The Effects of Acrylate Impregnation of Black Spruce Timber as Connectors Strength

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Chemical impregnation of black spruce was conducted to enhance the wood embedment capacity. The formulation was made of 1,6 hexanediol diacrylate, trimethylpropane triacrylate, and a polyester acrylate oligomer. A second formulation, same as the first but with 1% wt of SiO_2 nanoparticles, was selected to investigate the potential of nanoparticles and to improve the efficiency of the treatment. The wood embedment capacity was carried out by a dowel-bearing test, which was performed for the two treatments and for an untreated wood group. Both treatments showed an increase of strength of nearly 50% when compared to untreated samples. Micrograph views revealed that the impregnation solution penetrated into the wood only up to 100 µm. Hence, with low chemical consumption, the structural bearing capacity can be significantly increased.

Keywords: Impregnation; Wood; Black Spruce; Connector; Bearing strength

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INTRODUCTION

It is not without reason that architects try to expand the use of wood in tall buildings. This renewable material makes incredible structures with a warm and bright look. It also provides an answer to an increasing demand of environmentally friendly construction materials. Wood buildings can offer lower greenhouse gas emissions, less air pollution, and lower volumes of solid waste. The choice of products in buildings correlates to an environmental impact, and wood can help to reduce it. In the life cycle assessment (LCA) study of a building, wood shows significant advantages. Compared to functionally-equivalent products made from other materials, Werner and Richter (2007) reported that wood products are particularly favourable for the consumption of non-renewable energy and cumulated energy demand. The trees in a forest can absorb quantities of CO_2 and store it. Approximately 0.9 metric tons of CO_2 equivalent is stored in every cubic metre of wood. Furthermore, wood can help to reduce the carbon footprint by 1.1 t of CO_2 per $m^3$of wood in comparison with a steel or concrete structure, resulting in a total of 2 t of CO_2 per cubic meter of wood (Frühwald 2007). Replacing concrete by mass timber in tall buildings is a current target for architects (mgb ARCHITECTURE + DESIGN *et al.* 2012). As the size of the buildings grows, the fastener design is the significant limiting criterion of the structural design. Consequently, the constructive system tends to lead to an over-size timber structure, bringing limitations in the architectural design. New strategies need to be developed with the aim to reinforce the critical point of a structure, the fastener attachment area. Creating new types of connectors, or improving upon current ones, are popular topics in timber construction research, such as the work of Gattesco (1998), Loferski and Platt (1999), Mungwa *et al.* (1999), Pantelides *et al.* (2010), Pizzi *et al.* (2006), and Yeh *et al.* (2012). Compared to previous research, the aim of this study was to improve mechanical resistance of wood in the fastener area. Improvement of wood by increasing its water repellent properties, dimensional stability, or flame resistance was done by Mathias *et al.* (1991), Hazarika *et al.* (2012), Wang *et al.* (2014), Cai *et al.* (2007), Islam *et al.* (2012), and Sun *et al.* (2013). Furthermore, adding nanoparticles leads to better results, such as increase of hardness or dynamic Young's modulus (Cai *et al.* 2007, 2008; Shi *et al.* 2007; Islam *et al.* 2012). With the aim to improve the mechanical properties of wood, Bergman *et al.* (2009) used impregnation and coating. Most of the time, high chemical retentions are wanted to distribute the solution equally and fully in the wood. Preliminary studies showed that impregnation of low-permeable species, like black spruce, resulted in a noticeable increase in mechanical resistance without a high chemical retention. Therefore, surface treatments with limited penetration, such as brushing and pulverisation, were tested in preliminary work. It was concluded that these treatments were efficient in increasing the mechanical resistance in wood dowel bearing strength by 20%. However, with the impregnation process, the gain reached 50%. This last method was retained for further experiments, as it showed the highest potential. In the current study, black spruce was chosen, since it is extensively used to manufacture engineered wood products in Eastern Canada (Nordic Structures Inc. 2015). The treatment was based on acrylate monomers and oligomers. The impregnation program, vacuum, and pressure steps were adapted from the literature (Fuller *et al*. 1997).

