

The Effect of Ultrasound Pretreatment on Poplar Wood Dimensional Stability

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Dimensional stability is a key property of wood that significantly affects its applications. The effect of an ultrasound pretreatment on poplar wood (*Populus tomentosa*) dimensional stability was examined. During the pretreatments, wood samples were immersed in distilled water and treated ultrasonically under three different powers and frequencies. The samples were then analyzed by Fourier transform infrared spectroscopy (FTIR) and X-ray diffraction (XRD). The chemical transformation of the cell-wall material was studied and then associated with the change of water absorption and the swelling coefficient. The results showed that the water absorption decreased after the ultrasonic pretreatment. The axial and radial swelling coefficients of the pretreated samples decreased, while the tangential swelling coefficients increased. The volumetric swelling coefficient of pretreated specimens fluctuated near 4.48% (the volumetric swelling coefficient of untreated wood). Ultrasonic pretreatment increased the number of hydrophilic groups, such as the hydroxyl, acetyl, and uronic ester groups. Meanwhile, the pretreatment also increased the degree of crystallinity and reduced the available polar groups. These two factors together caused the change of the moisture absorption and the swelling coefficient of the pretreated wood. These conclusions suggest that the ultrasonic pretreatment is a promising method for further chemical modification of wood.

Keywords: Ultrasonic pretreatment; Dimensional stability; FTIR; XRD

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INTRODUCTION

Wood is sensitive to humidity because of the hydrophilic nature of its cell wall constituent polymers, cellulose, hemicelluloses, and lignin. As a result, the moisture content (MC) of wood varies with the surrounding humidity and temperature changes (Sun *et al.* 2010). MC changes below the fiber saturation point lead to dimensional variation, namely shrinkage and swelling. This phenomenon significantly affects the usability of wood in products. Small shrinkage values are an advantage for some wood species, especially in the products that are exposed daily to external conditions.

Ultrasound technology is based on mechanical waves at a frequency above the threshold of human hearing (> 20 kHz) (Chandrapala *et al.* 2013). Ultrasonic pretreatment involves the immersion of the material in water, a hypertonic aqueous solution, or other solutions to which ultrasound is applied (Fan *et al.* 2008). Sonication in liquid produces cavitation, which can explosively collapse and generate localized pressure (Wan *et al.* 1992). The rapid collapse of bubbles generates high instantaneous pressure that can cause physical damage to the solid surfaces in contact with the cavitation gas bubbles (Frederick

1965). Ultrasonic cavitation has been used to clean hard-to-reach surfaces, to homogenize immiscible liquids, to accelerate chemical reactions, and to deaerate liquids. Ultrasonic cavitation has been used as a pretreatment to successfully reduce the volumetric shrinkage of redwood upon drying (Erickson *et al.* 1970).

In recent years, ultrasound has been implemented as an alternative pretreatment method for wood drying; this pretreatment greatly reduces the overall processing time (He *et al.* 2012, 2014) due to the following factors: increased mass transfer rate (García-Pérez *et al.* 2009, 2011; Xu *et al.* 2009; Cárcel *et al.* 2011), increased effective water diffusivity (Bantle and Eikevik 2011; He *et al.* 2012), increased wood specific permeability coefficient (Tanaka *et al.* 2010), loss of cellular adhesion, formation of large cellular interspaces, ruptured cell walls, and formation of large channels (García-Pérez *et al.* 2009). However, few reports so far have addressed the influence of ultrasound pretreatment on the dimensional stability of wood.

To satisfy the increasing demand for forest products, fast-growing trees such as poplar are being seriously considered for future supply needs. Poplar is planted in large areas of China. However, planted poplar has dimensional instability and low density, which greatly decreases the value of the lumber (Cai *et al.* 2013). Therefore, efforts have been made to improve the dimensional stability of poplar. In this study, the influence of ultrasonic pretreatment on the dimensional stability of poplar was determined.

