



Two components based polyethylene glycol/thermosetting solid-solid phase change material composites as novel form stable phase change materials for flexible thermal energy storage application

Zhimeng Liu, Bo Wu, Xiaowei Fu, Peiyao Yan, Ye Yuan, Changlin Zhou*, Jingxin Lei*

State Key Laboratory of Polymer Materials Engineering, Polymer Research Institute of Sichuan University, Chengdu 610065, China

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ABSTRACT

In this study, two components based polyethylene glycol (PEG)/thermosetting solid-solid phase change material composites as novel form stable phase change material (FSPCM) were successfully prepared via free-radical polymerization. One of the components consists of PEG-based polyurethane acrylate prepolymer and extra pristine PEG. Another component is made up of 1,6-hexanediol diacrylate and benzoyl peroxide. The extra pristine PEG with low viscosity at melting state was used as diluents to decline the melting viscosity of PUA and served as phase change functional chains of the FSPCMs, endowing the FSPCM with high latent heats and flexible application ability for thermal energy storage. The chemical structure, crystalline performances, phase change properties and thermal reliability and stability of prepared FSPCMs were investigated by Fourier transform infrared spectroscopy, X-ray diffraction, polarizing optical microscopy, differential scanning calorimetry and thermogravimetry analysis, respectively. The leakage test results show that the maximum content of PEG in novel FSPCM without any leakage above the melting point of PEG is 85 wt%. The DSC analysis results show that the novel FSPCMs have high latent heats and the latent heats efficiency of novel FSPCMs is significantly higher than that of the traditional FSPCMs. The prepared FSPCMs exhibit good thermal reliability and stability from the thermal cycling test and TG analysis.

1. Introduction

In recent years, phase change materials (PCMs) with excellent energy storage-releasing capability and isothermal nature have been gained wide attention [1–5]. Among PCMs, some pristine organic PCMs including polyethylene glycol (PEG), fatty acids, fatty alcohol and paraffin waxes have been researched and applied extensively for thermal energy storage (TES) [6–10]. Nevertheless, the solid-liquid transition property of these organic PCMs restricts their direct application [11,12]. Therefore, the methods of using a special container/device to prepare form-stable PCMs (FSPCMs) and through chemical modification to synthesize solid-solid PCMs (SSPCMs) are essential to prevent leakage of these organic PCMs during the phase change process [13–16].

As typical organic PCMs, PEG based SSPCMs and FSPCMs have been extensively studied for TES [17,18]. Su et al. [19] reported the preparation of polyurethane block copolymer based thermoplastic SSPCM with PEG as soft segment, 4,4-diphenylmethane diisocyanate (MDI) and 1,4-butanediol as hard segment. Li et al. [20] synthesized thermosetting SSPCM via the two step condensation reaction of PEG with

pentaerythritol and MDI. Peng et al. [21] synthesized four thermosetting SSPCMs with different crosslinking density using PEG as the phase change functional chain, β -cyclodextrin and MDI as molecular framework. Fang et al. [22] prepared PEG/epoxy resin composite as FSPCM using casting molding method, where epoxy resin was served as supporting material to prevent the leakage of melted PEG. Karaman et al. [23] using diatomite as supporting material to prepared PEG/diatomite composite as FSPCMs.

In general, thermoplastic SSPCMs have inherent merit of good processibility [24]. However, the PEG contents in thermoplastic SSPCMs were greatly limited, which results in lower latent heats. The thermoplastic SSPCMs will get flow once the PEG contents exceed critical a value, where the temperature higher than the melting point of PEG [19,25,26]. Different from the thermoplastic SSPCMs, the thermosetting SSPCMs have both the merits of high PEG contents and good mechanical strength when the temperature over the melting point of PEG [27]. This is because that the crosslinking structures in SSPCMs strongly confine the flow of melted PEG and provide the high mechanical strength. Nevertheless, the tough processibility of thermosetting SSPCMs seriously restricts their widespread applications. To

* Corresponding authors.

E-mail addresses: chouscu@scu.edu.cn (C. Zhou), jxlei@scu.edu.cn (J. Lei).

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address this problem, our group has reported a two components thermosetting SSPCMs [28]. These PEG based two components, isocyanate-terminated prepolymer and tetrahydroxy prepolymer were used to endow the thermosetting SSPCMs with flexible application ability. However, both two components of isocyanate-terminated prepolymer and tetrahydroxy prepolymer have relatively high viscosity in the melting state, which results in difficulties of homogeneous mixing and sufficient reaction at the time of application. On the other hand, the latent heat density of previous prepared PCMs was unavoidably declined by the non phase change of hard segments and crosslinking structures in SSPCMs and supporting materials in FSPCMs [17,20, 29].

In this paper, two components based PEG/thermosetting SSPCM composites as novel FSPCM were successfully prepared via free-radical polymerization, endowing the novel FSPCM with high latent heats and flexible application ability for thermal energy storage. One component (component A) consists of PEG-based polyurethane acrylate prepolymer (PUA) and extra pristine PEG. Another component (component B) is made up of 1,6-hexanediol diacrylate (HDDA) and benzoyl peroxide (BPO). In component A, the PUA was synthesized via bulk polymerization using the high molecular weight PEG as phase change functional chains, 2-hydroxyethyl acrylate (HEA) as blocking agent and the MDI as coupling reagent. The extra pristine PEG with low viscosity at melting state was used as diluent to decline the melting viscosity of PUA and it was also served as phase change functional chains of novel FSPCMs. The HDDA in component B was acted as solvent to dissolve the BPO powder and then polymerized with PUA to form crosslink skeleton to prevent the flow and leakage of melting PEG. Therefore, both components A and B with low viscosity can be easily homogeneous mixed at melting state. In addition, the phase transitions of thermosetting SSPCM and extra PEG simultaneously occur in the novel FSPCMs, hence the latent heats of novel FSPCMs would be greatly increased compared with the traditional FSPCMs. In this study, we named novel FSPCMs as FPCMs. The chemical structure, crystalline performances, phase change properties, thermal reliability and stability of prepared FPCMs were investigated.