The aim of this study was to increase the mechanical performance of connectors in black spruce through the manufacture of a wood based product obtained from the impregnation treatment with or without silica nanoparticles. In order to quantify the treatment effect on the mechanical resistance of timber in a connection, an embedment test was performed to study the dowel bearing strength of timber according to ASTM D5764 (ASTM International 2013).

EXPERIMENTAL

Materials and Impregnation Process

Wood specimens

Black spruce *(Picea mariana* (Mill.) B. S. P.) was obtained from Les Chantiers Chibougamau Ltée in Chibougamau, Province of Quebec, Canada. This species accounts for 12% of Canada's total softwood inventory and is desirable for its straight lines, light weight, fiber density, and dimensional stability (Forintek Canada Corp. 2006). Wood block samples were cut to dimensions of 140 mm x 89 mm x 38 mm (L x T x R) and conditioned at 20 °C and 65% RH until constant mass at 12% humidity. Defect-free wood samples were selected. As is shown in Fig. 1, a 14 mm diameter hole was drilled in the longitudinal-tangential plane for each specimen. The 14 mm hole allows a 12.7 mm diameter bolt to be inserted; this is a common diameter for bolts used in construction. Samples were numbered t-1, t-2, t-m, …, t-30; i-1, i-2, i-m, …, i-30; n-1, n-2, n-m, …, n-30, where the maximum for m is 30 for the total number of repetitions by treatment. The letter t is for the control group, i is for the impregnation treatment group, and n is for the impregnation with nanoparticles group.

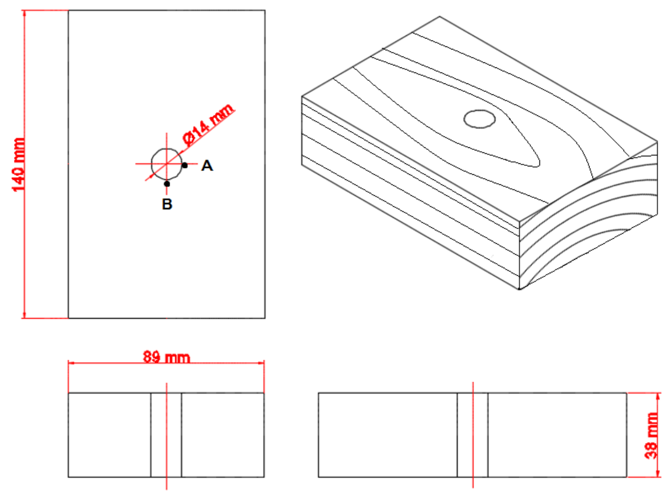

Fig. 1. Wood sample dimensions

Chemicals and formulations

The chemicals 1,6 hexanediol diacrylate (HDDA), trimethylpropane triacrylate (TMPTA), and a polyester acrylate oligomer (CN2262) were obtained from Sartomer Americas. Dispersion at 50 wt% of surface-treated silica nanoparticles in HDDA (Nanobyk 3605) was obtained from BYK Additives and Instruments (Germany). The thermal polymerization initiator, Vazo 67 (2,2′-azobis(2-methylbutyronitrile)) was supplied from DuPont Canada. The acrylate monomer HDDA was chosen because of its low viscosity and its ability to increase the hardness of wood (Cai and Blanchet 2011), while TMPTA brings three functional sites for three-dimensional polymeric network. The aim to include an oligomer was to obtain a less brittle polymer. The ratios of HDDA, TMPTA, and CN2262 were adjusted to obtain a 16 cP viscosity formulation. Blending of formulation was achieved using a Ragogna (Canada) high speed disperser at a 30 Hz frequency. Table 1 gives the details of the formulation. To obtain the second formulation, Nanobyk 3605 was added, resulting in 1 wt% of SiO_2. The reasoning to include nanoparticles was based on the work of Cai and Blanchet (2010), where nanoparticles have created a peak of density at the wood surface leading to a superior hardness because of the accumulation of particles limiting the flow of formulation into the samples.

