Effect of Modification with Phenol Formaldehyde Resin on the Mechanical Properties of Wood from Chinese Fir

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Samples of Chinese fir were treated with either low-molecular-weight or commercial phenol-formaldehyde (PF) resins. The macro- and micromechanical properties of the treated and untreated samples were determined. The average longitudinal tensile modulus of elasticity (MOE) was 30.88% larger for the samples treated with the low-molecular-weight PF resin than it was for the untreated samples. The average MOE of the samples treated with the commercial PF resin was 29.84% less than that of the untreated samples. The micromechanical properties of the samples were investigated through nanoindentation studies. For the samples modified with low-molecular-weight PF resin, the values of average MOE and hardness were 32.94 and 32.93%, respectively, greater than those of the untreated samples. In contrast, the average MOE and hardness values were 11.99 and 18.14%, respectively, greater for the samples modified with commercial PF resin compared to the untreated samples. It could be inferred that the low-molecular-weight PF resin was able to diffuse into the nanopores in the S_2 layer of the tracheid cell wall of the Chinese fir, thereby improving its macromechanical properties. Modification with low-molecular-weight PF resin was an effective way to enhance the longitudinal macromechanical properties of wood from the Chinese fir.

Keywords: Phenol-formaldehyde resin; Mechanical properties; Cell wall; Modulus of elasticity; Hardness

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INTRODUCTION

Phenol-formaldehyde (PF) resin was the first synthetic resin to be produced industrially, and it has now been used for over 100 years (Dong *et al*. 2009). PF resin possesses several advantages, including readily available raw materials (for instance, the raw materials of phenol and benzene could be obtained from petroleum), low cost, simple production process and equipment, excellent mechanical properties, dimensional stability, weathering resistance, flame retardancy, and moldability. Therefore, PF resin is indispensible in the manufacture of plywood, fiberboard, particleboard, adhesives, and coatings (Stamm and Seborg 1936; Rowell and Banks 1985; Ryu *et al*. 1991).

Water is used as the solvent for water-soluble PF resins, making such reactions inexpensive, nonhazardous, and difficult to ignite. Such reactions also meet economic and environmental requirements, so they have significant potential for application (Li *et al*. 2001). Much research has demonstrated that the treatment of wood-based materials with water-soluble, low-molecular-weight PF resin obviously improves their mechanical

properties, dimensional stability, resistance to biological deterioration, and fixation of compressive deformation. Therefore, treatment with PF resin is an important way to improve the utility of wood (Stamm and Seborg 1936; Rowell and Banks 1985; Ryu *et al*. 1991; Furuno *et al.* 2004; Liu *et al*. 2004; Wan and Kim 2008).

Early research focused on the macrophysical and macromechanical properties of wood modified with low-molecular-weight PF resin and various microscopic techniques have been used to observe the distribution of PF resin in wood (Inoue *et al.* 1993; Furuno *et al.* 2004; Wan and Kim 2008). However, studies on the mechanical properties of the cell walls of wood after modification with low-molecular-weight PF resin have been limited. To improve its mechanical properties and increase its range of applications, wood from the Chinese fir was impregnated with either low- or high-molecular-weight (*i.e.*, commercial) PF resin. The macro- and micromechanical properties of the treated and untreated Chinese fir samples were then compared in order to better understand the mechanism of resin modification.

MATERIALS AND METHODS

Materials

The four Chinese fir trees (*Cunninghamia lanceolata*) used in the experiment were 40 years old and were collected from the Huangshan Gongyi Forest Farm in Anhui province, China. Radial slices of approximately 100 × 12 × 1.5 mm (longitudinal × radial × tangential) were cut from the sapwood of the fir trees at a trunk height of 1.5 m, which was chosen because this region exhibits less variability than do other areas (Fig. 1). The slices were numbered by the sawing sequence. The total number of samples was 48.

The water soluble low-molecular-weight PF resin was prepared using a molar ratio of industrial phenol : formaldehyde (36.9%) : sodium hydroxide (analytically pure) of 1 : 2.1 : 0.2. Conditions such as reaction temperature, reaction time, and the addition of the catalyst were controlled to obtain low-molecular-weight PF resin. The prepared PF resin was an orange-yellow transparent liquid with a pH of 9.7, viscosity of 36.2 mPa·s (determined using a viscometer (Brookfield, AT85442, MA, USA)), and a solid content of 48%.

