

Viscoelastic and Dynamic Characteristics of Wood-melamine-urea-formaldehyde Composites Prepared by *In Situ* Polymerization

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The viscoelastic behavior and the dynamic mechanical properties were studied for wood-melamine-urea-formaldehyde composites prepared from fast-growing poplars using *in situ* polymerization. The changes in stress relaxation (SR), storage modulus (E'), and damping parameter ($\tan \delta$) were considered. The relationship between the dynamic mechanical properties and the modifier concentration was also studied. The SR value decreased as the modifier content increased in all the samples examined. The urea content had a greater impact than the melamine-urea-formaldehyde mixture content on the SR value of the wood samples. A similar trend was observed for E' . The modified wood samples exhibited a higher $\tan \delta$. The results confirmed that a better interfacial interaction between melamine-urea-formaldehyde and wood fiber contributed to the improvement of mechanical properties in the fast-growing poplar wood.

DOI: 10.15376/biores.17.1.1015-1030

Keywords: Viscoelastic properties; Copolymerization; Fibers; Mechanical properties

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INTRODUCTION

Over the past several decades, wood materials have received remarkable attention in the marketplace because of their advantages over established materials. Wood possesses higher specific strength and has a higher modulus of elasticity compared to other materials (Zhu *et al.* 2017; Düzkale Sözbir *et al.* 2021). However, natural wood has poor compatibility, a tendency to form aggregates during processing, and a low resistance to moisture, which reduces its use as an elastic material.

Chemical modification of wood is a common method used to reduce or eliminate its undesirable properties. Chemical treatments could permanently alter the nature of the fiber cell walls by grafting polymers onto the fiber surface, or by bulking or cross-linking within the fiber cell walls. Movement of chemicals in and out of the cell walls has important implications in wood treatments and their performances. Chemical modification could make the fiber cell walls more dimensionally stable, reduce water sorption, and increase resistance against fungal decay (Kumar 1994; Kaymakci and Bayram 2021). The chemicals used must impregnate into the cell lumen, cell wall, or the combination of both so that they can react with the available hydroxyl groups in the wood cell wall (Hon and Qu 1989). The polymer type used in wood modification is typically a thermosetting resin, such as phenol formaldehyde (PF), urea formaldehyde (UF), melamine formaldehyde (MF), or epoxy resins. Following the wood modification, the properties of wood, such as dimensional

stability, durability, mechanical properties, and hardness, are improved to meet specific end-use requirements (Tanimoto and Nakano 2012; Liu *et al.* 2014).

Polymeric materials are commonly classified as viscoelastic materials, meaning they exhibit time-dependent stress-relaxation properties under various strain loadings. An excessive relaxation of the stress in a polymer-based structural component could render its performance non-functional (Tanimoto and Nakano 2012). Wood is a unique composite that consists of three structural polymers, cellulose, hemicelluloses, and lignin (Kelley *et al.* 1987). At the macromolecular level, wood is described as a two-phase composite of elastic fibers, consisting of cellulose and hemicellulose, and a viscoelastic matrix substance comprised of lignin and hemicelluloses (Obataya *et al.* 1998).

Dynamic mechanical analysis (DMA) has become a widely used technique for determining the interfacial characteristics of these heterogeneous polymeric systems (George *et al.* 1996). The DMA is defined as a technique where the storage modulus E' (elastic response) and the loss modulus E'' (viscous response) of the sample under an oscillating load are monitored against time, temperature, or frequency of oscillation (Shinoj *et al.* 2011). The DMA measurements can be conducted over a wide range of temperature, helping to study the viscoelastic behavior of the polymer systems, in particular the glass transition region in the fiber-reinforced composites. Materials respond to the applied force by exhibiting either elastic behavior, viscous behavior, or a combination of both, called viscoelastic behavior. Viscoelastic behavior of wood is typically determined by the DMA and is important because it reveals the proper temperature setting that wood tolerates during specific processing operations. Several studies have focused on the DMA properties of wood after modification. Fabiyi *et al.* (2011) conducted a study on the viscoelastic properties of ethylene glycol plasticized Nigerian plantation grown teak (*Tectona grandis*) wood. Kuo *et al.* (2009) investigated the effects that material compositions have on the mechanical properties of wood-plastic composites (WPCs) manufactured *via* injection molding. The results showed that the mechanical properties and the E' values were the highest and the $\tan \delta$ values were the lowest when the SPF (spruce, pine, and fir) wood flour content was 47%. This suggests that a wood flour content of less than 50% could be optimal for the manufacturing of WPCs.

The aim of this study was to investigate the changes that occur in the dynamic modulus and the viscoelastic properties of fast-growing wood-melamine-urea-formaldehyde composites, compared to untreated woods. The authors evaluated the dynamic mechanical properties of the fast-growing polar wood *via* various parameters. The change of stress-relaxation over various wood modifier contents were analyzed. The results showed that the plasticity of wood improved after chemical modification.

EXPERIMENTAL

Materials

Formaldehyde (mass fraction 37% to 40%), melamine (99% purity), and ammonia were obtained from Beijing Chemical Reagent Ltd, China.

Fresh-cut fast-growing poplars (*Populus davidiana*) were obtained from Hebei Province, China. The poplar samples used for the study were approximately 7 years old and 35 cm in diameter. The boles were relatively straight and practically free of decay. The initial moisture contents of the natural wood samples ranged from 70% to 80%.

Preparation of Chemical Wood Modifier

The wood modifier was prepared as described by Lang *et al.* (2014). Urea (60% w/w), formaldehyde (25% w/w), and ammonia (15% w/w) were mixed in a reactor using a mechanical agitator to form the urea-formaldehyde (UF) pre-polymer. An adequate amount of melamine was added to the melamine-urea-formaldehyde pre-polymer (MUF), and then the mixture was heated at 55 to 60 °C for 2 h with stirring. The pH of this solution was adjusted to 7 to 8 by adding sodium hydroxide and hydrochloric acid solutions. The prepared pre-polymer had a density of 1.5 g/cm³, pH of 9.0, viscosity of 12 mPa·s, solid content of 57.84%, and free formaldehyde content of 0.05 mg/100 g. The chemical formulation for the wood impregnation is presented in Table 1.