Table. 1. Weight Percentages of Chemicals for the Two Formulations

	Ratio of chemicals (%wt)	
	Formulation 1	Formulation 2
HDDA	70	70
TMPTA	20	20
CN2262	10	10
Nanobyk 3605	0	1

Impregnation process

The mass of the samples was determined before and after treatment in order to monitor the chemical retention. Then, samples were isolated in a plastic container and immobilized with a heavy load on top. Samples were covered fully in chemicals and then placed into the impregnation cylinder. A vacuum of 27 mm Hg was created and maintained for 15 min to remove the air from the wood pores. Then, a pressure of 520 kPa was applied for 15 min. Samples were wiped off and placed in the oven at 85 °C for 24 h to achieve resin cure. After polymerization, all blocks were conditioned at 20 °C and 65% RH until constant mass was obtained in order to reach 12% of moisture content (MC). The impregnation process was defined as traditional (Fuller *et al.* 1997) to ensure a maximal retention, but the process parameters could be optimized.

Chemical retention

At constant mass, after being conditioned at 20 °C and 65% RH, samples were weighted to determine mass after impregnation. Chemical retention was defined as the mass of the impregnated specimens adjusted at 12% moisture content minus mass before impregnation at 12% MC over mass before impregnation at 12% MC.

Test Method

Dowel-bearing strength

According to ASTM D5764 standard, dowel-bearing strength (embedment) parallel to the grain was measured. The dowel-bearing behavior is the load-deformation behavior of wood under lateral loading by an assumed non-bending fastener. The dowel-bearing strength (σ_{max}) is defined as the yield load (P_{max}) obtained from the load-deformation curve divided by the dowel diameter (d) and specimen thickness (e) (seen in Eq. 1).

$$\sigma_{max} = \frac{P_{max}}{(d*e)} \tag{1}$$

A dowel of 12.7 mm diameter was inserted into the 14 mm diameter predrilled hole. A MTS Alliance RT/50 (MTS Systems Corporation, USA) testing machine with a 50 kN load cell was used. The rate of motion of the movable crosshead was 1 mm/min during the test with a maximal displacement of 5 mm. A testing device (seen in Fig. 2) was manufactured and assembled on the cross-head in order to carry out the test. Slipping of the samples was prevented by a surrounding basement.

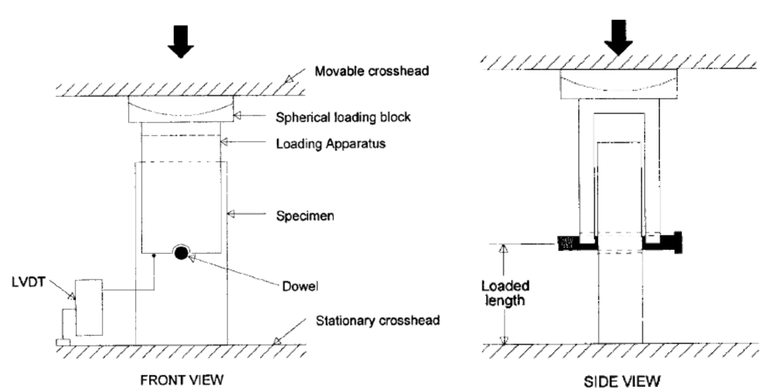

Fig. 2. Testing device to determine dowel-bearing strength (from ASTM D5764)

The displacement was considered to be the cross-head vertical movement, measured by TextWorks software (MTS Systems Corporation, USA). The ultimate loads and the load-displacement curves were monitored at 1 N accuracy. Tests were conducted in a controlled environment to maintain the 12% equilibrium moisture. Thirty repetitions were performed for each group of replicate samples.

Moisture content and oven-dry density

After the dowel-bearing test, samples were cut and weighted to determine the mass when conditioned at 20 °C and 65% HR. Then, they were placed in the oven at 103 ± 2 °C until reaching constant mass to determine oven-dry weight. Moisture content during dowel-bearing test is defined as the difference between the sample mass conditioned at 20 °C and 65% HR and the oven-dry mass divided by the oven-dry mass. The oven-dry dimensions of cut samples were also measured to determine the oven-dry density according to ASTM D2395 (ASTM International 2014).