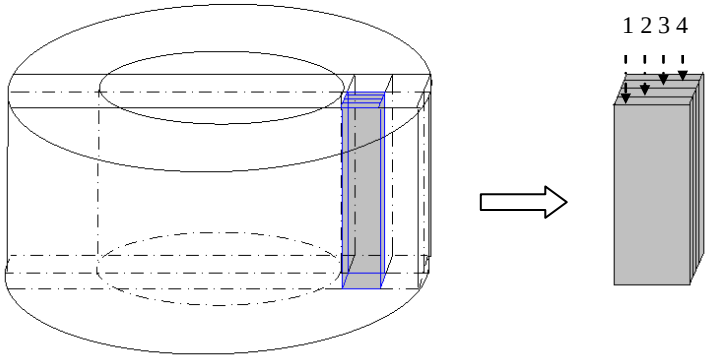
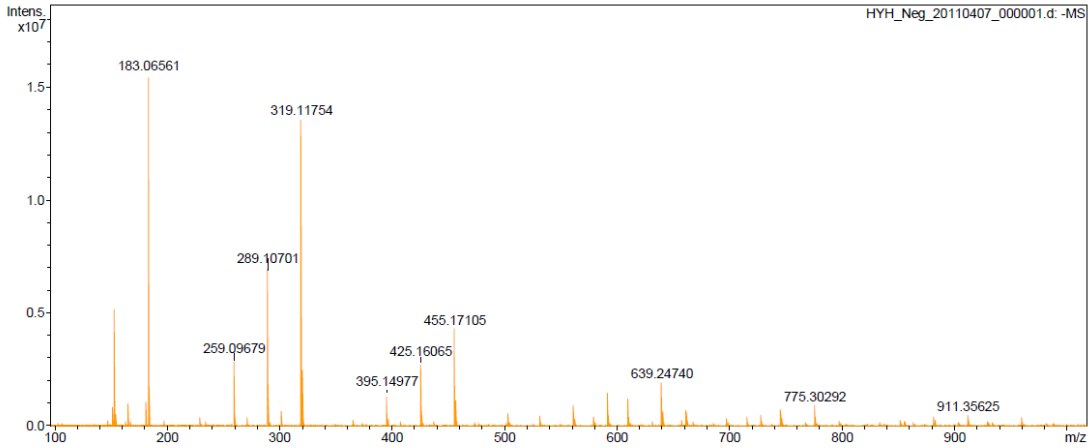


Fig. 1. Schematic diagram of the sample location, sawing method, and treatment sequence. (1) Sample treated with low- or high-molecular-weight PF resin, (2) untreated reference sample, (3) sample treated with corresponding low- or high-molecular-weight PF resin, and (4) untreated reference sample.



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
183.06561	1	C 9 H 11 O 4	100.00	183.06628	0.7	3.7	6.8	4.5	even	ok
259.09679	1	C 15 H 15 O 4	100.00	259.09758	0.8	3.1	12.4	8.5	even	ok
289.10701	1	C 16 H 17 O 5	100.00	289.10815	1.1	3.9	5.2	8.5	even	ok
319.11754	1	C 17 H 19 O 6	100.00	319.11871	1.2	3.7	2.7	8.5	even	ok
395.14977	1	C 23 H 23 O 6	100.00	395.15001	0.2	0.6	3.7	12.5	even	ok
425.16065	1	C 24 H 25 O 7	100.00	425.16058	-0.1	-0.2	6.4	12.5	even	ok
455.17105	1	C 25 H 27 O 8	100.00	455.17114	0.1	0.2	3.2	12.5	even	ok

Fig. 2. High resolution mass spectrum of the molecular weight distribution of low-molecular-weight PF resin.

High resolution electrospray ionization mass spectrometry showed that the molecular weight of the PF resin mainly ranged from 183 to 319, and that the main structures were monomers and dimers (Fig. 2).

The high-molecular-weight PF resin was purchased from Tier Chemical Corporation Ltd., Beijing, China. The high-molecular-weight PF resin was a brown-red transparent liquid with a pH 12.8, viscosity of 82 mPa·s, solid content of 44.3%, and molecular weight ranging from 2000 to 3000.

Treatment with PF Resin

To obtain the resin loading of each sample after treatment with PF resin, the weights of all oven-dried samples were measured before and after PF resin treatment using a balance with a resolution of 0.001 g. Before PF resin treatment, the samples were dried at less than 50 °C to avoid degradation. Before PF resin impregnation, the moisture content of all samples was set to 12% at a temperature of 20 °C and a humidity of 65%.

