

Sodium silicate coated wood

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Abstract

The objective of this study is to investigate the effect of treatment of wood with inorganic silicon compounds. “Water glass”, an alkali silicate has been shown to enhance the physical properties of wood, such as dimensional stability, hardness, decay resistance, and fire resistance. However, the high hygroscopicity of water glass also creates some drawbacks: 1) absorption of moisture, 2) reduction in bending strength due to the drying process, and 3) high chemical leaching when exposed to water. Using an appropriate drying process for water glass soaked wood showed high water repellency and high fire resistance. The author derived a drying process to treat sodium silicate solution impregnated southern pine (*Pinus spp.*) samples. The effect of cellulose nanofibrils added in the treating solution was also studied. The mechanical test results for the treated samples showed that the flexural modulus of rupture (MOR) and modulus of elasticity (MOE) increased significantly. The effect increased with increasing concentration of sodium silicate. The highest MOR and MOE of the treated samples were 170.4 ± 12.96 MPa and 18.76 ± 0.93 GPa while the controls’ were 97.62 ± 8.05 MPa and 10.96 ± 1.15 GPa, increased by 75 % and 71 %, respectively. A study of water vapor uptake indicated that the treatment decreased the moisture absorption of solid wood. The fracture surfaces of the flexural samples showed the deposition of silicate in the wood cell lumina. The presence of cellulose nanofibrils in the sodium silicate solution did not change the properties of the treated wood samples.

Keywords sodium silicate solution, solid wood, cellulose nanofibrils

Introduction

A wide variety of chemical compounds have been used to modify wood to improve durability. One of the most extensively studied types over last decade are silicon compounds (De Silva and Hillis 1980; Furuno et al. 1992; Yamaguchi 2002). Among silicon compounds, “water glass”, mainly consisting of potassium or sodium silicates or solutions thereof, is the most common. The process using water glass to treat wood is similar to the typical sol-gel process. In the first step, the wood products are impregnated with sodium silicate solution. The second step is to

precipitate the silicate within the wood structure. One of the methods is to precipitate the silicate using metal salt solutions such as aluminium sulphate, calcium chloride, barium chloride, borax (Furuno et al. 1991, 1992). Using this method, Furuno et al. (1992) found that the moisture excluding efficiencies (MEE) were negative due to the high hygroscopicity of the water glass and the unreacted metal salts in the lumina of wood cells. Leaching experiments also indicated that considerable amounts of chemical were washed out when exposed to water (Kartal et al. 2007). The bending strength of wood was significantly reduced by the water glass treatment while the dynamic and static moduli of elasticity (MOE) were changed only slightly (Furuno et al. 1992). The other method used to precipitate the silicate within the wood structure is curing the impregnated silicate through a suitable drying process. In US Patent 6,040,057, the author developed a series of appropriate drying processes to dry the water glass treated wood blocks (Slimark et al. 2000). The author proposed that under the appropriate conditions of heat and dehydration, sodium silicate undergoes a polymerization process. A water insoluble sodium silicate was formed on the wood surface. The author provided data to demonstrate the improved water repellency and fire resistance. The author also claimed that the water glass treatment can increase the strength, but strength data were not provided in the patent. Another study showed that strength properties depend on the curing temperature after impregnation (Matthes et al. 2002). No change or slightly increased strength was observed with air drying of treated samples. A significant strength reduction was observed for high temperature drying (103 °C). The author proposed that the strength reduction was caused by the hydrolysis of the cell wall polysaccharides at high pH and high temperature conditions.

In this study, the objective is to investigate the effect of wood treatment with inorganic silicon compounds. Different loading levels of cellulose nanofibrils used as a nano-reinforcement were added into the sodium silicate solution. Recently, nanotechnology as one of the evolving and promising new technologies has been applied to many fields of study. In the work, the effect of cellulose nanofibrils on the treated wood samples was investigated.

Materials and methods

Materials

Sodium silicate solution N[®] (37.5 wt. % concentration, weight ratio of SiO₂ to Na₂O is 3.22, pH=11.3, viscosity is 180 mPa·s) was provided by PQ Corporation. Cellulose nanofibrils suspension in 10 wt. % concentration was provided by J. Rettenmaier & Sohne, Germany. Southern pine (*Pinus spp.*) samples were purchased from a local lumber supplier. The moisture content of the samples was 7.31%.