Table 1. Formulation for Wood Modification

Sample Number	MUF (%)	Urea (%)	Catalyst (%)
Natural wood	0	0	0
A	15	15	5
B	20	15	5
C	25	15	5
D	15	20	5
E	20	20	5
F	25	20	5
G	15	25	5
H	20	25	5
I	25	25	5

Preparation of the Wood-melamine-urea-formaldehyde Composites

The dimensions of wood samples used for impregnation were 15 cm in diameter and 60 cm in length. The fresh poplar wood was impregnated using a vacuum machine under 0.9 MPa for 1.5 h. The impregnated samples were cut into blocks of 5 cm × 10 cm × 60 cm (T × R × L) for the drying process.

Stress Relaxation (SR) Analysis

Specimens of dimensions 1 cm × 1 cm × 1 cm (T × R × L) were scanned in a TAQ 800 (Mode of SR TTS; TA Instruments, New Castle, DE, USA) at 30 °C, with a relative humidity (RH) of 50%. The compression rate of the wood samples was approximately 10%. Five replicates were used for each treatment.

Dynamic Mechanical Properties

Rectangular natural and modified wood samples (50 mm × 10 mm × 5 mm) with a 10% compression ratio were examined on a DMA-242 analyzer (TA Instrument, New Castle, DE, USA) at ambient air atmosphere. The E' and $\tan \delta$ were measured with a three-point flexure test at a heating rate of 2 °C/min with a temperature range between 30 and 180 °C. The initial static force was 20 N, and the dynamic force was 10 N. Frequency sweeps were performed between 1 and 2 Hz. A static stress of 0 N, deformation amplitude of 50 µm, load of 4 N, and a scale factor of 1.3 were used. Five replicates were tested for each treatment and the error was less than 1%.

RESULTS AND DISCUSSION

SR Analysis

The SR analysis is an effective means of characterizing the dynamics of polymer systems and assists in demonstrating the transient network behavior. Figure 1 shows the SR spectra with various modifier contents. The SR value of the modified wood was successively lower than the natural wood, where the SR value decreased as the amount of MUF content was increased. Figure 1(a) shows that the initial SR value was relaxed to 92% (Sample A), 91% (Sample B), and 89% (Sample C) when the MUF content ranged from 15% to 25%. The SR value showed the same trend with an equal urea content. The value of SR decreased to 92% (Sample A), 90% (Sample D), and 87% (Sample G) as the urea content was increased, as shown in Fig. 1(d). The amount of urea demonstrated a remarkable effect that was greater than the effect that the MUF content had on the SR value of the wood samples. Sample G showed that the SR value decreased to 87%, while the SR value of sample I decreased to 75%.

Wood is a polymeric material in which hydrogen bonds serve as the connection between the macromolecular chains of wood fibers (Sreekala *et al.* 2001). The urea and the MUF are polar molecules that penetrate the amorphous structure of cellulose and form hydrogen bonds. The penetration of the modifier destroyed the macromolecular chains in wood and regenerated the hydrogen bonds. The urea and the MUF contained a large number of $-NH_2$ and $-OH$ groups, which increased the combination between the intermolecular hydrogen bonds and the wood modifier. The increased cohesion between the wood fibers was attributed to the residual stress value of the wood. Following the modification, the decreased SR value of wood confirmed that the wood modifier penetrated the wood cell and a chemical reaction occurred between the $-NHCH_2OH$ functional groups from the MUF and the wood carboxyl $C=O$ group. The $-OH$ groups and the $-NH_2$ groups from the MUF were *in situ* polymerized with the hydroxyl groups (wood). The covalently cross-linked network had potential for improving the dimensional stability (Lang *et al.* 2013). The lower SR value of the material indicated that the material had an uneasily-deformed stability and superior mechanical properties.

Storage Modulus (E') Analysis

Dynamic mechanical analysis is a useful technique that provides information on the mechanical behavior, molecular relaxations, as well as interactions taking place in the materials formed as the temperature varies (Liu *et al.* 2008; Kiziltas *et al.* 2011). Figure 2 shows the changes in the E' of wood over various concentrations of MUF and urea. This decreasing trend of modulus is characteristic of the viscoelastic behavior of wood (Morlat *et al.* 1999; Pothan *et al.* 2003). As the content of wood modifier was increased, the E' in the samples decreased gradually between 50 and 120 °C. Figure 2(a) shows the E' value of wood with 15%, 20%, and 25% MUF contents, which changed to 6700, 7200, and 7400 MPa, respectively. The E' value of the modified wood samples was lower than the natural wood sample at 120 °C.

Figure 2 shows that the glass transition occurred around 50, 75, 120, 80, and 70 °C for samples A, B, E, F, and G, respectively. The modulus value remained unchanged afterwards. There was no noteworthy transition that occurred in the samples C, D, H, and I. The decreasing rate of E' in samples C, D, H, and I were lower than samples A, B, E, F, and G, demonstrating that the transition temperature was related to the curing degree of wood modifier solution during the modification.

