In-situ preparation of a shape stable phase change material

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ABSTRACT

A shape-stable phase change material (PCM) compounded through polyethylene glycol (PEG) and triallyl isocyanurate (TAIC) was investigated, in which PEG was used as component of PCM and confined inside cross-linked TAIC. The microstructure of the PEG/TAIC composites was determined by Fourier Transform Infrared Spectroscopy (FT-IR) and X-ray Diffraction (XRD). The results indicated that there was no chemical interaction between PEG and TAIC and the composites remained the PEG crystalline. The size of the composite nanoparticles was about 150 nm. The thermal characteristic of the composites determined by Differential Scanning Calorimeter (DSC) indicated that the composites had excellent thermal stability and heat storage durability after 200 heating-cooling cycles. Moreover, the composites had typical solid-solid phase transition temperatures among the range of 31.16 ~ 57.14 °C and high latent heat enthalpy between 110.2 J/g and 136.9 J/g. The shape analysis suggested that the composites could be used under a high temperature 100 °C as thermal energy storage materials.

KEYWORDS

Phase change materials; Polyethylene glycol; Shape stabilized; Thermal stability; Latent heat thermal energy storage

1. Introduction

Nowadays, a variety of non-renewable natural resources and energy have been consumed massively, which has led to more and more serious environmental pollution.

The energy shortage and environmental protection has attracted worldwide attention. It is important to develop new types of high efficient green energy materials ^[1-3]. Phase change materials (PCMs) are considered as a satisfied candidate for thermal energy storage due to their high energy storage density and slight temperature variation during the absorbing and releasing heat processes ^[4-6].

PCMs can be divided into two types, inorganic PCMs and organic PCMs. Compared with inorganic PCMs ^[7-9], organic PCMs ^[10-14] have many advantages for thermal energy storage, such as little volume change during phase change process, no supercooling and phase separation during crystalline, non-toxic, non-corrosiveness, and so on. Among various organic PCMs, polyethylene glycol (PEG) is very promising for thermal energy storage due to its suitable phase change temperature ranging from 4.2 °C to 70 °C, high phase change latent heat ranging from 117.6 ~ 175.8 J/g, and excellent chemical stability ^[15]. However, PEG belongs to solid-liquid PCMs, it changes into liquid state and flows easily as the temperature is beyond its melting point (lower than 70 °C). In order to prevent the leakage of PEG during the phase transition, supporting materials ^[16-19] such as micro-encapsulation and porous materials were developed. Feng et al. ^[17] prepared PEG and mesoporous materials (active carbon (AC) and silica molecular sieves) composites as shape stabilized PCMs by a physical blending and impregnating method. The PEG/AC PCMs with 80 wt% of PEG had a large latent heat 102 J/g, a relatively low melting point, 50.1 °C, a little supercooling and high heat storage efficiency 79.44 %. Wang et al. ^[18] prepared PEG/SiO₂ composite as a shape-stable PCM, which exhibited a high phase transition

enthalpy 162.9 J/g and improved thermal conductivity. Li et al. ^[19] synthesized melamine/formaldehyde/polyethylene glycols crosslinked copolymers as solid-solid PCM with maximal latent heat enthalpies 109.4 J/g and 103.9 J/g during heating and cooling cycles, respectively. Obviously, previous works were focus on utilizing porous inorganic materials as supporting material, the main problem encountered was phase segregation during repeating thermal cycles because the PCMs were dispersed in porous inorganic materials only by physical interaction. Even if some previous work prepared PCMs through chemical interaction between PEG and the supporting materials, the PCMs were generally of low phase transition enthalpy. Moreover, the processing was difficult to perform.

Herein, we report a concise approach to prepare PCMs with typical solid-solid phase transition temperatures among the range of 31.16 ~ 57.14 °C and high latent heat enthalpy between 110.2 J/g and 136.9 J/g by choosing TAIC as the supporting material and PEG as the PCM. TAIC is a common and cheap crosslinking agent. By adding a small amount of initiator, the cross-linking polymerization of TAIC occurred under a low temperature 120 °C. A three-dimensional cross-linked network structure will be constructed by the cross-linking polymerization of TAIC, and hence the free movement of the PEG chains will be restricted in a finite inter space. The characteristics of the composite are investigated by FT-IR, WAXD, DSC, SEM and TGA, respectively. The results show that the PCM owns excellent phase change properties. It can be processed directly and is likely to be produced in large scale.

