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journal homepage: www.elsevier.com/locate/solmatPreparation and characterization of PEG/SiO₂ composites as shape-stabilized phase change materials for thermal energy storageJingruo Li^{a,b}, Lihong He^{a,b}, Tangzhi Liu^{a,b}, Xuejuan Cao^{a,b}, Hongzhou Zhu^{a,b,*}^a School of Civil Engineering and Architecture, Chongqing Jiaotong University, Chongqing 400074, PR China^b The National Joint Engineering Laboratories of Traffic Civil Materials, Chongqing Jiaotong University, Chongqing 400074, PR China

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ABSTRACT

A novel sol–gel method was adopted to prepare polyethylene glycol/silicon dioxide shape-stabilized phase change materials (PEG/SiO₂ ss-PCMs) with various PEG mass fractions. The gelatinization was carried out by adjusting temperature instead of adding coagulant. In PEG/SiO₂ composites, PEG acted as the phase change material and silica gel served as the supporting material to keep the stable shape of the composites during the phase transition. Various characterization techniques were employed to investigate the structures and properties of the composites. Results showed that the composites exhibited the stable core–shell structures by impregnating PEG into multi-mesoporous silica gel; they could remain in the solid form even if the temperature exceeded the melting point of PEG. It was physical adsorption between PEG and silica gel, and the crystal structure of PEG component was unaffected, so that PEG in the composites retained an excellent phase change performance. The enthalpies of the composites varied from 63.4 J/g to 128.4 J/g (PEG mass fractions: 50–80%), which was proportional to PEG content. The thermal conductivities were increased to 0.558 W m⁻¹ K⁻¹ by addition of graphite in mass fraction of 2.7%. Moreover, the composites presented excellent thermal stabilities and possessed a broad applicable temperature range, and they were suitable for thermal energy storage applications in building envelopes.

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1. Introduction

The use of phase change materials (PCMs) for storing and releasing large amounts of energy during phase change process is called latent heat thermal energy storage (LHTES), and PCMs are acknowledged to be critical for LHTES. Because of the demand in energy market and the development of new national and international policies, those various PCMs have a great development prospect. Owing to their high latent heat density, constant phase change temperature, small volume change, and no phase segregation [1–3], solid–liquid PCMs have become the most potential and practical materials. However, ease of leakage in the melting process as a drawback will reduce their energy storage efficiency and limit the applicability [4–6]. Nowadays, the shape-stabilized PCM composites which can maintain the solid form even when the temperature is higher than the melting point of PCMs, have become the focus of the studies [7–11]. This technique is one of the most effective ways to solve the leakage problems of PCMs.

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Recently, impregnating solid–liquid PCMs into porous carriers is becoming a promising encapsulation technique in the field of shape-stabilized PCM composites [12–14]. First, porous carriers with high porosity can reliably store large amounts of PCMs. Second, porous carriers can be easily divided into numerous stand-alone energy storage units, which also can improve the heat transfer efficiency of PCMs during phase change processes. Third, the tiny pores of porous carriers help to enhance the storage reliability of PCMs due to the sufficient capillary effect. Based on the diameter size of pores, the porous carriers are classified into three categories: macroporous carriers (i.e., > 50 nm), mesoporous carriers (i.e., 2–50 nm) and microporous carriers (i.e., < 2 nm). As a result of lack of sufficient capillary forces, macroporous carriers need additional encapsulation to prevent the leakage of PCMs, which leads to the increasing difficulty of encapsulation as well as the cost. For the high specific surface area and small pore space, microporous carriers cause the PCM chains not to aggregate and crystallize due to the limited movement of PCM molecules, and the phase change behaviors of PCMs which are confined into the tiny pores are different from the normal situation, such as the remarkably increase in phase change temperature [15,16]. Mehling et al. [17] pointed out composites could be formed by embedding PCMs into matrix materials. The order of magnitude of the composite structure should be microscopic. Otherwise, the properties of the composite would depend on the sample size, so the

composite could not be called a material anymore. Oh et al. [18] reported that the average pore diameter should be controlled to reduce the oozing rate of PCM out of carrier material so that the encapsulation efficiency is enhanced. Py et al. [19], Elgafy et al. [20] and Li et al. [21,22] reported that mesoporous materials were the promising candidates to absorb and confine PCMs with high latent heat capacity. Mesoporous materials were chosen as the supporting materials in the following research.

In the previous studies, we had already compared two sol–gel methods, coagulant-assisted sol–gel method and temperature-assisted sol–gel method. Both of them were used to prepare PEG/SiO₂ composites as shape-stabilized PCMs for thermal energy storage. It was concluded that the temperature-assisted sol–gel method was adopted for easy operation and low cost, and the as-prepared ss-PCMs had good thermal property and shape stability as well. In addition, ss-PCMs were potential candidates for thermal energy storage applications in building envelopes whose temperature is often as high as 60–70 °C in the continuous hot summer, since they had high enthalpies and proper phase change temperature.