As shown in Fig. 1, the odd number samples were impregnated with corresponding low- or high-molecular-weight PF resin, respectively. During impregnation, the vacuum chamber containing the samples was maintained at -0.095 MPa for 40 min. Aqueous low- or high-molecular-weight PF resin (25% concentration) was then added to the chamber. The tank was maintained at -0.095 MPa for at least 12 hours. The chamber was slowly pressurized to 0.2 MPa and held for 1 hour, then increased to 0.4 MPa and held for 2 hours, and lastly increased to 0.65 MPa and maintained for 4 hours. After the pressure had been released, the impregnated samples were soaked in the chamber for 24 hours and then placed in a well-ventilated area until a moisture content of about 16% was obtained.

The samples were dried in an oven that was heated under a gradient to prevent the high temperature from affecting the samples under alkaline conditions during the resin curing. The samples were dried in an oven at 45 °C for about 10 hours, 60 °C for 10 hours, 80 °C for 10 hours, and then 100 °C for about 0.5 hours so that the resin was completely cured. The whole impregnating and curing process was designed to be mild but effective enough to make the PF resin penetrate sufficiently into the cell walls of the wood. After impregnation, the samples were equilibrated at 20 °C and 65% humidity before their properties were tested.

Testing the Macromechanical Properties

The macroscopic longitudinal tensile modulus of elasticity (MOE) of the specimens was determined using an electronic universal material testing machine (Instron 5582, Instron Co., Grove City, PA, USA) equipped with a high-precision optical extensometer. To induce fracture in a suitable region during tension, four strengthening slices of wood, each with a length of 20 mm, were attached to both ends of each specimen. In the tests, the load sensor was ±10 kN, the gauge length of the dynamic extensometer was 25 mm, and the loading rate was 2 mm/min. To ensure that the results were reproducible, 13 and 11 groups containing samples treated with corresponding low- and high-molecular-weight PF, respectively, were tested.

Testing the Mechanical Properties of the Cell Wall

A novel nanoindentation technique was used to determine the microscopic mechanical properties of the cell wall. Spurr resin is traditionally used to embed samples for nanoindentation testing; however, it can penetrate the cell walls of wood and influence their mechanical properties. Therefore, nanoindentation tests with *in situ* imaging were performed directly on cross-sections of cell walls in the wood. Following macromechanical testing, blocks with a length of 10 mm were sawn from the samples. Each block was converted into a pyramid using handsaws and a sliding microtome. Each testing plane, with an area of 200 μm^2, was polished with an ultramicrotome mounted with a diamond knife. Each sample was then glued to an iron sample holder with a rapid curing adhesive. All the samples were placed on the magnetic sample table in the nanoindentation testing chamber, and then the temperature and humidity were equilibrated over a period of 4 hours.

The nanoindentation instrument (Triboindenter, Hysitron Inc., Minneapolis, MN, USA) with *in situ* imaging function possessed theoretical resolutions of load and displacement of 1 nN and 0.01 nm, respectively. The temperature and humidity in the testing chamber and laboratory were maintained at 22 °C and 40%, respectively. Indentations were only performed in the cell wall areas of latewood because their thickness made them convenient to locate. A load-displacement curve of a nanoindent was obtained after measuring. The nanoindentation hardness and longitudinal MOE could be calculated by the definite equations. The testing principles and method are described in detail in the literature (Yu *et al* 2007). Using a force-controlled mode, a Berkovich diamond indenter with a tip radius of less than 100 nm was loaded to a maximum of 250 μN at a rate of 50 μN/s, held at constant depth for 6 seconds, and then unloaded at the

same rate. The MOE of the cell wall was calculated from the slope of the unloading curve in the range of 50 to 95%.

RESULTS AND DISCUSSION

Wood is a natural polymer, and it is relatively soft and flexile. After impregnation with PF resin, the PF resin was cured to form a hard, brittle polymer in the voids of the cell lumen and the cell walls of the wood. The resulting wood-PF resin composite possessed mechanical properties different from those of untreated wood.