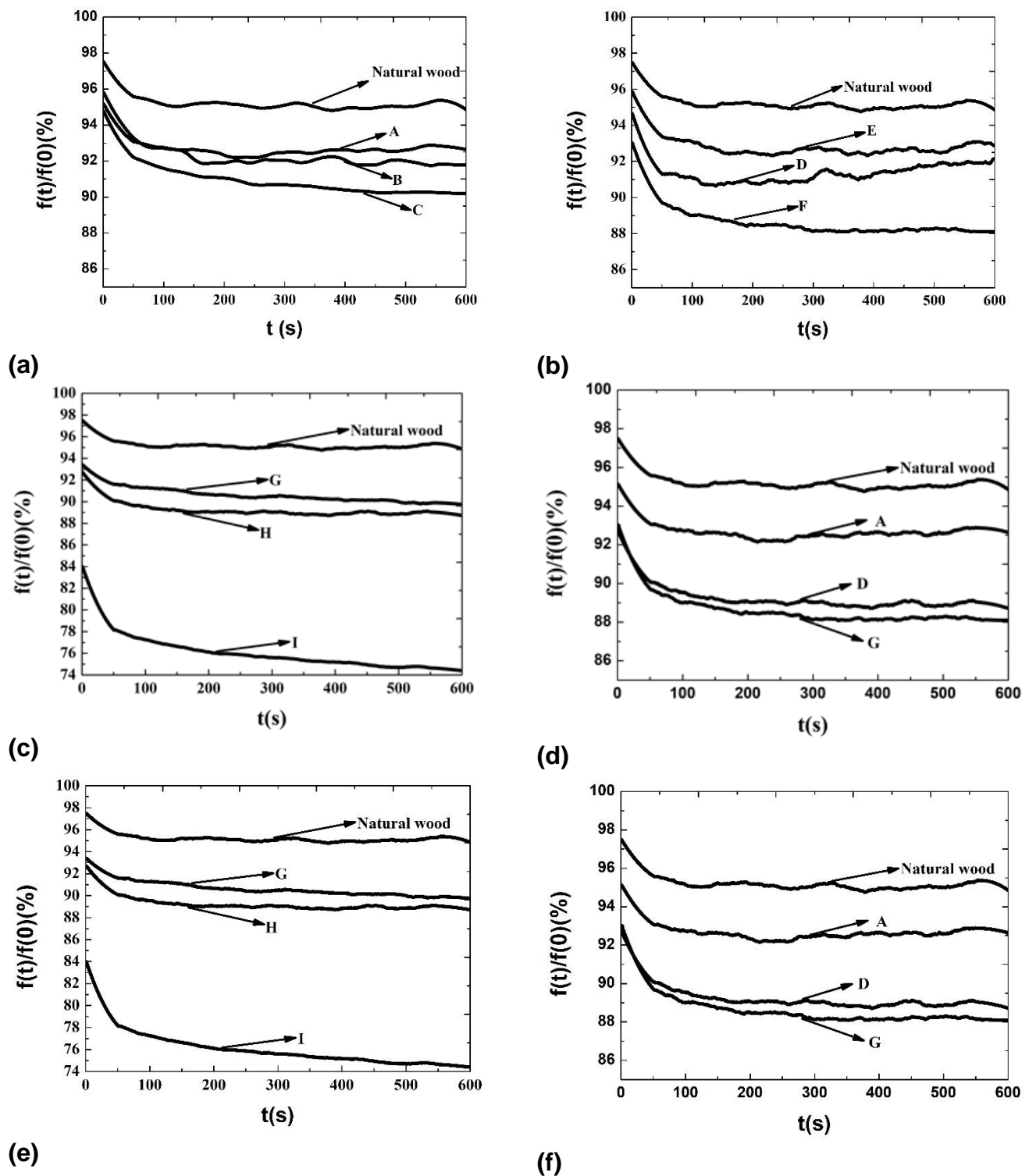
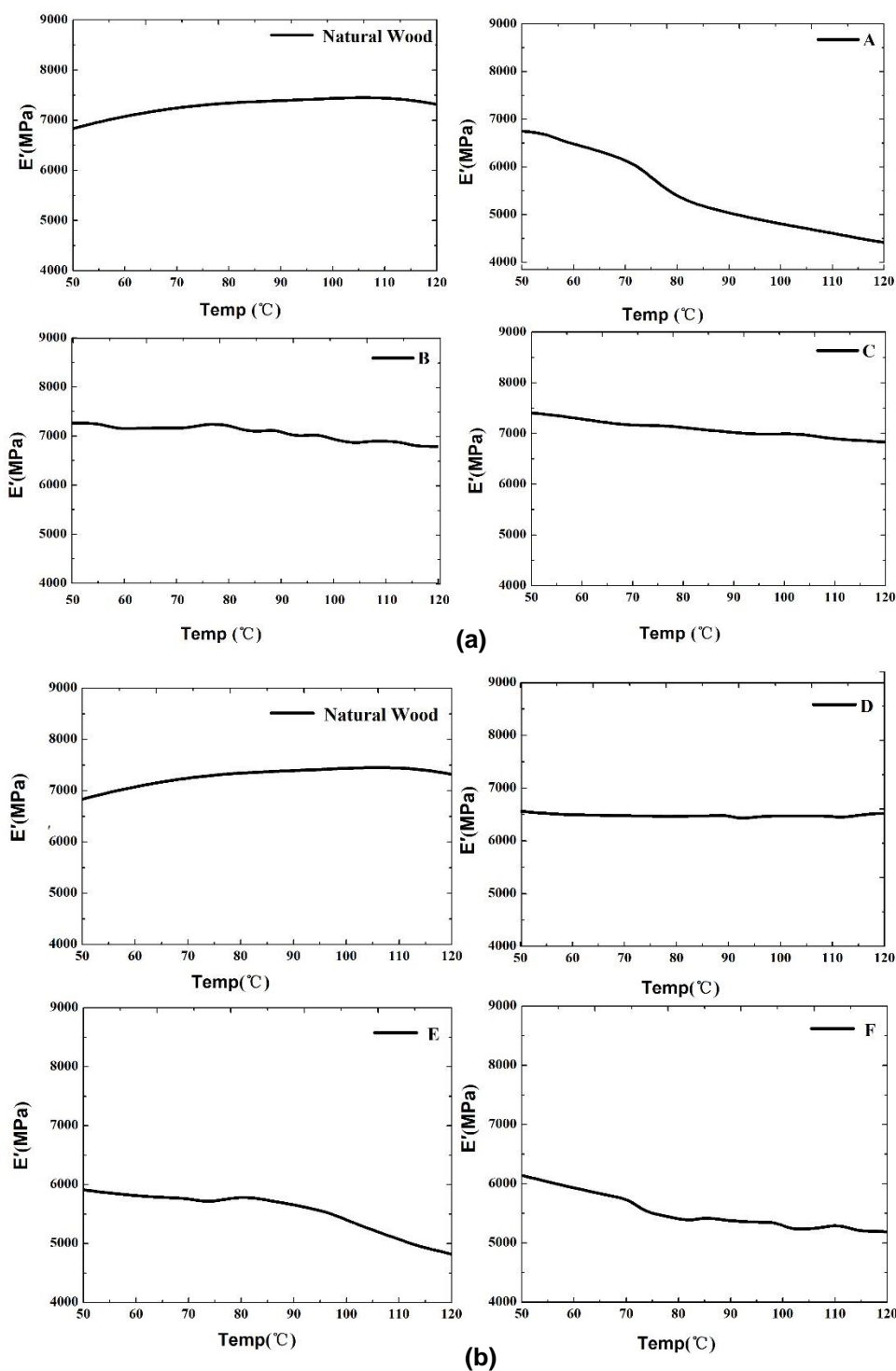