Scanning electron microscopy

The polymer depth of penetration in the wood was determined through scanning electron microscopy (SEM). Wood blocks of 1 cm^3 cut from samples of each group were prepared with a microtome on the transversal surface. Areas near the hole boundary were photographed with a scanning electron microscope JEOL JSM-6360LV (JEOL Ltd., Japan). Other 1 cm^3 blocks were cut to analyze the deformation zone close to the hole in the longitudinal-radial plane.

Statistical analysis

Results were statistically analyzed using SAS software program, version 9.4 TS (SAS Institute Inc., USA). One-way analysis of variance (ANOVA) was conducted with the general linear model procedure to determine any significant difference on the dowel-bearing strength between treatments. Linear contrasts were made between the untreated and both treated groups.

RESULTS AND DISCUSSION

Chemical Retention

Table 2 shows average chemical retention (CR) and oven-dry density of the tested group after treatment with and without nanoparticles.

Table. 2. Chemical Retention and Oven-Dry Density for Untreated, Treated, and Treated NP Groups of Samples (Mean of 30 Samples and Coefficient of Variation).

Wood samples	CR (COV) (%)	Density (COV) (g/cm3)
Untreated	-	0.46 (11%)
Treated	7% (49%)	0.46 (6%)
Treated NP	4% (55%)	0.45 (7%)

Chemical retention results were low, as expected according to the low permeability of the spruce species. For example, the intrinsic longitudinal permeability of white spruce (k_{iL}) is 2.8408 $m^3_{gaz} * m^{-1}_{wood}$ x 10^{-15} for duramen and 4.4589 $m^3_{gaz} * m^{-1}_{wood}$ x 10^{-15} for sapwood (Messaoud 2007). Tangential tracheids in the black spruce have diameters 25 to 30 μm, while tracheids in a balsam fir have diameters up to 50 μm (Brown 1949). Also, this refractory species tends to have low permeability due to the closure of pits during the drying process. Bordered pits are frequent and are characterized by the formation of a pit chamber after the constriction of part of the pit cavity. A torus can be created by the layer of microfibril strands over the pit aperture. This presence limits the passage of liquids, even more when an aspirated pit is formed; the torus is moved to the aperture to close it completely (Panshin and De Zeeuw 1980). This is why the non-porous structure of black spruce did not make this species popular for impregnation treatment. To compare the differences between treatments, chemical retentions were slightly higher for treated samples without NP group. As seen before in the work of Cai and Blanchet (2010), nanoparticles act as a barrier for chemical penetration in wood when they accumulated in the pores near the surface. Poor dispersion could lead to the formation of agglomerates that are too large to penetrate further into the wood. This tends to limit the flow of formulation into samples. As a surface treatment is intended, low chemical retention is adequate to offer the highest mechanical performance.

Dowel-bearing Strength

The dowel-bearing strength and stiffness, as determined according to ASTM D5764, are shown in Table 3 for untreated, treated, and treated with NP samples. These data are averages of 30 specimens. Using SAS one-way variance analysis, homogeneity groups were formed. Data with same letters are not significantly different. The percentage of change is also included to show the impact of the treatment. To find the bearing stress, load is divided by the projected surface of the bolt. Stiffness (k_e) was found using the slope of the linear area of the stress load-displacement curve.

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Table. 3. Bearing Strength and Stiffness for Untreated, Treated, and Treated NP Groups of Samples (Mean of 30 samples and Coefficient of Variation)

Wood samples	Bearing strength (COV) (MPa)	Change (%)	Stiffness (k_e) (COV) (kN/mm)	Change (%)
Untreated	27.2 ^{a} (10%)	-	10.7 ^{a} (11%)	-
Treated	40.4 ^{b} (6%)	+48%	13.6 ^{b} (7%)	+27%
Treated NP	39.2 ^{b} (7%)	+44%	13.5 ^{b} (9%)	+26%

Data with the same letter are not statistically different at a 95% level of confidence.

The treated samples without nanoparticles showed an increase of strength of 48% (40 MPa) compared to the untreated ones (27 MPa), while the increase with nanoparticles was 44% (39 MPa). Yielding was the failure mode for all groups; for the untreated one, a split could occur after crushing. Means for mechanical properties tested were not statistically different for the two treated groups, with and without nanoparticles. The means of bearing strength and stiffness for the treated group with nanoparticles was only slightly lower than without nanoparticles.

