Fabrication and Properties of Polyethylene Glycol-Modified Wood Composite for Energy Storage and Conversion

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Green fir wood (*Pseudotsuga menziesii*) was modified with polyethylene glycol (PEG) to produce wood composites for energy storage and conversion. The PEG-modified wood composites were evaluated based on their dimensional stability, durability, and thermal properties by various analytical methods. The differential scanning calorimetry (DSC) results showed the melting temperature and the latent heat of the phase change material (PCM) composite were 26.74 °C and 73.59 J/g, respectively. Thermal cycling tests and thermogravimetric analysis confirmed the composite exhibited good thermal stability, reliability, and chemical stability. All treated specimens were free from noticeable defects, and the addition of a surface varnish coating prevented PEG from leaching. The PEG-modified composites exhibited improved dimensional and thermal performance, which makes this material a potential candidate for economical and green, lightweight building materials.

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INTRODUCTION

Wood is a natural polymer composed of cellulose, hemicellulose, and lignin. It exhibits a high capacity for water absorption and desorption because of the quantity of hydroxyl groups present in the polymer (Fadl and Basta 2005). As a result, wood shrinks as it loses moisture and swells as it absorbs moisture. Defects, such as warping, may occur in the wood when exposed to environmental conditions with repeated fluctuations in relative humidity and temperature, which can reduce the service life of wood products.

To improve the dimensional stability and durability of natural wood, various methods of modifications have been utilized (Fadl and Basta 2005; Nicholson and Hoffman 2006; Park and Wilderman 2010), including physical treatment and chemical modification (Avramidis *et al*. 2011). Physical modification can be achieved through thermal treatment, which imparts excellent performance in water repellency and prevents the wood from deformation. Nevertheless, the large-scale commercial application of this method is limited because of the weakening of the wood's strength, the unsatisfactory dark color that results, and the high consumption of energy (Zhang *et al*. 2007a). Alternatively, practical chemical modification involves the treatment of wood with polymers, resins, or other functional agents to either fill in voids within the wood or trigger chemical reactions to boost the

dimensional stability of wood. The latter is banned in many countries because of its harmful effects on the environment (Chen *et al*. 2008); noxious wastewater is released during processing, resulting from chemical leakage.

Impregnation with polyethylene glycol (PEG) is a novel modification method of wood and is quickly evolving in the biomass research field. Polyethylene glycol is a latent heat storage resource that serves as a stabilizer and bulking agent. Latent heat is the energy released or absorbed during phase change, so the temperature in living space could be accommodated. Its impregnation is widely utilized in several fields because it adds to the dimensional stability wood product fabrication, archeological artifact preservation and imported carving protection. When green or air-dried wood is continuously soaked in PEG solution, the wood will not significantly shrink if dried during a later stage of processing. Furthermore, wood treated with PEG followed and dried slightly swells when exposed to high humidity. Environmentally, PEG is nontoxic, noncorrosive, odorless, and colorless (Mitchell and Wahlgren 1959; Juliane *et al*. 2014). Hence, it has been recommended for use as a favorable thermal energy storage material because it possesses a suitable phase change temperature, high latent heat capacity, and minimal volume change during the solid-liquid phase. In addition, PEG has high thermal and chemical stability after long-term service time (Karaman and Karaipekli 2011).

With a mean molecular weight of 1000, PEG dissolves easily in warm water and can be incorporated into porous materials through diffusion into fine structures (Ohkoshi 2002; Mortensen *et al*. 2007). The latter physically bulks the wood cell walls and fibers, thus preventing inner shrinkage and eliminating the development of destructive stress. The large molecular structure also displaces moisture in the lattice-like structure of wood fibers (Bjurhager *et al*. 2010).

Douglas-fir-based wood, abundantly available in North American, is broadly recognized by architects and engineers as one of the most utilized structural lumber for housing and building because of its dimensional stability, superior strength-to-weight ratio, and high specific gravity. Although some previous PEG modification studies have investigated the anti-shrinkage behavior of commercial and lab-made wood panels from Douglas-fir, however, fewer researchers have focused on evaluating the water uptake behavior of PEG-treated fast-growing timber and its phase change applications in light wood frame materials. Therefore, the objective of this paper was to examine the effect of treatment duration and PEG concentration on various parameters of wood, including the weight percentage gain (WPG), dimensional stability, thermal properties, and reliability.

