WOOD RESEARCH
64 (1): 2019
145-154

PHYSICAL AND MECHANICAL PROPERTIES OF MODIFIED POPLAR WOOD BY HEAT TREATMENT AND IMPREGNATION OF SODIUM SILICATE SOLUTION

Liu Ming-Li, Li Chun-Feng, Liu Yan-Long
Beihua University, Jilin Provincial Key Laboratory of Wooden Materials Science and Engineering
Jilin, China

(Received November 2017)

ABSTRACT

The objective of this study was to improve physical and mechanical properties of fast-growing Chinese white poplar wood (*Populus tomentosa* Carrière). To this purpose, the heat treatment and impregnation by sodium silicate solution were investigated. In experiments, four processes under four different conditions were applied on poplar wood samples: temperature treatment (T), solution treatment (J), first solution and then temperature treatment (J-T) and first temperature and then solution treatment (T-J). The results showed that all measured mechanical properties were improved under conditions of J process. The hardness, impact toughness, bending strength and modulus of elasticity were improved by 8.4%, 29.2%, 12.0% and 16.1%, respectively. Additionally, tested wood samples exhibited significant increasing of values some mechanical properties such as hardness (70.1%) and modulus of elasticity (80.4%) in comparison with values for untreated samples if treatment was conducted under J-T process conditions. Treated wood by this technology could be utilized as solid wood composite or material for flooring substrate.

KEYWORDS: Poplar, sodium silicate solution, impregnation, heat treatment, physical and mechanical properties.

INTRODUCTION

In recent years for the reason that timber resources are reduced increasingly, fast-growing wood species in wood industry are more important. However, compared with natural trees, fast-growing wood species have wood of lower quality with weaker mechanical properties, which limit their applications. In order to improvement their useable value, it is necessary to modify it.

Heat treatment is one of the commonly used methods of wood modification (Jiang and Lu 2012). Heat treatment can decrease hygroscopicity of wood and improve its dimensional stability (Chen et al. 2010, Feng and Zhao 2011), but also some mechanical properties of wood it may limited. Deng et al. (2009) used hot air and hot vegetable oil to treat Chinese fir (*Cunninghamia lanceolata*). Main results showed that the MOR (modulus of rupture), MOE (modulus of elasticity), compression strength parallel to grain and hardness of wood were significantly changed under examined process conditions. Barcík et al. (2014) deals with nail withdrawal strength of spruce (*Picea abies* L.) with a focus on its dependence on thermal modification. Results showed that the effect of thermal modification was clear: with increasing temperature, gradually decreasing values of nail withdrawal strength. Gašparík et al. (2015) studied the dependence of screw direct withdrawal load resistance on thermal modification of spruce wood. The thermal treatment was performed at three different temperatures: 140, 180, and 220 °C. Main results showed that the effect of the thermal modification was unambiguous: the screw direct withdrawal load resistance decreased with increasing modification temperatures. From this can be concluded that the use of heat modified wood may be restricted in some applications.

Sodium silicate solution (commonly known as water glass) is an inorganic raw material and it has a wide range of sources, low cost and it is friendly to environment. It has been used by domestic and foreign scholars to modify wood.

A research showed that the introducing inorganic substances into wood using the water glass (sodium silicate - boron) compound system results in an increase of 6-25% for bending strength in comparison with the untreated wood. The increase in the modulus of elasticity of such modified material was also 16-41%. The hardness in the cross section also showed a marked increase of 50-150% compared with the untreated wood (Furuno et al. 1996). Composite wood material made by means of sodium silicate, aluminium sulphate, calcium chloride and wood had also good flame retardant and antifungal and anti-swelling properties (Furuno et al. 1991, 1992) Therefore, the modification methods by sodium silicate solution had a greater impact on the performance of the wood by itself; but after the high temperature heat-treatment can be enhanced physical and mechanical properties (Kang 2011, Slimak et al. 2000) and also thermal stability of wood (Qu et al. 2019).

Therefore, this work tries to apply the method of impregnation by inorganic sodium silicate solution combined with high temperature heat-treatment and so to modify properties of fast-growing poplar (*Populus tomentosa* Carrière). The study is mainly aimed to develop of comprehensive improvement physical and mechanical properties of poplar and thus to expand its application field.

