Investigation of the Interfacial Compatibility of PEG and Thermally Modified Wood Flour/Polypropylene Composites Using the Stress Relaxation Approach

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Stress relaxation has been proven to be a good measure for studying the interaction between the constituents in wood flour/polymer composites by evaluating the internal bonding quantitatively. In order to investigate the combination effect of polyethylene glycol (PEG) impregnation and heat treatment on the interfacial compatibility of wood flour/polypropylene composites, the stress relaxation of PEG and/or thermally modified wood flour/polypropylene composites was determined at three temperatures (26, 40, and 60 °C). The apparent activation energy (ΔE) was also calculated according to Eyring's absolute rate reaction theory. The results showed that PEG treatment accelerated the stress relaxation rate of the composites and decreased the ΔE. However, heat treatment resulted in an alleviation of the increasing rate of stress relaxation caused by PEG modification and an increase in the ΔE of the composites. These results suggested that PEG treatment had a negative effect on the interfacial compatibility between wood flour and polypropylene in the composites, and heat treatment could compensate for this effect to a certain extent.

Keywords: Wood flour/polypropylene (PP); Polyethylene glycol (PEG); Thermal treatment; Interfacial compatibility; Stress relaxation

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INTRODUCTION

Incorporation of natural fibers into a polymeric matrix to produce stiff composite materials has received considerable attention from both academic and industrial quarters in recent years. Wood fibers are attractive reinforcements for thermoplastic polymers due to their low cost, low density, high specific strength, and stiffness (Ashori and Nourbakhsh 2008). Compared with plastic, these composites are more biodegradable, renewable, economically efficient, and easier to machine (Saheb and Jog 1999). However, wood fibers are not problem-free since they contain large amounts of hydroxyl groups, which make them hydrophilic and therefore incompatible with hydrophobic thermoplastics (Kabir *et al.* 2012). This incompatibility produces poor interfacial adhesion between the matrix and filler, which results in the reduction of mechanical properties because stress cannot be transferred properly from the matrix to the fibers (Kazayawok *et al*. 1999). Additionally, the incompatibility provides pathways for moisture uptake and biological attack (Pendleton *et al*. 2002). Therefore, finding a way to decrease the hydrophilic character of wood fibers by blocking or modifying their hydroxyl groups might make them more compatible with thermoplastics and improve the properties of composites.

Up to the present, the use of coupling agents such as silane (Abdelmouleh *et al*. 2007) and maleic anhydride grafted polypropylene copolymer (Arbelaiz *et al*. 2005) has been the most common way to improve compatibility. In addition, various modification methods for wood fibers such as acetylation (Segerholm *et al*. 2012), alkali treatment (Islam *et al*. 2012), hemicellulose extraction (Hosseinaei et al. 2012), and heat treatment (Ayrilmis *et al.* 2011) have been used to try to improve the interfacial bonding between wood fibers and polymers. The effectiveness of these approaches for improving interfacial compatibility has been checked by a variety of techniques. For example, scanning electron micrographs (SEM) were used to observe the fracture surfaces of wood fibre–polypropylene composites (Yuan *et al*. 2004). Dynamic mechanical analysis (DMA) was used to obtain the viscoelastic properties of wood flour/polymer composites, and the interaction between wood powder and polymer in the composites could be analyzed based on the position of the primary peaks in the tan δ curve (Qin *et al*. 2005). X-ray photoelectron spectroscopy (XPS) was used to investigate the reactivity of different coupling agents with surface hydroxyl groups on fibrous cellulose (Nakatani *et al*. 2011).

