Preparation of Cetyl Alcohol-Palmitic Acid-Lauric Acid/SiO_2 Composite Phase-Change Humidity-Control Material Postprint

Authors: Zhang Hao, Huang Xinjie, Liu Xiuyu

Date: 2023-03-18T00:00:00+00:00

Abstract

A cetyl alcohol-palmitic acid-lauric acid/SiO_2 composite phase-change humidity-control material was prepared using SiO_2 as the carrier and cetyl alcohol-palmitic acid-lauric acid as the phase change material, and characterized by isothermal moisture adsorption-desorption method, cooling curve method, Fourier transform infrared spectroscopy (FT-IR), scanning electron microscopy (SEM), laser particle size analysis (LPSA), thermogravimetric analysis (TG), and differential scanning calorimetry (DSC). The results show that SiO_2 has a good encapsulation effect on cetyl alcohol-palmitic acid-lauric acid, and the resulting cetyl alcohol-palmitic acid-lauric acid/SiO_2 composite phase-change humidity-control material not only exhibits good temperature control performance, but also possesses excellent humidity control performance.

Full Text

Chinese Journal of Materials Research, Vol. 29, No. 11, November 2015

Preparation of Hexadecanol-Palmitic Acid-Lauric Acid/SiO_2 Composite as Phase Change Material for Humidity Control

ZHANG Hao, HUANG Xinjie, LIU Xiuyu
School of Civil Engineering and Architecture, Anhui University of Technology, Ma' anshan 243032, China

Supported by National Natural Science Foundation of China No. 51206002.

ABSTRACT

A hexadecanol-palmitic acid-lauric acid/SiO_2 composite phase change material for humidity control was prepared using SiO_2 as a carrier and hexadecanol-palmitic acid-lauric acid as the phase change material. The performance, molecular structure, and morphology of the prepared composite were characterized by isothermal sorption, cooling curve analysis, FT-IR, SEM, LPSA, TG, DSC, and other methods. The results demonstrate that SiO_2 effectively encapsulated the hexadecanol-palmitic acid-lauric acid, and the resulting composite exhibited excellent performance in both temperature and humidity control.

KEY WORDS
composite, hexadecanol-palmitic acid-lauric acid, SiO_2, humidity controlling performance, temperature controlling performance

Low-cost porous inorganic nano-SiO_2 exhibits nanoscale effects, small average particle size, large specific surface area, high surface energy, and a three-dimensional network structure. Using it as a carrier [1] to composite with organic acids having suitable phase change temperatures for building applications can effectively immobilize fatty acids [2], which improves both the thermal stability of the phase change material and its compatibility with inorganic building materials. Current research has primarily focused on binary organic phase change materials composited with low-cost porous inorganic materials (SiO_2) [3-5], while studies on ternary organic phase change materials composited with SiO_2 remain relatively scarce [6]. Because little attention has been paid to the potential humidity control performance of the network pore structure of porous inorganic materials (SiO_2), the resulting SiO_2-based composite phase change materials can only improve indoor thermal comfort rather than humidity comfort. This paper employs SiO_2 as the carrier material and hexadecanol-palmitic acid-lauric acid as the phase change material to prepare hexadecanol-palmitic acid-lauric acid/SiO_2 composite phase change and humidity control materials using the sol-gel method [7]. FT-IR, SEM, LPSA, TG, and DSC were used to characterize the composition, structure, microstructure, particle size distribution, thermal stability, and thermal properties of the composite. The humidity control and temperature control performance were investigated through isothermal sorption and cooling curve methods.

1 Experimental Methods

Raw Materials: The main raw materials included tetraethyl orthosilicate ($Si(OC_2H_5)_4$), anhydrous ethanol (CH_3CH_2OH, analytical grade), hexadecanol ($C_{16}H_{34}O$, analytical grade), palmitic acid ($C_{16}H_{32}O_2$, analytical grade), lauric acid ($C_{12}H_{24}O_2$, analytical grade), hydrochloric acid (HCl, analytical grade), ammonia solution ($NH_3 \cdot H_2O$, analytical grade), and deionized water for all experiments.

