

Synthesis and Performance Analysis of a Novel Phase Change Hydrogel Polyethylene Glycol/Polyacrylamide/Silicon Dioxide for Thermal Energy Storage

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To study the composite phase change hydrogel, the shape stabilized composite phase change materials of polyethylene glycol/polyacrylamide/silicon dioxide with different mass fractions of polyethylene glycol were successfully prepared by the combination of in-situ polymerization and sol-gel method. The 3D network formed by polyacrylamide /silicon dioxide cross-linking has abundant pores, which is conducive to polyethylene glycol encapsulation and prevent polyethylene glycol leakage. At the same time, the hydrogel-based support can buffer the thermal stress caused by phase change. According to the analysis results of differential scanning calorimetry, composite phase change materials have a high phase transition enthalpy of 126.9 J/g. In addition, this new type of composite phase change materials has good thermal stability and remains stable in shape at 110 °C without any leakage. The composite phase change hydrogel provides an alternative approach for the investigation of phase change materials.

Keywords: composite phase change materials, nanocomposite hydrogels, PEG, cross-linked network, thermal properties.

1. INTRODUCTION

With the development of society and economy, due to the increasingly serious shortage of resources and the continuous increase of production and living, energy consumption, energy saving and emission reduction have received extensive attention [1–3]. Energy storage technology alleviates energy challenges by improving energy availability [4]. As we all know, phase change materials (PCMs) are excellent energy-saving materials with high energy storage density and a wide temperature range through latent heat energy storage [5–7]. Therefore, PCMs are widely used in temperature-regulated buildings, solar energy systems and other fields [8–10].

In general, among all PCMs, solid-liquid PCMs have the advantages of higher energy storage density, good thermal stability, safety, non-toxicity and higher application properties [11]. Among them, organic solid-liquid phase change materials have a more outstanding energy density, including paraffin, fatty acid, polyethylene glycol (PEG) and so on [12–14]. Costa et al. developed a novel composite PCMs with alginate-vermiculite minerals as the carrier and a mixture of paraffin and naphthenic oil as the working material. The optimized wax mixture has a solid-liquid temperature of 57.45 °C and a high enthalpy of 159.42 J/g. At the same time, the thermal properties of cement mortar were improved by wrapping vermiculite particles with diatomite filled with paraffin. The authors further studied PCMs with different volume ratios and evaluated the thermal properties of these materials [15]. Xu et al. have synthesized shape-stabilized phase change materials with a heat storage density of 131.2 J/g by binary eutectic of lauric and palmitic acids. Carbon nanofibers were also added to

enhance the thermo-conductivity [16]. Zhu et al. prepared a stereotyped PCMs by immobilizing PEG inside a natural rubber-expanded graphite skeleton by vacuum immersion. The PCMs was remarkably reusable and flexible, exhibiting a high latent heat of phase change of 144.11 J/g [17]. Currently, PEG has a wide range of applications because its phase transition temperature can be adjusted by its relative molecular mass and has also been widely studied for its high latent heat and bio-friendliness [18].

However, the leakage of working materials after phase transformation is the main factor limiting its application. To solve this problem, many researchers have provided different encapsulation strategies: core-shell (0D), longitudinal confinement (1D), interfacial (2D) and porous confinement (3D). Liu et al. successfully synthesized PCM nano-capsules composed of ZnO/SiO₂ and n-octadecane. The nanocapsules had a good core-shell structure with an enthalpy value of 95.0 J/g. ZnO also achieved better thermal conductivity in the modified shell material [19]. Fang et al. constructed a support network by ionic cross-linking between cellulose nanofibers and chitosan. Meanwhile, PEG was confined within the fiber network by hydrogen bonding to form a composite phase change fiber material. The addition of hydroxylated boron nitride enabled the composite to exhibit a high thermal conductivity of 4.005 W/m·K [20]. Zhang et al. synthesized graphene oxide (GO) composite paper in situ by embedding PEG molecules into GO sheets through hydrogen bonding, which had excellent thermo-storage and shape stability because the interfacial confinement was very powerful [21]. Although conventional encapsulation strategies can keep the macroscopic shape of PCMs stable, the low mechanical