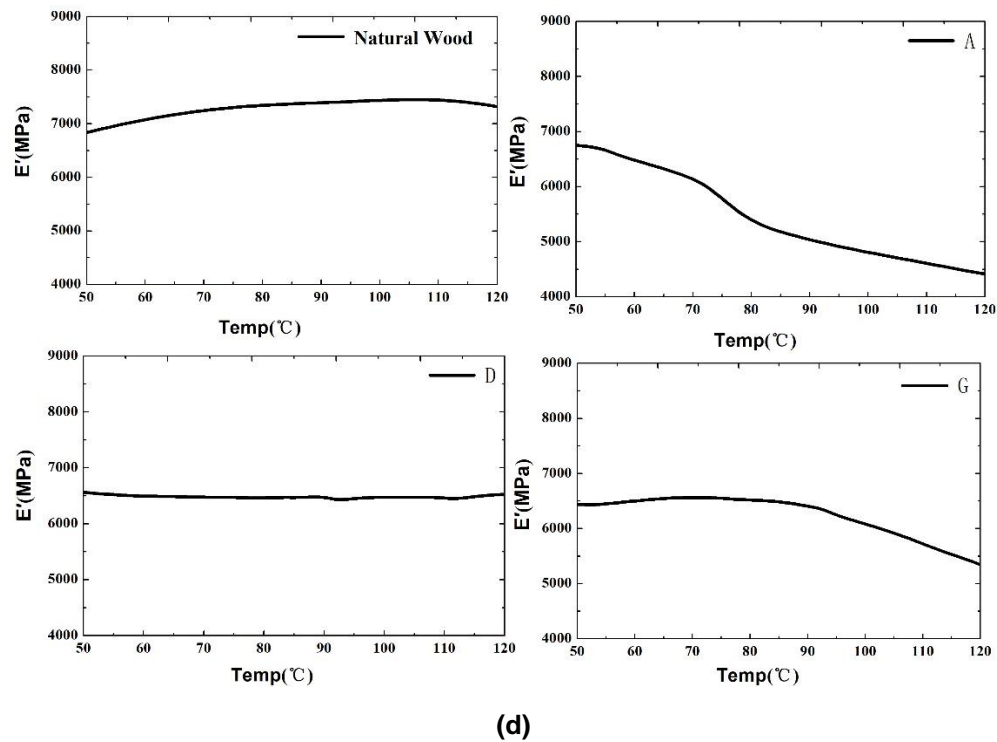
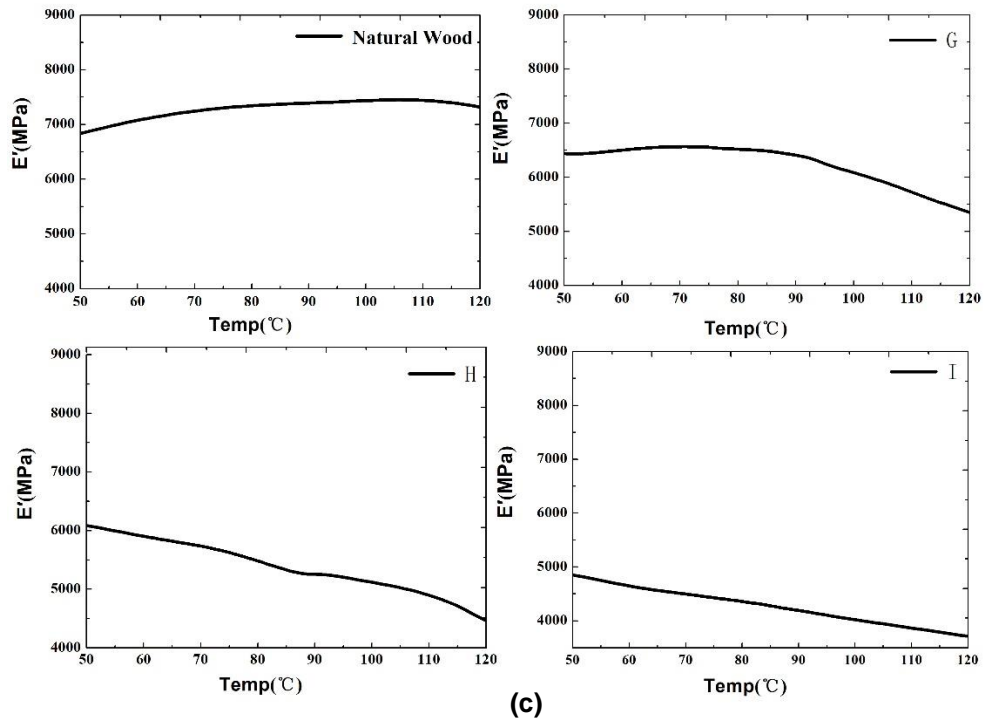