EXPERIMENTAL

Materials

Green wood (*Pseudotsuga menziesii*) was collected from Guangxi Province, China. The wood was 13 years old, with a diameter of 13.6 cm and mean initial moisture content (MC) of 72.14% to 88.72%. The specimens were sawn into samples with dimensions of 20 mm^3. Inclusion criteria for the samples were as follows: normal color, knot-free, and straight grain. Polyethylene glycol, with a molecular weight of 1000 g and melting point of 40 to 45 °C, was supplied by the E-KEAR Biotechnology Co., Shanghai, China. Polyurethane (PU) varnish was provided by the Jinke Technology Company, Ltd., Beijing, China.

Methods

Preparation of wood composites

To ensure proper penetration of the stabilizing agent into wood, the freshly cut test samples were wrapped in plastic membranes and immersed in distilled water to maintain the green condition before impregnation. The PEG-1000 powder was dissolved in distilled water at 40%, 60%, and 80% concentrations by weight. The mixtures were automatically stirred for 5 min and kept at 30 °C for 1.5 h in an electric-heated thermostatic water-bath. Three sets of marked specimens were simultaneously soaked in solution at room temperature (26.8 °C) and at atmospheric condition (1.015×10^5 Pa). The duration of impregnation lasted 2, 4, and 8 d, and there were three replicates per treatment group. After the impregnation period, specimens were towel-dried and then oven-dried at 36 °C at a constant temperature for 48 h. The dimensions in the tangential, radial, and longitudinal directions of the PEG-modified wood composites were recorded before and after immersion. The linear and volumetric shrinkage ratios were calculated in accordance with the method for determination of the shrinkage of wood (GB/T 1932 2009).

Thermal properties analysis

The surface morphological properties of the PEG-modified wood composites were studied by scanning electron microscopy (SEM; Questar, 450 Quanta^{TM}, America), and thermal properties were investigated by synchronous thermal analyzer (STA; STA 449 F3 Jupiter$^®$, Netzsch Co., Japan), respectively. All of the measurements were made at a constant heating and cooling rate of 10 °C/min, a flow rate of 20 mL/min, and constant nitrogen stream at atmospheric pressure. The temperature accuracy was estimated to ± 0.01 °C, and the heat flow repeatability was set to 0.2 μW. The onset temperature of differential scanning calorimetry (DSC) was calculated by drawing a line at the maximum slope of the leading edge of the DSC peak and extrapolating to baseline. For the melting and freezing points measurements, both temperatures were onset points of the heating and cooling peaks, respectively (Karaipekli and Sari 2008). The latent heat during the phase change was determined by numerical integration of the area under the peaks. The thermal stability of the PEG-modified wood composites was examined by thermal gravimetric analysis (TGA; Pyris6; Perkin Elmer). The analyzer was calibrated with calcium oxalate, and the analyses were conducted under a static air atmosphere at 30 to 600 °C and a heating rate of 5 °C/min.

Thermal cycling test and thermal performance

To determine the thermal reliability of the PEG-modified wood composites with respect to changes in the latent heat and phase temperature, thermal cycling tests were consecutively performed at up to 400 cycles using a synchronous thermal analyzer. To evaluate the thermal and chemical stability of the phase change material (PCM), DSC and TG analyses were repeated over various temperatures and recorded at the melting and freezing phases.

To investigate the thermal performance of the PCM, PEG-modified wood composites wallboards were molded into 100 mm × 100 mm × 10 mm composites. In the control group, ordinary untreated Douglas-fir wallboards were also molded. Initially, the molds were stored at room temperature for 24 h and then dried at 36 °C for 48 h. The test cells were separately assembled using the wallboards, with two halogen tungsten lamps (250 W) as the heat source. The temperature variation at the cell wall inner, center, and outer surfaces was measured by thermocouples (Pt-RhPt), with an accuracy of ± 0.18 °C.