Preparation Process: Hexadecanol, palmitic acid, and lauric acid were mixed at a mass ratio of 30%:20%:50% in a beaker and dissolved at 60°C under constant stirring for 2 hours to obtain a homogeneous mixture. For the SiO_2 sol, 0.15 mol of tetraethyl orthosilicate was combined with 0.783 mol of anhydrous ethanol and 1.3545 mol of deionized water in a beaker, then stirred at medium speed at 60°C for 10 minutes using a constant temperature magnetic stirrer. The mixture was dispersed in an ultrasonic cell disruptor for 15 minutes, after which the pH was adjusted to 2.68 using hydrochloric acid and ammonia solution. The solution was then ultrasonically dispersed for an additional 15 minutes to obtain the SiO_2 sol.

Subsequently, 0.0765 mol of the hexadecanol-palmitic acid-lauric acid mixture was added to the SiO_2 sol and stirred at high speed at 60°C for 15 minutes, followed by ultrasonic dispersion for 45 minutes to ensure uniform distribution and embedding into the SiO_2 carrier. The resulting hydrosol was aged at 60°C for 2 hours to form a gel, which was then dried at 80°C for 8 hours to obtain the final hexadecanol-palmitic acid-lauric acid/SiO_2 composite phase change and humidity control material.

Performance Testing: Humidity control performance was evaluated using the isothermal sorption method [8-9] with a high-precision analytical balance (0.1 mg accuracy). Samples of 5 g were dried in an oven until three consecutive measurements at 24-hour intervals showed a mass difference of less than 0.1%, indicating complete dryness. The dried samples were placed above saturated salt solutions in desiccators at specific relative humidity levels. When three consecutive measurements at 24-hour intervals showed a mass difference of less than 0.1%, the sample was considered to have reached moisture absorption equilibrium. The samples were then moved to another relative humidity environment, and this process was repeated across seven humidity conditions (Table 1) ranging from 32.78% to 97.30% RH. Desorption tests followed the same procedure. The equilibrium moisture content (g/g) was calculated as $(m - m_0)/m_0$, where m_0 is the mass of the dry sample and m is the mass after moisture sorption/desorption.

Temperature control performance was evaluated using the cooling curve method [10-12] with a thermocouple. A 2.5 g sample was placed in a test tube with the thermocouple probe immersed in the sample. The tube was placed in a 40°C water bath until the sample temperature reached 35°C, then transferred to a 10°C water bath to cool to 15°C. Temperature readings were recorded every 5 seconds during the cooling process from 35°C to 15°C, and the cooling curve was plotted. The time required for this temperature drop was used to evaluate temperature control performance.

Characterization: The composition and structure were analyzed using a BRUKER VECTOR22 FT-IR spectrometer. Microstructure was examined using a JSM-6510LV scanning electron microscope (SEM). Particle size distribution was measured using a NANOPHOX laser particle size analyzer (LPSA). Thermal stability was evaluated using a TA Instruments Q600 thermogravimetric analyzer (TG). Thermal properties were analyzed using a TA 2910 differential scanning calorimeter (DSC).

2.1 FT-IR Analysis of Hexadecanol-Palmitic Acid-Lauric Acid/SiO_2 Composite

Figure 1a [Figure 1: see original paper] shows the FT-IR spectrum of SiO_2, revealing characteristic peaks at 1056.47 cm^{-1} (asymmetric stretching of cyclic Si-O-Si), 792.75 cm^{-1} (symmetric stretching of Si-O-Si), and 933.75 cm^{-1} (bending vibration of Si-OH). Figure 1b presents the spectrum of hexadecanol-palmitic acid-lauric acid, showing peaks at 2917.05 and 2849.35 cm^{-1} (C-H stretching from $-CH_3$ and $-CH_2$ groups), 1464.88 and 939.35 cm^{-1} (O-H in-plane and out-of-plane bending), and 1706.44 cm^{-1} (C=O stretching). Figure 1c displays the spectrum of the composite material, which exhibits absorption peaks from both SiO_2 and the organic phase change material at 2917.34, 2849.75, 1710.27, 1466.16, 1057.93, 938.40, and 800.03 cm^{-1}, with only slight shifts and intensity changes. This indicates that no significant chemical reaction occurred between hexadecanol-palmitic acid-lauric acid and SiO_2; the components are physically embedded rather than chemically bonded.