MATERIAL AND METHODS

Materials

Poplar (*Populus tomentosa* Carrière) was collected from Jiaohe forest farms of Jilin province. The density of wood was 0.43 $g \cdot cm^{-3}$ and moisture content in range of 8 ~ 12%. Specimens were from sapwood with dimensions 400 × 150 × 25 mm. In experiments, 12 samples were used for each run. Sodium silicate solution was used as follows: modulus (SiO2: Na2O) was 2.3 (M2.3), 2.8 (M2.8) and 3.3 (M3.3), respectively. The mass fraction was 35 ~ 40% and pH = 12.

Equipment

Heat chamber, impregnation vessel, universal mechanical testing machine, scanning electron microscope (SEM) was used.

Determination of modulus of sodium silicate

The modulus of sodium silicate solution is higher; the content of effective SiO_2 is higher. Based on the results of the preliminary test, if modulus of sodium silicate solution is greater than 3.3, the viscosity is very large, flow ability is poor and the solution is penetrated not easily inside the wood. If modulus is less than 2.3, the content of SiO_2 is low; the wood modification effect is limited. Therefore, the study was carried out with modulus of sodium silicate solution between 2.3 ~ 3.3.

Combined process of impregnation and heat treatment

The overall process can be briefly described as follows: samples weighing → impregnation → weighing → air drying → drying → heat treatment.

First, after weighting, the sample was inserted into impregnation vessel and evacuated in the impregnation vessel at vacuum 0.1 MPa during 30 ~ 60 min. Next, sodium silicate solution was sucked off and pressurized to 2 MPa and kept approximately 60 ~ 120 min. Then specimens were modified by liquid again and weighed. Next, the impregnated specimens were drying in air during 24 h. Finally, heat treatment was carried out for 2 h in 210°C.

Process comparison

The term wood modification or treatment summarizes the physical and chemical processes applied to extend the service life of wood by decelerating biological degradation and improving the wood properties without biocide treatment. It is known that thermal modification of wood between 160°C and 260°C leads to an increased biological resistance and dimensional stability (Mahnert et al. 2012). For effect of thermal treatment of wood aimed on physicochemical properties are in literature often recommended conditions: T = 180 - 240°C, t = 4 - 5 h (Cademartori et al. 2013). Additionally, it has been reported that heat treatment of wood close by T = 200°C and t = 3 - 5 h had significant effect on mechanical properties of wood. However, if temperature is higher than 220°C and time is longer than 5 h, the negative effect on mechanical properties was observed (Deng et al. 2009). Process parameters temperature (T = 210°C) and time (t = 2 h) were selected due to sodium silicate modifier needs to react at temperature higher than 130°C. According to (Kang 2011), the temperature over 200°C is better for process.

In this study four kinds of treatment processes were designed and compared. They can be described as follows: single heat treatment (T), single impregnation treatment (J), combined process with impregnation treatment and heat treatment (J-T), and combined process with heat treatment and impregnation treatment (T-J). The process parameters are shown in Tab. 1.

Tab. 1: Treatment processes for poplar wood.

Process category	Symbol	Process parameters
Single heat treatment	T	T = 210°C, t = 2 h
Single impregnation treatment	J	samples evacuated at p = 0.1 MPa, keeping at 30 ~ 60 min; then sodium silicate solution is sucked off and pressurized to 2 MPa, and keeping at 60 ~ 120 min
Combined impregnation treatment and heat treatment	J-T	First J and then T treatment (parameters are the same as above)
Combined heat treatment and impregnation treatment	T-J	First T and then J treatment (parameters are the same as above)

Performance test on physical and mechanical properties

The equilibrium moisture content of the sample was about 5% after heat treatment. Tests were performed according to GB/T 1934.1-2009: Method for determination water absorption of wood, GB/T 1936.1-2009: Method for testing bending strength of wood, GB/T 1936.2-2009: Method for testing bending elastic modulus of wood, GB/T 1940-2009: Method for testing impact toughness of wood and GB/T 1941-2009: Method for testing hardness of wood.