EXPERIMENTAL

Materials

Poplar (*Populus tomentosa*) trees, harvested in the forest region of northeast China and air dried for one year, was machined to a size of 20 mm × 20 mm × 20 mm (length, tangential, and radial). All of the samples with the initial moisture content of 150 ± 5% were cut from a log. The ultrasonic pretreatment was performed in distilled water by an ultrasonic cleaner (VGT-2200A, Cheng-Cheng Ultrasonics, Beijing, China). The ultrasonic frequencies were 25 kHz, 40 kHz, and 59 kHz, and the power was 180 W, 300 W, and 400 W. All specimens were treated for 1 h, and 30 replicates were prepared to form a sample of the mentioned variables. After the pretreatment, all specimens were oven-dried at 103 ± 2 °C. Ten specimens of each sample were selected and ground in a laboratory grinder. The obtained wood flour was dried at 103 ± 2 °C for 24 h. The wood powder passing through an 80-mesh screen but retained by a 100-mesh screen was prepared for XRD analysis, while the wood powder passing a 100-mesh screen was selected for FTIR detection.

Methods

Measurements of FTIR spectra

FTIR spectra were recorded on a Perkin Elmer Spectrum 2000 FTIR spectrometer (Waltham, MA, USA) in the range of 400 cm⁻¹ to 4000 cm⁻¹, with a resolution of 4 cm⁻¹. Wood meal was dispersed in a matrix of KBr and pressed into form pellets. Due to the difficulty in determining a reference spectral band remaining completely invariable to conduct quantitative measurements, all comparisons were made qualitatively (Mohammed *et al.* 2005).

XRD analysis

Wide-angle XRD measurements were taken with a Shimadzu XRD-6000 diffractometer (Tokyo, Japan). The X-ray generator was equipped with a copper tube operating at 40 kV and 40 mA, and the sample pre-stabilized to the laboratory conditions was irradiated with monochromatic radiation with a wavelength of 0.154 nm. XRD spectra were acquired at room temperature over the 2θ range of 5° to 40° at 0.04° intervals, with a scanning speed of $2^\circ/\text{min}$.

Estimation of dimensional stability

The swelling coefficient and water absorption tests were carried out according to GB/T 1934.2 (2009). Specimens with a size of $20\text{ mm} \times 20\text{ mm} \times 20\text{ mm}$ were oven-dried and subsequently stored in a climate controlled chamber at 65% relative humidity (RH) and 20°C to reach the equilibrium moisture content (EMC). The specimen dimensions and weight were measured before and after the conditioning. The swelling coefficient and water absorption were calculated using Eqs. 1 and 2,

$$S (\%) = (D_v - D_0)/D_0 \quad (1)$$

where $S (\%)$ is the swelling coefficient (axial, radial, tangential, and volumetric), D_0 is the initial dimension of the specimen, and D_v is the dimension after conditioning,

$$\text{WA} (\%) = (W_v - W_0)/W_0, \quad (2)$$

where $\text{WA} (\%)$ is the absorption of water, W_0 is the initial weight of the specimen, and W_v is the weight after conditioning.

Evaluation of crystal properties

According to the peak height method developed by Segal *et al.* (1959) for native cellulose, the XRD crystallinity index (CI_{XRD}) was calculated from the following ratio,

$$\text{CI}_{\text{XRD}} (\%) = \frac{I_{002} - I_{am}}{I_{am}} \times 100 \quad (3)$$

where I_{002} is the intensity of the 002 crystalline peak around the 22° , and I_{am} is the height of the minimum (I_{am}) between the 002 and the 101 peaks.

RESULTS AND DISCUSSION

FTIR Results

Fourier transform infrared (FTIR) spectroscopy is an appropriate method in establishing the variations introduced by different treatments on the chemical structure of the isolated samples. Figure 1 compares the FTIR spectra of untreated wood meal with wood pretreated by sonication at 25 kHz under different powers. The intensity of the dominant peak of OH- stretching at approximately 3400 cm^{-1} increased after the ultrasonic pretreatment, which may cause higher hygroscopicity in the wood. This modification may be explained by the effect of an acoustic cavitation of high frequency ultrasound in the formation, expansion, and implosion of microbubbles in an aqueous solution. The violent collapse induces microjets and shock waves on the samples, breaking the relatively weak interfaces among the cell wall constituent polymers, which interact mainly by hydrogen bonds (Chen *et al.* 2011).