Because wood exhibits a relatively high permeability to water, experiments were conducted to determine whether a varnish finish could serve as a semi-permeable membrane to retain the PEG-1000 within the composites under leaching conditions. The specimens were treated with a 60 wt.% PEG-1000 solution for 4 d. The treated and control samples were modified with a PU varnish coating. After drying, the specimens were weighed and fully immersed in water for 2 wks. The treatment groups were impregnated/coated, unimpregnated/coated, and unimpregnated/uncoated.

RESULTS AND DISCUSSION

Dimensional Stability

The SEM images of the samples before and after impregnation are shown in Fig. 1. The majority of lumens and voids within the composites were filled, and the micro-voids in the wood structure were filled with PEG. This indicates that the atmospheric impregnation was adequate for exchanging with interior water and producing a satisfactory bulking effect.
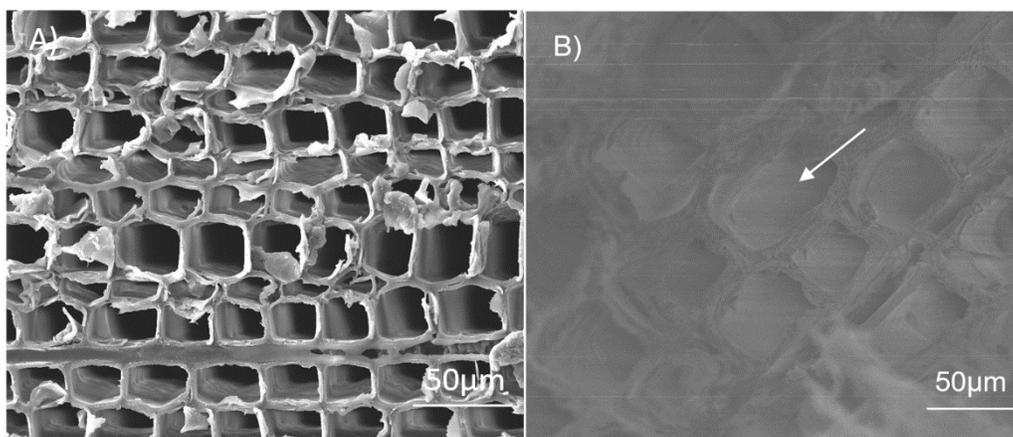
Fig. 1. Scanning electron microscope images of the sample A) before and B) after PEG-1000 impregnation

Table 1 shows the effect of treatment time and PEG concentration on the average weight percentage gain (WPG). The maximum amount of PEG reached 36.18 wt.% and remained stable during the tests; therefore, this composite was selected as the PCM stable-form. After 4 d of impregnation, the WPG of the treated samples increased by 24.14%, 34.25%, and 33.90% with 40 wt.%, 60 wt.%, and 80 wt.% PEG solutions, respectively. Furthermore, when treated with 60 wt.% PEG, the WPG increased by 27.12%, 34.25%, and 36.18% after treatment impregnation periods of 2, 4, and 8 d, respectively. The WPG rose rapidly during the initial 4 d and then slowly declined thereafter. In addition, it was found that simply increasing the PEG concentration did not necessarily raise the PEG absorption of the wood. This indicated that the wood structure was fully impregnated with PEG to saturation in 4 d of impregnation, especially for samples immersed in highly concentrated PEG solutions. For the latter, solid PEG may form on the wood's surface, blocking the passage of further amounts of PEG into the interior of the wood.

Table 1. Effect of Time and PEG Amount on Weight Percentage Gain (WPG)