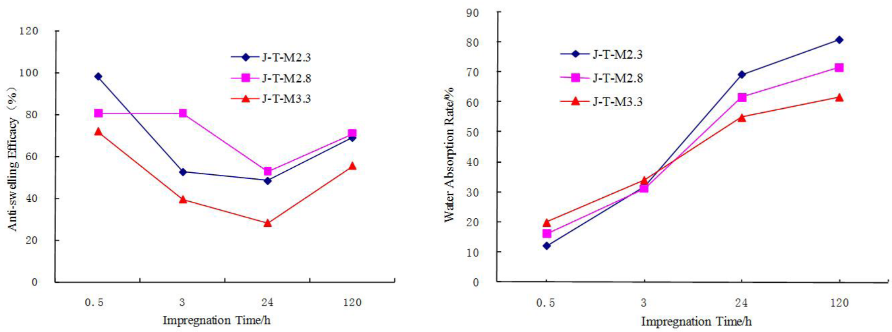
In samples were detected the water absorption rate, bending strength, elastic modulus, the impact toughness and hardness.

RESULTS AND DISCUSSION

Effect of the sodium silicate solution on dimensional stability

Fig. 1 shows dimensional stability of impregnated poplar wood by different solutions of sodium silicate. Results here represent combined impregnation and heat treatment.

From Fig. 1(a) can be seen that the increase the impregnation time, the anti-swelling efficiency (%) of modified samples by different sodium silicate solution was decreased. In addition, Fig. 1(a) also shows slight increase of this property after impregnation time 24 h because the water in wood is saturated by sodium silicate; the anti-swelling efficiency (%) of treated poplar wood tends to be increasing.



(a) *(b)*

Fig. 1: Dimensional stability of poplar wood samples modified by different sodium silicate solutions.

The Fig. 1(b) shows the water absorption rate (%) of treated poplar samples by different sodium silicate solutions as a function of time. After impregnation time of 120 h, water absorption rate (%) of poplar samples was the lowest if the solution with the highest modulus (M3.3) was used for impregnation. This fact is caused probably by the higher content of SiO_2 in solution than that of the others and it can more effectively block channels in wood for water.

Effect of sodium silicate solution on mechanical properties

The Fig. 2 represents the effect of different sodium silicate solutions on mechanical properties of poplar wood samples.

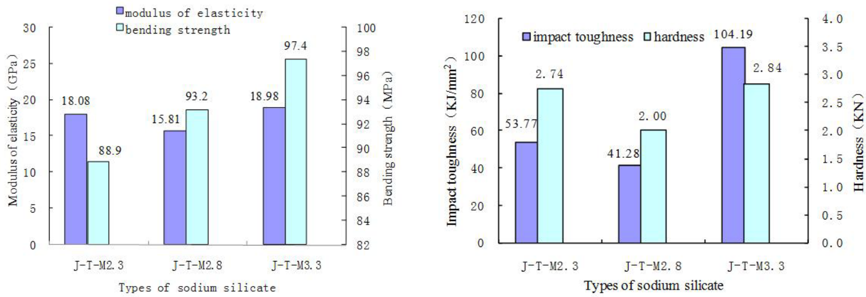


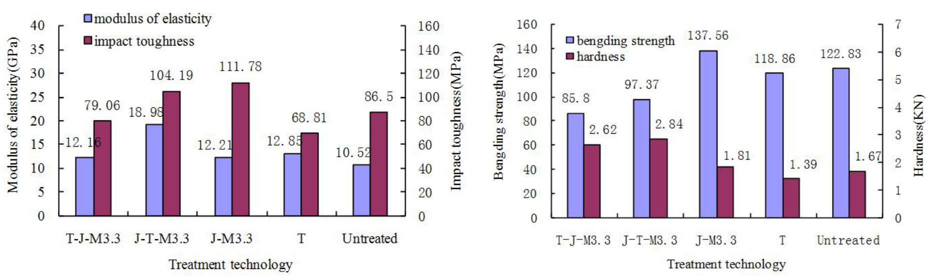
Fig. 2: Mechanical properties of poplar wood samples modified by different sodium silicate solutions.