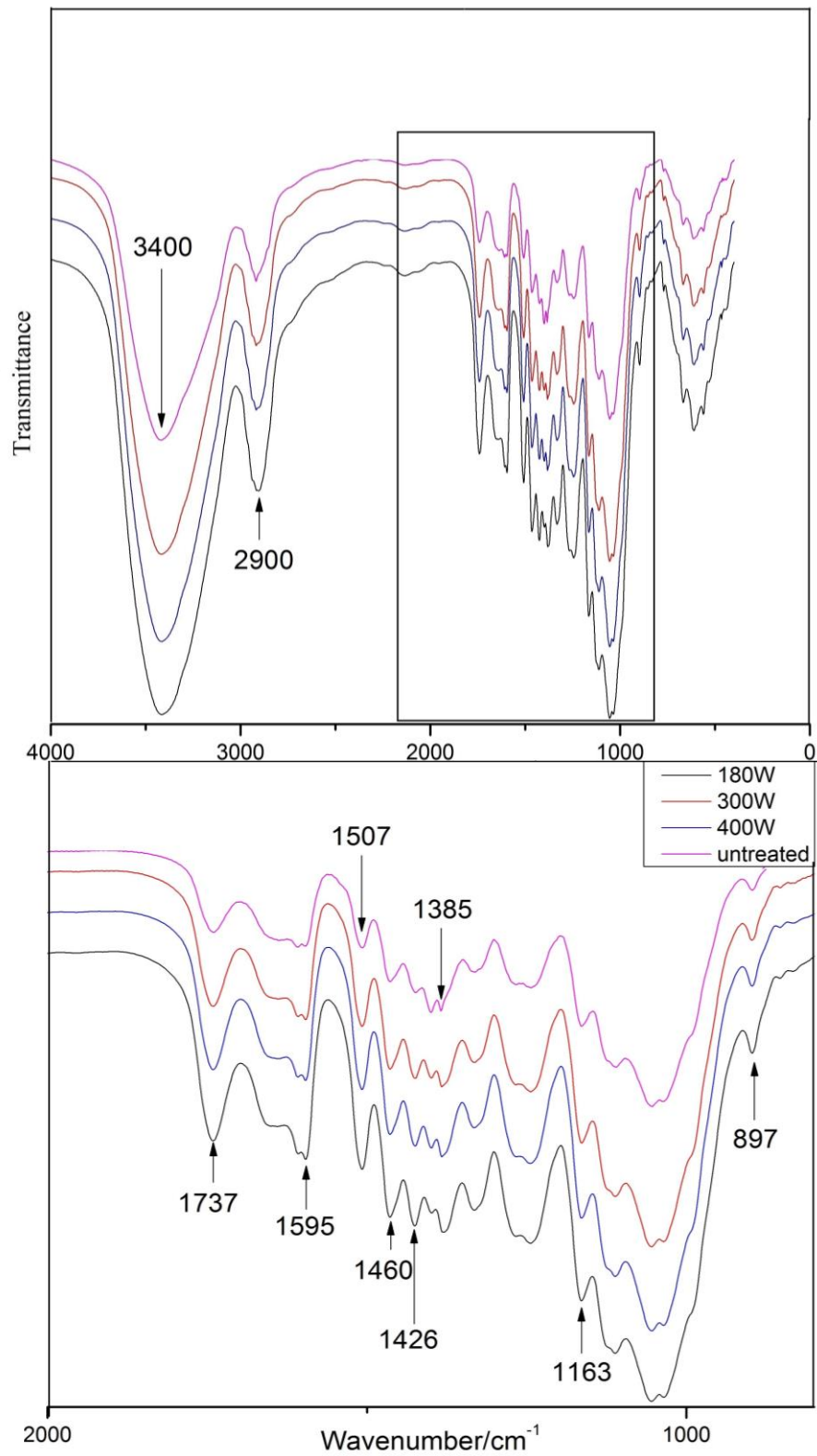


Fig. 1. FTIR spectra of untreated and pretreated samples

The breaking of hydrogen bonds leads to the increasing of hydroxyl groups. The band at 2900 cm^{-1} shows the CH stretching vibration. The prominent peak at 1737 cm^{-1} is attributed either to the acetyl and uronic ester groups of the hemicellulose or to the ester linkage of the carboxylic groups of the ferulic and p-coumeric acids of lignin and/or hemicellulose (Sun *et al.* 2000; Sain and Panthapulakkal 2006). This band became more intense, indicating an increase in the acetyl or carboxylic acid derived mainly from lignin (Colom *et al.* 2003). This increase may then increase the amount of moisture that is absorbed by samples. The ultrasonic treatments resulted in the modification of the hemicellulose structure, disrupted some of the linkages between the hemicellulose and the cellulose or lignin, and increased the 1737 cm^{-1} signals. The 1595 cm^{-1} , 1507 cm^{-1} , and 1460 cm^{-1} peaks representing the aromatic ring vibration and the C-H deformation vibration of lignin, respectively (Sun *et al.* 2000; Mohammed *et al.* 2005; Sain and Panthapulakkal 2006), were significantly enhanced in the ultrasonic pretreated samples. This result reflects the combination of localized high pressure and the high temperature generated by sonication, which destroyed lignin-carbohydrate bonds and broke the bonds between the lignin subunits (Koutsianitis *et al.* 2015). As a consequence, the lignin was released and re-deposited on the surface. The intensity of the band at 1426 cm^{-1} , which was assigned to the CH_2 bending vibration in the crystallized cellulose I and the amorphous cellulose mixture (Colom *et al.* 2003), increased after sonication. The band at 1385 cm^{-1} , which was assigned to the conformational changes in the glycosidic bridge, was strengthened after the ultrasonic pretreatment. The band at 1163 cm^{-1} , which was associated with the asymmetrical bridge at the C-O-C stretching for crystallized cellulose (Lionetto *et al.* 2012), increased in the pretreated samples. The band at 897 cm^{-1} , which was assigned to the C-O-C stretching at the β -glucosidic linkages in the cellulose and hemicellulose, was also increased. These phenomena could be interpreted as changes in the structure of cellulosic components in the pretreated samples.

