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Alkylated Phase Change Composites for Thermal Energy Storage Based on Surface-Modified Silica Aerogels

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Synthesis of Silica Aerogels and Phase Change Materials

The chemicals for this work are purchased from the following sources: hexamethyl disilazane (HMDZ), palmitic acid (PA) and octadecanol (OD) from Sinopharm Chemical Reagent Co., Ltd; tetraethoxysilane (TEOS) and n-hexane from Xilong Chemical Co. Ltd; ammonia water from Beijing Tongguang Fine Chemical Co. and anhydrous ethanol and oxalic acid from Beijing Chemical Factory. All the reagents are analytically pure and used without further purification.

The silica aerogels are prepared through the sol-gel method¹, in which 10 mL TEOS, 15.7 mL ethanol and 2 mL 0.1 mol/L oxalic acid solvent are stirred until the mixture was homogeneous, and followed by hydrolysis for 12 h. The sol transformed to alcogels after dispersing 5 drops of ammonia water and standing for 5 h. In order to prevent the inner pore structure from collapsing during the drying process, we used carbon dioxide supercritical dry (from Hua'an Science Instrument Co.) to produce the hydrophilic silica aerogels. The alcogels are aged at 50 °C for 24 h and the ethanol is exchanged to n-hexane. Then, the gel is immersed in HMDZ for 48 h for the surface modification process to make the silica aerogels hydrophobic. Finally, the product is washed by n-hexane and dried at 80 °C for 2 h to yield surface-modified silica aerogels, denoted as M-SiO₂ aerogels.

The phase change composites (PCCs) were fabricated by means of the vacuum impregnation method^{2,3}, where the SiO₂ aerogel and M-SiO₂ aerogel were infiltrated separately with PA and ODA in sealed containers. To fabricate the PCCs, the air in the aerogels was vacuumed out and the mixtures heated to above the melting points of the PCMs, after which, the porous aerogels were immersed into the melted PCMs. Then, air was allowed to enter the container to force the PCMs penetrate into the pore space of aerogels for a 4 h duration. The excess PCMs on the surface of the PCCs were removed prior to being used.

Further, the M-SiO₂ aerogels are carbonized at 900 °C for 5 h under an Ar protective atmosphere. The carbonized aerogels (C-SiO₂ aerogels) are also used to similarly encapsulate PA and OD by the vacuum impregnation method. The as-prepared composites are then pulverized in an agate mortar and compressed into the square block (2.5 cm×1 cm×0.3 cm) by a steel mold for the light-to-thermal conversion experiments.

Additional Characterization Results

The pore-size distribution are measured using a nitrogen gas adsorption method carried out using a Quantachrome Autosorb-iQ gas adsorption analyzer. The aerogels are treated at 200 °C for 5 h under vacuum with a heating rate of 10 °C/min before gas adsorption. The N₂ adsorption and desorption isotherms were collected at 77 K in a liquid nitrogen bath. The pore size distribution of the samples is calculated by the density functional theory (DFT) using 21 partial pressure points for both adsorption and desorption isotherms. X-ray diffraction (XRD) patterns are recorded on a Rigaku Dmax/2400 diffractometer using Cu K α radiation ($\lambda=1.5406\text{\AA}$) and operated at 40 kV and 100 mA. The XRD data is collected at a scanning rate of 4 °/min over an angular range of 3-60° (2 θ). Fourier transform infrared (FT-IR, ECTOR22, USA) spectrometer is used to analyze the structure between 400 cm⁻¹ and 4000 cm⁻¹ at 4 cm⁻¹ resolution in KBr pellets. Scanning electron microscope (SEM) characterization for the microstructure is carried out using a field-emission microscope (Hitachi-S4800) operating at 10 kV.

Differential scanning calorimetry (DSC, Setaram DSC 131 evo) and thermo-gravimetric analysis (TGA, Q600 SDT TGA-DTA-DSC thermal analyzer) are used to determine the thermal properties. For DSC measurements, the sample weights used vary from 1-5 mg and the temperature change rate is 5 °C/min from 0-100 °C under an Ar atmosphere. For TGA measurements, the sample weights are from 1-5 mg and the heating rate was 10 °C/min from room temperature to 400 °C under a protective N₂ atmosphere.