Fig. 1. Stress relaxation curves of natural and modified wood: (a) 15% urea; (b) 20% urea; (c) 25% urea; (d) 15% MUF; (e) 20% MUF; and (f) 25% MUF contents

The decreasing trend in E' resulted from the increased molecular mobility of the wood cell wall polymers at high temperatures (Ou *et al.* 2014). The increase in E' was observed for the modified wood. The modifier molecules cross-linked the growing linear chains during the curing process and thus tightened the structure of wood and added restrictions to the molecular motions in the wood. The $-OH$ groups from the MUF molecules increased the hydrogen bond formations and restricted the molecular rotations at lower temperatures.





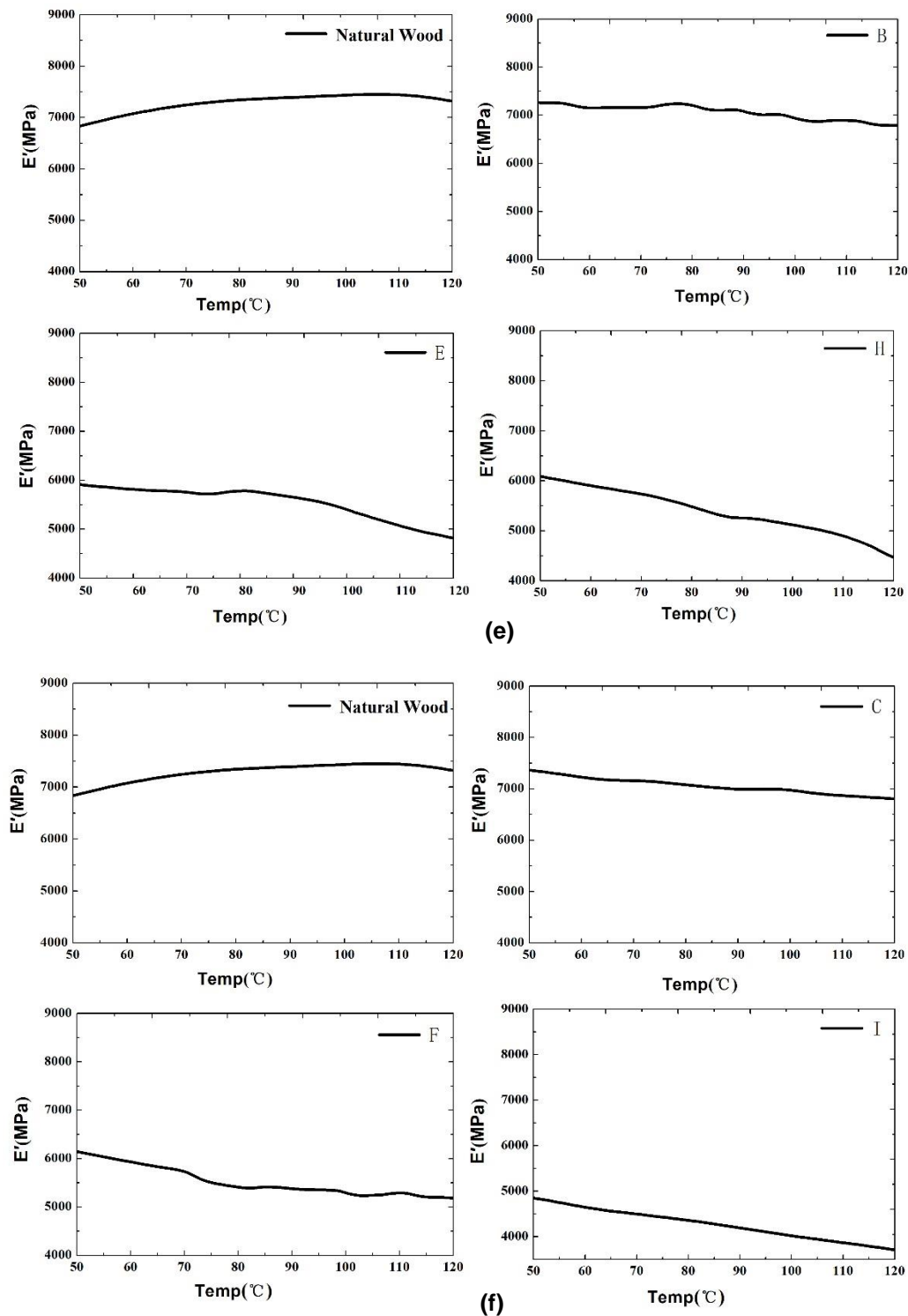


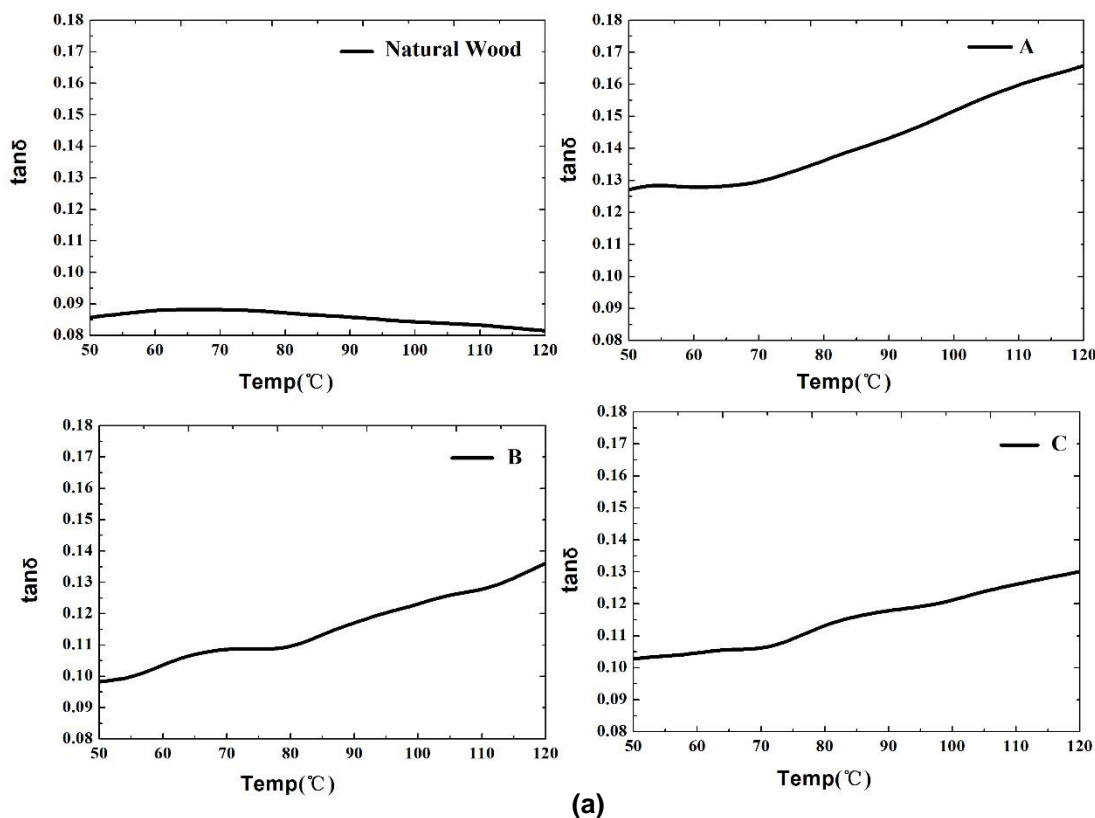
Fig. 2. Storage modulus curves of natural and modified wood: (a) 15% urea; (b) 20% urea; (c) 25% urea; (d) 15% MUF; (e) 20% MUF; and (f) 25% MUF contents