Crystal Structure Analysis by X-ray Diffraction (XRD)

The crystallinity of the cellulose in the samples is a key factor in determining their dimensional stability. XRD studies of the pretreated and untreated samples were conducted to investigate the crystalline behavior of the specimens. Figure 2 shows that all diffractograms exhibited sharp peaks around $2\theta = 16^\circ$ and 22° , which were believed to represent the typical cellulose I form (Chen *et al.* 2011). Overall, the peak intensity of the cellulose crystal of the ultrasonic pretreated poplar did not show much difference from that of the untreated specimens. This result indicates that the crystal structures changed very little during the ultrasonic pretreatment. Nevertheless, the CI_{XRD} of most pretreated specimens increased, as listed in Table 1 and Fig. 3. The crystalline fraction of wood is given only by cellulose because the other two main components, hemicellulose and lignin, are amorphous. The increased CI_{XRD} may be attributed to the degradation caused by acoustic cavitation, which reduces the amorphous fractions of wood, and consequently, enriches the relative crystalline content. An ultrasonic process can be used in the generation of nano-size material slurries, dispersions, and emulsions because of the potential for deagglomeration and the reduction of primaries (Hielscher 2007). Ultrasound enhances mass transfer and facilitates the penetration of formed radicals into the lignin-carbohydrate matrix to soften the surface of the matrix. Enhanced diffusivity helps to degrade amorphous cellulose, improve the extractability of adjacent molecules or fragments, and increase the crystalline region (Koutsianitis *et al.* 2015). Sulman *et al.* (2011) suggested that ultrasonic treatment of pine fibres facilitates lignin extraction, especially in an aqueous medium.