2.2 Microstructure of Hexadecanol-Palmitic Acid-Lauric Acid/SiO_2 Composite

Figure 2a [Figure 2: see original paper] shows the SEM image of SiO_2, revealing a sponge-like structure with particles aggregated and connected by short necks to form a three-dimensional network with numerous well-defined, small pores. Figure 2b presents the SEM image of the composite material, which appears as irregular particles resembling multiple small spheres strung together, with some degree of agglomeration. This morphology demonstrates that hexadecanol-palmitic acid-lauric acid effectively intercalated into the SiO_2 pores through the sol-gel process, achieving good encapsulation and shape stabilization.

2.3 Particle Size Distribution

Figure 3a [Figure 3: see original paper] shows the LPSA results for SiO_2, with d_{10} = 224.16 nm, d_{50} = 382.31 nm, and d_{90} = 698.64 nm, consistent with the SEM observations (Figure 2a). This good dispersion is attributed to the strong polarity of both deionized water (the dispersion medium) and SiO_2, following the principle of "like dissolves like." Figure 3b presents the LPSA results for the composite material, showing d_{10} = 1551.88 nm, d_{50} = 2570.29 nm, and d_{90} = 4612.13 nm, which aligns with the composite' s microstructure shown in Figure 2b. The good dispersion in deionized water further confirms the effective encapsulation of hexadecanol-palmitic acid-lauric acid by SiO_2.

2.4 Thermal Stability

Figure 4a [Figure 4: see original paper] shows the TG curve for SiO_2, which exhibits only 10.50% weight loss at 700°C due to evaporation of adsorbed water and residual organic groups. Figure 4b presents the TG curve for hexadecanol-palmitic acid-lauric acid, showing a major weight loss between 150-400°C, with over 95% weight loss by 400°C. Figure 4c displays the TG curve for the composite material, which begins to lose weight at approximately 120°C, with the primary weight loss occurring between 120-400°C due to volatilization of the organic phase change material. The total weight loss at 700°C is 64.70%, indicating that the mass fraction of hexadecanol-palmitic acid-lauric acid in the composite is approximately 54.20%. The composite shows no decomposition or weight loss below 120°C, demonstrating good thermal stability suitable for building applications.

2.5 Thermal Properties

Figure 5a [Figure 5: see original paper] shows the DSC results for hexadecanol-palmitic acid-lauric acid, revealing a phase transition temperature of 28.18°C and enthalpy of 173.36 J/g. Figure 5b presents the DSC results for the composite material, showing a phase transition temperature of 27.05°C and enthalpy of 91.35 J/g. The small temperature difference of 1.13°C indicates that SiO_2 as an encapsulating material has minimal effect on the phase transition temperature, which is primarily determined by the properties of hexadecanol-palmitic acid-lauric acid. Based on the enthalpy values, the mass fraction of hexadecanol-palmitic acid-lauric acid in the composite is calculated to be approximately 52.69%, which is consistent with the value of 54.20% estimated from TG analysis.

2.6 Humidity and Temperature Control Performance

Figure 6a [Figure 6: see original paper] shows the humidity control performance of SiO_2. At 97.30% RH, the equilibrium moisture content reaches 0.1786-0.1808 g/g, while in the comfortable humidity range of 40-65% RH, the adsorption equilibrium moisture content is 0.0608-0.0977 g/g and the desorption equilibrium moisture content is 0.0692-0.1108 g/g. This demonstrates good humidity control performance due to SiO_2' s large pore volume and specific surface area, which facilitate water molecule adsorption. Figure 6b presents the humidity control performance of the composite material, showing an equilibrium moisture content of 0.1755-0.1763 g/g at 97.30% RH, with adsorption and desorption equilibrium moisture contents of 0.0883-0.1045 g/g and 0.0990-0.1153 g/g, respectively, in the 40-65% RH range. The composite exhibits similar humidity control performance to pure SiO_2, further confirming the effective encapsulation and indicating that the humidity control properties are primarily determined by the SiO_2 carrier.