During this research, we characterized the various properties of shape-stabilized PEG/SiO₂ composites prepared by temperature-assisted sol–gel method, such as composite structures, thermal properties and thermal stabilities, crystal structures and crystallization properties, and chemical compatibilities. They were investigated by Transmission polarization microscope, Simultaneous thermal analyzer (DSC/TG), X-ray powder diffractometer (XRD) and Fourier transformation infrared spectroscopy (FTIR), correspondingly. In addition, the pore structures of silica gel were detected by Surface area and porosimetry analyzer.

2. Experimental

2.1. Materials

For the preparation of shape-stabilized composites, PEG, as the latent heat storage material, was in commercial grade with an average molecular weight of 2000 and produced by Jiangsu Hai'an Petroleum Chemical Plant in China. PEG was desiccated with a molecular sieve drier for 48–72 h before use. Silica sol, as the silica source, was supplied from Zhejiang Yuda Chemical Co. Ltd. in China.

2.2. Preparation of PEG/SiO₂ composites

The temperature-assisted sol–gel method was adopted to prepare PEG/SiO₂ composites by impregnating PEG into silica-gel porous network structure. Firstly, PEG was dissolved into silica sol at room temperature in mass fractions of 30%, 40%, 50%, 60%, 70%, 80%, and 90%. Then these PEG/SiO₂ blended sols were heated by water bath with quick continuous stirring at 70 °C, 60 °C, 50 °C, 40 °C, 30 °C and room temperature, correspondingly. The gelatinization of PEG/SiO₂ blended sols occurred in 10 min or so. In the same time, PEG had been adsorbed and confined into the network of porous silica gel. Afterwards, PEG/SiO₂ shape-stabilized composites were obtained after a drying procedure in an oven at 80 °C for 12–24 h. Finally, PEG/SiO₂ composites were heated at a constant temperature above the melting point of PEG for 3 days. The composites without melted PEG leakage were considered as shape-stabilized composite PCMs. However, when the PEG mass fraction reached 90%, there was a slight leakage of melted PEG. Thus, we mainly discuss the composites with 30–80% of PEG in this paper.

2.3. Characterization of PEG/SiO₂ composites

The pore size of silica gel were determined by a V-Sorb 2800P Surface Area and porosimetry Analyzer manufactured by Gold APP Instrument Corporation in China. The composite structures of ss-PCMs were observed by a XP-213 transmission polarization microscope equipped with a heating stage and a digital camera, which was manufactured by Jiangnan Novel Optics Co., Ltd. in China.

The thermal properties and thermal stabilities of ss-PCMs were measured by a STA 449C Simultaneous thermal analyzer (DSC/TG) made by German Netzsch Company. The protective gas and purge gas were high purity nitrogen, and their flow velocities were 20 ml/min and 40 ml/min, respectively. DSC measurements were performed at 10 °C/min heating rate and 2 °C/min cooling rate with a temperature program of 20 °C→200 °C→20 °C→130 °C→20 °C→130 °C. Besides, in TG measurements, the heating rate was taken as 10 °C/min with temperature range from 25 °C to 600 °C.

The chemical compatibilities between PEG and silica gel were characterized by a PE 100 Fourier transform infrared spectroscopy (FTIR) which was manufactured by PerkinElmer Company in USA. Samples were ground sufficiently and mixed with potassium bromide. The mixtures were compacted to provide the smooth test surfaces and then pressed into pellets. FTIR spectra were obtained in the range of 400–4000 cm⁻¹ with 20 scans.

The crystal structures and crystallization properties of PEG in ss-PCMs were investigated by XRD-6000 model of X-ray powder diffractometer, which was manufactured by Shimadzu in Japan. It was operated at 40 kV and 30 mA with the Cu K α radiation ($\lambda=1.54$ Å, wavelength). The scanning angle 2θ , from 10° to 45°, was swept at a speed of 4°/min, and the scanning step size was 0.02°.

The thermal conductivities were measured using YBF-2 thermal conductivity coefficient analyzer manufactured by Hangzhou Dahua Apparatus Manufacture Co., Ltd. in China.

3. Results and discussion

3.1. Pore structures of silica gel and composite structures of PEG/SiO₂ composites

Table 1 illustrates the average pore size, BET surface area, and total pore volume of the silica gel. As Table 1 indicates, as a mesoporous material, silica gel possessed highly porous structure, and its pores were open to the surface. It could be used as supporting material to absorb and confine PCMs because of its high specific surface area and large pore volume. Fig. 1 presents the composite structures of ss-PCMs in the molten state. In this image, it can be clearly observed that a three-dimensional network structure was formed by the dark silica gel framework and the melted PEG had the same color as background. This image demonstrates a functional core–shell structure in which silica gel served as carrier material and PEG as latent heat storage material. In this composite structure, due to the unique network porosity and high surface affinity of silica gel, PEG was well impregnated and trapped into the pores of silica gel. After many heating and melting processes, there was no seepage of melted PEG, which suggested that silica gel played a very critical role in wall materials

Table 1
Pore features of silica gel.

Sample	D_{pore} (nm)	S_{BET} (m ² /g)	V_{pore} (cm ³ /g)
Silica gel	7.478	528.178	0.719

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