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strength of the usual rigid encapsulation systems can't effectively deal with the thermal stresses caused by phase transitions, which can provoke rupture and collapse leading to leakage of working materials and reduce the energy storage. To promote its wide application, the research of composite phase change materials (CPCMs) with good toughness and high energy storage density has become the focus of research [22]. At present, many research groups have modified materials by constructing cross-linked structures in CPCMs. For example, Mu et al. constructed a structurally stable cross-linked network by using poly (acrylonitrile-co-itaconate) as a substrate and preparing new CPCMs by mixing it with PEG. The cross-linked network could not only load a large amount of PEG, but also prevented the leakage of PEG during the phase transition [23]. This was because the flexible network formed by cross-linking makes the support material had good toughness and fracture stress, which reduced the rigidity of the system and thus prevented it from fracture and collapse. At the same time, the porous structure formed by cross-linking could increase the load of PCMs and improve the energy storage density. Many investigations have demonstrated that nanocomposite hydrogels are a very prospective support material with excellent toughness and load performance, which is suitable for loading large amounts of PCMs [24–26].

In this research, polyethylene glycol (PEG)/polyacrylamide (PAM)/silicon dioxide (SiO_2) nanocomposite hydrogels CPCMs were successfully prepared by in situ doping and sol-gel process. The introduction of functionalized SiO_2 nanoparticles strengthened the PAM hydrogel network and adsorbed a large amount of PEG through physical interaction. The CPCMs had high enthalpy of phase transition (117.6 J/g for crystallization and 126.9 J/g for melting) and retained shape stability during the phase transition. This paper characterized the microstructure, shape stability, thermal stability and thermal storage properties of the prepared PEG/PAM/ SiO_2 CPCMs. The influence of the interaction between PEG and the network framework on the energy storage performance was also investigated. This paper provided a new method for the preparation of CPCMs with cross-linked network structure.

2. EXPERIMENTAL PROCEDURE

2.1. Materials

All experiments were of analytical grade and used without further purification. Acrylamide ($\text{C}_3\text{H}_5\text{NO}$, purity $\geq 99\%$) and Ammonium persulfate ($\text{H}_8\text{N}_2\text{O}_8\text{S}_2$, purity $\geq 98\%$) in powder form were purchased from Aladdin Co., Ltd. Polyethylene glycol ($M_n = 6000$) and liquid Vinyltriethoxysilane ($\text{C}_8\text{H}_{18}\text{O}_3\text{Si}$, purity $\geq 97\%$) was produced by Macklin Co., Ltd.

2.2. Preparation of silica nanoparticles

The transparent dispersions of vinyl hybrid silica nanoparticles (VSNPs) were obtained by adding 3.8 g Vinyltriethoxysilane (VTES) to 30 g deionized water under vigorous stirring and keeping the stirring for 12 h until all oil droplets disappeared.

2.3. Preparation of PEG/PAM/ SiO_2 composite PCMs

PEG/PAM/ SiO_2 CPCMs with different PEG loads were prepared by in situ doping and sol-gel methods. The synthesis procedure was as follows: 1) 3 g Acrylamide and 0.015 g dispersion of VSNPs was added to 12 g deionized water and stirred at room temperature for 30 min; 2) Heated the system to 60 °C and mixed PEG and stirred until PEG completely dissolved; 3) Add 0.02 g APS and stir for 30 min; 4) After magnetic stirring, free-radical polymerization proceeded at 55 °C for 30 h; 5) At last, the prepared samples were dried completely at room temperature to obtain PEG/PAM/ SiO_2 composite PCMs. The drying process was also the isolation and purification of products. The mass fraction of PEG in the CPCMs was calculated based on the total mass of the samples after drying. The samples with different weight of PEG were denominated as CPCM-X ($X = 1, 2, 3, 4$ and 5) as described in Table 1.