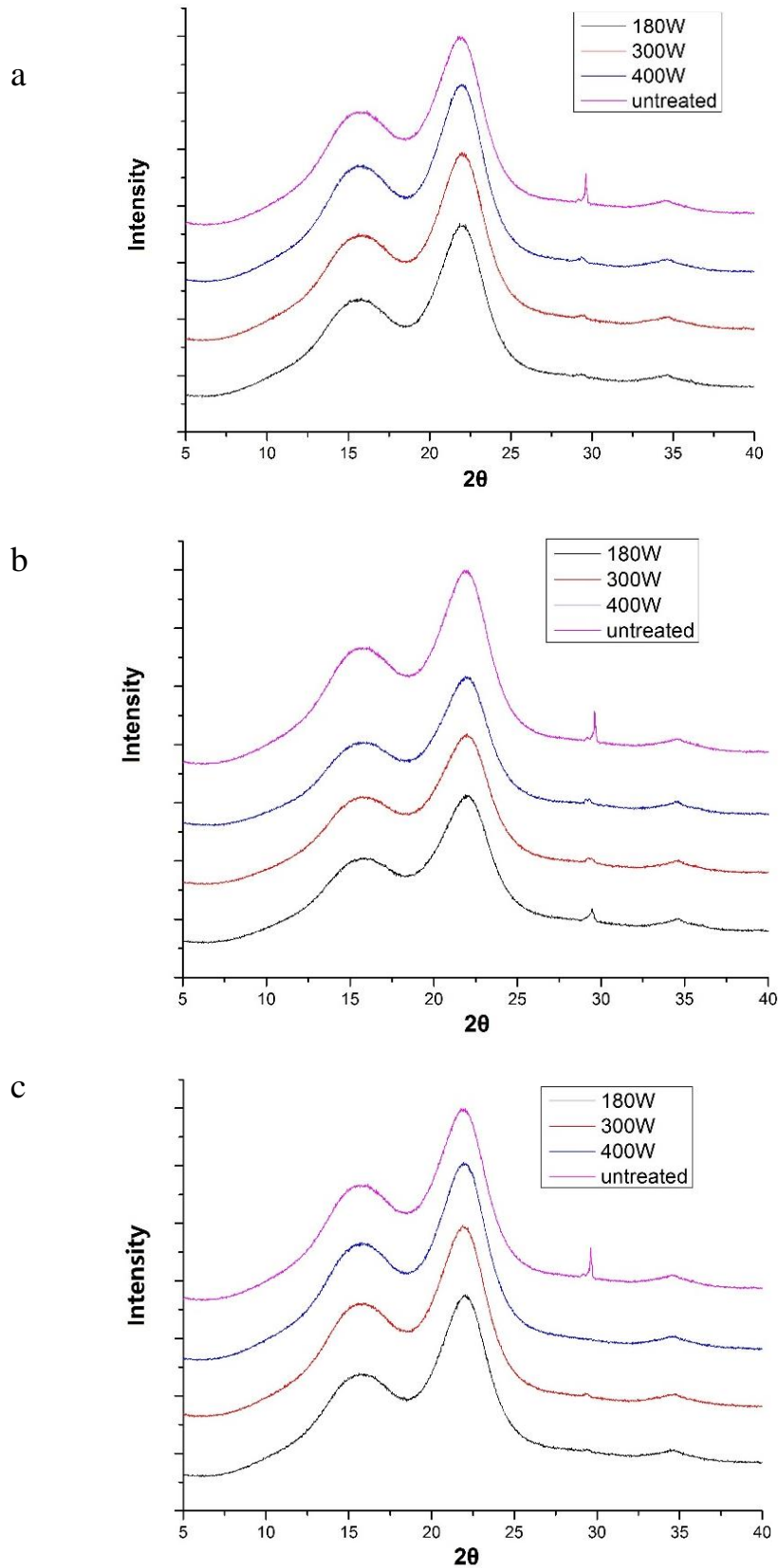
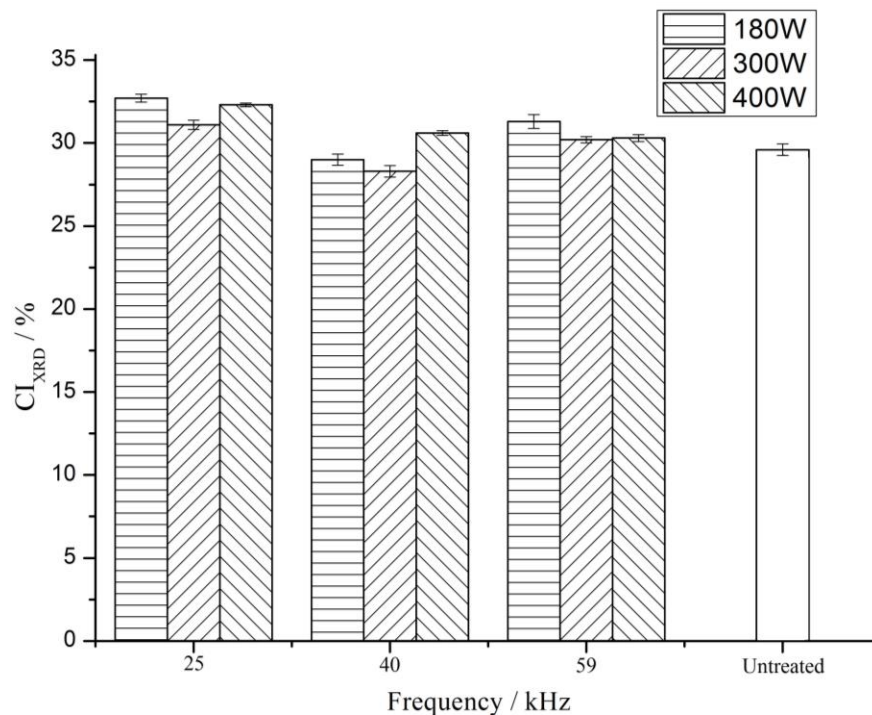