Figure 7a [Figure 7: see original paper] shows the temperature control performance of SiO_2, with the cooling curve displaying linear behavior, confirming that SiO_2 has no latent heat capacity. Figure 7b presents the temperature control performance of the composite material, which shows a distinct phase transition plateau between 24-28°C with a relatively long duration, demonstrating effective temperature control capability.

Conclusions

1. The hexadecanol-palmitic acid-lauric acid/SiO_2 composite phase change and humidity control material prepared by the sol-gel method using SiO_2 as the carrier and hexadecanol-palmitic acid-lauric acid as the phase change material exhibits good temperature control performance with a phase transition temperature of 27.05°C and enthalpy of 91.35 J/g, as well as excellent humidity control properties.
2. FT-IR analysis revealed no significant chemical interaction between hexadecanol-palmitic acid-lauric acid and SiO_2 in the composite, indicating only physical embedding. SEM observations showed that the composite consists of irregular particles with some agglomeration, and that the phase change material was effectively encapsulated within the SiO_2 matrix, achieving good shape stabilization.
3. Using deionized water as the dispersion medium, the composite material demonstrated good dispersibility. The primary cause of weight loss in the composite is volatilization of the organic phase change material, from which the mass fraction of hexadecanol-palmitic acid-lauric acid was estimated to be approximately 54.20%.

References

1. F. He, X. D. Wang, D. Z. Wu, "New approach for sol-gel synthesis of microencapsulated n-octadecane phase change material with silica wall using sodium silicate precursor," *Energy*, 67, 223 (2014).
2. A. Sharma, V. V. Tyagi, C. R. Chen, "Review on thermal energy storage with phase change materials and applications," *Renewable and Sustainable Energy Reviews*, 13(2), 318 (2009).
3. ZHANG Hong, WU Xiaohua, WANG Xiaolei, WANG Qianqian, "Structure and properties of lauric acid/cetyl alcohol/silicon dioxide composite phase change material," *Journal of Materials Science & Engineering*, 28(5), 672 (2010).
4. B. X. Li, T. X. Liu, L. Y. Hu, L. N. Gao, "Fabrication and properties of microencapsulated paraffin@SiO_2 phase change composite for thermal energy storage," *Acs Sustainable Chemistry & Energy*, 1(3), 374 (2013).
5. FU Lujun, DONG Faqin, YANG Yushan, HE Ping, "Preparation and characterization of binary fatty acid/SiO_2 composite phase change energy storage materials," *Journal of Functional Materials*, 44(4), 1 (2013).
6. HUANG Zishuo, YU Hang, ZHANG Meiling, "Humidity-control materials and their humidity absorption and desorption rate variation," *Journal of*

Tongji University (Natural Science), 42(2), 310 (2014).

7. ZHANG Hao, HUANG Kai, HUANG Xinjie, “Study on degradation performance of Ce-TiO_2 photocatalyst coating on formaldehyde solution and its prediction model,” *Chinese Rare Earths*, 35(4), 18 (2014).
8. G. Y. Fang, Z. Chen, H. Li, “Synthesis and properties of microencapsulated paraffin composites with SiO_2 shell as thermal energy storage materials,” *Chemical Engineering Journal*, 163(1-2), 154 (2010).
9. LI Kuishan, ZHANG Xu, HAN Xing, ZHU Dongming, “Experimental research of isothermal sorption curve of building materials,” *Journal of Building Materials*, 12(1), 81 (2009).
10. SHANG Jianli, LI Qiaoming, WANG Zhengjun, “Preparation and thermal performance tests of microencapsulated gypsum-based phase change building material,” *Acta Energiae Solaris Sinica*, 33(12), 2140 (2012).
11. YU Yongsheng, JING Qiangshan, SONG Fangfang, “Study on ternary phase change system of H/PA/LA,” *Journal of Building Materials*, 16(1), 97 (2013).
12. SHANG Jianli, WANG Si, DONG Li, “Preparation and research of energy storage effect and humidity-control performance of PAR/POL/SOD composite-wall microencapsulated materials,” *Journal of Functional Materials*, 44(8), 1141 (2013).

Note: Figure translations are in progress. See original paper for figures.

Source: ChinaXiv —Machine translation. Verify with original.