2. Materials and methods

2.1. Materials

PEG ($M_n = 4000$) was purchased from Sinopharm Chemical Reagent Co. Ltd. Triallyl isocyanurate (TAIC) (liquid state) was obtained from Shanghai Farida Chemical Co. Ltd. Benzoyl peroxide (BPO) was purchased from Tianjin Fu Chen Chemical Reagents Factory. All these agents were used as received.

2.2. Preparation of the shape stabilized PEG/TAIC composites

PEG and TAIC were mixed uniformly by stirring under 70 °C in a flask. Then 3 wt% BPO (mass ratio of BPO to the mixture) was added and the mixture was slowly heated up to 120 °C and stirred for 1 h. Finally, the composites were dried at 50 °C for 6 h in vacuum oven to obtain PEG/TAIC composites (PCMs). The synthesized composites with the mass ratio of PEG/TAIC being 3/1, 6/1, and 9/1 were marked as PCM-1, PCM-2 and PCM-3, respectively.

2.3. Characterization

The chemical composition and structure of the PCMs were characterized by FT-IR spectrophotometer (U.S. Company Perkin Elmer). The spectral analyses were carried out by using KBr pellets among wave number range from 400 cm^{-1} to 4000 cm^{-1} . The X-ray diffraction (XRD) measurements were recorded by D/max-3B diffractometer (Rigaku Corporation) with 2θ ranging from 10 ° to 70 ° at scanning rate of 10 °/min with Cu K α radiation. The thermal energy storages of the pure PEG and the PCMs were determined by using differential scanning calorimeter (DSC Q2000) with nitrogen as purge gas. Thermal cycling tests were carried out by hot

stage (STC 200, Instec) within temperature range of 20 ~ 80 °C. The morphology and microstructure of PEG/TAIC composites were measured by scanning electron microscope (SEM, JSM-5600, JEOL, Japan). The shape-stabilized properties of the pure PEG and PCMs were examined by heating panel. At every given temperature the sample was maintained for 10 min, the states of the samples were recorded through digital camera. Thermogravimetry analysis was carried out by using TGA50 heating the samples from 30 °C to 550 °C with linear heating rate of 10 °C/min under nitrogen atmosphere. The thermal conductivity of the CFSPCMs was measured by using thermal conductivity testing instrument (TC3100, XIATECH, accuracy: $\pm 2.0\%$).

3. Results and discussion

3.1. Structural characterization of PEG/TAIC composites

The FT-IR spectra of PEG, TAIC and PCMs (PCM1-3) are shown in Fig 1. From the profile of the pure PEG as shown at the top in Fig 1, it is obvious that there is a peak at wave number 3435 cm^{-1} which comes from stretching vibration of functional group of -O-H group, the peaks at wave numbers 2888 cm^{-1} , 962 cm^{-1} and 842 cm^{-1} are assigned to the stretching vibration of the -CH₂ group, the strong peak at wave number 1108 cm^{-1} belongs to the stretching vibration of C-O-C group. The FT-IR spectrum of TAIC is shown on the second profile in Fig 1. It shows the stretching vibration of C=C group at wave number 1638 cm^{-1} , and the stretching vibration of C=O at wave number 1717 cm^{-1} in TAIC. Compared the FT-IR profiles with those of PEG and TAIC, the profiles of the prepared PCMs as shown in the lower part in Fig 1 clearly show the individual peaks of PEG and TAIC but no significant new peaks,

which indicates that there are no chemical interaction between the PEG and TAIC, there may be only physical interactions between the components of the composites in nature. PEG is closed in the cross-linked TAIC network, and hence leakage of the melted PEG from the composites is prevented.