Fig. 2. XRD patterns of pretreated and untreated samples when the frequency of ultrasound was (a) 25 kHz, (b) 40 kHz, and (c) 59 kHz

Table 1. Crystallinity Index (CI_{XRD}) of Samples

Sample	Frequency (kHz)	Power (W)	CI_{XRD} (%)	ΔCI_{XRD} (%)
Pretreated	25	180	32.70±0.24	10.47
	25	300	31.10±0.28	5.07
	25	400	32.30±0.11	9.12
	40	180	29.00±0.33	-2.03
	40	300	28.30±0.35	-4.93
	40	400	30.60±0.14	3.38
	59	180	31.30±0.42	5.74
	59	300	30.20±0.19	2.03
	59	400	30.30±0.21	2.36
Untreated	-	-	29.60±0.35	-

**Fig. 3.** Crystallinity Index (CI_{XRD}) of Samples

The CI_{XRD} of the pretreated samples first decreased and then increased with the increasing power of ultrasound under different frequencies. Ultrasound degraded the amorphous components of wood and increased the crystallinity. However, a higher intensity pretreatment caused degradation of the crystallites, leading to reduced CI_{XRD} . If the ultrasound power continues to be strengthened, the ultrasonic will produce large amounts of useless bubbles, which form a sound barrier and increase the scattering attenuation, and consequently, reduce the intensity of cavitation. This reduction weakens the destruction of ultrasonic wave on the crystalline region and increases the CI_{XRD} .

Dimensional Stability

The dimensional stability of the pretreated specimens was estimated by the water absorption (WA) and swelling coefficients (*S*). Table 2 shows the axial, radial, tangential, and volumetric swelling coefficients (AS, RS, TS, and VS, respectively) and the water absorption of the pretreated and untreated samples.

The water absorption in pretreated wood samples decreased from about 0% to 7.05%, compared with that of untreated wood. Thus, in the same ambient conditions, the ultrasonic pretreated wood absorbed less water and, consequently, had less changes in the dimensional stability. No obvious variation law about the WA were found between the samples treated under different ultrasonic powers and frequencies. The FTIR and XRD analyses showed that ultrasonic pretreatment caused a great change in the wood cell wall constituent polymers. The hydroxyl, acetyl, and uronic ester groups of the pretreated specimens increased, and the CI_{XRD} of pretreated samples also increased.