2.4. Material characterization

The chemical compatibility of PAM/ SiO_2 and CPCMs was analyzed by Fourier transform infrared spectroscopy (FTIR, 5700, Nicolet, USA). The wavenumber tested was in the range of 4000–400 cm^{-1} . The leakage properties were investigated by taking photographs of CPCMs on a heating platform with a digital camera and the observation condition was to heat the sample from 25 °C to 110 °C at a rate of 5 °C/min. The microscopic morphology of the surface layers and faults of CPCMs was studied by scanning electron microscopy (ESEM, Quanta 200FEG, FEI, USA) at an accelerating voltage of 20 kV. The thermal storage properties of CPCMs were analyzed by differential scanning calorimetry (DSC, 8000, PerkinElmer, USA). The samples were heated and cooled at a rate of 10 °C/min between 10 °C and 80 °C under a nitrogen atmosphere. A thermogravimetric analyzer (TGA, 4000, PerkinElmer, USA) was used to investigate the thermal stability of CPCMs. During the measurements, the samples were heated at a heating rate of 20 °C/min from 100 °C to 700 °C in a nitrogen atmosphere.

3. RESULTS AND DISCUSSION

3.1. Preparation principle of PEG/PAM/ SiO_2 CPCMs

VTES as a precursor was hydrolyzed to obtain vinyl hybrid silica nanoparticles (VSNPs) (Fig. 1). After the addition of acrylamide (AM) and ammonium persulfate (APS), polyacrylamide (PAM) polymerized from acrylamide monomer was connected with VSNPs through an addition reaction, which formed a PAM/ SiO_2 hydrogel network framework. The ability of pure PAM hydrogel to cope with thermal stress was relatively weak, so the insertion of silica nanoparticles provided reliable cross-linking sites for the PAM hydrogel network to cope with greater stresses and became an excellent support material [27, 28].

Table 1. The composition of CPCMs samples and critical parameters after the drying procedure

Samples	PEG, g	AM, g	H ₂ O, g	APS, g	VSNPs, g	Total weight after drying, g	PEG mass fractions, wt.%
PAM/SiO ₂	0	3	12	0.02	0.015	4.257	0
CPCM1	6.51	3	12	0.02	0.015	10.751	60.5
CPCM2	8.13	3	12	0.02	0.015	12.801	63.5
CPCM3	10.02	3	12	0.02	0.015	14.561	64.8
CPCM4	12.31	3	12	0.02	0.015	18.250	67.4
CPCM5	15.04	3	12	0.02	0.015	20.597	73.0