Treatment Time (days)	WPG (%)		
	PEG 40 wt%	PEG 60 wt%	PEG 80 wt%
0	0	0	0
2	17.21	27.12	29.83
4	24.14	34.25	33.90
8	27.89	36.18	34.36
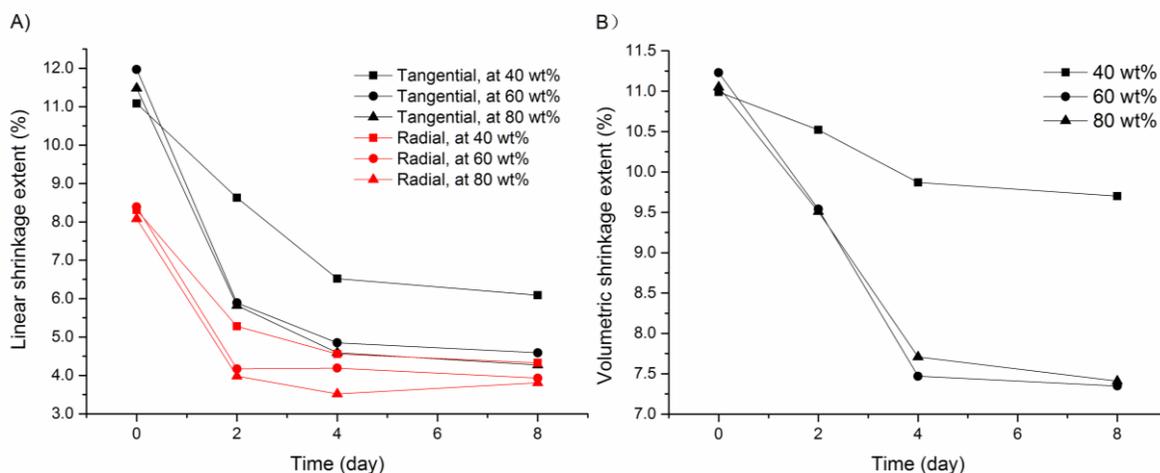
Fig. 2. Relationship between the impregnation treatment duration at different PEG-1000 concentrations with A) linear and B) volumetric shrinkage ratios

The relationships between the treatment periods at various PEG concentrations as a function of the linear and volumetric shrinkage ratios are depicted in Fig. 2. The linear and volumetric shrinkage ratios decreased rapidly during the first 4 d, and then remained fairly constant thereafter. In comparison with the control group at 2, 4, and 8 d, the tangential shrinkage ratio of the treated samples decreased by 49.30%, 60.02%, and 62.80%, respectively, and the radial shrinkage ratio decreased by 50.74%, 56.44%, and 52.85%, respectively. The volumetric shrinkage ratio declined by 34.55%, and the linear and volumetric shrinkages ratios notably declined with increasing PEG concentration, then reached a steady-state at the 60 wt.% PEG concentration.

Impregnation of the wood with PEG efficiently reduced shrinkage and enhanced the dimensional stability. Shrinkage of the cellular walls under humid conditions was sufficiently minimized after the voids and lumens were filled with solid PEG. The PEG served as an excellent bulking medium for the wood structure (Håfors 1990; Rowell and Barbour 1990). In addition, the deposition of solid PEG in the cell walls inhibited the outward movement of water molecules, decreasing both the linear and volumetric shrinkage rates and improving the dimensional stability of the wood composites (Moré 1968; Wallström and Lindberg 1995). After impregnation, the PEG penetrated the cell walls and then successively provided a thin, semipermeable layer to prevent the movement of water molecules inside. As a result, heavily bulking composites with PEG retains their indefinite dimensions to prevent wood from critical shrinkage, swelling, or warping, regardless of the atmospheric humidity (Stamm 1964).

Thermal Properties of Wood Composite

Figure 3 displays the DSC curves of pure PEG and the stable-form PEG/Douglas-fir composite. The melting and freezing temperatures for pure PEG were 29.81 and 34.23 °C, respectively, and 26.74 and 36.14 °C, respectively, for the form-stable composite. The physical interaction between PEG and the inner wood structure might be responsible for the slight difference in the phase change temperatures.

Phase change material composites, with phase transition temperatures of 18 to 26 °C, are feasible for heat-regulated building utilization (Khudhair and Farid 2004; Zhang *et al*. 2007b). The prepared composites exhibited phase change temperatures ranging from 27 to 36 °C, which was larger than that corresponding to human comfort. However, these composites can still be utilized as thermal energy storage material for exterior wallboard in the wood frame, which would absorb heat from the surrounding air and solar radiation during daytime, subsequently releasing stored heat back into the room overnight. This would achieve considerable energy conservation, especially in cold countries.

