THE INFLUENCE OF DIFFERENT IMPREGNATION FACTORS ON MECHANICAL PROPERTIES OF SILICA SOL-MODIFIED *POPULUS TOMENTOSA*

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Abstract. The low density of many fast-growing plantation species results in poor mechanical and flexural properties that limit their usefulness. Supplemental impregnation may represent one method for improving wood properties to create new applications for these materials. The potential for using varying silica sol impregnation processes to improve hardness and flexural properties was investigated on plantation-grown *Populus tomentosa*. Silica sol-gel impregnation resulted in significant improvements in hardness, MOR, and MOE. The results suggest that these supplemental processes have the potential to create modified woods with a broader range of potential applications.

Keywords: *Populus tomentosa*, fast-growing wood, silica sol, hardness, MOE, MOR.

INTRODUCTION

Because of its high strength-to-weight ratio, outstanding thermal insulation characteristics, ease of processing, carbon neutrality, recyclability, and beauty, wood is frequently used in construction and decorative applications (Brischke 2020). Nevertheless, increasing global demand for wood products will place increasing importance on the development of fast-growing plantations to fill supply shortages. One negative aspect of this material is that the resulting wood tends to have lower density which results in lower physical and mechanical properties that preclude many more valuable uses. As a result, the wood needs to be modified to improve its usefulness (Cheng 2017).

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Most of the research in modification has concentrated on Chinese fir (*Cunninghamia lanceolata*), eucalypts, and pines (Yang et al 2020), but fast-growing poplars also have potential in this space. *Populus tomentosa* has a straight bole, fine-grained wood, and one of the shortest rotations for a timber species. Poplars are widely used for construction, furniture, and decorative products (Zhang 2012); however, low density, softness, and poor physical and reduced mechanical properties limit their applications (Lang 2016). Modifying wood properties may represent one approach to increasing the utilization of these resources.

Wood modification can be either physical or chemical. Physical modification densifies the cell structure per unit volume to improve mechanical properties (Yang et al 2020), but the improvements via these processes are limited. Gao et al (2019) explored surface compression and heat treatment of poplar. While heat treatment had no appreciable impact on the mechanical properties, the simultaneous use of pressure to densify the wood increased hardness.

Chemical modification can be accomplished by bulking the wood with monomers that polymerize in situ to increase wood properties or by using chemicals that react with cell wall constituents to form covalent bonds with the active components of wood (Lang 2016). Chemical modification can improve mechanical properties, but it can also introduce specialized properties such as hydrophobicity, flame retardancy, or corrosion resistance (Qiu et al 2018). The primary method for chemical modification is to impregnate the wood with a reactant that either reacts at room temperature or the process can be accelerated by heating (Qin et al 2021). The process can occur by simply soaking or fluid uptake can be expedited using combinations of vacuum and pressure to “force” the reactant into the wood (Bao 2021). For example, Wang and Wang (2019) vacuum impregnated fast-growing poplar with unsaturated polyester resin and found that the vacuum level significantly impacted mechanical properties. Similarly, Hou et al (2016) found slight improvements in the wear resistance of modified poplar.

Impregnation with silica-magnesium gel improved the physical and mechanical characteristics of Chinese fir, including hardness flexural properties and compression strength (Zhang et al 2022). Liu et al (2019) used heat treatment and impregnation with a water glass solution to improve the physical and mechanical qualities of poplar. Furfuryl alcohol has also been used to densify the wood surface while also improving dimensional stability and improve mechanical properties (Zheng et al 2022). Liu et al (2021) used a glucose-urea-melamine resin/sodium silicate compound modifier to enhance wood properties.

Silica sol is primarily composed of water and hydroxyl groups, has a low viscosity, small particle size, is nontoxic, and has minimal negative environmental attributes. The silica sol penetrates the wood pores creating a spatial network structure after gelation and drying that can enhance the mechanical and aesthetic qualities of wood (Sun et al 2021).