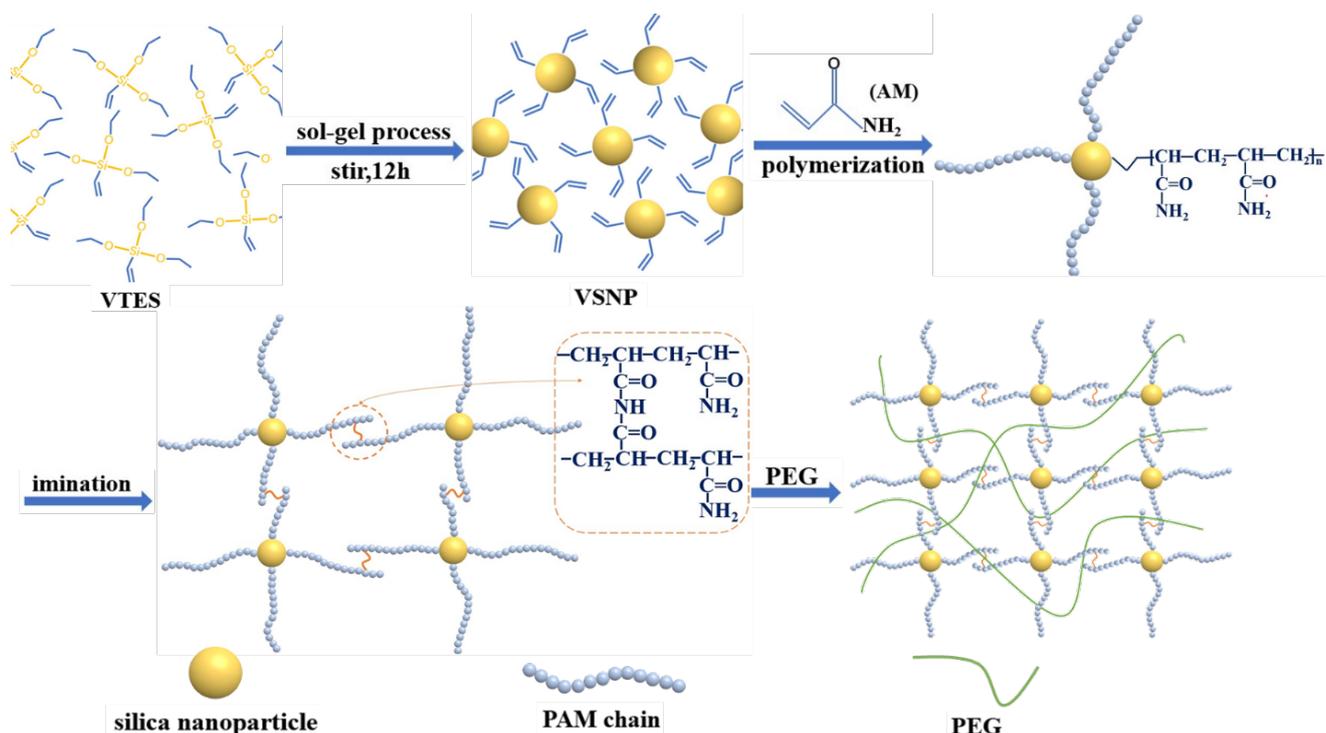


Fig. 1. Synthesis principles schematic of PEG/PAM/SiO₂ CPCMs

In addition, during the sol-gel process, the PAM/SiO₂ network immobilized the long-chain PEG within the system through physical interactions such as adsorption, chain entanglement and hydrogen bonding, which constituted a composite phase change hydrogel with good load performance.

3.2. Leakage test of PEG/PAM/SiO₂ CPCMs

PEG and as-prepared PEG/PAM/SiO₂ composite PCMs were placed on a heating platform and continuously heated in the range of 25 °C to 110 °C. The leakage properties and shape stabilization of the samples exhibited during the process were observed and recorded macroscopically with a digital camera (Fig. 2).

When the temperature reached 110 °C, CPCM1, CPCM2, CPCM3 and CPCM4 did not observe obvious leakage of PEG, while CPCM5 had slight leakage because the PEG content exceeded the adsorption capacity of the stent network, so CPCM4 was the best sample. Therefore, in the following research, CPCM4 was the key research object. All CPCMs maintained shape stability at 110 °C. The above observations indicated that the results of the

leakiness tests were promising and PEG/PAM/SiO₂ CPCMs had excellent and reliable shape stability during the phase transition.

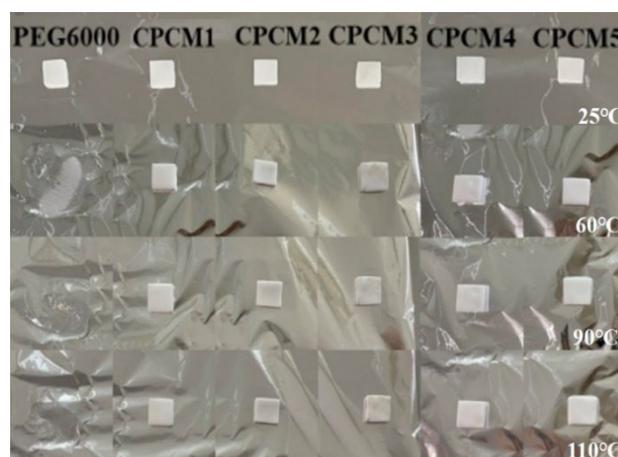


Fig. 2. Heating test of CPCMs with different masses of PEG

The good shape stability came from the excellent structure of the PAM/SiO₂ crosslinking network and the

multiple physical effects between the PEG long chain and support network. The introduction of SiO₂ nanoparticles not only strengthened the PAM gel network and made it more stable at high temperature, but also provided adsorption sites for PEG. PEG was effectively immobilized in PAM/SiO₂ support system through the chain entanglement between PEG long chain and crosslinking network, hydrogen bonding and physical adsorption with SiO₂ nanoparticles. Therefore, it was proved that PEG/PAM/SiO₂ CPCMs still had good shape stability at high temperature.