PEG
melting point: 29.81℃
freezing point: 34.23℃

Composite
melting point: 26.74℃
freezing point: 36.14℃
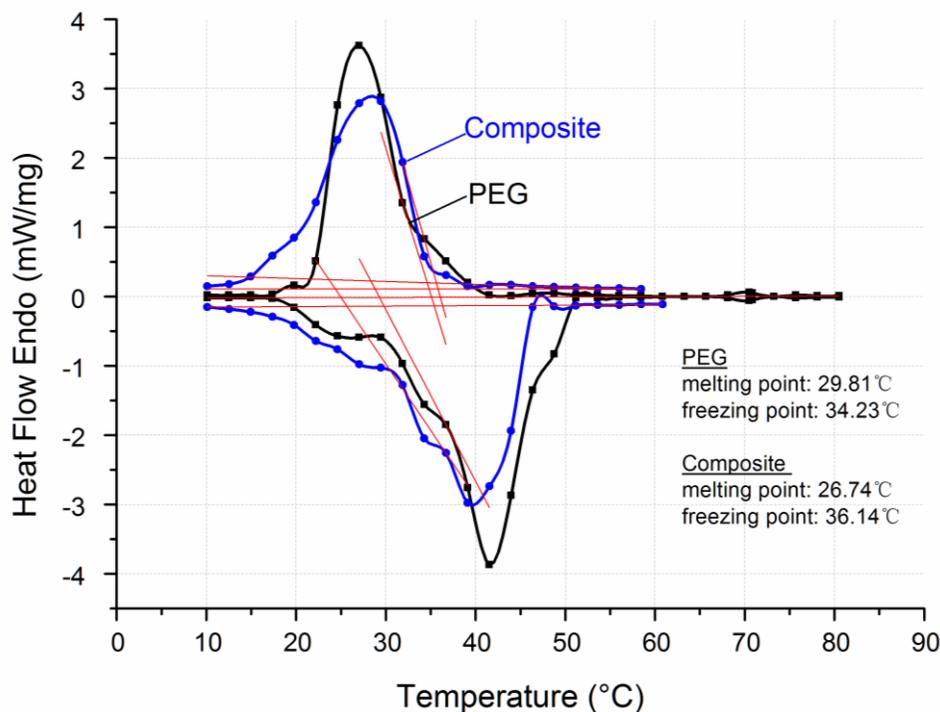
Fig. 3. Differential scanning calorimetry curves of PEG and the stable-form PEG/Douglas-fir composite

Table 2 shows a comparison of the thermal properties of the stable-form PCM composite with the phase change composites in the literature. It should be noted that the melting and freezing latent heats were 128.98 to 157.82 J/g, respectively, for pure PEG, and 73.59 to -85.65 J/g, respectively, for the stable-form PEG/Douglas-fir composite. The results show that the PEG-modified composite is suitable for heat storage application in wooden buildings.

Table 2. Thermal Energy Storage Characteristics of Phase Change Material Composites

Composite PCM	Melting point(℃)	Freezing point (℃)	Latent heat (J/g)	Reference
Butyl stearate (25–30 wt%)/gypsum	18.0	21.0	30.0	[Feldman *et al.* 1995]
Capric–lauric acid+fire retardant (25–30wt%)/gypsum	17.0	21.0	28.0	[Feldman *et al.* 1995]
Dodecanol/pumice concrete block	12.0	14.9	12.7	[Feldman *et al.* 1995]
Lauric–stearic acid (38 wt%)/gypsum	34.0	--	50.3	[Feldman *et al.* 1995]
Propyl palmitate (25–30 wt%)/gypsum	19.0	16.0	40.0	[San *et al.* 2008]
Dodecanol (25–30 wt%)/gypsum	20.0	21.0	17.0	[San *et al.* 2008]
Methyl palmitate–stearate (26.6 wt%)/wallboard	22.5	23.8	41.1	[San *et al.* 2008]
Capric–lauric acid (26 wt%)/gypsum	19.1	--	35.2	[Karaipekli 2007]
Capric–stearic acid (25 wt%)/gypsum	23.8	23.9	49.0	[Shilei *et al.* 2006]
Capric–myristic acid(20 wt%)/VMT	19.8	17.1	27.0	[Karaipekli 2009]
RT20/montmorillonite	20.8	--	53.6	[Feldman *et al.* 1995]
Capric–palmitic acid (25 wt%)/gypsum	22.9	21.7	42.5	[Sari *et al.* 2008]
n-nonadecane (50 wt%)/cement	31.9	31.8	69.1	[Karaipekli 2007]
Emerest2326/gypsum	16.9	19.3	35.0	[Karaipekli 2009]
Capric–myristic acid (55 wt%)/expanded perlite	21.7	20.7	85.4	[Karaipekli 2008]
Capric–myristic acid (25 wt%)/gypsum	21.1	21.4	36.2	[Karaipekli 2008]
PEG(60 wt% 4 days)/Douglas-fir*	26.7	36.1	73.6	**Present study**