Stress relaxation is a viscoelastic response where for a fixed strain the resultant stress decays with time. Wood is a viscoelastic material and it exhibits time-dependent mechanical behavior such as stress relaxation (Wang *et al*. 2011). Previous literature shows that the stress relaxation approach has been applied to study the rheological behaviour of chemically treated woods such as preservative-treated wood (Cao *et al*. 2006; Yu *et al*. 2010), glycerine impregnated wood (Yan *et al*. 2011), and dimethylsulfoxide-swollen wood (Xie and Zhao 2004). This approach has proven successful for analysis of the interaction between the wood components and wood-modifying agents. Stress relaxation is not only useful for investigating the interaction between wood and wood-modifying chemicals, but is also an indicator of the interface properties of natural fiber-plastics composites. Varghese *et al*. (1994) found that the stress relaxation process in sisal fiber-nature rubber composites was influenced by the fiber-rubber interface. George *et al*. (1998) studied the stress relaxation behavior of pineapple fiber-reinforced polyethylene composites, and found that chemical treatment of fiber with coupling agents reduced the relaxation rate, which was associated with improved interfacial adhesion. Wang *et al*. (2012) measured compressive stress relaxation at three temperatures (26, 40, and 60 °C) and calculated the apparent activation energy (ΔE) to investigate the interfacial compatibility of wood flour/polypropylene composite. The results showed that ΔE of wood flour/polypropylene composites without a coupling agent at a wood content of 40% was the highest, suggesting that the best interfacial compatibility between wood and PP occurred at 40% wood content.

In a previous study, the combination of polyethylene glycol (PEG) impregnation and heat treatment was applied to modify wood flour to improve the dimensional stability and water resistance of wood flour/polypropylene composites (Luo *et al*. 2012). The compound modification of wood flour greatly reduced water uptake and decreased the thickness swelling of the composites, which is very promising considering the possible application of these composites in humid or water-contact environments. However, the flexural strength of the composites after modification was decreased evidently. Therefore, it is of interest to investigate the effect of PEG and/or thermal modifications on the interfacial compatibility between wood flour and polymer in the composites. In this study, two concentration levels of PEG (20% and 30%) and a heat treatment at 140 °C were used to modify the wood flour, then the modified wood flour was mixed with PP to prepare wood flour/PP composites for different treatments. The compressive stress relaxation curves of the composites were obtained at temperatures of 26, 40, and 60 °C, and thereafter the apparent activation energy was calculated for further investigation.

EXPERIMENTAL

Materials

Wood flour of poplar (*Populus tomentasa* Carr.) with a size over 100 mesh and polypropylene (PP) (K8303; Sinopec Chemical Products Sales Company, China) with a density of 0.9 g/cm^3 were used as the raw materials to prepare wood flour/PP composites. The PP, purchased from Beijing Yanshan Petrochemical (China), had a melting point around 165 °C and a melt flow index of 1.5 to 2.0 g/10 min at 230 °C. Polyethylene glycol (PEG) with an average molecular mass of 1000 and a melting point around 40 °C was purchased from Guangdong Xilong Chemistry (China). Polyfluotetraethylene (PTFE) membranes (270 × 270 × 3 mm) were used as demoulding materials to avoid sticking of the board during hot-pressing. Samples were sorted into six groups (Table 1).

Table 1. Test Groups of Wood Flour with Different PEG Modifications and Heat Treatments with a Wood Content of 40%

Labeling of Test Groups	Concentration of PEG Aqueous Solution (w/w %)	Heat Treatment (^{o}C)
C	0	—
CH	0	140
P2	20	—
P3	30	—
P2H	20	140
P3H	30	140

Treatment of Wood Flour

Wood flour was oven-dried and weighed before being immersed in different concentrations of PEG solution for 13 h. In this study two PEG concentration levels (20% and 30%) were used and labeled as P2, P3, with water-saturated fibers (C) as the control. After immersion, the wood flour was removed from the solution, air-dried at room temperature for 4 h, and then oven-dried at 105 ºC until constant weight. The samples in the groups of P2H, P3H, and CH were heated at 140 °C for 4 h in a modified drying oven connected to a water vapor supplying system, which used a big container to hold water and then produce water vapor as the protecting medium.