Under the same process conditions, the hardness, impact toughness, modulus of elasticity and bending strength of the sample modified by M3.3 sodium silicate solution were the highest. These results can be explained by heating temperature above 130°C. The Si-OH bond is dehydrated and associated with to each other and they form Si-O-Si bonds. Next, a three-dimensional structure system with excellent water resistance is created (Kang 2011). Therefore, the effective content of SiO_2 of M3.3 sodium silicate solution is higher than that of the other solutions, more Si-O-Si bonds are formed and thus improving effectively mechanical properties of treated wood. Coefficients of variation for mechanical properties after experiments were in ranges as follow: hardness 6.1 - 10.2%; modulus of elasticity 6.4 - 9.1%; impact toughness 11.7 - 13.1% and bending strength 9.3 - 12.5%.

Considering the effect of sodium silicate modulus on the dimensional stability and mechanical properties of poplar wood, the M3.3 sodium silicate solution was used in subsequent tests.

Effect of tested treatment processes on mechanical properties of poplar wood

Fig. 3 shows the results from experiments where poplar wood samples were treated under conditions of four treatment processes (Tab. 1). In this section, mechanical properties of untreated and treated poplar samples were measured and compared with results of other researchers.



(a) *(b)*

Fig. 3: Mechanical properties of poplar wood samples after different treatment processes.

As can be seen from Fig. 3, the hardness, impact toughness and modulus of elasticity of J-T treated wood samples in comparison with untreated wood were improved by 70.1%, 20.5% and 80.4%, respectively. Peng et al. (2010) in their study also showed that modulus of elasticity was increased significantly. Authors reported that the effect was increased with increasing concentration of sodium silicate. The highest modulus of elasticity reached the value 18.76 ± 0.93 GPa while the control's was 10.96 ± 1.15 GPa. It represents 71.2% increase. Authors used southern pine (*Pinus* spp.) samples instead of poplar samples and similar experimental conditions.

Samples from T-J treatment process showed other values of mechanical properties than samples from J-T treatment process. For instance, here only hardness was significantly improved by 56.9% in comparison with untreated sample. Modulus of elasticity was also improved but only by 15.6%. However, it must be stated that these values are lower than values from J-T treatment process.

Moreover, enhanced mechanical properties were also measured on samples from J treatment process (without thermal treatment). The hardness, impact toughness, bending strength and modulus of elasticity were improved by 8.4%, 29.2%, 12.0% and 16.1%, respectively. This process is suitable to use if all above measured properties have to be enhanced.

For instance, bending strength on Fig. 3(b) increased from 122.83 MPa (untreated sample) to 137.56 MPa (treated sample by sodium silicate). According to Peng et al. (2010), for untreated samples in their experiments, no depositions of material were found in the lumen of the cells. All the treated samples showed depositions of sodium silicate in the lumen of the wood cells. This was confirmed by scanning electron microscopy. In compliance with Kim et al. (2009), modification of wood by SiO_2 mineralization first occurs through the decomposition of some cell walls and the subsequent deposition of silicon dioxide on these walls. In accordance with Hill (2006), artificial oversaturation of wood with silicates leads to structural changes similar to those in natural mineralization. The mineral solution penetrates into the wood, where it remains in lumens, or penetrates the cell walls where it can chemically react with some components. According to Akahane et al. (2004) and Borůvka et al. (2016) the first attempt to carry out mineralization under laboratory conditions was published in 1968. The process of modification of wood by sodium silicate solution may be describes as follows: silicone molecules penetrate well into the wood structure. Due to low stability of silicone molecules they first react with water molecules and produce silanes, which subsequently react with the OH groups of wood (Hill 2006, Donath et al. 2006). In work of Peng et al. (2010) is reported that the function of the sodium silicate depositions is to reinforce the solid wood like rebar in reinforced concrete. This can partially explain the improved bending strength property of the treated wood samples without temperature treatment.

According to Fig. 3(b), the hardness in our results was enhanced in T-J treated (2.62 kN) and J-T treated (2.84 kN) samples in comparison with untreated poplar wood (1.67 kN). This effect is in accordance with the research of Slimak and Slimak (2000). In addition, by course of Neyses et al. (2017) sodium silicate to fill the cell lumen and subsequently polymerise and stabilise the densified wood and increase its hardness.