Thermal Cycling Reliability

During the service life of wood composites, it is necessary for PCM composites to maintain stability in terms of thermal and chemical properties over a great number of melting and freezing cycles. Thermal cycling tests were performed to examine the relationship between the thermal properties and chemical structure of the stable-form PEG/Douglas-fir composites as a function of the number of thermal cycles.

The DSC curves of the stable-form PEG/Douglas-fir composite, before and after thermal cycling, are shown in Fig. 4. After 400 thermal cycles, the melting temperature of PEG varied minimally by 2.54 °C and the freezing temperature by 1.39 °C. This could be caused by the porous structure that blocks the heat transfer within the composites. The changes in the melting and freezing temperatures after thermal cycling are minimal, which qualifies its utilization for thermal energy storage.

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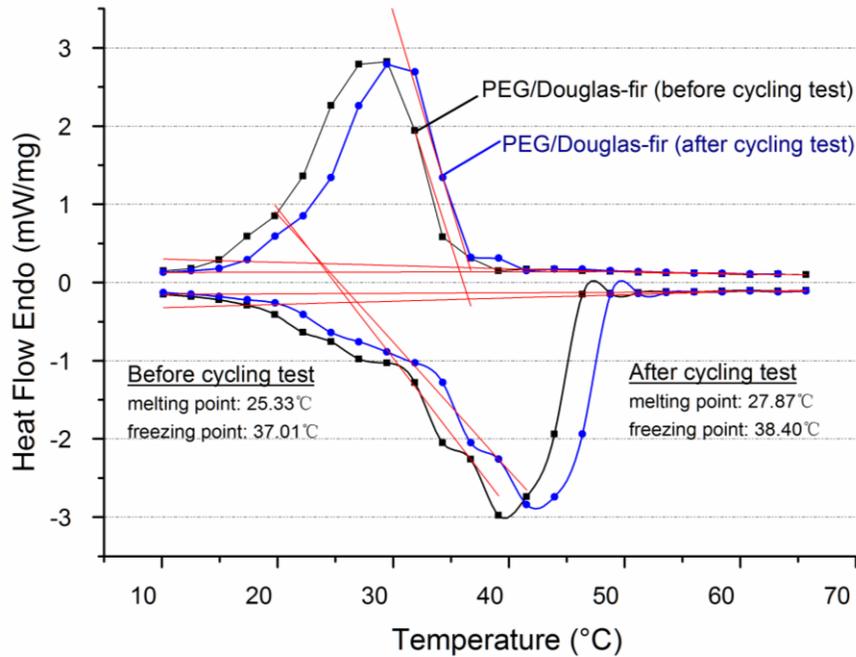
Fig. 4. Differential scanning calorimetry curves of stable-from PEG/Douglas-fir composite before and after thermal cycling

The effects of heat cycling on latent heat production in the PEG/Douglas-fir composite during the cycling test are shown in Fig. 5. After 400 repeated thermal cycles, the latent heat of the melting phase decreased by 9.06% and the latent heat of the freezing phase decreased by 8.44%. Also, it could be seen that the latent heat slowly dropped during the first 200 cycles and then stabilized at approximately 145.0 J/g. The decline in latent heat was probably caused by thermal expansion, as well as leakage of PEG from the surface and in the exterior pores during the melting process. Hence, it could be concluded that the PEG/ Douglas-fir PCM composite exhibited good thermal stability.
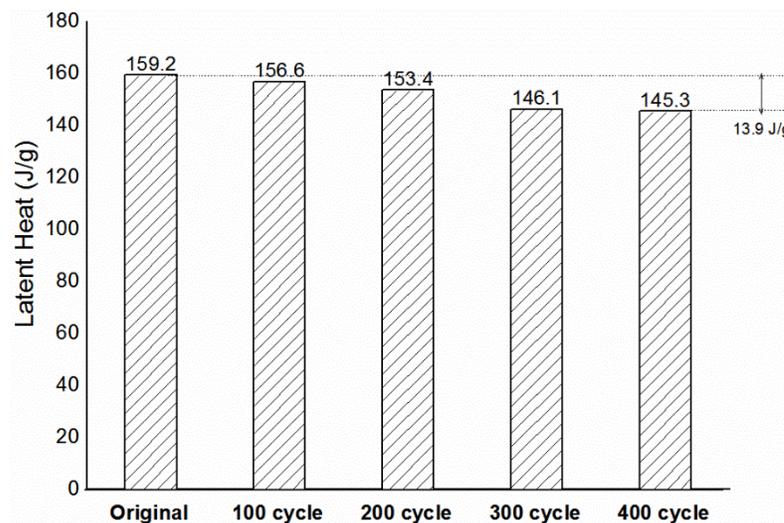
Fig. 5. Changes in latent heat of the PEG/Douglas-fir composite during cycling test

