PAPER

Fabrication of thermal energy storage wood composite based on shape-stable phase change material

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Abstract
In this work, a novel phase change energy storage wood (PCESW) was fabricated by impregnating solid-solid phase change materials (SSPCM) into delignified wood. The SSPCM was prepared by using polyethylene glycol (PEG) 1500 as the thermal energy storage ingredient, hexamethylene diisocyanate (HDI) as the cross-linking agent and castor oil (CO) as the skeleton material. The chemical composition and structure of SSPCM and PCESW were confirmed by fourier transform infrared spectroscopy (FTIR); the crystalline structure of PEG and SSPCM were tested by x-ray diffraction (XRD); the microstructure of PCESW was observed by scanning electron microscopy (SEM); thermal properties and durability of SSPCM and PCESW were investigated by differential scanning calorimetry (DSC) and thermogravimetric analysis (TG). The results showed that: (1) the porosity of wood was improved obviously after delignification and SSPCM mainly distributed in wood vessels; (2) PCESW-15 exhibited superior temperature-regulating performance and thermal stability; (3) PCESW-15 presented outstanding shape stability and no liquid leakage occurred during the phase transition. The great thermal performance of PCESWs suggested that it can be used as energy-saving building materials for indoor temperature regulating.

1. Introduction

With the growth of economy and population, the demand for energy is also rising. In response to these needs, the focus of research has shifted to the large-scale development of renewable resources. However, renewable resources such as solar, wind, geothermal and hydropower are not enough to meet the overall energy demand in the future. In recent years, the energy crisis and environment deterioration have made the utilization of renewable energy and the development of environment-friendly materials become a crucial subject [1–3]. Thermal energy storage (TES) can offset the increasing demand for energy management and conservation in some ways [4–6].

Phase change materials (PCMs) as a kind of TES is the most potential method owing to its isothermal nature and high-energy storage density [7–9]. Solid-liquid PCMs (SLPCMs) and solid-solid PCMs (SSPCMs) are two common types of PCMs [10–12]. SLPCMs has a series of problems such as leakage, volume change during the phase change process. While SSPCMs get the advantages of no liquid or gas generation even above phase change temperature, which are environment-friendly and having the ability to process the material into the desired shape [13]. It could be prepared by grafting the solid-liquid PCM as soft segments grafted to the hard segments of another macromolecular framework [6], the hard segments are introduced to the molecular chains of the SSPCMs as supporting materials to keep the material in solid state during phase change processes, the SSPCM prepared by this method showed good properties.

Polyethylene glycol (PEG) is known as a versatile PCM with high latent enthalpy, wide transition temperature range, thermal and chemical stability, ease of chemical modification, good biocompatibility, low vapor pressure, non-toxic and non-corrosive nature [14]. The latent enthalpy and transition temperature of
PEG are varying with molecular weight, so it can be used to prepare PCMs with controlled phase change temperature. Li et al. [15] prepared a SSPCM using PEG 10000, pentaerythritol (PE) and 4, 4’-diphenylmethane diisocyanate (MDI), which phase change temperature is higher than 50 °C. Xia et al. [16] prepared a series of SSPCM by using PEG 8000 as the phase change ingredient, MDI as the cross-linking agent, and graphene oxide (GO) as the skeleton material, and their phase change temperature were in the range of 37 °C–67.5 °C. The other SSPCM such as PEG/HDiT [17], PEG/MDI/DETA [18], PEG/HDiB/HNTs [19], PEG/PVP/CNTs [20], PEG/SMA [21] et al were all used high molecular weight PEG as the heat storage unit, and these materials always had high phase change temperature which limited its application for indoor temperature regulation. In this study, we intend to synthesize a SSPCM with low phase change temperature by using low molecular weight PEG.

Castor oil (CO) is a valuable renewable resource due to its biodegradable and sustainable and low toxicity property [22]. The major ingredient of CO, 12-hydroxy-cis-9-octadecenoic acid, is a hydroxyl containing fatty acid ester, and an average number of hydroxyl per molecule is 2.7. It can be used for preparing polyurethanes without further modification [23]. Due to the high content of hydroxyl and the stable structure of CO, it was used as the skeleton material reacting with isocyanate group to obtain SSPCMs. Wood is an important building material with good performance, such as high strength, low density, and great heat insulation [24–26]. There are many interconnected pores in wood, which provides possibility for introducing PCM into wood. Andrea et al. [27] prepared a kind of green and multi-function energy storage material by impregnation PEG into thin beech laminae and interleaved with thin foils of thermoplastic starch (TPS), PEG improved the performance of heat storage, tensile and Charpy impact strength of the laminates. Xu et al. [28] prepared a composite phase change material by incorporating silica-stabilized PEG with wood, the SiO2 network on liquid phase leakage of PEG during phase transition, the thermal enthalpy of composite materials could reach 46.7 J g⁻¹, it is a kind of potential energy saving building material. Céline et al. [29] prepared a kind of transparent wood for thermal energy storage (TW–TES) by encapsulated PEG into a delignified wood substrate, TW–TES showed good heat-storage and thermal insulation properties. The other composite materials such as TDCW [30], TESW [31], Micro-PCM PCESW [32] et al were all used wood combined with phase change materials to get great thermal performance which can be used in the fields of thermal insulation, storage and building energy conservation.