Preparation of PP/Wood Flour Composites

Wood flour/polypropylene composite panels with dimensions of 270 × 270 × 3 mm and a target density of 1.0 g/cm^3 were prepared on a lab-scale hot press. Prior to manufacturing, the wood flour was dried at 103 °C for 4 h. The pure or modified wood flour and PP were weighed at a ratio of 4:6 and then blended in a high-speed mixer. The mixture was then dried in an oven at 105±1 °C for 2 h and taken out for hand matting. A hot press (SYSMEN-II, made by the Chinese Academy of Forestry) was used to compress the mat at 180 °C and 4 MPa for 6 min. After hot-pressing, the formed mat was pressed at 4 MPa for another 6 min at room temperature in a cold press.

Stress Relaxation Tests

The compressive stress relaxation tests were performed on the self-assembled equipment composed of an electric system, a mechanical system (including the compression plates), and heating, cooling, and control systems. For details of the equipment, please refer to Cao *et al* (2006). The stress relaxation of these composites was studied at temperatures 26±1, 40±1, and 60±1 °C. The test for each sample of 10 × 10 × 3 mm (thickness) lasted 3000 s. The compressive strain is 20% of its original thickness. The average stress relaxation value based on tests on three specimens per test category was accepted as the representative result for that category.

RESULTS AND DISCUSSION

Stress Relaxation Analysis at Room Temperature

In order to eliminate the influence of initial stress, the compressive stress relaxation curves of the composites were expressed as the change in ratio of instantaneous stress $\sigma(t)$ to initial stress $\sigma(0)$ over time. As shown in Fig. 1, the stress relaxation of all samples increased with increasing time at room temperature. The most significant stress relaxation was observed at 30% PEG modified wood flour/PP composites, revealing that PEG modification increased the long-term deformation of wood flour/PP composites. After heat treatment, PEG modified wood flour/PP composites exhibited lower stress relaxation at three concentrations (0%, 20%, 30%) compared with those without heat treatment, indicating that heat treatment was a positive factor in inhibiting long-term deformation.

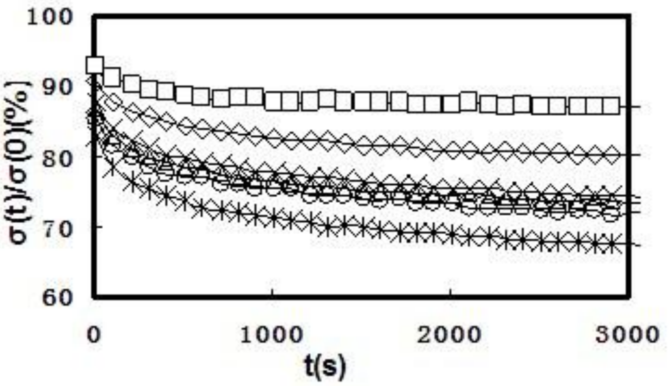

Fig. 1. Compressive stress relaxation curves of modified wood flour/PP composites at room temperature □: CH，◇: C，×: P2H，∆: P3H， ○: P2，*: P3

Stress Relaxation Analysis at Various Temperatures

The stress relaxation curves of wood flour/PP composites with different modifications at temperatures 26, 40, and 60 °C are shown in Fig. 2. The stress relaxation was more obvious at higher temperatures for all the samples, suggesting that modified wood flour/PP composites were sensitive to temperature. The stress relaxation results show an expected temperature dependence, a consequence of the PP phase in the composite.