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Figure 6 shows a comparison of the TG curves of pure PEG and PEG/Douglas-fir composite before and after thermal cycling. The PEG/Douglas-fir composite degraded over the temperature range of 245 to 430 °C before thermal cycling and from 238 to 427 °C after thermal cycling. Conversely, degradation of pure PEG began at 265 °C and ended at 410 °C. The change in thermal gravity was minimal (17 to 27 °C), which indicates that after the impregnation of PEG, Douglas-fir composite exhibited favorable thermal stability after 400 thermal cycles.
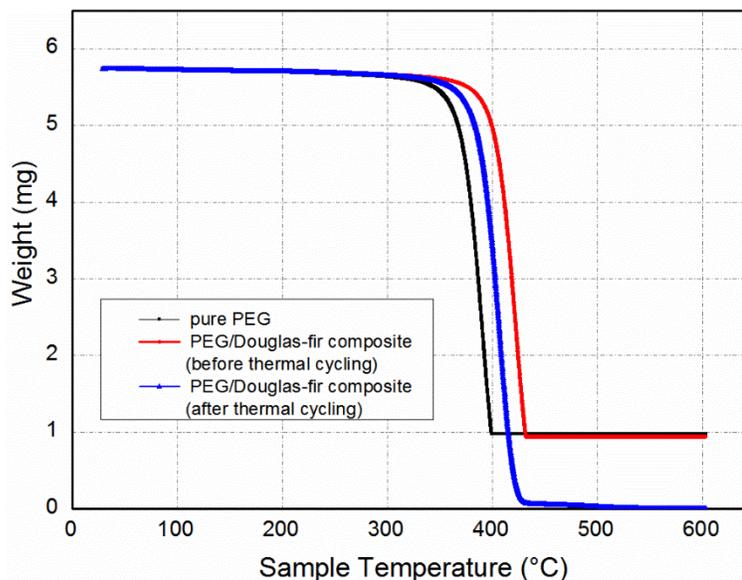
Fig. 6. Thermogravimetric analysis curves of PEG and the PEG/Douglas-fir composite before and after thermal cycling