3.3. FT-IR spectra of PEG/PAM/SiO₂ CPCMs

The chemical compatibility of PEG, PAM/SiO₂, CPCM1, CPCM3, CPCM4, CPCM5 was analyzed by FT-IR. The results were shown in Fig. 3. -CH₃ (2933 cm⁻¹), -NH₂ (3359 cm⁻¹), C-N (1661 cm⁻¹), Si-CH₂- (1327 cm⁻¹), Si-O-Si (1211 cm⁻¹) was observed in the FT-IR spectrogram of PAM/SiO₂. No C=C absorption peak was observed in the 1600–1700 cm⁻¹ band, indicating that the polymerization of acrylamide monomer and the crosslinking between the long chain of polyacrylamide and VSNPs were accomplished [29, 30].

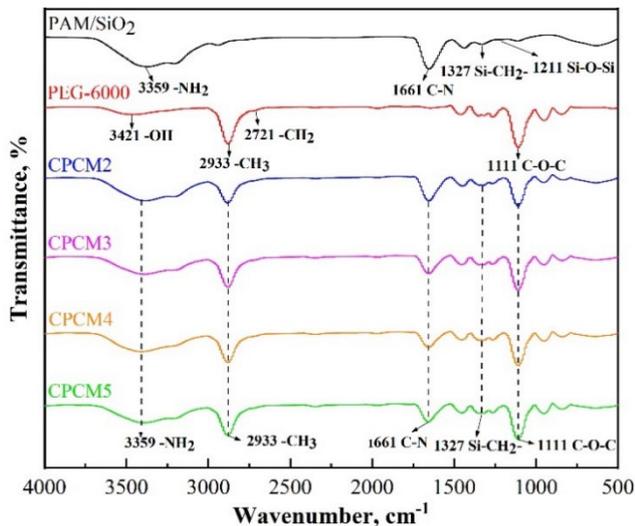


Fig. 3. FT-IR spectra of the PEG6000, PAM/SiO₂, and CPCMs

Meanwhile, as the characteristic peak of C-N was observed at 1661 cm⁻¹, it indicated that imide bonds had been generated and imide crosslinks had been formed between the long chains of PAM, suggesting that cross-linked network structure was synthesized in the PAM/SiO₂ hydrogel. The typical characteristic peaks of PEG such as -OH, -CH₂, C-O-C were observed at 3421 cm⁻¹, 2721 cm⁻¹,

1111 cm⁻¹, respectively. The FT-IR curves of PEG/PAM/SiO₂ composites also observed the characteristic band of C-N without the characteristic band of C=C, indicating that the cross-linked network was also successfully formed in the CPCMs. The CPCMs had the characteristic peaks of PEG and PAM/SiO₂ without generating new characteristic peaks, indicating that the PEG long chains and the PAM/SiO₂ network were combined by physical rather than chemical interaction. In addition, the characteristic peaks of -NH₂ and C=O functional groups at 3359 cm⁻¹ moved to lower wave numbers, which indicated the existence of hydrogen bonds between PEG and PAM/SiO₂ networks and once again proved that the PEG and PAM/SiO₂ networks were physically bonded to each other.