The minor impact of nanoparticles can be explained by the low penetration of formulation in the wood and, consequently, the poor density of nanoparticles in the wood. However, there was a strongly significant (α = 1%) strength gain between the untreated and treated samples. The resin itself seems to have created a thin layer of higher density on the surface of the predrilled hole. This layer increases strength of wood in the connector area under the microscale. A hypothesis could be that this layer distributes the stress more efficiently than the untreated samples. Further work using digital image correlation could be used to confirm this hypothesis. Stiffness was less influenced by the treatment than the bearing strength. This could be explained by the increase of the strength in the plastic zone, resulting in a much higher bearing capacity and a moderately improved stiffness. For the treated samples and samples treated with NP, the increase of stiffness was 27% (14 kN/mm) and 26%, respectively (14 kN/mm). Again, the treatment effect was statistically significant (α=1); the homogeneity groups are shown in Table 3. The load-displacement curves for untreated, treated, and treated with nanoparticles are shown in Fig. 3.

SEM photos at 150X of untreated before test (Fig. 3a), untreated after test (Fig. 3b), and treated without nanoparticles after test (Fig. 3c) are included in the curves. The loading area at the edge of the hole is shown (point B in Fig. 1.). The deformation of the fibers of treated wood compared to untreated wood is more important in the yielding.

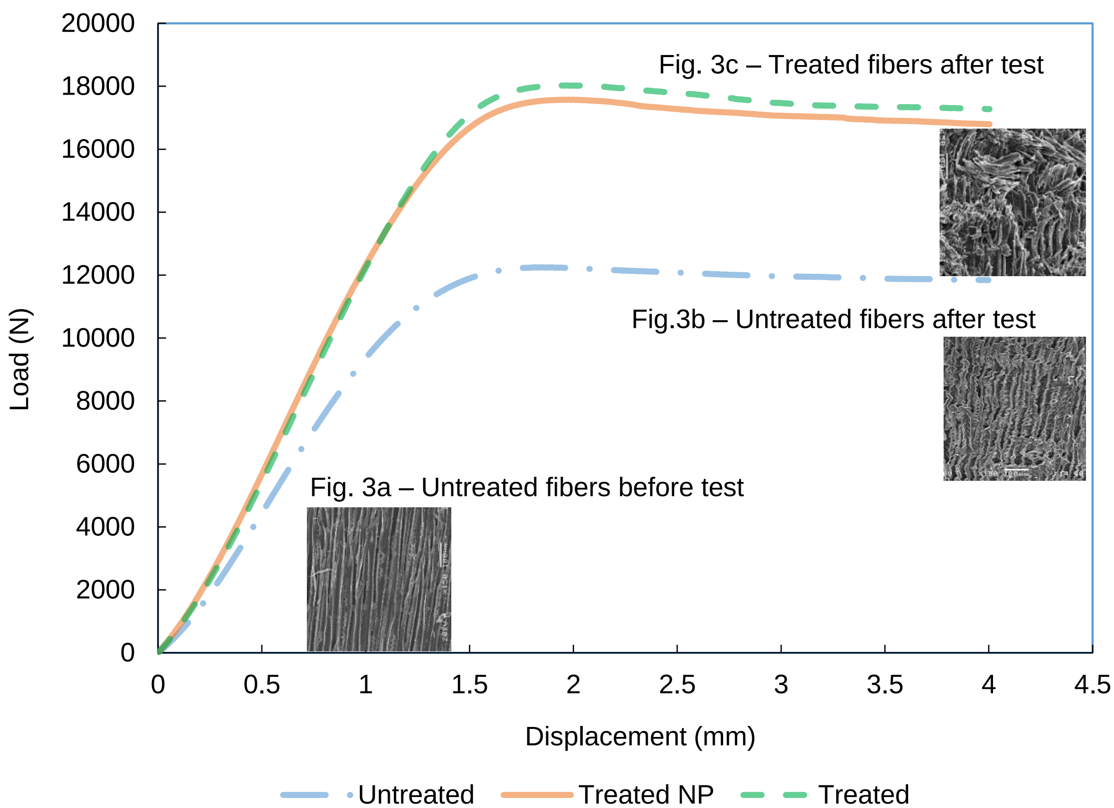


Fig. 3. Load-displacement curve in dowel bearing strength test (according to ASTM 5764) for treated samples (impregnation with and without NP) and for untreated samples. (a) – Untreated fibers before test; (b) – Untreated fibers after test (folded fibers); (c) – Treated fibers after test (crushed and broken fibers)