Thermal Energy Storage Performance

Figure 7 illustrates the variation in temperature at the inner and outer wall surfaces of the fabricated test cells during the heat loading and cooling processes. During the heating period, a temperature difference of 3 °C was observed between the optimal inner surface temperatures of the cell fabricated by Douglas-fir wallboard and that of the composite. This suggests that a portion of the loading heat was absorbed by PEG. Conversely, the maximum indoor temperature of the Douglas-fir cell and the composite cell were approximately 29 and 27 °C, respectively. The latter validates the feasibility of PEG/Douglas-fir wallboard for the purpose of cooling peak load reduction and effective energy storage. Additionally, the thermal performance of the stable-form PEG/Douglas-fir composite in the simple cell was better than that of the various PCM composites in the literature (Shilei *et al*. 2006; Fang *et al*. 2008; San *et al*. 2008).
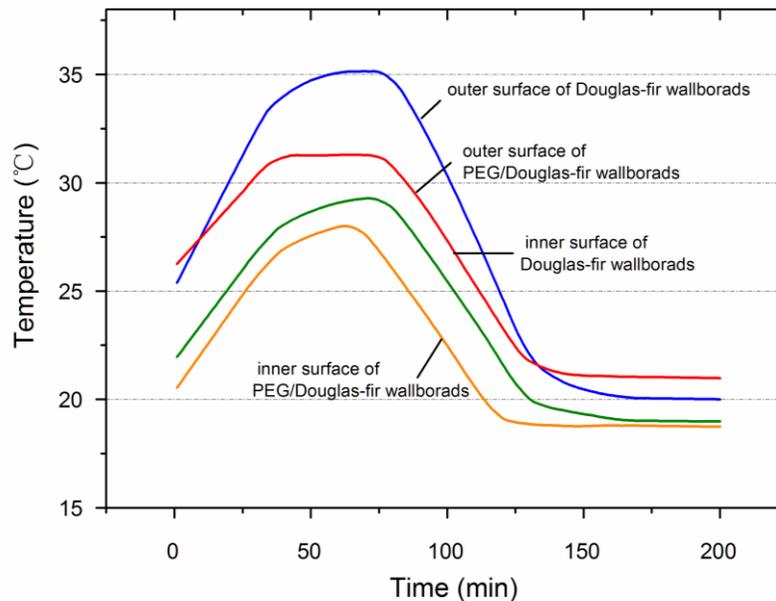
Fig. 7. Temperature variations at the inner and outer surfaces of the front walls of test cells

In terms of wood durability, tests performed after 60 d revealed that the wood samples retained their dimensions. The dimensions of the composites did not deteriorate substantially and the PEG did not leach out (Mortensen and Hvilsted 2009). Three weeks after the tests, the specimens were placed in a climate chamber with 90% relative humidity and then transferred to a 65% relative humidity room. Two days after the transfer, all untreated controls began to develop V-shaped checks. Meanwhile, the samples soaked in 40 wt.% PEG solution developed slight internal radial checks that did not extend into the periphery and all of these composites were free of observable checks.

Table 3. Weight Gain of Leached Test Samples

Weight increase (%)	Treated and coated	Untreated but coated	Untreated and uncoated
One week	1.5	29.7	38.9
Two weeks	2.6	53.2	57.8

Table 3 lists the weight gains of leached test samples after being washed for 2 weeks. The average increase in weight of the unimpregnated/nonsurface-coated specimens after 2 weeks was 57.8%, and that of the unimpregnated/surface-coated specimens was 53.2%. Conversely, the mean increase in weight of the impregnated/surface-coated specimens was 2.6%, indicating less deformation. Under the present experimental conditions, the PEG-1000 did not leach out to any measurable degree. Consequently, specimens were oven-dried, weighed, and measured. The impregnated and surface-coated specimens lost less than 15% of the initial PEG content. The unimpregnated/uncoated specimens blistered in drying. Hence, even thought that PEG could leach out from the inner

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wood structure, the coating on the surface of the specimens retained most of the PEG in the composites.

These findings provide a reference for further study involving dimensionally stable wood structure and high-efficiency green energy storage building materials. In the future, the mechanical performance and regulation effects of composites for building applications under different climates conditions should be investigated.

CONCLUSIONS

1. Green Douglas fir (*Pseudotsuga menziesii*) was impregnated with PEG under atmospheric conditions. The melting and freezing temperatures and latent heat of the stable-form composites were measured at 26.74, 36.14, and 73.59 J/g, respectively. The thermal cycling test indicated that the stable-form composite exhibited satisfactory thermal and chemical reliability, with respect to changes in thermal properties and chemical structure after 400 melting and freezing cycles. The thermogravimetric analysis showed that PEG within the composite displayed good thermal stability as the pure PEG, even after 400 thermal cycles.

2. A portion of the loading heat was absorbed by PEG inside of the Douglas-fir composite, and the maximum indoor center temperature was recorded at 27.45 °C in the composite cell. The thermal performance test showed that the fabricated PCM composite could be applied in fields, such as temperature room regulation and overheating accommodation in wood frame building.

3. The leaching test revealed that PEG impregnated and surface-coated specimens, exhibiting less than 15% in PEG loss. Therefore, PEG did not leach out of the composites to any measurable degree, and the addition of the surface varnish coating retained most of the PEG in the wood.

ACKNOWLEDGMENTS

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