Table 1. Size and Longitudinal Mechanical Properties of Untreated Chinese Fir and Chinese Fir Treated with Low-Molecular-Weight PF Resin

Group	Sample	Width	Thickness	Max load	MOE	Resin loading	Increase of MOE
		(mm)	(mm)	(N)	(GPa)	(%)	(%)
1	Treated	12.48	1.56	1402.80	12.75	75.68	15.91
	Untreated	12.44	1.52	1201.77	11.00		
2	Treated	12.49	1.56	1367.20	13.07	79.27	51.62
	Untreated	12.36	1.55	1040.09	8.62		
3	Treated	12.33	1.56	1124.45	14.35	76.38	38.65
	Untreated	12.29	1.53	1400.83	10.35		
4	Treated	12.18	1.56	1447.43	11.39	80.29	1.42
	Untreated	12.22	1.55	1367.05	11.23		
5	Treated	12.43	1.57	1000.34	12.78	90.06	53.24
	Untreated	12.44	1.49	746.92	8.34		
6	Treated	12.45	1.54	897.62	8.28	92.21	1.60
	Untreated	12.46	1.49	941.55	8.15		
7	Treated	12.49	1.50	1104.90	12.76	96.23	64.01
	Untreated	12.48	1.57	1023.70	7.78		
8	Treated	12.39	1.52	1501.90	13.93	66.94	21.34
	Untreated	12.36	1.55	1754.52	11.48		
9	Treated	12.38	1.55	1928.77	17.08	61.45	23.23
	Untreated	12.42	1.55	1943.74	13.86		
10	Treated	12.43	1.54	2036.28	14.22	61.80	13.40
	Untreated	12.43	1.52	1918.52	12.54		
11	Treated	12.46	1.49	1961.35	15.71	60.98	1.09
	Untreated	12.50	1.51	1806.48	15.54		
12	Treated	12.51	1.50	1716.77	14.76	63.96	52.16
	Untreated	12.51	1.51	2083.57	9.70		
13	Treated	12.45	1.56	2062.38	15.61	61.78	63.80
	Untreated	12.49	1.54	1833.23	9.53		

Macromechanical Properties

The resin loading of the treated samples was closely related to their macromechanical properties. Generally speaking, the impregnation of PF resin into the wood pores should be greater at higher resin loadings, which can obviously improve the mechanical properties of samples. After treatment with the low-molecular-weight PF resin, the resin loading of the samples increased significantly, with a maximum loading of 96.23% and an average loading of 73.97% (Table 1). The average resin loading was 26.55% higher than those of samples treated with high-molecular-weight PF resin. However, the variability of the former was larger, with a range of 35.25% between minimum and maximum loadings (Table 2). This indicated that the PF resin of low-molecular-weight was better at infiltrating the wood pores than was the high-molecular-weight PF resin, although the distribution of resin in the wood was not uniform.

Table 2. Size and Longitudinal Mechanical Properties of Untreated Chinese Fir and Chinese Fir Treated with High-Molecular-Weight PF Resin

Group	Sample	Width	Thickness	Max load	MOE	Resin loading	Increase of MOE
		(mm)	(mm)	(N)	(GPa)	(%)	(%)
1	Treated	12.46	1.61	1580.93	8.85	50.00	-36.51
	Untreated	12.30	1.60	1681.98	13.94		
2	Treated	12.39	1.57	1731.17	4.82	47.46	-56.62
	Untreated	12.27	1.60	1619.44	11.11		
3	Treated	12.23	1.63	1549.77	8.20	50.56	-21.08
	Untreated	12.12	1.44	1346.86	10.39		
4	Treated	12.48	1.48	1470.17	11.77	47.63	-1.67
	Untreated	12.48	1.45	1770.84	11.97		
5	Treated	12.52	1.52	1637.29	9.75	41.01	-25.29
	Untreated	12.52	1.49	1766.57	13.05		
6	Treated	11.73	1.56	1759.71	4.56	47.61	-65.64
	Untreated	11.76	1.48	2079.48	13.27		
7	Treated	11.90	1.26	1376.51	13.11	47.58	-21.21
	Untreated	11.99	1.40	1498.25	16.64		
8	Treated	12.13	1.51	1706.30	11.37	50.38	-19.65
	Untreated	12.36	1.49	1894.78	14.15		
9	Treated	12.20	1.46	1933.83	9.37	48.71	-41.03
	Untreated	12.43	1.48	1634.92	15.89		
10	Treated	12.54	1.53	2331.38	12.95	47.95	-7.76
	Untreated	12.57	1.59	1861.87	14.04		
11	Treated	12.57	1.53	2269.15	8.91	42.74	-31.83
	Untreated	12.58	1.62	1620.11	13.07		

The increase in the longitudinal MOE of the samples treated with low-molecular-weight PF resin was significant, with an average increase of 30.88%. However, the increase also varied significantly, with a range of 1.42 to 64.01% (Table 1). Compared with control samples, the average longitudinal MOE of the samples treated with high-molecular-weight PF resin was 29.84% lower. This decrease also showed large variation, with values ranging between 1.67 and 65.61% (Table 2).