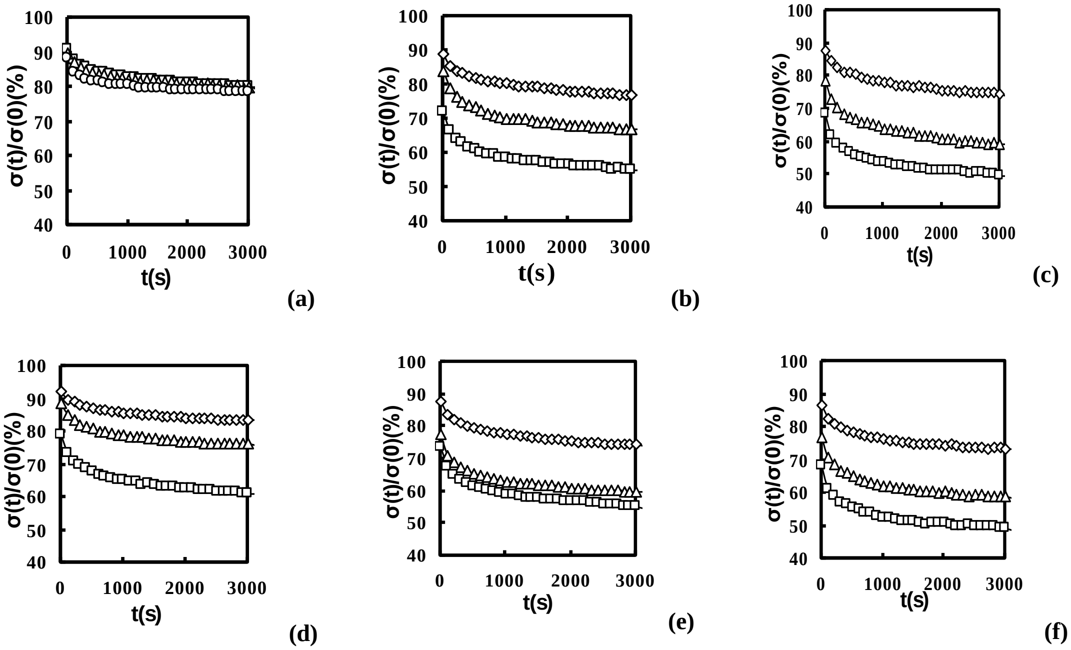

Fig. 2. Compressive stress relaxation of wood flour/polypropylene composites without a coupling agent at 26 °C (◇), 40 °C (Δ), and 60 °C (☐)
(a) C； (b) P2； (c) P3； (d) CH； (e) P2H； (f) P3H

In order to make it clearer, the rate of stress relaxation is defined as the slope of the double logarithmic curve of $\sigma(t)$ / σ(0) versus time (Cao *et al*. 2006; Yu *et al*. 2010; Wang *et al*. 2012). Before a relaxation time of 1000 s, the linear relationship was very clear and showed little sign of deviation. Therefore, the slope of the double logarithm curve of the stress relaxation curve (*t*: 10 to 1000 s) was finally taken as the rate of stress relaxation. As shown in Fig. 1, the stress decreased over time in an exponential trend, which corresponded to a linear relationship when plotted in the double logarithmic form. The rates of stress relaxation and the values of R^2 were calculated to investigate the effect of PEG modification and heat treatment on the relaxation behavior of composites. The results are shown in Table 2. It was found that R^2 values ranged from 0.96 to 0.99, suggesting a good linearity between the double logarithm of stress and time.

As shown in Table 2, the trend of stress relaxation rates of wood flour/PP composites at each temperature was consistent. At each temperature, the rate of stress relaxation increased with increasing PEG concentration, and the fastest relaxation appeared at the 30% PEG concentration without heat treatment. This might be due to the fact that numerous PEG molecules accumulated on the wood flour, resulting in a homogeneous interfacial structure and weak compatibility between the wood flour and the PP. It was also found that at the same PEG concentration, the rate of stress relaxation was slower after heat treatment. With temperature increasing, stress relaxed faster, and the increment in stress relaxation rates of thermally modified wood flour/PP composites at three PEG concentrations was less than that without heat treatment. For example, at the 30% PEG concentration, the rates of stress relaxation increased by 0.0291 as the temperature ranged from 26 °C to 60 °C. By contrast, after heat treatment the rates of stress relaxation increased by 0.0270 in the same temperature range. These results indicated that heat treatment could slow down the increase of stress relaxation rates caused by PEG modification.