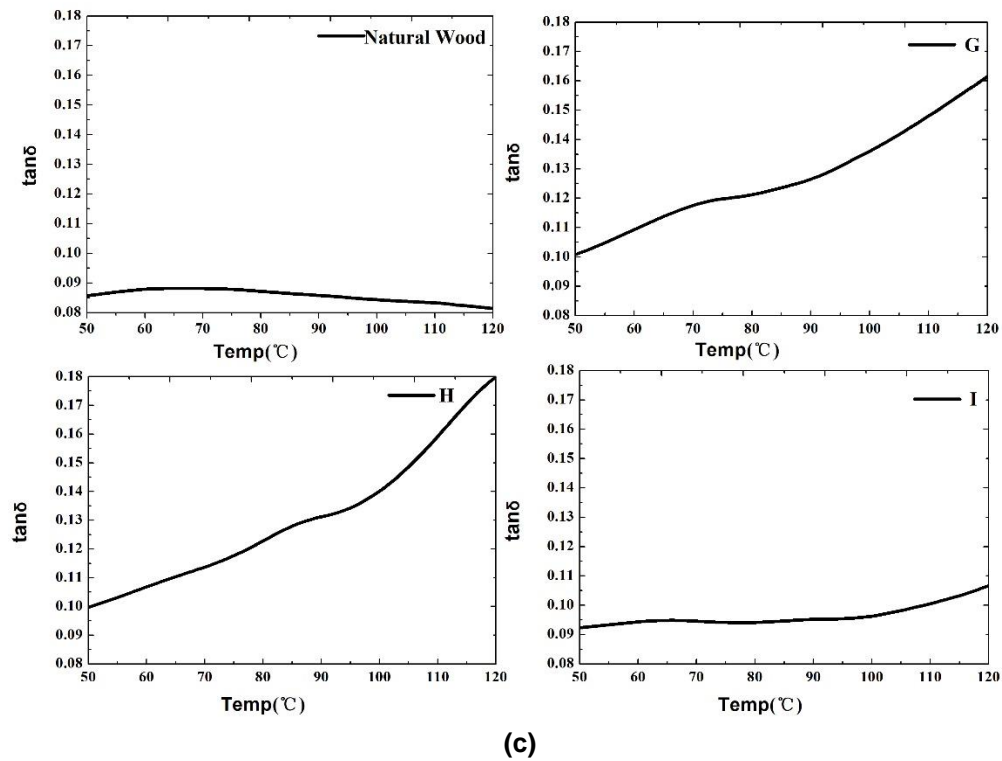
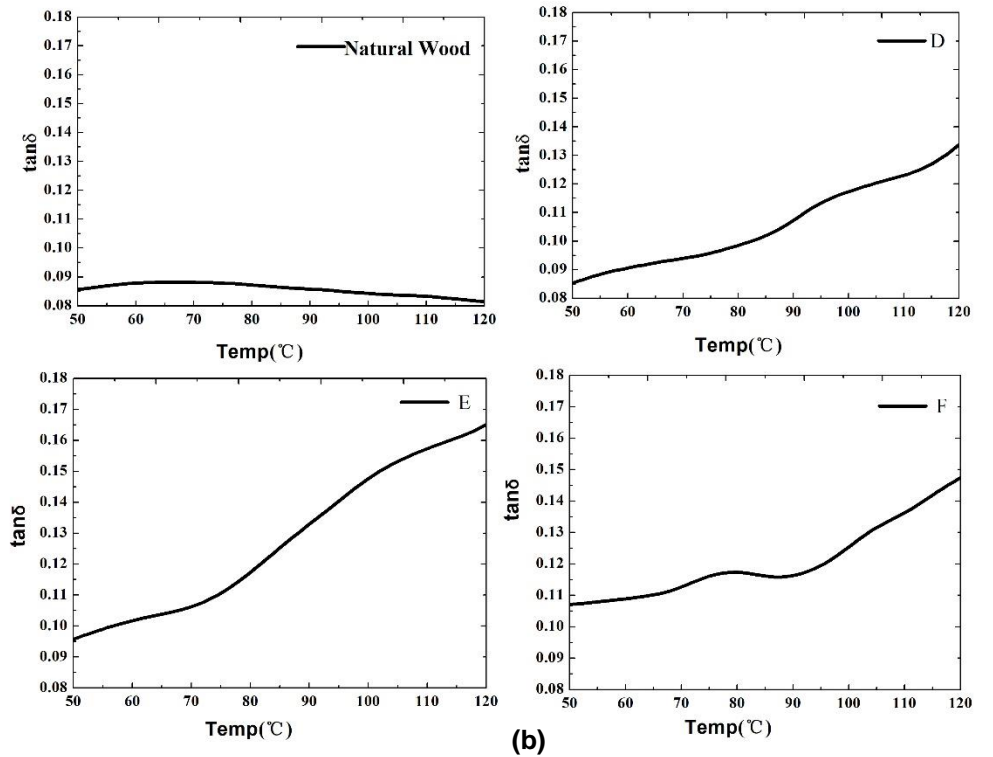
A superior interfacial adhesion between the wood fiber and the wood modifier occurred, which allowed a greater stress transfer at the inter-phase (Cai *et al.* 2008; Islam *et al.* 2012; Wang *et al.* 2017). The E' was associated with the elastic response of the