All of the tested samples were selected from the area of the sapwood (mature wood) with consistent properties, and the treatment and reference radial slices were obtained in an identical manner (Fig. 1). Because the properties of the samples were very similar, the influences of the microfibril angle and density of the wood were not necessary to consider. Therefore, the changes in the mechanical properties of the samples were almost completely caused by the PF resin. Many researchers have confirmed that water-soluble low-molecular-weight PF resin improves the mechanical properties of wood (Miroy *et al* 1995; Liu and Wang 2004; Wan and Kim 2008). The prepolymer solution of PF resin contained a large number of aromatic moieties that are similar in morphology to the main components in wood. According to the principle of "like dissolves like", the PF resin has a propensity to infiltrate wood. After infiltration, the uniformity of the wood and the interaction between the wood and the PF resin are enhanced. Both of these factors improve the mechanical properties of the wood treated with PF resin (Qian *et al.* 2001). The research presented here also confirmed that the longitudinal MOE of wood from the Chinese fir improved significantly after treatment with low-molecular-weight PF resin. Although the high-molecular-weight PF resin also improved the properties of the wood from the Chinese fir, the large molecules of PF resin had difficulty infiltrating the pores in the wood. This prevented a continuous, cross-linked network from forming in the wood to limit deformation (which will be discussed further in the following section). Furthermore, the pH of the high-molecular-weight PF resin was 12.8 because NaOH was added during the preparation of the resin, so its solution was strongly alkaline. Hemicelluloses and other chemical components in the wood could be destroyed by exposure to such alkaline conditions, which reduced the longitudinal MOE of the wood treated with high-molecular-weight PF resin (Curling *et al*. 2001; He *et al*. 2008).

Mechanical Properties of the Cell Wall

As shown in Fig. 3, after treatment with low-molecular-weight PF resin, the average longitudinal MOE and hardness of the cell walls of the Chinese fir tracheids were significantly improved compared with those of the reference samples, increasing by 32.94 and 32.93%, respectively. The cell walls treated with high-molecular-weight PF resin did not show such obvious improvements in longitudinal MOE and hardness. Compared with the control samples, the average increases in longitudinal MOE and hardness for the samples treated with high-molecular-weight PF resin were 11.99 and 18.14%, respectively (Fig. 4).

The penetration of resin into the wood occurred on two or more levels. Micropenetration occurred through the cell lumens and pits, while nanopenetration was observed in the cell walls (Kamke and Lee 2007; Konnerth *et al*. 2008). As early as 1971, Smith and Cote, using electron microscopy and energy-dispersive X-ray analysis,

discovered that brominated PF penetrated the cell wall near the bond interface. Rapp *et al.* (1999) used electron energy loss spectroscopy to confirm that melamine-formaldehyde (MF) resin was present in all layers of lignified cell walls. Furuno *et al.* (2004) observed by electron probe X-ray microanalysis that PF resins with molecular weights of 290 and 470 easily infiltrated the tracheid cell walls of Japanese cedar wood, whereas those with a molecular weight of 820 did not. The molecular weight of PF resin used in this study was mainly less than 400, so the PF resin could diffuse into the nanopores in the S_2 layer of the cell walls of Chinese fir tracheids. The nanoindentation results presented here further confirmed that the low-molecular-weight PF resin reinforced the cell wall of wood tracheids. This finding is also supported by Gindl *et al.* (2002a), who used MF resin to modify the tracheid cell walls of spruce wood, increasing the longitudinal MOE and hardness of the cell walls by 33 and 115%, respectively. Gindl *et al.* suggested that covalent crosslinking between the resin and cell walls reinforced the cell walls and improved mechanical properties (Gindl *et al*. 2002a, 2002b, 2007, and 2010). However, Laborie (2002) confirmed that low-molecular-weight PF resin penetrates the cell walls of wood and forms a continuous crosslinked polymer network, but that PF resin and the components of the wood cell walls do not react directly. It is proposed that the formation of a polymer network of PF resin is the direct cause of the improved macro- and micromechanical properties of the wood from the Chinese fir in this study.