Light-to-heat conversion is carried out under simulated sunlight provided by a solar simulator (Neuport Thermo Oriel 91195A-1000). The samples are placed in a plastic box and irradiated under the stimulant sunlight. The temperature change of a sample is recorded via a thermal sensor (ST-50, RKC, Japan) attached to its surface and read by a thermometer (DP-700B, RKC, Japan).

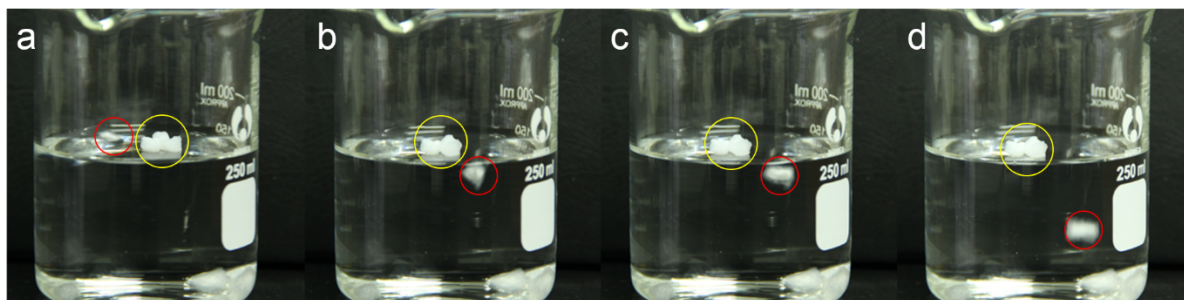


Fig. S1 The hydrophobicity of the alkylated aerogel. Red circles showing that the hydrophilic aerogel suck water into its pores, while red circles illustrated that the hydrophobic aerogel floating on the water.

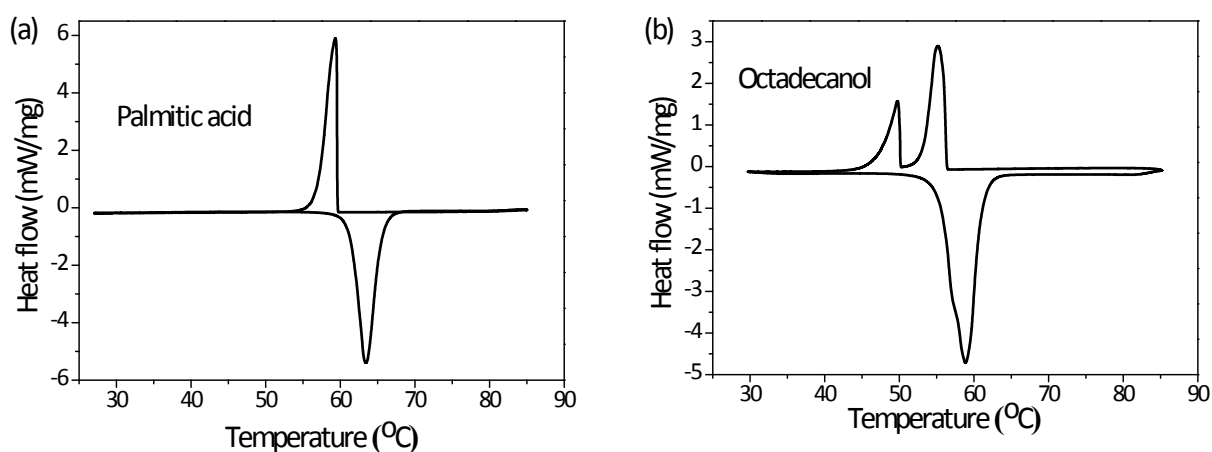


Fig. S2 DSC curves of pure palmitic acid and octadecanol. (a) DSC curve of pure palmitic acid. (b) DSC curve of pure octadecanol.