Table 2. The Compressive Stress Relaxation Rates of Modified Wood Flour/PP Composites*

Labeling of Test Groups	Rate of Stress Relaxation ($\times 10^{-2}$)		
	26 °C	40 °C	60 °C
C	2.91(0.98*)	3.81(0.98)	4.95(0.97)
P2	3.98(0.96)	5.66(0.98)	6.93(0.99)
P3	4.89(0.99)	7.60(0.99)	7.80(0.98)
CH	2.84(0.98)	3.34(0.97)	4.45(0.97)
P2H	3.82(0.99)	5.15(0.99)	6.67(0.98)
P3H	4.34(0.98)	6.64(0.99)	7.04(0.98)

* The values in parentheses represent the R^2 of the regression linear curves of the double logarithm curve of $\sigma(t)$ / σ (0) versus t (10 ~ 1000 s).

Apparent Activation Energy Analysis

The apparent activation energy can be calculated to quantitatively evaluate the internal bonding according to Eyring's absolute rate reaction theory (Rawat *et al*. 1998). The relaxation time (τ_m) of each sample was defined as the corresponding time of the point that the stress relaxation curve started to parallel the abscissas, and the stress relaxation at each temperature has a corresponding relaxation time. A plot was made with the logarithm of the relaxation time ($\ln\tau_m$) as the ordinate and the reciprocal of the temperature ($1/T$) as the abscissa, and the apparent activation energy (ΔE) could be calculated from the rate of this curve according to Eq. 1,

$$\Delta E = R\frac{d\ln\tau_{m}}{d(1/T)} \tag{1}$$

where, τ_m is the average relaxation time (s); R is the gas constant, R = 8.314 J/(mol•K); and T is the absolute temperature (K). Higher apparent activation energy means the requirement of more energy to get the stress relaxed, which suggests a better internal bonding and compatibility between the molecules in the composites. As shown in Fig. 3, the ΔE value of modified wood flour/PP composites decreased as PEG was incorporated,

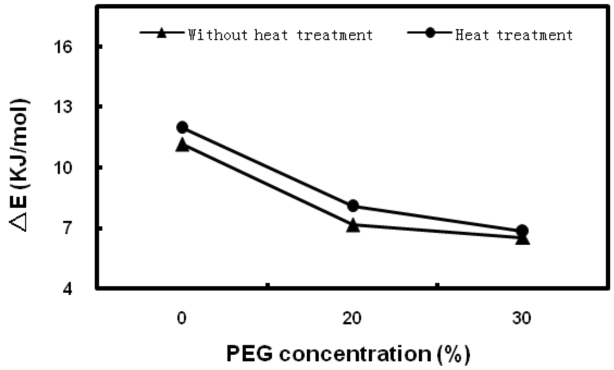

Fig. 3. The apparent activation energy of modified wood flour/PP composites

indicating that PEG modification had a negative effect on the interfacial compatibility between modified wood flour and PP. However, Δ*E* of composites after heat treatment was much higher than that without heat treatment, suggesting that the interfacial compatibility would be improved by heat treatment. It is known that heat treatment could decrease the hygroscopic properties of wood fiber (Tjeerdsma and Militz 2005). The improvement may partly be attributed to a more uniform dispersion of wood fibers, which leads to better interfacial bonding between wood flour and polymer, and eventually the improvement of the mechanical properties of the composites.