Only a very small fraction of the high-molecular-weight PF resin penetrated the wood samples on a nanometer scale, so it mostly formed separate domains from the wood (Laborie 2002). Therefore, high-molecular-weight PF resin had a limited ability to improve the mechanical properties of the cell walls of Chinese fir. The longitudinal MOE and hardness of the S_2 layer cell walls measured by nanoindentation testing are highly dependent on the microfibril angle and lignin content in the matrix of wood (Yu *et al*. 2006), but the alkaline PF resin solution only degraded the hemicelluloses. Therefore, the alkaline PF resin solution had little effect on the mechanical properties of the cell wall, but had a negative effect on the macromechanical properties of the samples.

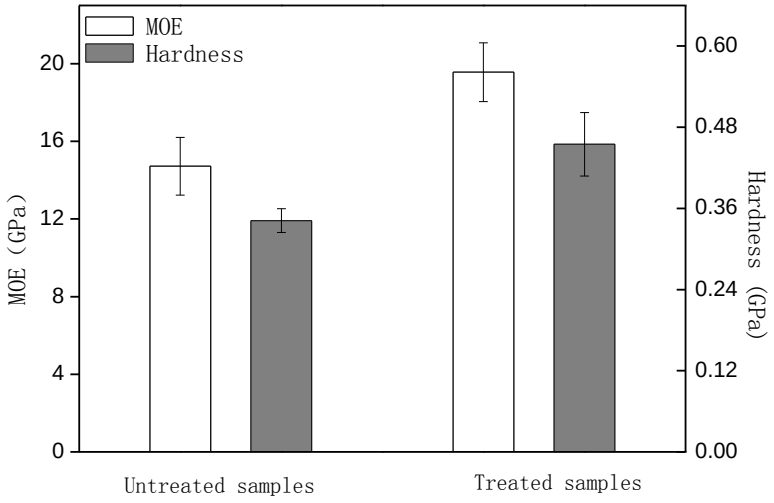


Fig. 3. Cell wall average MOE and hardness of Chinese fir tracheids untreated and treated with low-molecular-weight PF resin.

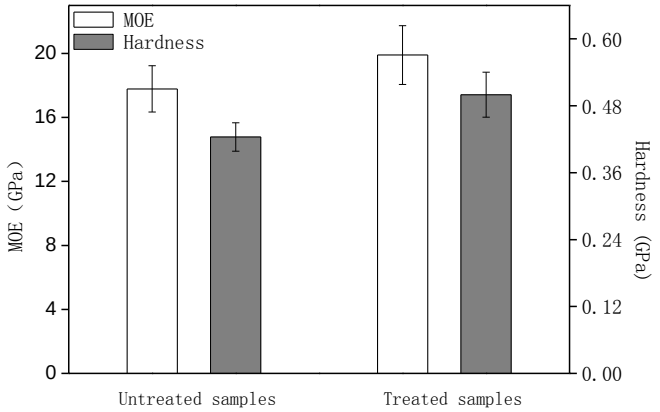


Fig. 4. Cell wall average MOE and hardness of Chinese fir tracheids untreated and treated with high-molecular-weight PF resin.

The improved mechanical properties of the wood from Chinese fir that was treated with the low-molecular-weight PF resin resulted not only from the filling and reinforcing of the cell cavities and walls with resin, but also from the decreased amount of water in the modified cell walls. Overall, treatment with water-soluble low-molecular-weight PF resin was an effective way to improve the longitudinal macromechanical properties of the wood from the Chinese fir. The modified Chinese Fir wood with the low-molecular-weight PF resin could be utilized in applications for several purposes such as garden furniture, outdoor flooring, outdoor building, and decoration.

CONCLUSIONS

1. Treatment with water-soluble low-molecular-weight PF resin significantly improved the MOE of samples from Chinese fir. The average increase due to treatment was 30.88%. In contrast, treatment with high-molecular-weight PF resin decreased the average MOE by 29.84%.
2. Nanoindentation testing showed that the average MOE and hardness of the S_2 layer of the tracheid cell wall were improved by treatment with low-molecular-weight PF resin, with increases of 32.94 and 32.93%, respectively. Smaller increases of 11.99 and 18.14%, respectively, were obtained following treatment with high-molecular-weight PF resin.
3. It could be inferred that the low-molecular-weight PF resin diffused effectively into the nanopores in the cell walls of Chinese fir tracheids and formed continuous crosslinked polymer networks. These networks enhanced the mechanical properties of both the cell walls and macroscale of the wood of Chinese fir. It is also inferred that the large size of the high-molecular-weight PF resin limited its ability to form a continuous crosslinked polymer network and improve the micromechanical properties of the wood. Furthermore, the strong alkalinity of the high-molecular-weight PF resin solution caused the MOE of the samples from Chinese fir to decrease.

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