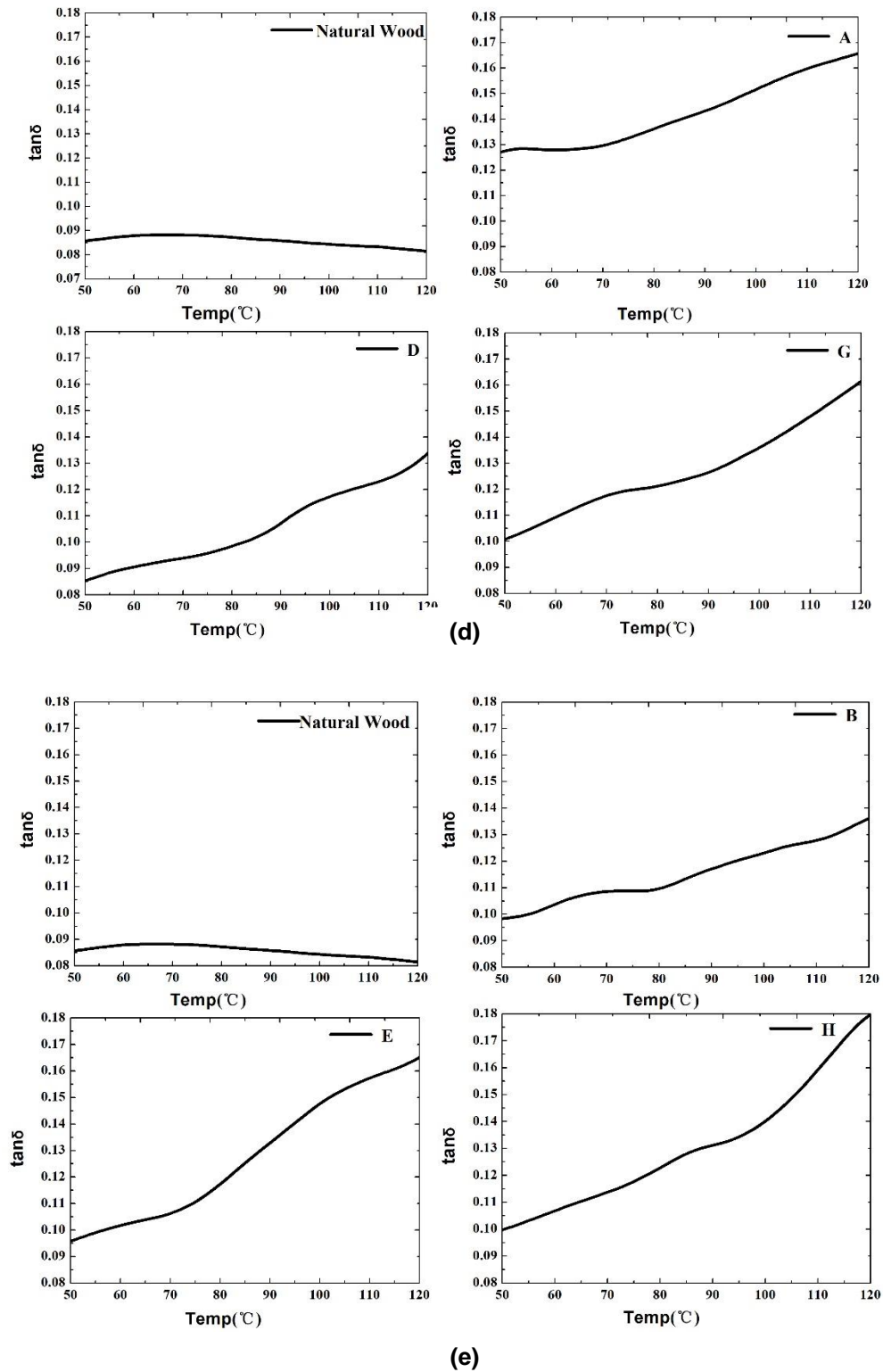
composite, and it indicated the stiffness of the material. The plasticity of wood was enhanced with modification.

Damping Parameter ($\tan \delta$) Analysis

$\tan \delta$ was associated with the degree of molecular mobility in the polymeric material. A high $\tan \delta$ indicated a high molecular mobility, while a low $\tan \delta$ value indicated less molecular mobility in the material. The $\tan \delta$ curves of the modified wood specimens are presented in Fig. 3. The glass transition temperatures of the natural and modified wood samples were identified between 60 and 90 °C. The transitions were caused by the microscopic Brownian movement in the main chain motion of the amorphous region.