An improvement of mechanical properties can be explained by physical and chemical changes of wood samples after impregnation process. In addition, it can be assumed that SiO_2 from solution can interact with structure of wood under process conditions and to create some physical or chemical bonds with components of wood.

If samples were modified only temperature without sodium silicate solution, the values of mechanical properties such as impact toughness, bending strength, hardness were decreased as shows Fig. 3(a) and Fig. 3(b). One explanation of this fact may be that the structure of wood after temperature treatment above 200°C is less strong and more fragile (Poncsák et al. 2006). Temperature above 200°C are generally regarded as the boundaries at which wood structure changes are the greatest, leading to a reduced wood strength (Gašparík et al. 2015). From our data of mechanical properties, only modulus of elasticity at these experimental conditions was increased from 10.52 GPa (untreated) to 12.85 GPa (treated sample).

In addition, it must be reported that temperature affects on results a significant way. It depends on when and also how is temperature applied on poplar wood. After all tested treatments, J-T process seems to be the best but only if bending strength property is not necessary to improve. It can be concluded that parameters such as temperature, time and alkaline pH of sodium silicate solution have strong impact on structure of poplar wood during the treatments. In terms of a structure of wood and its individual components, there are many other factors that may affect on mechanical properties of wood, e.g. crystallinity index of cellulose, amount of lignin, physical interaction, hydrolysis reactions and other chemical reaction that may occur during the treatment. For detail elucidation in this way is needed further research.

CONCLUSION

In this study, the affects of sodium silicate solution and heat treatments on mechanical properties of poplar wood were investigated. After all experiments, it can be concluded that the best treatment for poplar wood was J process where high temperature treatment was not applied. Here all tested mechanical properties were improved. The hardness, impact toughness, bending strength and modulus of elasticity were improved by 8.4%, 29.2%, 12.0% and 16.1%, respectively. From processes where were used the temperature, J-T process seems to be the best. Here only bending strength property was not improved. The hardness, impact toughness and modulus of elasticity of treated wood in comparison with untreated wood samples were improved by 70.1%, 20.5% and 80.4%, respectively. Further research is needed for detail elucidation of parameters that may have an effect on mechanical properties of poplar wood.

ACKNOWLEDGMENTS

This work were supported by Key Laboratory of Bio-based Material Science & Technology (Northeast Forestry University), Ministry of Education, National Natural Science Foundation (31300477), Special program for young teachers and promotion program of Beihua University, Jilin Provincial Key Laboratory of Wooden Materials Science and Engineering, Strategic alliance project of industrial technology innovation in Jilin Province (20140309019NY).

REFERENCES

1. Akahane, H., Furuno, T., Miyajima, H., Yoshikawa, T., Yamamoto, S., 2004: Rapid wood silicification in hot spring water: An explanation of silicification of wood during the Earth's history. Sedimentary Geology 169(3): 219-228.
2. Barcík, Š., Gašparík, M., Horejš, P., 2014: Influence of thermal modification on nail withdrawal strength of spruce wood. BioResources 9(4): 5963-5975.
3. Borůvka, V., Ziedler, A., Doubek, S., 2016: Impact on silicon-based chemicals on selected physical and mechanical properties of wood. Wood Research 61(4): 513-524.
4. Cademartori, P.H.G., Santos, P.S.B., Serrano, L., Labidi, J., Gatto, D.A., 2013: Effect of thermal treatment on physicochemical properties of Gympie messmate wood. Industrial Crops and Products 45: 360-366.