In this study, we prepared a phase change energy storage wood (PCESW) by using balsa wood and SSPCM. The SSPCM was fabricated by using PEG as the phase change ingredient, HDI as the cross-linking agent, and CO as the skeleton material. The prepared composites were characterized by Fourier transform infrared spectroscopy (FTIR), x-ray diffraction (XRD) and scanning electron microscopy (SEM), differential scanning calorimetry (DSC) and thermogravimetric analysis (TG) to investigate the effect of SSPCM on the performance of PCESW, including chemical composition and microstructure, crystalline, thermal reliability and thermal properties. Practical thermal performance of PCESW was evaluated by a house model.

2. Materials and methods

2.1. Materials

The agents used for PCESW fabrication including polyethylene glycol (PEG, analytical grade), castor oil (CO, analytical grade), hexamethylene diisocyanate (HDI, 99.6% pure) and N, N-dimethylformamide (DMF, analytical grade) were all provided by Shanghai Aladdin biological technology Co., Ltd. China. Sodium hydroxide (NaOH, ≥96%) and sodium sulfite (Na₂SO₃, ≥97%) were provided by Shanghai Chemical Co., Ltd. China. Balsa wood (Ochroma lagopus swartz) with an average density of 0.138 g cm⁻³ was purchased from Shanghai Feiheng Trading Co., Ltd.

2.2. Synthesis of solid–solid PCMs

The fabrication was carried out via a two-step polymerization and the corresponding synthetic route is shown in figure 1. The reaction was carried out in a 500 ml three necks round bottom flask equipped with reflux condenser, mechanical stirrer and constant pressure funnel. Firstly, PEG and HDI (molar ratio of PEG to HDI was 1:2) were dissolved in DMF and stirred at 60 °C for 5 h. Secondly, castor oil (molar ratio of CO to HDI was 1:3) was slowly dropped into the prepolymer and stirred at 70 °C for 10 h. After that the SSPCM was obtained in DMF solution. The synthetic materials based on PEG 1500, PEG 1000 and PEG 800 as the phase change ingredient were named as SSPCM-15, SSPCM-10 and SSPCM-8, respectively. The nomenclature used for different composites is given in table 1.
2.3. Delignification of wood
Balsa wood (40 × 20 × 2 mm) was immersed in the NaOH (2.5 mol l⁻¹) and Na₂SO₃ (0.4 mol l⁻¹) mixed solution and boiled for 36 h to remove lignin. Then the wood was washed by hot deionized water to remove the residual alkaline substances and preserved in DMF solvent for further use.

2.4. Impregnation of solid–solid PCMs
The delignified wood was immersed into the SSPCM prepolymer mixture and vacuumized for 30 min, then the vacuum was removed to let SSPCM fill the wood. This process was repeated three times and followed by over-drying at 80 °C. The SSPCM was fabricated inside of the wood after over-drying and the prepared phase change energy storage wood named as PCESW. The nomenclature used for different composites is given in table 1.

2.5. Characterization
The chemical composition and structure of SSPCM were qualitatively confirmed by a Spectrum One-B model FT-IR (NICOLET iS10, USA) within the range of 4000–400 cm⁻¹. The crystalline structure of PEG and SSPCM were detected by using an automatic powder diffractometer (D8 Advance, Switzerland) at room temperature with Cu Kα radiation at 35 kV and 30 mA. The data were conducted in the 2θ ranging from 5° to 50° at a scanning rate of 1° min⁻¹.

The morphology of PCESW was observed by a scanning electron microscope (6380LV, Japan Electronics Corporation, Japan) at 3 kV. All samples were glued on the sample stages and sprayed with gold powder prior to the examination.

The latent heat and phase change temperature of SSPCM and PCESW were measured by a Q 2000 (TA, USA) DSC analyzer. The analysis was carried out under nitrogen atmosphere at a heating or cooling rate of 5 °C min⁻¹ and the temperature range was 0 °C–60 °C. The particle size of sample flour was about 40–60 mesh.

Thermal stability of SSPCM and PCESW were analyzed by TG (Q5000IR, USA) in the temperature range of 30 °C–600 °C at a heating rate of 10 °C min⁻¹ under nitrogen atmosphere.