Lin (2008) found that silica sol impregnation of poplar improved flexural properties but at the expense of reduced compressive strength. Zhang et al (2021) used silica sol to enhance the mechanical properties of wood while also improving phenolic resin durability. Sun et al (2019) varied silica sol solution strength with an ambient pressure treatment to improve the mechanical properties and flame-retardance capabilities of poplar.

Shi et al (2019) impregnated poplar with acid and alkaline silica sols and found that acid silica sol-modified material had higher flexural resistance, whereas alkaline silica sol only improved MOR. Hong et al (2022) found significant improvements in the compressive strength of polyvinyl acetate (PVA)-nano silica sol-impregnated composite wood.

Although soaking treatments can result in high loadings of permeable wood species, combinations of vacuum and pressure can produce higher, more uniform uptakes. Jun et al (2022) found that thermal modification before treatment introduced channels into the wood that facilitated solution uptake, resulting in improved hydrophobicity, dimensional stability, thermal stability, and surface hardness.

The extensive prior research highlights the potential for enhancing the properties of low-density poplar, but a large number of variables complicate process optimization. The objective of our study was to explore a limited number of variables to improve silica sol modification of *P. tomentosa* using an orthogonal test design.

MATERIALS AND METHODS

Materials

Populus tomentosa was provided by Dehua Tubao Decoration New Material Co., Ltd. (Deqing, China). The material was treated for 2 h at

120°C with superheated steam and then dried to 0-3% MC before being cut into eighty 50 mm × 50 mm × 70 mm samples for hardness tests and eighty 20 mm × 20 mm × 300 mm (T × R × L) beams for flexural properties. Silica sol was purchased from Jinan Yinfeng Silicon Products Co., LTD (Jinan, China), and had a solids content of 20%, an average particle size of 8-15 nm, pH of 9.3, and a viscosity of 7.0 MPa. The materials were then allocated to one of nine groups of eight samples each for the hardness and bending tests and conditioned to constant mass at 65% RH and 25°C to an MC of approximately 12%. One group of eight hardness and bending samples was left untreated to serve as a control while the others were assigned to one of eight treatments. The high number of possible variables made complete replication of all possible treatment combinations difficult. Instead, an orthogonal test design was used where variables and treatments were chosen at random among the wide array of choices.

The variables examined were vacuum level (-04, -06, or -0.08 MPa), vacuum time (10, 20, or 30 min), pressure level (0.8, 1.0, or 1.2 MPa), and pressure time (1, 2, or 3 h) (Table 1). Samples were immersed in the silica sol and subjected to the desired vacuum/pressure conditions. The treated materials were conditioned at 25°C and 65% RH for 7 d, then dried at 50°C for 12 h, then 60°C for 12 h, and finally 90°C for 12 h. The material was then reconditioned to constant mass at 65% RH and 25°C. The silica sol treatment altered the EMC to between 9% and 15% MC.

The hardness and flexural elasticity of the two groups of samples were measured by the same superheated steam pretreatment, vacuum pressure impregnation, and dry aging treatment. In the hardness measurement, the MC of the test-modified material was adjusted, and the MC of the nine groups was balanced (close to 12%), which was converted to a value under 12% MC. In MOE and MOR measurements, the flexural modulus and flexural strength of the sample with 12% water content could be calculated when the water content of the sample was in the range of 9-15%.

Hardness Tests

The effect of silica sol treatment on hardness was assessed according to Chinese Standard GB1941-2009 where a 5.64 mm diameter steel indenter was pressed at two points into the radial, tangential, or cross-section of each 50 mm × 50 mm × 70 mm long sample using a universal mechanical testing machine (LX-WN-LL2T, Lixiong, Dongguan, China, UTM) The indenter was pressed at a rate of 3 mm/min or 6 mm/min to a depth of 5.64 mm, and the load required to achieve this depth was recorded (±10 N). After the test, a 20 mm × 20 mm × 20 mm sample was cut from the indented area, weighed, oven-dried

Table 1. Effect of silica sol treatment on the hardness of *Populus tomentosa*.