In the untreated sample, fibers are simply compressed while treated fibers are crushed and broken. Surely, the gap between the maximal loads for each group of samples is responsible for this change in the wood behavior. After reaching maximal load, the curve of treated sample showed a diminution of load more accentuated than for the untreated sample.

The fibers (Fig. 3c) could have broken just after the maximal load and released energy causing the load decrease, as can be seen in the load displacement curve. This event does not occur in the untreated samples due to the inferior loading causing a simple folding of fibers.

However, past the maximal load, the treatment appears to transfer the load more equally around the connector zone, resulting in ductile behavior. Gaining such behavior is required, especially in tall wooden buildings, for ductile failure modes. A digital image correlation could be considered a tool to better understand the loading transfer of treated load-bearing connectors. Moreover, only the parallel-to-grain direction was tested. Due to wood anatomy, penetration of the formulation is easier in the direction of fibers. The hypothesis that perpendicular-to-grain dowel-bearing strength would show a lower increase after treatment could be tested. The orientation of testing needs further investigation. Despite a thin layer of reinforcement at the surface of the drilled hole, the assembly with treated wood was nearly 50% stronger in the dowel-bearing strength parallel to grain.

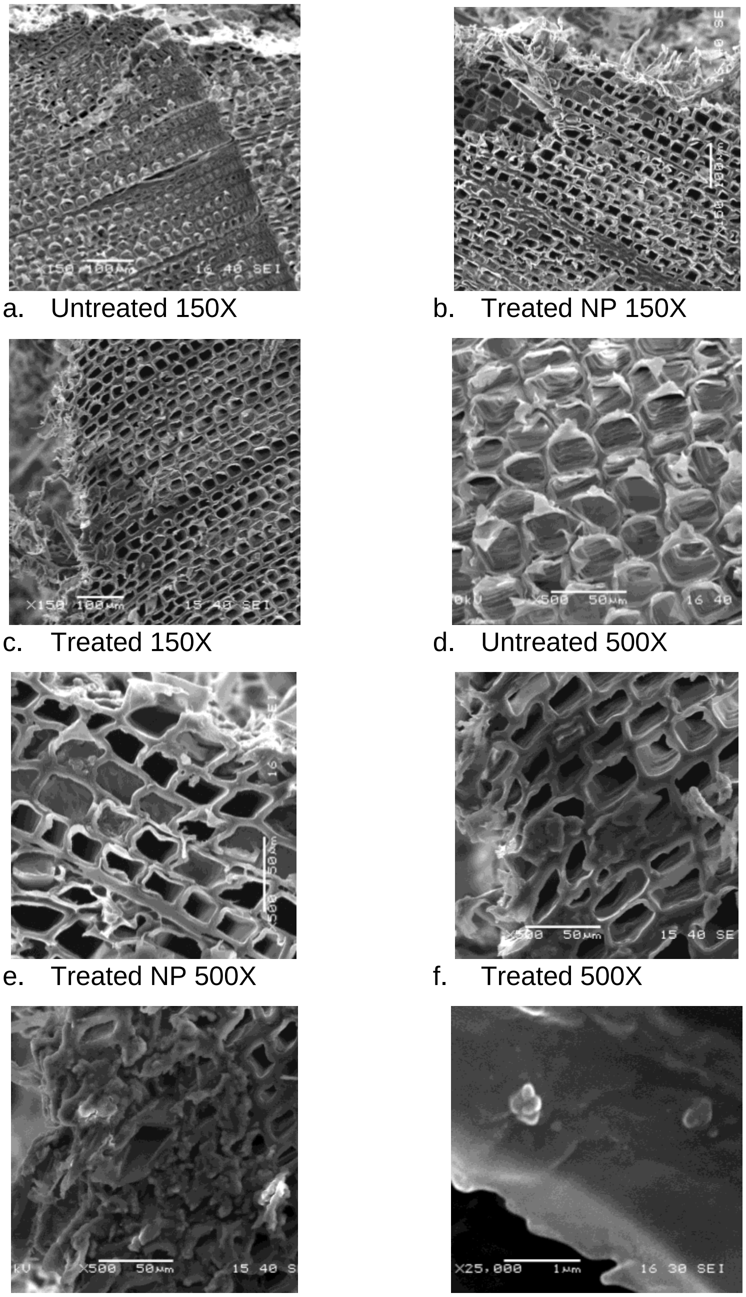
Scanning Electron Microscopy

Figure 4 presents micrographs of untreated, treated and treated wood with nanoparticles in the transversal plane. Areas at point A in Fig. 1 were cut for each treatment group. Figure 4 (a, d) shows untreated black spruce, while Fig. 4 (b, e) presents few lumen cells full of treatment formulation in a treated NP sample and Fig. 4 (c, f) in a simply treated sample.

In Fig. 4 (d), emptiness of lumens in the untreated wood is hard to detect due to the oblique surface cut of the sample. In Fig. 4 (f, g), acrylate deposits are visible, not inside the cells, but on top of the lumen and cell walls as a deposit layer on treated wood. This phenomenon appears only on the hole edges, at both sides of the sample. This may result in an incomplete polymerization, while monomers and oligomer are migrating to the wood surface.

The impregnation process with black spruce led to low chemical retention and non-uniform penetration into samples. Only partial rows of cells were impregnated. The penetration depth could be evaluated to 100 μm, corresponding to approximately four rows of cells.