DISCUSSION

Physical and mechanical properties of wood flour/PP composites have been investigated in previous research (Luo *et al*. 2012). For a better understanding of the effects of PEG and heat treatment on wood flour/PP composites, stress relaxation results shown above were discussed here in combination with the physical and mechanical performance of composites. In the previous study, water absorption tests of composites revealed that the 30% PEG treatment reduced the water uptake and thickness swelling of the composites for 24 h by 34.4% and 64.6% (Luo *et al*. 2012). Mechanical tests of the composites showed that the 20% PEG treatment resulted in a 36.7% and 51.2% reduction in the flexural modulus of rupture (MOR) and flexural modulus of elasticity (MOE). As shown in Fig. 3, the Δ*E* of composites after 30% PEG modification decreased compared with those untreated, indicating the decrease in interfacial compatibility. This explained why flexural properties decreased after PEG treatment. These results indicated that PEG treatment of wood flour remarkably improved the dimensional stability of composites. Heat treatment provided an extra 28.6% and an extra 12.1% reduction in 24 h thickness swelling as compared with composites made with fiber treated with 20% and 30% PEG solution alone. Thus, the combination of PEG treatment and heat treatment was more effective in improving the dimensional stability of the composites as compared with the use of either of them alone. On the other hand, both flexural MOR and MOE were improved after heat treatment. For example, heat treatment led to a 28% and 38% increase in MOR and MOE of 20% PEG modified wood flour/PP, and 7.7% and 4.9% in 30% PEG modification. The trends of alteration in the flexural properties were basically consistent with the stress relaxation analysis. The increase of mechanical properties was due to improved adhesion between the fiber and matrix, which was confirmed by the increase of Δ*E* in stress relaxation tests. Previous literature also showed that a high compatibility at interfaces leads to better stress transfer between the two components and the improvement of mechanical and physical properties of the materials (Youssef *et al*. 2008). It was conclusively demonstrated that the combination of PEG and heat treatment could alleviate the increasing stress relaxation rate and the decreasing strength caused by PEG impregnation, especially in the case of low-concentration PEG treatment. Therefore, in the case of the application situation where the strength is not strictly defined, the combination of PEG and heat treatment is a quite valid method for wood flour used for wood flour/PP composites.

As shown in Fig. 3, heat treatment led to an increase of 12.81% and 4.86% in the Δ*E* of composites using wood flour impregnated by 20% and 30% PEG, respectively, indicating that the compatibility of wood flour/polymer interface was improved. But the improvement was not obvious compared with the reduction caused by PEG impregnation. Temperature is a crucial factor for heat treatment. In our study, 140 °C was applied to heat-treated wood flour and the temperature was relatively low. Yan *et al*. (2011) studied the chemical compositions of glycerin-pretreated wood at different temperatures and found the hemicelluloses began to degrade at 120 °C and degrade violently at 160 °C. This was attributed to the glycerin being adsorbed into hydroxyl groups through hydrogen bonding in the amorphous region after pretreatment; as a consequence, the wood was in a swollen state and the molecules were easily subjected to slippage. It is known that the main components of wood flour are cellulose, hemicelluloses, and lignin. Hosseinaei *et al*. (2012) found that the absence of hemicelluloses improved the tensile strength and water resistance of wood-plastic composites, which might indicate a better compatibility, and interfacial bonding between the filler and matrix. Therefore, in the presence of PEG, which is also a polyalcohol, raising the temperature of the heat treatment might help to accelerate the degradation of hemicelluloses and further improve the interfacial compatibility of composites.

CONCLUSIONS

1. The increased stress relaxation rate and decreased ΔE of composites made from PEG-impregnated wood flour indicated that PEG modification might lead to a reduction of interfacial compatibility. In addition, the interfacial compatibility likely decreased with increasing PEG concentration.
2. The quantitative analysis of stress relaxation also showed that thermal treatment of wood flour improved the interfacial compatibility and could compensate for the decrease of compatibility caused by PEG modification to a certain extent, but the effect was not significant enough.
3. In general the PEG treatment reduced the shrinkage and swelling of the composites, but caused negative effects in certain mechanical properties. PEG concentration could be adjusted with the purpose of finding a compromise among the resulting physical and mechanical properties of composites for use in practical applications.

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