						Hardness at 12% MC (N)		
No.	Vacuum (min)	Vacuum (MPa)	Time (h)	Pressure (MPa)	MC (%)	End face	Chord plane	Diameter surface
0		Untreated			9.8	4.39 (±0.01)	3.02 (±0.02)	2.96 (±0.02)
1	10	−0.04	1	0.8	10.7	5.64 (±0.02)	4.22 (±0.01)	3.89 (±0.01)
2	10	−0.06	2	1.0	10.8	6.23 (±0.01)	4.80 (±0.02)	4.13 (±0.02)
3	10	−0.08	3	1.2	10.8	6.33 (±0.02)	4.78 (±0.02)	4.36 (±0.01)
4	20	−0.04	2	1.2	11.2	5.85 (±0.01)	4.43 (±0.01)	4.03 (±0.01)
5	20	−0.06	3	0.8	11.4	5.98 (±0.01)	3.90 (±0.02)	4.39 (±0.02)
6	20	−0.08	1	1.0	11.3	6.81 (±0.02)	4.91 (±0.02)	4.22 (±0.02)
7	30	−0.04	3	1.0	10.9	5.44 (±0.01)	4.43 (±0.02)	4.28 (±0.01)
8	30	−0.06	1	1.2	10.8	5.91 (±0.02)	4.14 (±0.01)	4.19 (±0.01)
9	30	−0.08	2	0.8	11.1	6.22 (±0.01)	5.42 (±0.01)	5.04 (±0.02)

Values represent the means of eight replicates per treatment group, whereas values in parentheses represent 1 SD (sample dimensions were 50 mm × 50 mm × 70 mm [T × R × L]).

(105 ± 2°C), and weighed to determine wood MC at the time of testing.

Flexural Testing

The conditioned 20 mm × 20 mm × 300 mm long beams were subjected to third point loading on the UTM according to Chinese Standard GB-1936-1-91 (*test method for MOE*) and GB1936-2-91 (*test method for MOR of wood*). The load was applied continuously at the center span of each beam using a 30 mm diameter load head at a rate of 3 mm/min. Load/deflection was continuously recorded as a failure. The linear portion of the load-deflection curve was used to calculate MOE, while the load at failure vs the beam dimension at that point was used to calculate MOR. The moisture content at the time of testing was determined by cutting a 20 mm cube from the failure zone of each beam. The section was weighed, oven-dried (105 ± 2°C), and weighed again to determine MC. These data were used to correct for slight moisture variations in individual beams.

Statistics

Statistical Package for the Social Sciences (SPSS) was used for auxiliary experimental design, data analysis and to generate the L16(44) orthogonal table to investigate the effects of prevacuum time (A), prevacuum pressure (B), impregnation pressure (C), and impregnation time (D), four influencing factors. The experimental conditions producing the best-modified wood were obtained by using the variance analysis and the range analysis models of SPSS.

The data were subjected to an analysis of variance to determine whether differences between the treatments were significant ($p < 0.1$).

RESULTS AND DISCUSSION

End surface hardness of pretreated and impregnated *P. tomentosa*-modified material increased by 24-55%; chord surface hardness increased by 29-79%; and radial hardness increased by 31-70%, indicating that silicon sol impregnation improved hardness *on all wood surfaces* (Table 1). Improved hardness might reflect the reinforcement of the wood cell wall or filling voids as well as physical, chemical, and mechanical interactions due to bonding.

The bending strength of the modified wood after pretreatment and impregnation increased by 23-70%; while MOE improved by 22-57% (Table 2). Increased MOR and MOE suggest that the silica sol not only bulks the wood but also interacts with the wood cell walls to improve properties. One interesting observation was that MOR and MOE both increased with the initial treatment and then declined with further pressure increases. In the increase of impregnation pressure, the weight gain rate of composite wood also increased, so the elastic modulus also increased.

Table 2. MOR and MOE of untreated and silica sol-modified *Populus tomentosa*.

