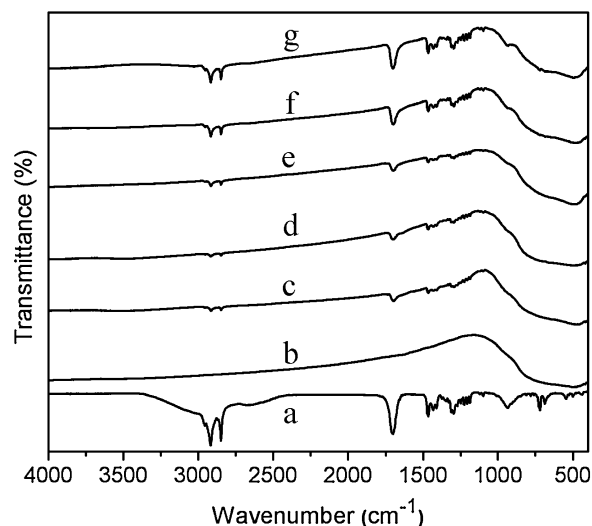
Stearic acid (Reagent grade, Jiangsu Huakang Chemical Reagent Company) was used as thermal storage material. Titanium dioxide (Reagent grade, Jiangsu Yonghua Chemical Reagent Company) acted as the supporting material and deionized water was used as solvent.

### 2.2. Preparation of the SA/TiO<sub>2</sub> composites

The SA and deionized water were mixed together in a beaker with different mass ratios, as showed in Table 1. The experimental parameters used for this preparation process were determined according to preliminary experimental results and our previous research [34]. The mixture was mixed uniformly by stirring at 75 °C for 30 min with a magnetic stirrer in order to form an oil/water (O/W) emulsion. Then titania powder was added into the emulsion while the stirring was kept until the emulsion became viscous. Finally, the composites were dried at 48 °C for 24 h in vacuum oven. Five kinds of the SA/TiO<sub>2</sub> composites were acquired, and were named as CPCM1, CPCM2, CPCM3, CPCM4 and CPCM5.

### 2.3. Characterization of the SA/TiO<sub>2</sub> composites

The chemical structure analysis of the SA/TiO<sub>2</sub> composites was conducted on a Fourier transformation infrared spectroscope (FT-IR, Nicolet Nexus 870, spectra from 400 to 4000 cm<sup>-1</sup> with a resolution of 2 cm<sup>-1</sup> using KBr pellets). The crystalloid phase of the CPCM5 was measured by an X-ray diffractometer (XRD, D/MAX-Ultima III, Rigaku Corporation, Japan) with continuous scanning mode at a rate of 5° (2θ)/min and operating conditions of 40 kV and 40 mA. The morphology and microstructure of the SA/TiO<sub>2</sub> composites were measured by a scanning electron microscope (SEM, S-3400NII, Hitachi Inc., Japan) at room temperature condition. Before the SEM test, the composites were dried in vacuum oven. During the SEM test, the sample space was vacuumized. Then, the morphology and microstructure of the composites could be observed. The thermal properties of the SA/TiO<sub>2</sub> composites was determined by differential scanning calorimeter (DSC, Pyris 1 DSC, Perkin-Elmer) under a constant



**Fig. 1.** FT-IR spectra of the (a) SA, (b) TiO<sub>2</sub>, (c) CPCM1, (d) CPCM2, (e) CPCM3, (f) CPCM4 and (g) CPCM5.

stream of argon. The accuracy of temperature measurements was ±0.2 °C and the enthalpy accuracy was ±5%. Indium was used as standard for temperature calibration. The samples were put in aluminium pans that were hermetically sealed before being placed on the calorimeter thermocouples. The sample space was cooled by a two-stage compression refrigeration system. The cooling rate was 5 °C/min from 90 °C to 10 °C, the heating rate was 5 °C/min from 10 °C to 90 °C. At first, the DSC cell containing a CPCM sample was cooled to a lower temperature than the melting temperature of the sample. The heating block was heated at a constant rate, the temperature of the reference sample pan also increased at a constant rate. If there was no phase change in the CPCM sample pan, the temperature difference between the CPCM sample and the reference sample pan produced an almost horizontal straight line. If there was a phase change in the CPCM sample pan, the temperature difference between the two pans followed a curve that deviated from the straight line. In DSC test, the melting and solidifying temperatures and their corresponding latent heats of the CPCM5 are not affected by heating and cooling rate. The heating and cooling rate with 10 °C/min or 20 °C/min is usually used. Therefore, the heating and cooling rate with 5 °C/min is appropriate in this experiment, which can ensure the accuracy of measurement. Lower heating and cooling rate such as for 2 °C/min or 0.5 °C/min may improve DSC resolution, but the testing time is longer. The thermal stability of the SA/TiO<sub>2</sub> composites was investigated by a thermogravimetric analyzer (Pyris 1 TGA, Perkin-Elmer) from 25 °C to 700 °C with a linear heating rate of 20 °C/min under a constant stream of nitrogen.

## 3. Results and discussion

### 3.1. FT-IR analysis of the SA/TiO<sub>2</sub> composites

Fig. 1 displays the FT-IR spectra of the SA, TiO<sub>2</sub>, CPCM1, CPCM2, CPCM3, CPCM4 and CPCM5. The spectrum of the SA is showed in Fig. 1a, and two strong absorption peaks at 2917 cm<sup>-1</sup> and 2849 cm<sup>-1</sup> are attributed to the asymmetric and symmetric stretching vibrations of its -CH<sub>2</sub> group. The stretching vibration of C=O group corresponds to the strong absorption peak at 1700 cm<sup>-1</sup>. The absorption peaks around 1471 cm<sup>-1</sup> presents the bending vibration of -CH<sub>3</sub> groups. The absorption peaks from 1400 cm<sup>-1</sup> to 1180 cm<sup>-1</sup> present a series of long-chain aliphatic characteristic peaks of the rocking vibration of -CH<sub>2</sub> group. In Fig. 1b, the

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