Wood sample preparation

Wood samples were prepared to do the static bending and water vapor uptake tests. Static bending testing samples were cut into a rectangular shape to provide the final dimensions of 12.7 mm (thickness) by 12.7 mm (width) by 203 mm (length). The final dimensions of water vapor uptake testing samples were 10 mm (thickness) by 13 mm (width) by 125 mm (length). The long dimension of all the specimens was parallel to the grain whereas the thickness was in the direction of radial growth. For the particular material property test, all the samples were cut from the same board to obtain consistency.

Treating solution preparation

Five different treating solutions were prepared. The formulae are shown in Table 1. The asterisk represents the treating solutions that were prepared. The concentrations of cellulose nanofibrils and sodium silicate shown in Table 1 are solution base. The mixture of cellulose nanofibrils and sodium silicate solution was prepared using a high shear mixer with corresponding weight percent shown in Table 1. A model L4RT-A of Silverson high shear mixer was used to mix the cellulose nanofibrils into the sodium silicate solution at room temperature for 8 min at 5500 RPM.

Treating process

A modified full-cell process typically used in wood preservation processes was employed to impregnate the wood samples. Before impregnation, wood samples were oven dried at 105 °C for 24 hours. The sample weights were measured as W_1 . After drying, the samples were placed in the pressure vessel for impregnation. A plastic net was used to prevent the contact among the samples. The prepared treating solution was then poured into the pressure vessel. An appropriate amount of weight submerged the samples under the treating solution. The pressure vessel was closed and 25 in Hg vacuum was applied for 30 minutes. After the vacuum treatment, 586 kPa pressure was applied for another two hours. The treated samples were then removed from the vessel and the surface was cleaned. The following drying procedures were derived from US Patent 6,040,057. First, samples were left in open room for three days and then dried in an oven at 200 °C for another 6 minutes. After the initial drying process, the treated samples were put in plastic bags for a week. Before material property testing, all samples were dried at 105 °C for 24 hours and then were cooled to the room temperature. The weights of the samples were measured as W_2 . The weight percent gains (WPGs) of the treated samples were calculated as $100 \times (W_2 - W_1)/W_1$. For the treated wood samples, the nomenclature of “T-5-1” represents the samples treated with solution including 5 wt. % of sodium silicate and 1 wt. % cellulose nanofibrils. The treated samples are designated as “T”. The first number represents the weight percentage of sodium silicate and the second number means the weight percent of cellulose nanofibrils in the treating solution.

Mechanical testing

Flexural properties of the treated samples were determined using an Instron machine (Model 8801) with a three point bending fixture. The test method used in this study is derived from ASTM D 143 with the span of 178 mm (7 in) and a testing rate of 1.3 mm/min (0.05 in/min). A linear variable differential transformer (LVDT) was used to measure the true deflection on the tension side of the wood samples. At least eight specimens were tested for each treatment. The control samples, cut directly from the board, were tested and compared to the treated samples.

Water vapor uptake

The water vapor uptake tests were conducted by placing the dried samples (weight W_2) in a conditioning room at a temperature of 20 ± 3 °C and relative humidity of 50 ± 5 %. When a constant weight (W_3) was reached, the moisture absorption was calculated using $100 \times (W_3 - W_2)/W_2$.

Fracture surface examination

Studies of the morphologies of the fracture surfaces of flexural test specimens were carried out using an AMR 1000 (AMRay Co.) scanning electron microscope. All samples were sputter-coated with gold before the microscopic observations were obtained. To investigate the effect of water glass treatment on the morphologies of the composites, the fracture surfaces of control wood samples were also prepared for SEM study.

Results and Discussion

Mechanical testing

Weight percent gains (WPGs) of the water glass treated samples are shown in Table 2. The statistical analysis indicated that higher WPG was obtained with higher sodium silicate content in the treating solution. The samples treated with 25 wt. % sodium silicate solution had the highest WPG of 47 wt. %. At the same concentration (15 wt. %) of sodium silicate, the WPGs of the samples treated with 1 and 2 wt. % cellulose nanofibrils was higher than that treated without cellulose nanofibrils. The mechanical testing results are shown in Table 2 and Figure 1. From Figure 1, it can be seen that the effect of different levels of sodium silicate is more dramatic than that of cellulose nanofibrils addition. The flexural modulus of elasticity (MOE) and modulus of rupture (MOR) are shown in Table 2. The statistical analysis was applied to the MOE and MOR data. All the treated samples have significantly higher MOE and MOR than that of control samples. The MOE and MOR increased with increasing loading levels of sodium silicate. There was no significant difference with respect to the MOR and MOE for the same content of sodium silicate with different levels of cellulose nanofibrils. The highest MOR and MOE values were 170.4 ± 12.96 MPa and 18.76 ± 0.93 GPa, respectively. Compared with the control wood samples, the MOR and MOE increased by about 75% and 71% for the treatment combined with 25 wt. % of sodium silicate and 1 wt. % cellulose nanofibrils. The normalized deflections (deflection/thickness) at maximum load are also shown in Table 2. The decreased deflection indicated that the treated material became more brittle than that of neat solid wood.