No.	Vacuum (min)	Vacuum (MPa)	Time (h)	Pressure (MPa)	MC (%)	MOR (MPa)	MOE (GPa)
0		Untreated			8.9	65.7	7.29
1	10	−0.04	1	0.8	9.8	97.6	10.03
2	10	−0.06	2	1.0	9.6	95.2	9.43
3	10	−0.08	3	1.2	10.2	111.9	11.39
4	20	−0.04	2	1.2	11.3	99.9	10.15
5	20	−0.06	3	0.8	10.5	108.8	11.44
6	20	−0.08	1	1.0	9.9	103.6	10.41
7	30	−0.04	3	1.0	10.5	95.2	10.11
8	30	−0.06	1	1.2	10.6	95.5	9.99
9	30	−0.08	2	0.8	11.1	81.1	8.92

Values represent the means of eight replicates per treatment, whereas figures in parentheses represent 1 SD (sample dimensions were 20 mm × 20 mm × 300 mm [T × R × L]).

Table 3. Variance analysis and significance test of the hardness of modified *Populus*.

Properties	Factor	Sum of squares	DF	*F* value	Critical value	Pr > F
End hardness (N)	Prevacuum time	0.193	2	2.573	19	
	Prevacuum pressure	0.984	2	13.120	19	
	Immersion time	0.075	2	1.000	19	
	Immersion pressure	0.069	2	0.920	19	
	error	0.07	2			
Flat hardness (N)	Prevacuum time	0.101	2	1.000	19	
	Prevacuum pressure	1.037	2	10.267	19	
	Immersion time	0.478	2	4.733	19	
	Immersion pressure	0.113	2	1.119	19	
	error	0.10	2			
Edge hardness (N)	Prevacuum time	0.233	2	2.044	19	
	Prevacuum pressure	0.345	2	3.026	19	
	Immersion time	0.152	2	1.333	19	
	Immersion pressure	0.114	2	1.000	19	
	error	0.11	2			

Table 4. Variance analysis and significance test of designed parameters on bending strength and MOE of modified *Populus tomentosa*.

Properties	Factor	Sum of squares	DF	*F* value	*F* critical value	Pr > F
MOR (MPa)	Prevacuum time	308.936	2	39.801	19	*
	Prevacuum pressure	7.762	2	1.000	19	
	Immersion time	262.776	2	33.854	19	*
	Immersion pressure	67.909	2	8.749	19	
	error	7.76	2			
MOE (GPa)	Prevacuum time	1.506	2	25.525	19	*
	Prevacuum pressure	0.059	2	1.000	19	
	Immersion time	3.304	2	56.000	19	*
	Immersion pressure	0.443	2	7.508	19	
	error	0.06	2			

* - significant effect.

While higher pressures increased solution uptake, they could also be associated with increased damage to the wood thereby reducing bending strength.

The analysis of variance examining the effects of prevacuum time, prevacuum pressure, impregnation time, and impregnation pressure on mechanical properties showed that prevacuum pressure affected the end surface and chord surface hardness of the modified *P. tomentosa* (Table 3). The hardness of the radial surface was not significantly affected.

Prevacuum time and immersion time significantly affected the MOR and MOE of the modified *P. tomentosa*. Although both treatment elements were significant, prevacuum time appeared to have a greater effect on flexural properties.

Optimal Modification Process of *P. tomentosa*

Optimum hardness was obtained when the modification parameters were 30 min prevacuum pressure at −0.08 MPa, impregnation for 2 h, and an impregnation pressure of 1 MPa. Optimum improvement in flexural properties was found with a 20 min prevacuum time at −0.06 MPa, and a 3 h impregnation at 1.2 MPa (Table 4). The use of orthogonal comparisons allowed us to reduce the number of variables needed to identify the processes that produced the most improvement in physical and mechanical properties.

CONCLUSIONS

The use of vacuum/pressure treatment with silica sol significantly improved the hardness and flexural properties of *P. tomentosa* although some reductions in flexural properties were noted with some treatments. The use of an orthogonal comparisons approach allowed us to reduce the number of variables examined while still optimizing property improvements within the parameters examined. The results illustrate the potential for improving the properties of fast-growing, low-density plantation *P. tomentosa*.

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