Thermal reliability of the PCESW were tested with 100 consecutive cooling/heating processes. It was the PCESW was placed in a refrigerator at −20 °C for 30 min to carry out the freezing process, and immediately it was placed in an oven at 60 °C for 30 min to suffer the melting process. The changes in thermal properties after cycling processes were investigated by DSC.

Two house models (in figure 2) were built to evaluate the practical thermal performance of PCESW, the internal layer (50 × 50 × 50 mm) was made of pure wood and PCESW-15, respectively. Cystosepiment

| Table 1. Materials analyzed. |
|-----------------------------|
| Material       | Specification        |
| SSPCM-15       | PEG1500/HDI/CO       |
| SSPCM-10       | PEG1000/HDI/CO       |
| SSPCM-8        | PEG800/HDI/CO        |
| PCESW-15       | Wood/PEG1500/HDI/CO  |

Figure 1. Illustration of PCESW fabrication.
(20 mm) was used as the insulation layer and the outside wall was balsa (4 mm) wood. Temperature change of each house was recorded by a multi-channel temperature inspection instrument (JK-16C, China) and its temperature sensor was placed in the middle of each house. The heating process was carried out in the drying oven at 80 °C and the cooling process was tested in a fridge at −20 °C.

3. Results and discussion

3.1. FTIR analysis of SPCCM and PCESW

Figure 3 shows the FTIR curves of SPCCM and PCESW. The characteristic peaks of castor oil at 3425 cm\(^{-1}\) were corresponding to the stretching vibration of O–H group, the stretching vibration of C=O and C–H rocking vibration appeared at 1746 cm\(^{-1}\) and 1165 cm\(^{-1}\), respectively. Salient neat peaks of PEG in figure 3(b) were observed at 3426 cm\(^{-1}\) and 1102 cm\(^{-1}\), corresponding to the stretching vibration of O-H and the symmetrical stretching vibration of C–O–C, while the absorption peaks at 2885 cm\(^{-1}\), 1456 cm\(^{-1}\), 951 cm\(^{-1}\), 843 cm\(^{-1}\) belong to the C–H bonds [32]. The characteristic peak of HDI was observed at 2273 cm\(^{-1}\) in figure 3(c) corresponding to the N=O=C. As clearly shown in figure 3(d), the characteristic peaks at 1538 cm\(^{-1}\) and 3343 cm\(^{-1}\) were derived from the –NHCOO– and N–H, and these characteristic peaks of C=O stretching/bending vibrations (2856 cm\(^{-1}\), 1465 cm\(^{-1}\), 952 cm\(^{-1}\) and 842 cm\(^{-1}\)), C–O–C stretching vibration (1104 cm\(^{-1}\)) and O–H stretching vibration (3424 cm\(^{-1}\)) were also appeared in SPCCM-15. It was obvious that the characteristic peak of N=C=O disappeared completely and newly formed characteristic peaks of –NHCOO– and N–H appeared in figure 3(c) suggested the reaction among CO, PEG and HDI.

Figure 3(e) shows the FTIR curve of Balsa wood, it was easy to find the characteristic peaks of O–H at 3343 cm\(^{-1}\), which was due to the large number of fibers, hemicellulose and lignin in wood. The FTIR of
3.2. Crystalline property of PEG and SSPCMs

The crystalline property of pure PEG 1500 and SSPCMs are shown in figure 4. All samples had two sharp and strong diffraction peaks around 19.5° and 23.7°, indicating that SSPCMs had the same crystal structure with PEG. But the diffraction peak height of SSPCM-15 was lower than that of pure PEG 1500, which was resulted by the reduced crystal region of PEG fragment after cross-link reaction. The diffraction peak of SSPCM-8 was declined significantly compared with that of SSPCM-15, which was attributed to the short molecular chain of PEG 800. Furthermore, the changes of molecular chain in PEG led to the reduction of crystalline region in synthetic material.

3.3. Microstructures of wood and PCESW-15

Figure 5 shows the microstructure of pure wood, delignified wood and PCESW-15. Delignified wood (figure 5(b)) had a honeycomb-like structure as same as pure wood (figure 5(a)), but its porosity was higher and a little deformation appeared after the lignin removal. This loose structure of delignified wood provided more potential for SSPCM-15 impregnation. Figures 5(d)–(e) shows the cross and radial section SEM images of PCESW-15. It is clear that SSPCM-15 had got into the delignified wood successfully and filled the wood cells.
While it just distributed in most big vessels rather than all wood cells due to the long molecular chains of SSPCM-15. And this may result to a heterogeneous thermal transfer speed during heating or cooling process.