Table 2. Water Absorption and Swelling Coefficient of Samples

Sample	Frequency(kHz)	Power(W)	WA (%)	AS (%)	RS (%)	TS (%)	VS (%)
Pretreated	25	180	12.94±0.31	0.15±0.18	2.03±0.39	3.10±0.34	5.38±0.53
	25	300	12.83±0.30	0.10±0.14	1.93±0.30	2.90±0.30	4.99±0.50
	25	400	13.07±0.38	0.02±0.14	1.78±0.31	2.89±0.26	4.74±0.31
	40	180	12.39±0.28	0.05±0.13	1.57±0.49	2.58±0.49	4.24±0.79
	40	300	12.68±0.34	0.16±0.18	1.37±0.27	2.34±0.42	3.91±0.55
	40	400	12.97±0.32	0.25±0.16	1.43±0.41	2.66±0.42	4.39±0.57
	59	180	13.05±0.38	0.08±0.15	1.39±0.33	2.53±0.31	4.04±0.46
	59	300	13.05±0.32	0.04±0.11	2.07±0.35	2.62±0.27	4.78±0.56
	59	400	13.33±0.42	0.01±0.16	2.03±0.26	2.55±0.43	4.62±0.50
Untreated	-	-	13.33±0.35	0.12±0.20	2.04±0.40	2.50±0.33	4.48±0.75

The wood cell wall is mainly composed of polymers with hydroxyl and oxygen-containing groups that attract moisture through hydrogen bonding. As water is added to the cell wall, the wood volume increases nearly proportionally to the volume of the water added (Stamm 1964). However, only some of these hydrophilic groups are accessible to water. Stamm (1964) estimated that 65 percent of the cellulose in wood is crystalline and therefore not accessible to water. Although ultrasonication increased the number of hydrophilic groups (hydroxyl, acetyl, and uronic ester groups) in wood, the increasing CI_{XRD} caused by the ultrasound reduced the available polar groups. The joint effect of the increased amount of hydrophilic groups and an increased crystallinity index led to the lower hygroscopicity of wood.

However, the volumetric swelling coefficients of the pretreated samples were not decreased proportionally as expected. The VS of the untreated samples was 4.48%, and the VS of the pretreated wood fluctuated around this value. Moreover, the axial and radial swelling coefficients of the pretreated samples were almost reduced, while the tangential swelling coefficients were increased. Water absorption and swelling are often used to evaluate the dimensional stability of wood. However, water repellency and dimensional stability are not the same. Water repellency is a rate phenomenon, and dimensional stability is an equilibrium phenomenon. Water repellency is defined as the ability to prevent or

control the rate of liquid water uptake, and dimensional stability is defined as the ability to reduce or prevent the swelling and shrinking resulting from moisture pickup (Rowell and Banks 1985). Therefore, the volumetric swelling coefficients of the pretreated specimens did not reduce as the water absorption did. The difference of the swelling coefficient changes in the three directions needs further study.

The ultrasonic pretreatment decreased not only the water absorption but also the axial and radial swelling coefficient of poplar. In industrial production, sawing wood in a radial orientation greatly reduces the dimensional change. However, the physiochemical effects of ultrasound disrupted the bonds between the lignin and the carbohydrate in the lignin-carbohydrate complex, which increased the number of functional groups available to react with various chemical modification reagents. Lee *et al* (2005) applied ultrasonic treatment for the preservative treatment of two softwood species. They found that both the retention and penetration depth were continuously increased with increasing the treatment time up to 120 hours. The results of electronic microscopic observation showed that the improved retention capacity could be attributed to air deflation, wood extractive deflation working of ultrasonic wavelength, and destruction of wood pits which served as the pathway of preservatives. Kim (2008) suggested that ultrasound treatment can assist in reducing the amount of chemical usage and shorten the reaction time when surface modify wood fiber. Therefore, ultrasound pretreatment prior to further chemical modification has great potential.

CONCLUSIONS

1. Ultrasonic pretreatment is an effective method for reducing the water absorption rate of fast-growing poplar wood. Nevertheless, the dimensional stability of the pretreated wood was not increased proportionally. The axial and radial swelling coefficients of the pretreated specimens were reduced, while the tangential swelling was increased after the ultrasonic treatment. The volumetric swelling of the pretreated wood was near the value measured in untreated samples.
2. The FTIR and XRD spectra indicated that ultrasonic pretreatment changed the cell wall component polymers of poplar, which caused changes in the water absorption and volumetric coefficient.
3. Ultrasonic pretreatment is a promising technique for chemical modification of wood.

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