5. Chen, R.W., Li, H.M., Yan, T., 2010: Dimensional stability of heat-treated lumber. China wood industry 24(5): 48-49.
6. Deng, S., Yang, W., Chen, R., Lin, J., 2009: Responses of main wood mechanical properties in Chinese fir plantation to different heat treatment process. Scientia Silvae Sinicae 45(12): 105-111.
7. Donath, S., Militz, H., Mai, C., 2006: Creating water-repellent effects on wood by treatment with silanes. Holzforschung 60(1): 40-46.
8. Feng, D.J., Zhao, J.F., 2011: Heat treatment of wood hygroscopicity and dimensional stability. Journal of Northwest Forestry University 26(2): 200-202.
9. Furuno, T., Aono, S., Uehara, T., 1996: Mechanical properties of wood-mineral composites using the water glass-boron compound system. Journal of the Society of Materials Science 45(6): 687-693.
10. Furuno, T., Shimada, K., Uehara, T., Jodai, S., 1992: Combination of wood and silicate II. Water-mineral composites using water glass and reactants of barium chloride, boric acid, and borax, and their properties. Mokuzai Gakkaishi 38(5): 448-457.
11. Furuno, T., Uehara, T., Jodai, S., 1991: Combinations of wood and silicate I. Impregnation by water glass and applications of aluminum sulfate and calcium chloride as reactants. Mokuzai Gakkaishi 37(5): 462-472.
12. Gašparík, M., Barcík, Š., Borůvka, V., Holeček, T., 2015: Impact of thermal modification of spruce wood on screw direct withdrawal load resistance. BioResources 10(1): 1790-1802.
13. GB/T 1934.1-2009: Method for determination water absorption of wood.
14. GB/T 1936.1-2009: Method for testing bending strength of wood.
15. GB/T 1936.2-2009: Method for testing bending elastic modulus of wood.
16. GB/T 1940-2009: Method for testing impact toughness of wood.
17. GB/T 1941-2009: Method for testing hardness of wood.
18. Hill, C.A.S., 2006: Wood modification: Chemical, thermal and other processes. Chichester, John Willey & Sons, 239 pp.
19. Jiang, J., Lu, J., 2012: Research process in effect of heat treatment on wood strength. Journal of Nanjing Forestry University (Natural science edition) 36(2): 133-136.
20. Kang, Y., 2011: The analysis of the solidification mechanisms and water resistance improvement accesses of water glass. Foshan Ceramics 15(5): 18-21.
21. Kim, K.W., Yoon, CH. J., Kim, P.G., Lee, M.B., Lim, J.H., 2009: Fine structure and X-ray microanalysis of silicified woods from a tertiary basin Pohang, Korea by scanning electron microscopy. Micron 40: 519-525.
22. Mahnert, K.C., Adamopoulos, S., Koch, G., Militz, H., 2012: Topochemistry of heat-treated and N-methylol melamine-modified wood of koto (*Pterygota macrocarpa* K. Schum.) and limba (*Terminalia superba* Engl. et. Diels). Holzforschung 67(2): 137-146.
23. Qu, L., Wang, Z., Qian, J., He, Z., Yi, S., 2019: Effect of combined aluminum-silicon synergistic impregnation and heat treatment on the thermal stability, chemical components, and morphology of wood. BioResources 14(1): 349-362.
24. Neyses, B., Rautkari, L., Yamamoto, A., Sandberg, D., 2017: Pre-treatment with sodium silicate, sodium hydroxide, ionic liquids or methacrylate resin to reduce the set-recovery and increase the hardness of surface-densified Scots pine. Biogeosciences and Forestry 10: 857-864.
25. Peng, Y., Han, Y., Gardner, D.J., 2010: Sodium silicate coated wood. In: Proceedings of the International Convention of Society of Wood Science and Technology and United Nations Economic Commission for Europe. Timber Committee. October 11-14, 2010, Geneva, Switzerland.

26. Poncsák, S., Kocaefe, D., Boazara, M., Pichette, A., 2006: Effect of high temperature treatment on the mechanical properties of birch (*Betula papyrifera*). Wood Science and Technology 40(8): 647-663.
27. Slimak, K.M., Slimak, R.A., 2000: Enhancing the strength, moisture resistance a fire-resistance of wood, timber, lumber, similar plant derived construction and building materials and other cellulosic materials. United States Patent. No. 6,146,766.

Liu Ming-li, *Li Chun-feng, Liu Yan-long
Beihua University
Jilin Provincial Key Laboratory of Wooden Materials Science and Engineering
Jilin 132013
China
*Corresponding author: lichunfeng17@163.com