3.4. Morphology of PEG/PAM/SiO₂ CPCMs

The ESEM images of the surface and cross-section of CPCM4 were shown in Fig. 4. As shown in. The network structure of the composite phase change materials was studied by SEM. Continuous 3D network structure and interconnected pore structure were successfully observed from the surface morphology of PEG/PAM/SiO₂ CPCMs. CPCM5 (Fig. 4 c and d) had fewer pores than CPCM4 (Fig. 4 a and b) because it loads more PEG so it had less bearing space for PEG volume change during phase transition, which was one of the reasons why CPCM5 had slight leakage. Fig. 4 e, f and g, h showed the transverse section and longitudinal section of CPCM4 respectively, showing the transverse network like pores and the longitudinal layered structure, and more comprehensively showing the internal morphology of the composite phase change material. It can be seen that the long chain of PEG was interspersed and dispersed in the PAM/SiO₂ network and microlayer, and the physical effects such as chain entanglement prevented the leakage of PEG. At the same time, CPCMs still had abundant pores, so they could cope with the volume change of PEG before and after phase transformation, and maintain good load performance.

3.5. Thermal storage performance of CPCMs

The thermal storage performance of PEG6000 and CPCMs with different PEG masses were shown in Fig. 5. The endothermic and exothermic temperatures and enthalpies of CPCMs derived from the DSC plots were also listed in Table 2. Through the two-point correction method, the baseline optimization, sample temperature correction, furnace temperature correction and energy correction were carried out for the equipment before use.

Table 2. Thermal storage parameters of the CPCMs and PEG6000

Samples	Endotherm			Exotherm		
	Onset temperature, °C	Peak temperature, °C	Latent heat, J/g	Onset temperature, °C	Peak temperature, °C	Latent heat, J/g
PEG-6000	56.6	63.6	181.7	44.5	40.1	175.3
CPCM1	42.4	53.2	84.3	42.3	30.7	82.4
CPCM2	43.0	53.8	99.0	43.3	39.5	91.5
CPCM3	54.6	63.7	118.7	42.7	37.5	111.9
CPCM4	55.8	62.7	126.9	43.5	40.1	117.6
CPCM5	54.4	62.3	136.2	43.6	39.8	127.3