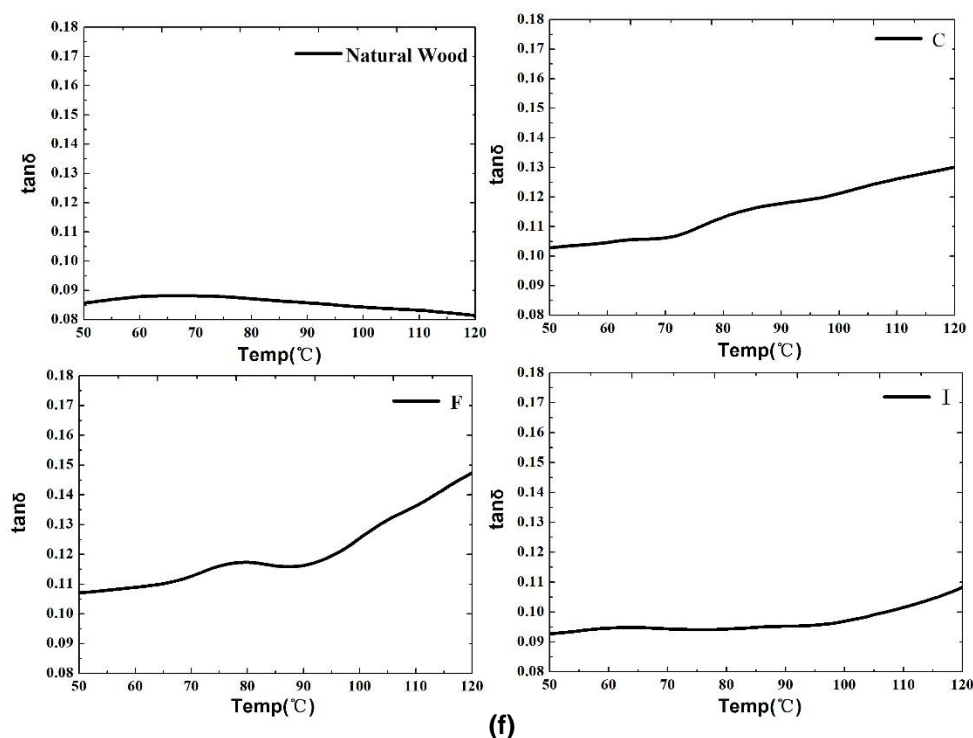


Fig. 3. Loss tangent curves of natural and modified wood: (a) 15% urea; (b) 20% urea; (c) 25% urea; (d) 15% MUF; (e) 20% MUF; and (f) 25% MUF contents

Altering the concentration of the MUF caused an apparent difference in $\tan \delta$. Figure 3 shows that the higher concentration of MUF yielded a higher $\tan \delta$ value, compared to the natural wood sample. The modified wood samples showed a higher $\tan \delta$ peak temperature for the temperature position of $\tan \delta$. The transition occurred around 65 °C for the natural wood sample, while the temperatures were 65 °C, 76 °C, 72 °C, 88 °C, 75 °C, 77 °C, 74 °C, and 85 °C for samples A, B, C, D, E, G, and H, respectively. The change in $\tan \delta$ confirmed that the chemical reaction occurred in the region, where the interaction between the molecular chains was enhanced, which reduced the relaxation process of the molecules in wood. When the MUF was impregnated into the wood, a polymer composite was formed, and a well-defined platelet-MUF inter-phase was created. This inter-phase was sensitive to the surface modification of the modifier, the compatibility between the wood fiber, and the polymer matrix. The functional groups of the MUF were more compatible with the wood fiber, and stronger polymer-wood adhesion was formed, so a better performance was obtained for the modified wood samples (Mohanty *et al.* 2006). The more compatible the wood fiber and the modifier were, the better the mechanical properties of the wood, including surface hardness, modulus elasticity, abrasion resistance, water repellency, and dimensional stability. The bonding strength increased as the wood modifier content was increased, confirming that a chemical reaction *in situ* polymerization reaction occurred during the modification. The effect that the concentration of urea had on the $\tan \delta$ value was not obvious, demonstrating there was no strong chemical connection between the urea, the wood, and the newly formed hydrogen bond.

Mechanism of Wood Modifications by MUF

Impregnation of solid wood with water-soluble MUF resin has led to a markable improvement of surface hardness and modulus of elasticity (MOE) (Cai *et al.* 2007). Melamine as a modifier has been used to form melamine-urea-formaldehyde (MUF) resin with higher mechanical properties and water resistance (Ding *et al.* 2020, 2021). The synthesis of MUF resins is a two-step process consisting of the methylation stage of amino groups and condensation of methylols to form polymeric chains (Siimer *et al.* 2008). MUF resins have a complicated structure, which contains two monomers reactive with formaldehyde in the polymer. A large variety of structural moieties can provide reactive sites for the modification of wood. The preceding work confirmed that the MUF not only can fill the wood cell walls and the amorphous region of cellulose fibrils, but it can also form bonds with the groups of wood cell due to the abundant active groups in the MUF by the FTIR analysis (Cai *et al.* 2010; Lang *et al.* 2014). Based on the authors' previous report, the absorbances corresponding to major functional groups changes at 3590 to 3300 cm^{-1} , 2950 to 2840 cm^{-1} , 2986 cm^{-1} , 1610 cm^{-1} , and 1160 to 1052 cm^{-1} , can be assigned as NH_2 stretching vibration, aliphatic CH_2 stretching vibration, C-NH_2 stretching vibration, NH bending vibration, and NH bending vibration, respectively. This is due to the stronger bonding between triazine carbons of MUF and wood cells (Lang *et al.* 2014). Meanwhile, the studies also showed the reaction mechanism of MUF and wood by ^{13}C NMR quantitative analysis (Liang *et al.* 2020). After modification by MUF, the wood samples displayed higher storage modulus even at higher temperature (Nagamadhu *et al.* 2020). Therefore, the cross-linking reaction between the MUF pre-polymer and wood fiber improved the viscoelastic and dynamic properties of wood.

CONCLUSIONS

1. The stress relaxation (SR) and the dynamic mechanical properties were highly sensitive to the concentrations of the melamine urea formaldehyde (MUF) and the urea. The SR of the modified wood samples indicated a lower value than the natural wood samples.
2. The storage modulus (E') value of the modified wood samples demonstrated a similar trend. The higher $\tan \delta$ value revealed a greater plasticity of the wood after the chemical modification.
3. The results confirmed that the wood modifier penetrated into the wood cell and a chemical reaction occurred between the functional groups of $-\text{NHCH}_2\text{OH}$ (methylolurea groups) and the carboxyl $-\text{C}=\text{O}$ group (wood). The hydroxyl and the amino groups from the MUF modifier was *in situ* polymerized with the hydroxyl groups (wood). The covalent *in situ* polymerization network improved the viscoelastic and dynamical mechanical properties.

ACKNOWLEDGMENTS

The research was supported by the China Postdoctoral Science Foundation (2017M621234), Heilongjiang Province Natural Science Foundation (LH2020C002), Heilongjiang Province Postdoctoral Science Foundation (LBH-Z17029), and the Youth Talent Foundation of Northeast Agricultural University (17QC31).

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Article submitted: August 16, 2021; Peer review completed: October 4, 2021; Revised version received: November 26, 2021; Accepted: November 28, 2021; Published: December 16, 2021.

DOI: 10.15376/biores.17.1.1015-1030