3.2. Crystallization properties of PCM1-3

Fig 2 displays the XRD profiles of the pure PEG and PCM1-3. As a comparison, XRD curve of TAIC self-crosslinked polymer (TAIC-P) is also displayed in Fig 2. Apparently, the composites have similar crystal form with that of the pure PEG. There are two main diffraction peaks appear at 19.1° and 23.3° on all XRD curves. It suggests that the crystal structure and crystal cell type of PEG is not damaged in the experiment process ^[19-22]. The main differences between the XRD curves of the pure PEG and the composites are diffraction peak intensity and half width of the peaks. As the content of PEG decreases from PCM-3 to PCM-1, the diffraction peaks of the composites become lower than those of pure PEG. It indicates that with the increase of the content of TAIC, PEG suffered binding force increases.

3.3. Thermal energy storage properties of PCM1-3

The DSC curves of the pure PEG and PCM1-3 are presented in Fig 3a-3b. The thermal energy storage data obtained from the DSC curves are given in Table 1. The melting points (T_m) of the pure PEG and PCM1-3 are 57.17°C , 54.90°C , 55.85°C and 57.14°C , respectively, as shown in Fig 3a and Table 1. The crystallizing temperatures (T_c) are 39.21°C , 34.20°C , 32.69°C and 31.16°C , respectively, as shown in Fig 3b, and Table 1. It can be found that the phase transition temperature of

PEG shifts from 31.16 °C of the pure PEG toward higher temperature 39.21 °C in PCM1-3. Because TAIC forms a three-dimensional network microstructure during the in-situ cross-linking process, the free movement of the PEG chains is restricted in a finite inter space. The PEG chains are unable to arrange freely to crystallize, which results in the crystallization integrity of PEG is compromised ^[16, 23]. It is worth noting that the PCM1-3 have appropriate phase change temperatures in the process of phase transformation, and therefore the prepared PCM1-3 can be used as thermal energy storage materials.

On the other hand, as shown in Fig 3a-3b and Table 1, the latent heat values are 161.5 J/g, 111.1 J/g, 127.3 J/g and 136.9 J/g for heating processes and 160.3 J/g, 110.2 J/g, 126.5J/g and 135.7 J/g for cooling processes of the pure PEG and PCM1-3, respectively. From which, we can find that the phase change values of PCM1-3 increase with the PEG content increasing, that is, the phase change values of PCM1-3 decrease with the TAIC content increasing. It is expected that TAIC does not have phase transformation function, but destroys the crystallization of PEG chains to some extent.

In addition, Table 2 shows the comparison of energy storage properties of PCM1-3 with that of other composite PCMs in literatures ^[17, 23-28]. In which the enthalpy efficiency (ψ) is defined as the ratio of practical and theoretical loading in mixtures. By considering these data it is appreciably noted that the ψ value of PCM1-3 in present work are much higher than other composites in literatures.

As PCM-2 is selected as a typical sample, we examine the DSC curves of it after 200 thermal cycles, as shown in Fig 4. The thermal data from DSC are summarized in Table 3. As can be seen from Fig 4 and Table 3, after 200 thermal cycles, the T_m and T_c of PCM-2 are measured as 56.06 °C and 32.70 °C, respectively, the latent heats of it are 125.1 J/g and 124.8 J/g, respectively. Obviously, there are insignificant changes in phase change temperatures and enthalpies after 200 thermal cycles. Therefore, the composites PCM-2 have good thermal energy storage reliability after thermal cycles.

3.4. Shape-stabilized properties of PCM1-3

The states of the pure PEG and PCM1-3 recorded by digital camera depending on the temperature are shown in Fig 5. Obviously, the pure PEG colored white and the composites colored faint yellow show different thermal shape stability with temperature changing from 20 °C to 100 °C. As seen from Fig 5, as the temperature rises to 60 °C, the pure PEG changes into liquid. However, even if the temperature rises to 100 °C, the composites PCM1-3 still keep the shape perfectly and have no liquid leakage on the surface. The results suggest that the composites PCM1-3 are shape-stable, and the absence of liquid leakage reveals their effective utilization in building and clothing applications.