Nanoparticles of SiO_2 were rare and possibly detected in Fig. 4 (h). Agglomerates of SiO_2 measured nearly 500 nm, while mean particle size was 20 nm diameter. Thus, a narrow layer of polymer in the connector areas creates enough density increase to give major uptakes in dowel bearing strength. The need of a low volume of impregnation agent to obtain such results is an important economic advantage to full cell impregnation consuming high volumes of chemicals.



a. Untreated 150X
b. Treated NP 150X
c. Treated 150X
d. Untreated 500X
e. Treated NP 500X
f. Treated 500X
g. Treated 500X
h. SiO2 aggregate 25 000X

Fig. 4. SEM photographs of transversal plane in untreated, treated and treated NP samples

CONCLUSION

The dowel-bearing strength of acrylate impregnated black spruce was determined with and without SiO_2 nanoparticles. Despite low chemical retentions and minimal penetration depth, the dowel-bearing strength parallel to grain was about 50% stronger than untreated wood, according to the ASTM standard embedment tests. This surface treatment could appear more attractive for industrialists because of its low demand in chemicals and energy needed to cross-link and solidify the impregnation agent. No significant differences were found in the samples treated with and without nanoparticles. A slightly lower chemical retention was observed for the treated NP sample group due to the accumulation of nanoparticles acting as a barrier at wood surface. Normally, this could lead to better densification of the external layer of the wood, but in this case mechanical performances were the same. There is no need to add nanoparticles and increase the cost of chemicals. Further research needs to achieve a better understanding of treated wood. Digital camera image analysis could be performed in future mechanical tests to have a deeper understanding of the effect of the treatment. Moreover, perpendicular-to-grain dowel-bearing strength should be tested to characterize the impact of treatment in this direction, as engineered wood products are not only loaded in the parallel-to-grain direction when used as a connector element. Due to the low chemical penetration, the impregnation process could be reviewed in order to reduce the processing time and the required amount of chemicals. A surface application, instead of soaking complete wood pieces into solution, could be considered. To optimize the process, the connector areas only should be targeted in the treatment. Treatment of wood should be done while processing the structural elements; it needs to be time-effective to be involved in the production line. With this increase of connector strength in structural timber, the design (*i.e.* section of a beam) would be reviewed to reduce the wood volume or the number of connectors required. Material cost would be decreased with assembly time on site. Besides, the environmental footprint of the building would be reduced as more wood structure solutions could be considered by the architects.

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