2. Experimental

2.1. Materials

Polyethylene glycol (PEG, $M_n = 8000$ g/mol, from Chengdu Kelong Chemical Reagent Co. Ltd., China) was dried under vacuum at 110 °C for 3 h before use. 2-Hydroxyethyl acrylate (HEA, analytical grade, from Chengdu Kelong Chemical Reagent Co. Ltd., China) was dried using 5 Å molecular sieve for a week followed by distillation before use. 1,6-Hexanediol diacrylate (HDDA, 100% pure, from Shanghai Curease Chemical Co. Ltd. China), diphenylmethane diisocyanate (MDI-50, from Wanhua Chemical Group Co. Ltd., China), benzoyl peroxide (BPO, analytical grade, from Chengdu Kelong Chemical Reagent Co. Ltd., China) and dibutyltin dilaurate (DBTDL, analytical grade, from Guangdong Wengjiang Chemical Reagent Co. Ltd., China) were used as received.

2.2. Synthesis of polyurethane acrylate prepolymer (PUA)

The synthesis of PUA was carried out under nitrogen atmosphere and the synthetic route is shown in Fig. 1. Firstly, 80 g (0.01 mol) PEG was introduced into a 250 ml three necks round bottom flask and then 5 g (0.02 mol) MDI-50 was added until the PEG melting completely at 80 °C. The mixture was stirred for homogenization and reacted at 80 °C for 3 h to obtain NCO-terminated prepolymer. Secondly, 2.32 g (0.02 mol) HEA was added and reacted at 80 °C for 1 h, then 0.05 wt% of DBTDL (based on the total mass) as catalyst were introduced and the reaction was further carried out at 80 °C for 2 h. The PUA was obtained.

2.3. Synthesis of FPCMs

The FPCMs were prepared by directly thermal curing the mixtures of two components. Fig. 1 and Table 1 illustrate the synthetic process and recipes of FPCMs, respectively. The calculated amount of PUA and extra PEG with 0, 60, 70, 80, 85 and 90 wt% contents (based on the total mass of FPCMs) were introduced into a 100 ml round bottom flask. The mixture was homogenized until the PUA and PEG completely melting at 70 °C to obtained the component A. Then, the predetermined HDDA with 2 wt% of BPO (based on the mass of HDDA) as component B was added into the flask, after quickly stirring for homogenization, the mixture was poured into a mould and the thermal curing was conducted in the nitrogen atmosphere at 90 °C for 4 h to obtain the FPCMs. Before the measurements, leakage test was performed to confirm the form-table performance of all prepared FPCMs. The leakage test was conducted by putting the samples into a oven and heated at 80 °C. The FPCMs that do not observe leakage or flow of melted PEG form the samples were defined as form stable PCMs.

2.4. Instruments and analysis

2.4.1. Fourier transform infrared spectroscopy (FTIR)

Infrared spectrogram of all samples was obtained using an Infrared Spectrophotometer (Nicolet-560, Nicolet Co., USA) with 4 cm^{-1} resolution and 32 scans were collected.

2.4.2. X-ray diffraction (XRD)

The crystalline structure of PEG and FPCMs were detected by using an automatic powder diffractometer (X'pert pro MPD, Netherlands) at room temperature with Cu K α radiation at 35 kV and 30 mA. The data were collected in an angle range of 5–50° with step width of 0.03°/min.

2.4.3. Polarizing optical microscopy (POM)

A POM (XPR-500D, China) was used to observe the crystalline morphology of PEG, SSPCM and FPCMs at room temperature. The specimens were putted between a microscope glass and a cover slip.

2.4.4. Differential scanning calorimetry (DSC)

Dynamic DSC scans of PEG, SSPCM and FPCMs were conducted using a differential scanning calorimeter (DSC 8500 PerkinElmer, USA) under nitrogen atmosphere with heating or cooling rate of 10 °C/min. Before the formal measurements, the first heating scan was carried out from 30 to 100 °C and the sample was kept at 100 °C for 5 min to eliminate the thermal history.

2.4.5. Thermogravimetric analysis (TG)

The thermal stability of PEG, SSPCM and FPCMs were investigated on a thermal gravimetric analyzer (SDTQ600, USA) from room temperature to 600 °C in a nitrogen atmosphere with a heating rate of 10 °C/min.

2.5. Accelerated thermal cycling test

Accelerated thermal cycling test was conducted in a high-low chamber with 100 consecutive thermal cycling from 20 to 90 °C to affirm the thermal reliability and reusability of the FPCMs. Afterwards, the variations of phase transition properties and chemical structure of heat treated FPCMs were respectively confirmed by DSC and FTIR.

3. Results and discussion

3.1. Leakage test of FPCMs

Leakage test for FPCMs with different PEG contents was performed to ensure the form stable performance. The photos of PEG, FPCM85 and FPCM90 which are heated at 80 °C for different time are shown in

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