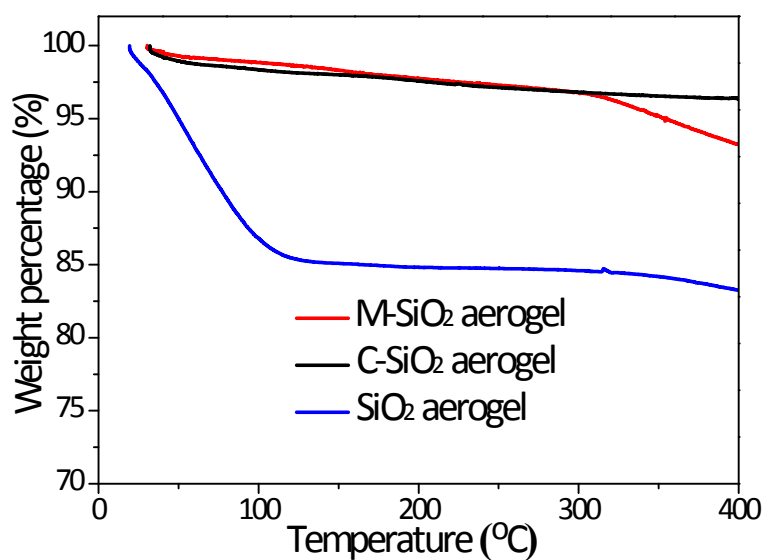


Fig. S3 TG curves of M-SiO₂ aerogel, SiO₂ aerogel, and C-SiO₂ aerogel.

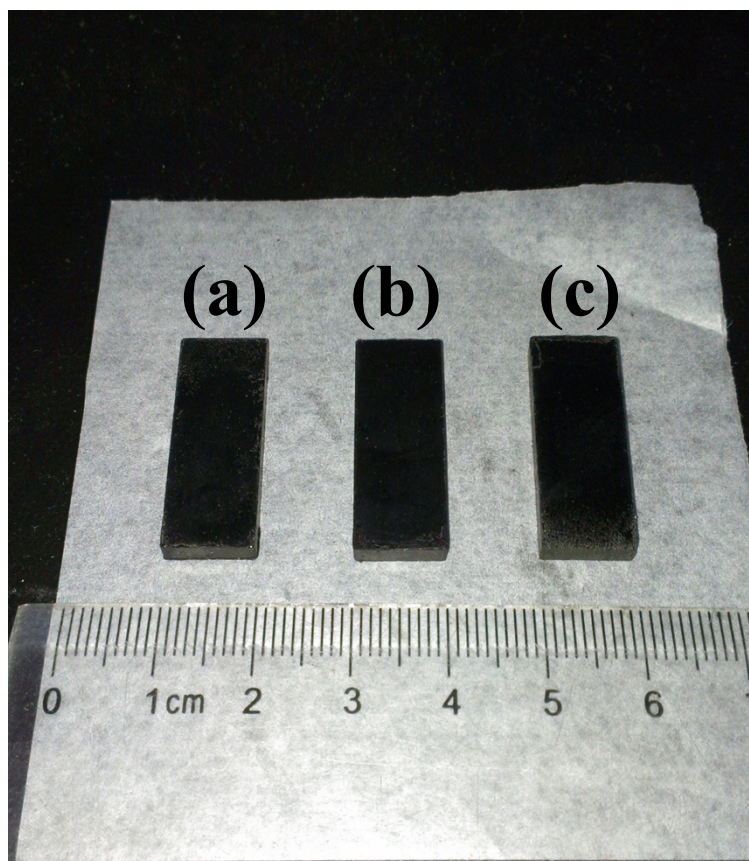


Fig. S4 Pictures of C-SiO₂ aerogel phase change composites for light-to-heat conversion. (a) and (b) are C-SiO₂ aerogel-PA composite. (c) C-SiO₂ aerogel-ODA composite