3.5. Morphologies of PCM1-3

Typical morphologies observed by SEM of TAIC self-crosslinked polymer (TAIC-P) and PCM1-3 are shown in Fig 6. Fig 6a shows the SEM image of the pure TAIC-P, which exhibits a uniform morphology. With blending with PEG, the morphology changes with the content of PEG. As the content of PEG is PEG/TAIC =

3/1, the component PEG forms spherical dispersed phase, as shown in Fig 6b. From the inserted magnified image in Fig 6b, one can determine the spherical phase size is 100-200 nm. As the content of PEG is PEG/TAIC = 6/1, the size of dispersed PEG phase is increasing, as shown in Fig 6c. When the content of PEG is PEG/TAIC = 9/1, the PEG forms continuous phase, as shown in Fig 6d.

Together with the above characteristics of the PCM1-3, it is rationally that when the content of TAIC is high, such as PEG/TAIC = 3/1, PEG is wrapped in the cross-linked network of TAIC, so as to form the nanospheres similar to the microcapsule structure ^[29, 30]. However, PEG is difficult to be wrapped in the cross-linked network of TAIC along with the reduction of TAIC content, such as PEG/TAIC = 9/1. But the PEG can be encapsulated and embedded still inside the crosslinked TAIC network ^[23]. Therefore, it keeps its shape unchanged as the temperature is higher than the PEG's melting point 57.17 °C.

3.6. Thermal stability of PCM1-3

TGA curves are shown in Fig 7a to assess the thermal stability of the pure PEG, TAIC-P, and PCM1-3, and the corresponding DTG curves are shown in Fig 7b to determine the maximum rate of weight loss in the composites PCM1-3. As seen from Fig 7a and 7b, the pure PEG exhibits one-step thermal degradation process, in which the beginning degradation temperature (T_d) is 377 °C and the degradation peak occurs at 408 °C. For TAIC-P, it displays two-step thermal degradation process. The minor weight loss ~ 10% occurs between 192 °C and 262 °C, which attributes to the loss of adsorbed water or some volatile component in TAIC. The major weight loss ~ 75%

starts at 452 °C and the degradation peak occurs at 461 °C. However, the composites PCM1-3 show one-step thermal degradation among approximate similar temperature range, between 50 °C to 550 °C. The degradation of PCM1-3 occurs at 384 °C, 390 °C, and 392 °C, respectively. That is, the as prepared composites remain thermal stable under 384 °C. Together with the shape analysis as shown in Fig 5, one can deduce that the composites can be used under a relatively high temperature 100 °C. Accordingly, the as prepared PCMs have potential usability in solar energy storing, smart housing, and phase change energy storage fibers [2, 5, 31].

3.7. Heat transfer properties of PCM1-3

Thermal conductivity of PCM can be considered as one of important parameters in thermal energy storage applications as well as its phase transition temperature and latent heat capacity. This parameter significantly affects the heat charging and discharging performance of a thermal energy storage system integrated with PCM. Table 4 presents the thermal conductivity of the PEG and PCMs. As seen from Table 4, the thermal conductivity of the PEG and PCM1-3 measured as 0.152, 0.144, 0.147 and 0.149 W m⁻¹ K⁻¹, respectively. The thermal conductivity of the PCMs increased with increasing mass fraction of PEG. Moreover, the thermal conductivity of PCMs comparing with PEG does not significant changed. This indicates that the as prepared PCMs have good thermal conductivity.

4. Conclusions

In this study, shape-stabilized PEG/TAIC composites PCMs were prepared by in-situ reactive blend PEG and TAIC, in which PEG functioned as a PCM, and TAIC

functioned as a supporting material by self-crosslinking. The PCMs kept solid state at high temperature, eg. 100 °C and showed excellent phase change properties, a suitable solid-solid phase transition temperature among the range of 31.16 ~ 57.14 °C, and high latent heat storage capacity of more than 135 J/g. Moreover, they had excellent thermal stability and heat storage durability after 200 heating-cooling cycles. Accordingly, the as prepared PCMs could be potentially used as shape-stabilized PCMs for thermal energy storage.

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Table and Figure Captions

Table 1. DSC data for the pure PEG and PCMs.

Table 2. Thermal energy storage characteristics of different shape stabilized PCMs in literatures.