3.4. Phase change properties of SSPCMs and PCESW-15

The DSC curves of PEGs, SSPCMs and PCESW-15 are shown in figure 6. The specific parameters are summarized in table 2. As can be seen in figure 6, PEGs and SSPCMs showed similar endothermic and exothermic peaks in the range of 5 °C–55 °C, which indicated that the phase change temperatures of SSPCMs were mainly influenced by PEGs. SSPCMs showed lower latent heats in the freezing/melting process than corresponding PEGs because of the crystalline regions of PEG segments were limited by the crosslinking structure of SSPCM, this result is similar to the previous conclusion reported [33–35].

Natural wood had no freezing/melting peaks, but it appeared after the combination with SSPCM, indicating that the heat storage ability of PCESW was mainly benefited from PCMs. However, both of freezing/melting latent heat (51.92 J g⁻¹, 58.24 J g⁻¹) and temperature (24.1 °C, 36.1 °C) of PCESW-15 were lower than that of SSPCM-15, this reason could be attributed to SSPCM-15 was the only working substance in composite phase
change material and wood has no contribution on enthalpy. SSPCM-15 was a polymer with long chains, it would be broken after incorporated with wood and the destroyed crystal structure led to a low phase change temperature.

3.5. Thermal stability of SSPCMs and PCESSW-15

The TG and derivative thermogravimetric (DTG) test results are shown in figure 7. As can be seen in figure 7(b), PEG just had one weight loss stage in the temperature range of 300 °C–410 °C, while SSPCM-15 and PCESSW-15 had two stages. SSPCM-15 appeared the degradation in the range of 270 °C–435 °C, the first step due to the decomposition of urethane bonds, and the second was belonging to the thermal degradation of the PEG chains. The degradation temperature range of PCESSW-15 occurred from 240 °C to 450 °C. The first step was corresponding to the decomposition of urethane bonds and hemicelluloses in wood, and the second was the thermal degradation of PEG chains and cellulose. There was nearly no weight loss of PCESSW-15 under the temperature of 240 °C, which indicated that the composites had an excellent thermal energy storage stability when used at room temperature.

3.6. Thermal durability of the PCESW

The DSC curves of PCESSW-15 before and after 100 thermal cycling are shown in figure 8. The specific parameters are summarized in table 3. Both of freezing/melting latent heat and temperature of PCESSW just changed a little compared with the freezing/melting latent heat and temperature before 100 thermal cycling, which are unnoteworthy for thermal energy storage applications. These results showed that the synthesized PCESSW had good thermal durability in terms of phase transition properties after thermal cycles.

3.7. Curves of temperature-time change in two house models

The practical thermal performance of PCESSW is shown in figure 9. It is obvious that the temperature change of PCESSW-15 changed slower than control group, indicating that PCESSW-15 had a good temperature regulating ability. In the heating process, there was an obvious temperature platform in the curves of PCESSW-15 which lasted about 13 min in the range of 30 °C–39 °C. This was corresponded to the phase change process of SSPCM. In this stage, PCESSW absorbed a lot of heat and prevented the increase of temperature, so there presented a constant temperature stage. And in cooling process, it also appeared in the range of 21 °C–26 °C and sustained about 16 min. So the superior thermal performance of PCESSW-15 means that it can be used as a temperature regulating material in buildings.

![Figure 7. TG (a) and DTG (b) curves of PEG 1500, SSPCM-15, delignified wood and PCESSW-15.](image)

| Sample   | Phase transition | $\Delta H_m$ (J g$^{-1}$) | $T_m$ (°C) | $\Delta H_f$ (J g$^{-1}$) | $T_f$ (°C) |
|----------|------------------|--------------------------|------------|--------------------------|------------|
| A-PCESW-15 | solid–solid     | 58.24                    | 36.1       | 51.92                    | 24.1       |
| B-PCESW-15 | solid–solid     | 57.82                    | 37.3       | 51.47                    | 27.6       |

Note: A is before 100 thermal cycles; B is after 100 thermal cycles.
4. Conclusions

In this study, a kind of phase change energy storage wood was synthesized successfully by using PEG 1500 as the phase change material and Balsa wood as the carrier. DSC test indicated that SSPCM-15 and PCESW-15 had a suitable phase change temperature and high freezing/melting enthalpy. SEM images showed that the delignified wood had a loose porous structure and SSPCM was mainly distributed in the vessels. TG test indicated the SSPCM-15 and PCESW-15 had nice thermal stability when used at room temperature. Thermal cycling test showed that PCESW-15 had good thermal durability. And the practical thermal performance of PCESW-15 demonstrated that it had an outstanding thermal performance when suffered temperature change. In view of the great thermal performance of PCESW-15, we suggest it as an energy-saving building materials for regulating indoor temperature.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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