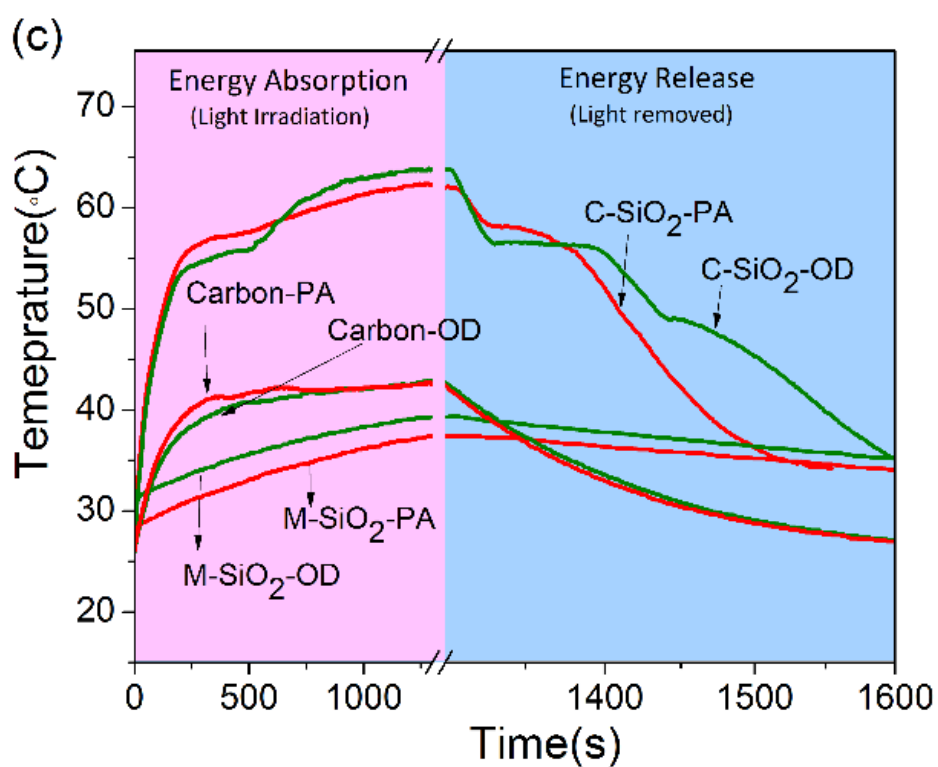


Fig. S5 Thermal energy storage and release curve

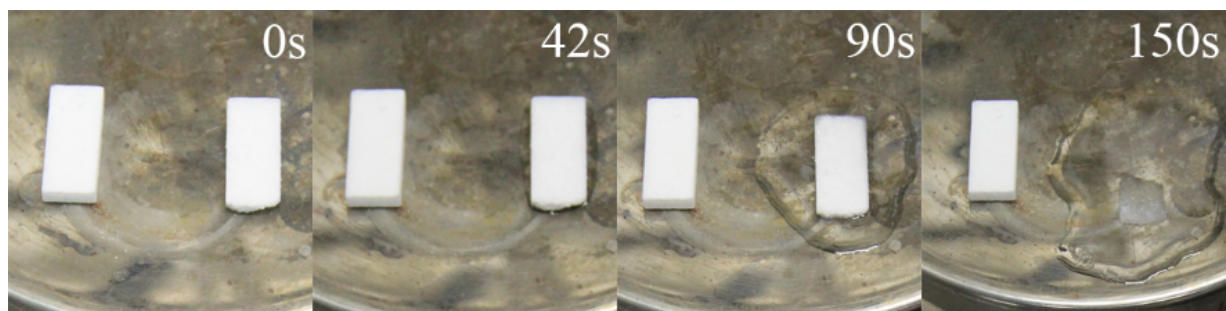


Fig. S6 Pictures of heating the composite and pure PCM with a hot plate.

Additional References

- 1 J. L. Gurav, D. Y. Nadargi and A. V. Rao, *Appl. Surf. Sci.*, 2008, **255**, 3019-3027.
- 2 D. Zhang, J. Zhou, K. Wu and Z. Li, *Sol. Energy*, 2005, **78**, 471-480.
- 3 D. Zhang, S. Tian and D. Xiao, *Sol. Energy*, 2007, 81, 653-660.