Water vapor uptake test

Water vapor uptake results are shown in Figure 2. The control samples used in this study were also dried in an oven at 105 °C for 24 hours. The WPGs of the samples are similar with the samples used for mechanical testing. The statistical analysis of moisture absorption properties indicated that all the treated samples have lower moisture absorption than the control samples. The samples treated with 25 wt. % of sodium silicate have the lowest moisture absorption value of 3.92 ± 0.67 %. This decreased by 48% with respect to the control samples. No obvious effect was observed by adding the cellulose nanofibrils in the treating solution.

Fracture surface examination

The fracture surfaces of tested samples scanned by SEM are shown in Figure 3. The image A in Figure 3 portrayed the fracture surface of the control wood. No depositions of material were found in the lumina of the cells. All the treated samples showed depositions of sodium silicate in the lumina of the wood cells. The function of the sodium silicate depositions is to reinforce the solid wood like rebar in reinforced concrete (Image B in Figure 3). This can partially explain the improved bending properties of the treated wood samples. The fibrous shape of the deposited

chemicals can be clearly seen in the SEM images in Figures 3. According to images in Figure 3, the deposited sodium silicate was presumed to be polymerized silicate. In image B, the deposited fibrous shaped sodium silicate still remains while the wood fiber was fractured. The fracture surface of deposited sodium silicate at low concentration (5 wt. %) were much coarser than the samples at higher concentrations of sodium silicate (15 and 25 wt. %) (See the arrow in image C of Figure 3). In addition, the fracture cross-section of dried sodium silicate at 15 wt. % differs from that at 25 wt. %. At 15 wt. % of sodium silicate, most of the formed reinforcement is located nearly in the center of the wood cell lumina and they are solid (Image D in Figure 3). There is considerable free space between the dried sodium silicate and cell lumina compared with the samples treated with 25 wt. % sodium silicate (Image E in Figure 3). The deposition formed in the 25 wt. % sodium silicate treated samples is hollow. In image F of Figure 3, a replica of the cell wall pit was formed on the surface of dried sodium silicate (the white arrow). This phenomenon indicated the decreased moisture absorption for the treated samples. The deposited dried sodium silicate blocked the pit connecting the two wood cells, preventing the migration of moisture in the wood cell through the pit. However, no cellulose nanofibrils were observed in the treated sample through the SEM examination. This may indicate that the cellulose nanofibrils did not penetrate into the wood samples. The long length of cellulose nanofibrils may be responsible for the lack of cell penetration through the pit.

Conclusions

Southern pine samples were impregnated by several different solutions combined with sodium silicate and cellulose nanofibrils. The mechanical test results indicated that the flexural modulus of rupture (MOR) and modulus of elasticity (MOE) increased significantly for the treated samples. This increase was related to increasing concentrations of sodium silicate. The highest MOR and MOE of the treated samples were 170.4 ± 12.96 MPa and 18.76 ± 0.93 GPa while the controls' were 97.62 ± 8.05 MPa and 10.96 ± 1.15 GPa, increased by about 75 % and 71 %, respectively. The study of water vapor uptake properties indicated that the treatment decreased the moisture absorption of the wood. The fracture surfaces which were examined using scanning electron microscopy (SEM) showed the deposition of silicate in the wood cell lumina. No significant difference was observed between the treated wood samples with cellulose nanofibrils and that without cellulose nanofibril in the solution.

Table 1. Formulae of treating solutions.