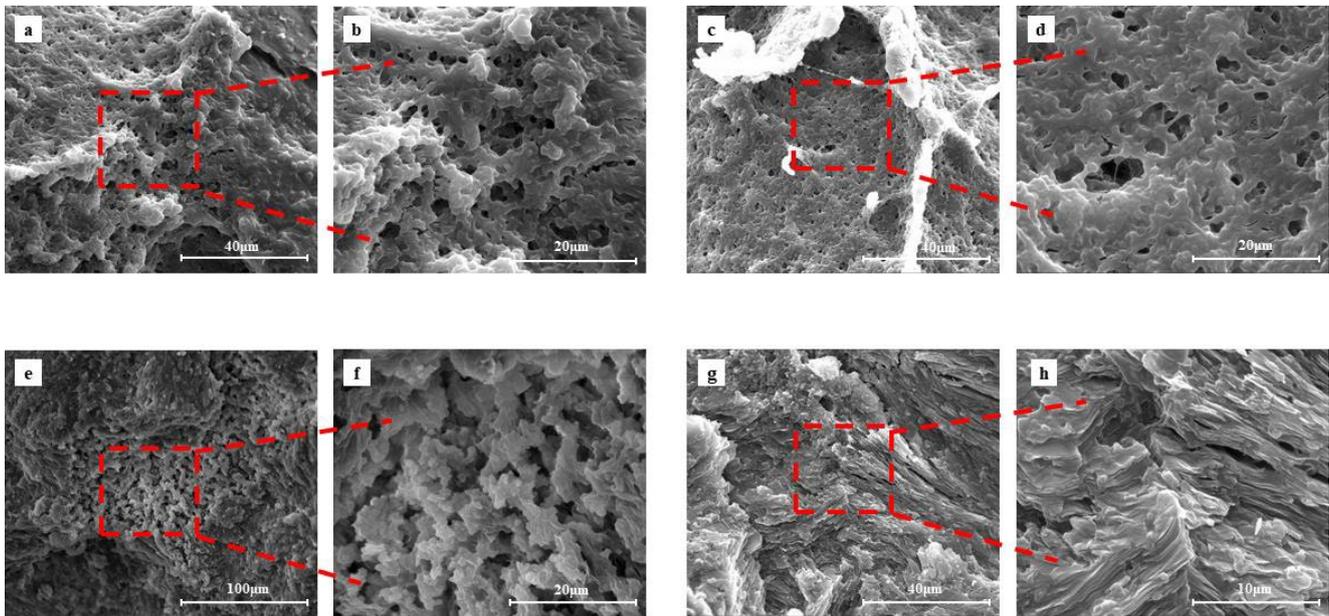


Fig. 4. SEM images: a, b –CPCM4; c, d–CPCM5; e, f– transverse section of CPCM4; g, h–longitudinal section of CPCM4

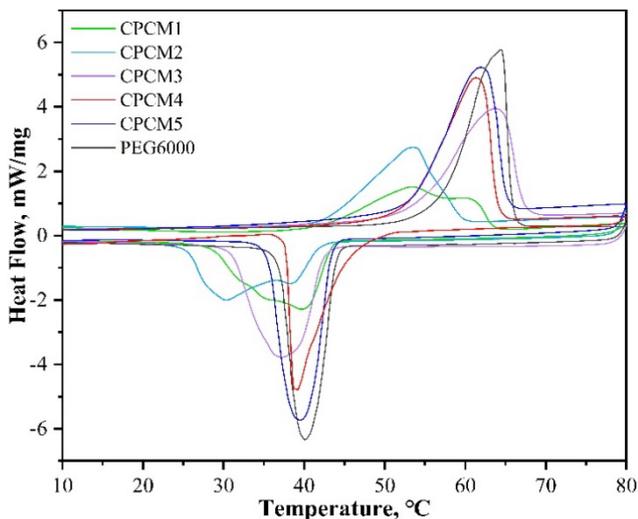


Fig. 5. DSC curves of CPCMs and PEG6000

As can be seen from the figure, the DSC curves of PEG and CPCMs both had endothermic and exothermic peaks indicating that they had similar thermal storage performance. Pure PEG6000 started to melt for endotherm and solidify for exotherm at 56.6 °C and 44.5 °C respectively with enthalpies of melting and solidification of 181.7 J/g and 175.3 J/g. However, compared with pure PEG6000, the temperature curves of CPCMs moved to low temperature in varying degrees. The phase transition temperature varied from 42.4 °C to 55.8 °C with the mass fraction of PEG. The energy storage densities of CPCM1, CPCM2, CPCM3, CPCM4, and CPCM5 had reached respectively 91.5 J/g, 84.3 J/g, 118.7 J/g, 126.9 J/g, and 136.2 J/g. The results showed that the introduction of PEG significantly improved the energy storage density of the composites. Because PEG was the main substance for energy storage during the melting or crystallization of PEG/PAM/SiO₂ CPCMs. Meanwhile, the phase transition temperature and enthalpy of CPCMs were almost

proportional to the PEG content. However, the enthalpy of melting and enthalpy of crystallization of CPCMs were both lower than that of PEG6000.

Because the PAM/SiO₂ network restricted the crystallization of PEG chain segments and the mass percentage of PEG in the composites was much lower. In conclusion, it was the melting and solidification of PEG that enabled the CPCMs to have heat storage capacity. Therefore, the changes in phase transition temperature and enthalpy might be associated with the crystallinity of PEG. While the weak physical interaction between the PAM/SiO₂ network was affecting the crystallinity of PEG. Surprisingly, CPCMs had low undercooling and high enthalpy of phase transition, which might be due to the weak physical interaction of PAM/SiO₂ network on the PEG long chain, which limited the movement of its chain segments to a lesser extent, to better retain the crystallization behavior of PEG. In similar studies, the enthalpy of CPCMs was only 119.6 J/g with 70 wt.% of PEG, which was lower than the result in this study [30].

The decomposition temperature thermostability of PEG6000 and CPCMs was investigated by the TGA characterization. The results of TGA analysis were presented in Fig. 6 and Table 3.