Table 3. DSC data of PCM-2 under different thermal cycles.

Table 4. Thermal conductivity of PEG and PCMs.

Fig 1. FT-IR spectra of the samples.

Fig 2. XRD patterns of the samples.

Fig 3. DSC curves : (a) PEG and PCM-2, (b) melting and (c) crystallization curves of PCMs.

Fig 4. DSC curves of PCM-2 before and after 200 cycles.

Fig 5. The shape changing of the pure PEG and PCMs with temperature.

Fig 6. SEM images of (a) TAIC-P, (b) PCM-1, (c) PCM-2, and (d) PCM-3.

Fig 7. TGA (a) and the corresponding DTG (b) curves of the pure PEG, TAIC-P and PCMs.

Table 1. DSC data for the pure PEG and PCMs.

Sample	T_c (°C)	ΔH_c (J/g)	T_m (°C)	ΔH_m (J/g)	Phase behavior
PEG4000	39.21	160.3	57.17	161.5	Solid-liquid
PCM-1	34.20	110.2	54.90	111.1	Solid-solid
PCM-2	32.69	126.5	55.85	127.3	Solid-solid
PCM-3	31.16	135.7	57.14	136.9	Solid-solid

Table 2. Thermal energy storage characteristics of different shape stabilized PCMs in literatures.

Sample*	T_c (°C)	T_m (°C)	Theoretical enthalpy (J/g)	Latent heat (J/g)	Enthalpy efficiency (%)	Reference
PEG/PLA (66.67)	43.6	63.7	111.87	74.7	66.77	[24]
PET/ADOE (57.14)	45.0	48.2	93.86	66.5	70.85	[25]
PEG/AC (70)	27.8	49.0	112.4	81.3	72.33	[17]
PEG/AC (80)	--	48.3	128.4	102	79.44	[17]
PEG/cellulose (60)	37.05	58.51	125.14	84.63	67.28	[26]
PEG/agarose (70)	43.36	57.73	146	110.86	75.93	[26]
PEG/chitosan (80)	44.76	57.18	166.86	152.16	91.19	[26]
PEG10000/PAA (90)	44.2	56.3	161.1	135.1	83.86	[27]
PEG10000/EcoA 15 (75)	46.6	59.9	134.25	119.2	88.79	[27-28]
P(AN-co-IA)/PEG4000 (73)	35.55	53.9	134.9	118.56	87.89	[23]
PCM-1 (75)	34.20	54.90	121.13	111.1	91.72	Present
PCM-2 (85.71)	32.69	55.85	138.43	127.3	91.96	Present
PCM-3 (90)	31.16	57.14	145.35	136.9	94.19	Present

* The numbers in blanket denote the mass content of the PEG in PCMs.

Table 3. DSC data of PCM-2 under different thermal cycles.

Sample	T_c (°C)	ΔH_c (J/g)	T_m (°C)	ΔH_m (J/g)	Phase behavior
1 cycle	32.69	126.5	55.85	127.3	Solid-solid
200 cycles	32.70	124.8	56.06	125.1	Solid-solid

Table 4. Thermal conductivity of PEG and PCMs.

Sample	Thermal conductivity at 20 °C (W m ⁻¹ K ⁻¹)
PEG4000	0.152
PCM-1	0.144
PCM-2	0.147
PCM-3	0.149

Fig 1. FT-IR spectra of the samples.

Fig 2. XRD patterns of the samples.

Fig 3. DSC curves: (a) melting and (b) crystallization curves of the PEG and PCMs.

Fig 4. DSC curves of PCM-2 before and after 200 cycles.

T(°C) Sample	20 °C	40 °C	60 °C	80 °C	100 °C
PEG
PCM-1
PCM-2
PCM-3

Fig 5. The shapes change of the pure PEG and PCMs with temperature.

Fig 6. SEM images of (a) TAIC-P, (b) PCM-1, (c) PCM-2, and (d) PCM-3. The scale bar indicates 1 μ m.

Fig 7. TGA (a) and the corresponding DTG (b) curves of the pure PEG, TAIC-P and PCMs.

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