Weight percent of sodium	5 wt. %	15 wt. %	25 wt. %
Weight percent of cellulose			
0		*	
1 wt. %	*	*	*
2 wt. %		*	

Table 2. WPGs and mechanical properties of treated

Sample	WPGs (%)	MOR (MPa)	MOE (GPa)	Deflection/thickness
Control	0	97.61±8.05	10.96±1.15	38.23±6.77
T-5-1*	6.69±1.36	123±11.5	12.13±1.01	36.35±3.06
T-15-0*	22.35±2.53	136.39±11.27	14.14±1.25	33.71±2.65
T-15-1*	26.01±1.63	133.59±14.04	14.05±1.12	35.47±4.53
T-15-2*	26.89±1.56	140.09±9.52	14.49±1.13	33.62±3.04
T-25-1*	46.53±2.12	170.4±12.96	18.76±0.93	31.38±3.14

* T indicates treated, the second value represents wt. % of sodium silicate and the third number represents the wt. % of cellulose nanofibrils.

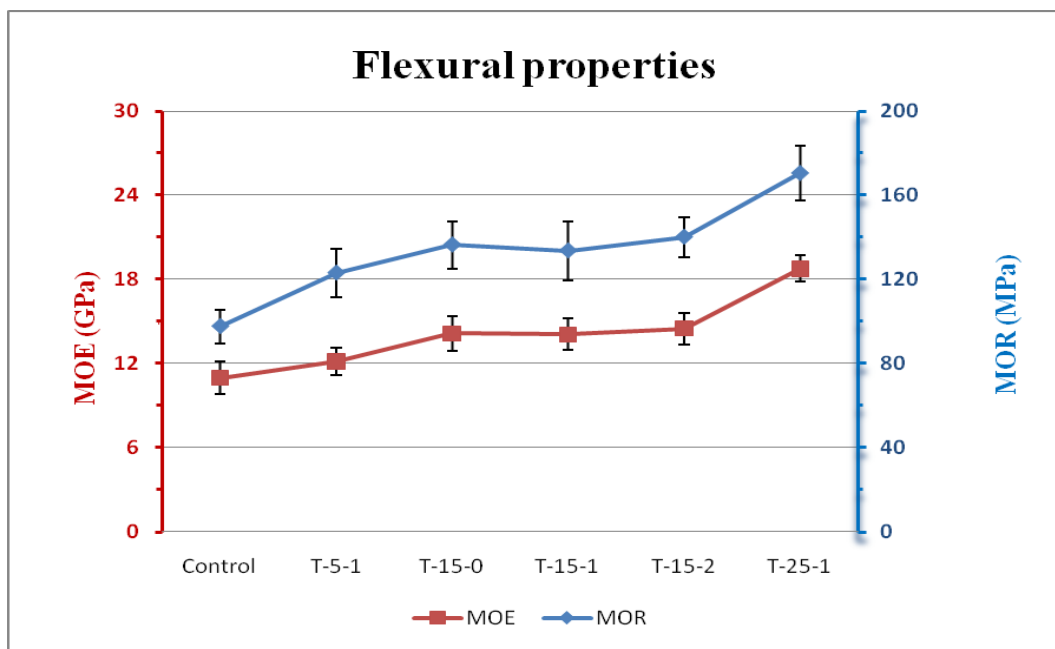


Figure 1. Flexural MOE and MOR of control and treated wood

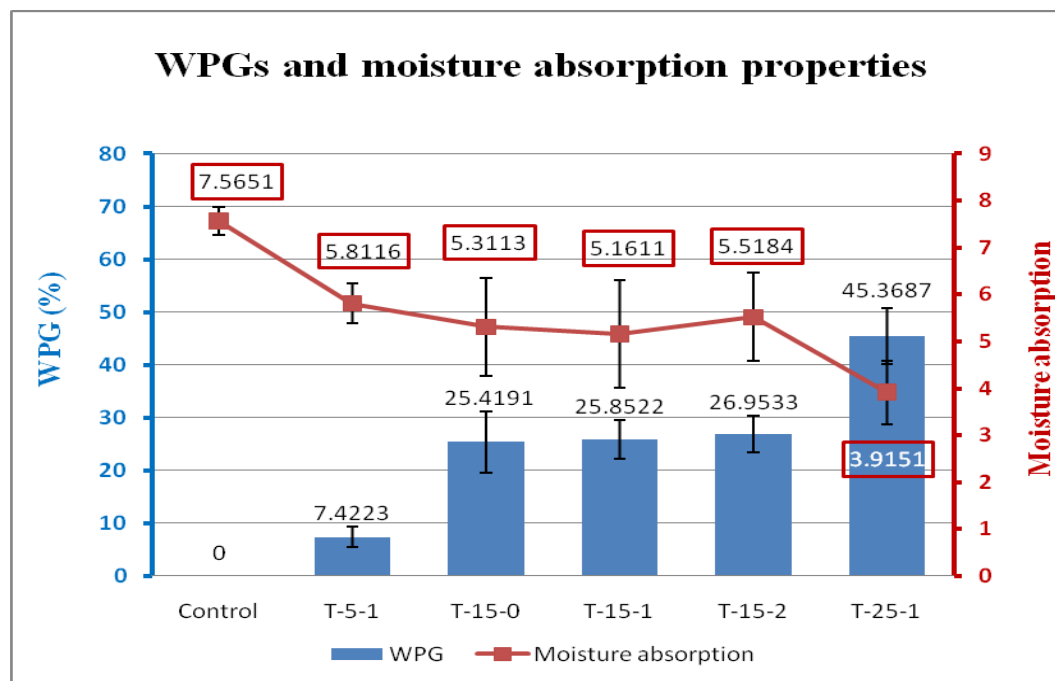
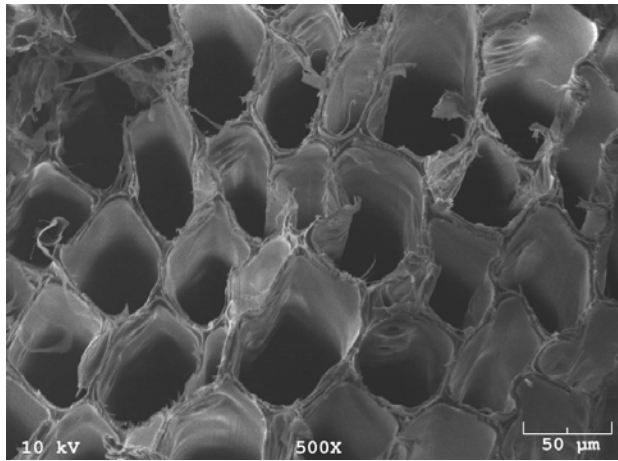
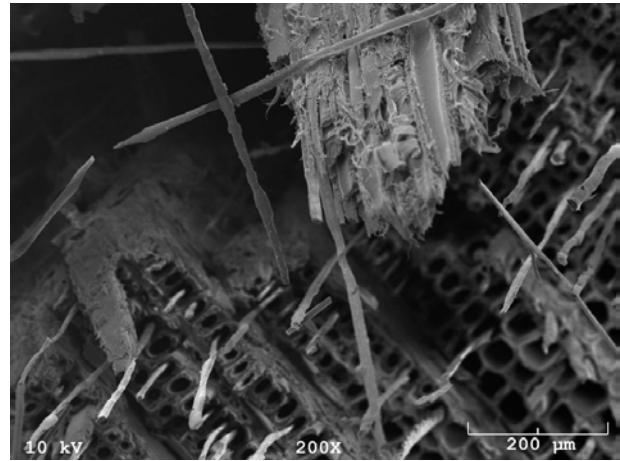