Table 3. Thermal stability parameters of the CPCMs and PEG6000

Samples	5 wt.% weight loss temperature, °C	Maximum degradation temperature, °C	Residual mass, wt.%
PEG-6000	322.8	443.7	2.9
PAM/SiO ₂	144.9	484.6	18.9
CPCM3	196.4	446.4	10.5
CPCM4	239.2	448.1	9.1
CPCM5	283.1	450.9	8.1

Thermal stability was a very important performance indicator for the application of CPCMs in thermal energy storage. The 5 wt.% weight loss temperature and the

maximum degradation temperature of PEG were 322.8 °C and 443.7 °C respectively.

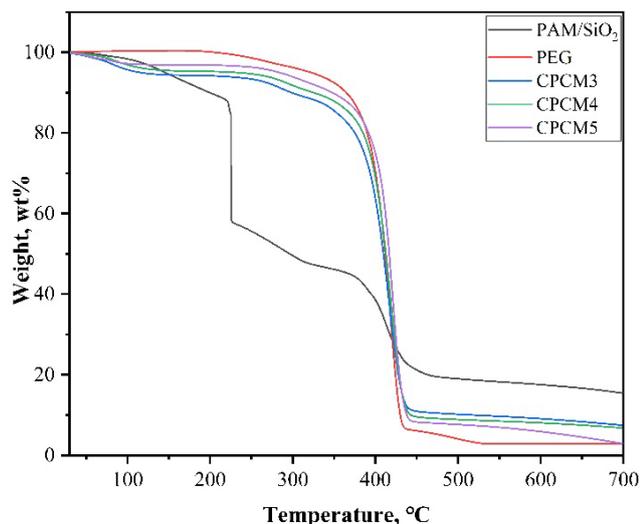


Fig. 6. The TGA curves of PAM/SiO₂, PEG-6000 and CPCMs

The 5 wt.% weight loss temperatures of PAM/SiO₂, CPCM3, CPCM4 and CPCM5 were 144.9 °C, 196.4 °C, 239.2 °C and 283.1 °C respectively. Compared with pure PEG6000, the maximum decomposition temperature of CPCMs was significantly higher. In the meantime, the introduction of PEG also apparently increased the decomposition temperature. The 5 wt.% weight loss temperature of CPCMs was in good agreement with the loading rate of PEG. It was known that the heat resistance of CPCMs was better than that of PEG and PAM/SiO₂. This might be because PEG was uniformly dispersed in the hydrogel network, which required more energy to destroy chain entanglement, intermolecular hydrogen bonds and other forces. And PEG/PAM/SiO₂ CPCMs showed strong thermal stability at 240 °C. The results indicated that PEG/PAM/SiO₂ CPCMs were useful for heat storage in a very wide temperature range.

4. CONCLUSIONS

In this study, PEG/PAM/SiO₂ composite phase change materials were prepared by in-situ free radical polymerization and sol-gel method. PEG was successfully fixed in the hydrogel network formed by the crosslinking of PAM and SiO₂ through chain entanglement, hydrogen bond and other physical effects. The abundant pores not only ensured the high loading rate of PEG, which made CPCMs have a high energy storage density of 126.9 J/g, but also gave the network scaffold the ability to buffer the volume change during the phase change of PEG. And the good toughness could cope with thermal stress, which made it exhibit good shape stability and thermal stability. PAM hydrogel had been widely used in the medical field due to its good structural characteristics and bio-friendliness, but little research had been done in the field of phase change materials. In this study, PEG was innovatively introduced into the PAM hydrogel system modified by SiO₂ nanoparticles to synthesize new composite phase change materials. To verify the porous network structure of the sample from multiple angles, the microscopic morphology of the transverse and longitudinal sections of the sample was

observed for the first time, which provided a powerful image and theoretical basis for subsequent research. The excellent mechanical and thermodynamic properties of PEG/PAM/SiO₂ CPCMs played an essential role in the application of this material in many fields and also provided ideas and methods for the preparation of new composite phase change materials.

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