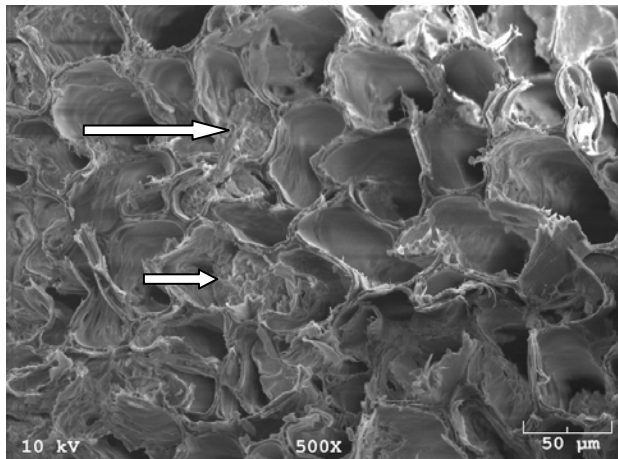
Figure 2. WPG and moisture absorption



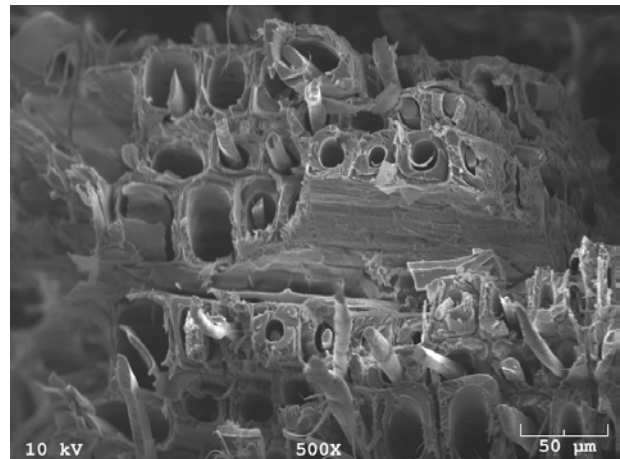
A: Control



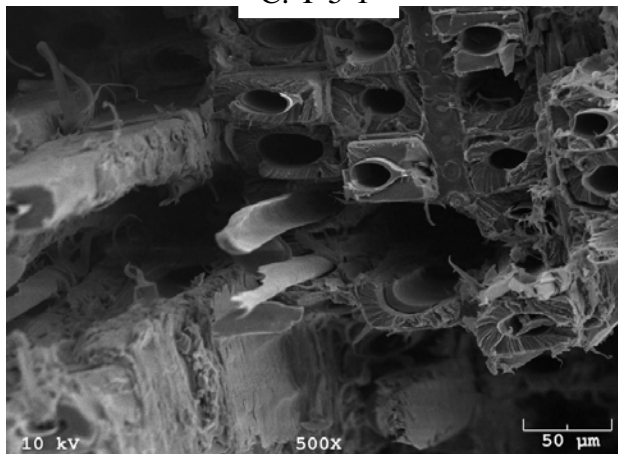
B: T-15-2



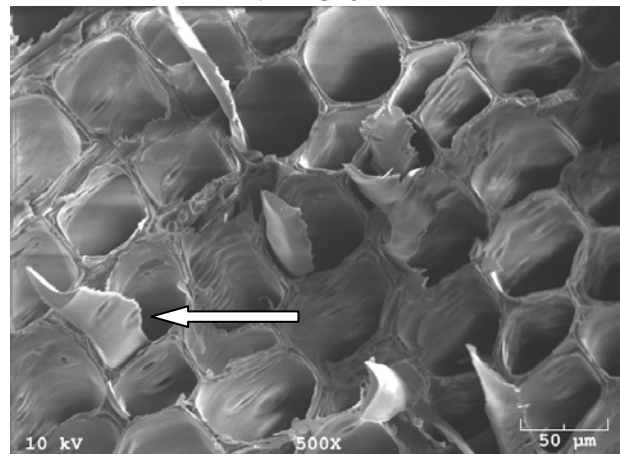
C: T-5-1



D: T-15-0



E: T-25-1



F: T-15-1

Figure 3. Fracture surface of the flexure samples.

References

De Silva, D. and Hillis, W. E. 1980. The contribution of silica to the resistance of wood to marine borers. *Holzforschung* 34: 95-97.

Furuno, T., Shimada, K., Uehara, T. and Jodai, S. 1992. Combinations of wood and silicate. 2. Wood-mineral composites using water glass and reactance of barium chloride, boric acid, and borax and their properties. *Mokuzai Gakkaishi* 38: 448-457.

Furuno, T., Uehara, T. and Jodai, S. 1991. Combinations of wood and silicate. 1. Impregnation by water glass and applications of aluminium sulfate and calcium chloride as reactants. *Mokuzai Gakkaishi* 37: 462-472.

Kartal, S. N., Hwang, W., Yamamoto, A., Tanaka, M., Matsumura, K. and Imamura, Y. 2007. Wood modification with a commercial silicon emulsion: effects on boron release and decay and termite resistance. *International Biodeterioration & Biodegradation* . 60(3): 189-196.

Mai, C. and Militz, H. 2004. Modification of wood with silicon compounds. Inorganic silicon compounds and sol-gel systems: a review. *Wood Sci Technol* 37: 339-348.

Matthes, R., Nehring, H. O. and Dellith, W. 2002. Wasserglas-Holzschutz im Holzbau. In: Proc "Intergrierter Umweltschutz im Bereich der Holzwirtschaft" January 22-24, 2002, Gottingen, pp 104-108.

Slimak, R. A., Haudenschild, C.C. and Slimak, K. M. 2000. US Patent 6040057: Enhancing the strength, moisture resistance, and fire-resistance of wood, timber, lumber, similar plant-derived construction and building material, and other cellulosic materials.

Yamaguchi, H. 2002. Low molecular weight silicic acid-inorganic compound complex as wood preservative. *Wood Sci Technol* 36: 399-417.