

Dimensional Stability and Water Repellency of European Aspen Improved by Oxidized Carbohydrates

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Small samples from European aspen (*Populus tremula* L.) were impregnated with carbohydrates oxidized by Fenton's reagent using water in a vacuum, followed by heating in an oven at 103 °C. An antismelling efficiency (ASE) of around 45% for wood treated with oxidized glucose and 35% for wood treated with oxidized sucrose was obtained. Samples treated with oxidized carbohydrates gave water repellent effectiveness (WRE) values over 35%. The decrease in cell wall thickness during impregnation was about 18% less in the presence of oxidized glucose than samples only treated with Fenton's reagent. An ASE of 20% for the wood samples that had been treated with oxidized glucose was obtained after 7 days of soaking in water. The reasons for the improvement in dimensional stability are discussed in this work.

Keywords: Antismelling efficiency (ASE); European aspen; Fenton's reagent; Oxidized carbohydrates; Water repellent effectiveness (WRE)

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INTRODUCTION

Wood modification is an attractive area of study within the wood industry to improve the properties of wood for outdoor structure applications. However, these applications face many technical difficulties. For example, the use of chromated copper arsenate (CCA) as a wood preservative for residential or domestic construction is forbidden in Europe because of the probability of releasing arsenic and chromium into the ecosystem and the lack of green techniques to recycle it (Hill 2006). The development of environmentally friendly and renewable materials to improve the dimensional stability of wood is of great interest.

Many studies have focused on using acetic anhydride, carboxylic acids, isocyanates, epoxides, aldehydes, and furfuryl alcohol to chemically modify wood (Hill 2006). Treatment of wood with formaldehyde can increase the dimensional stability of wood at quite a low WPG, but the major concerns are the health risks associated with formaldehyde vapor. The focus is to shift to non-formaldehyde cross-linking agents like glyoxal and glutaraldehyde (Yasuda and Minato 1994; Xiao *et al.* 2010). Xiao *et al.* (2010) suggested that glutaraldehyde forms a strong bond (with itself or with wood) inside the cell wall during impregnation and subsequent heating, showing the potential to replace formaldehyde with less harmful dialdehyde as a modifying chemical. Šefc *et al.* (2009) claimed that citric acid has the potential to be a non-formaldehyde cross-linking reagent by forming ester bonds, but the extent of cross-linking is still unknown.

Fenton's reagent consists of hydrogen peroxide and a ferrous/ferrous iron catalyst. It is an effective oxidant and is applied mostly to treat organic waste and pollutants

(Barbusinsky 2009). The generated hydroxyl radical is a very active oxidant, capable of attacking most organic compounds, including lignin and carbohydrates. However, when it reacts with wood, it involves complex reactions that are still not fully understood. Westermark *et al.* (2011) suggested that oxidized carbohydrates containing a dialdehyde functional group plays an important role in the bonding with the lignocellulosic material. In previous studies, Karlsson and Westermark (2002) used hydrogen peroxide and a catalyst to oxidize the wood particles, and then a hot press to create resin-free particleboard. Slight springback was achieved, and the internal bonding (IB) was reported to be excellent. Furthermore, a lower extent of thickness swelling in water was reached when the amount of oxidant was higher.

It has been reported that glyoxal, gluconic acid, ribose, glucitol, glycolate, glyceraldehyde, and formic acid are generated from the oxidation of carbohydrates with hydrogen peroxide (Emery and Schroeder 1974; Maksimovic *et al.* 2006; Manini *et al.* 2006; Morelli *et al.* 2003; Westermark *et al.* 2011; Widsten *et al.* 2003). Some of these products could function as cross-linkers. For example, glyoxal has a bi-functional group (dialdehyde) that can react with hydroxyl groups bonded to wood polymers. Westermark *et al.* (2011) suggested that the oxidized carbohydrates with one extra aldehyde and one or two keto groups are the most active reactants when used for increasing the wet strength of paper.

As far as can be seen in the current literature, oxidized sugars have not been used to improve the dimensional stability of solid wood. In this study, glucose oxidized with Fenton's reagent was used to treat solid wood. Fenton's reagent is one way of treating waste-water and can thus be used in large quantities due to its high potential to oxidize organic material. Even though production is relatively low, acetylation and furfurylation are common chemical modification methods of wood (Hill 2006). Using Fenton's reagent may be a more technically favourable way to modify carbohydrates than production of furfuryl alcohol from pentose-based carbohydrates used in furfurylation of wood (Hill 2006). Using water solutions of oxidized carbohydrates for modification of wood may also be a technically easier process to modify wood than for example acetylation, which is done using organic solvents. Though the composition of the reagent solution (oxidized glucose) is complex, bi-functional groups formed during oxidation may improve the dimensional stability of the wood by forming stable bonds with wood components.

EXPERIMENTAL

Materials

European aspen (*Populus tremula* L.) sapwood was obtained from XL-Bygg Norra, Skellefteå, Sweden. Samples with dimensions of 10 (longitudinal, L) × 20 (tangential, T) × 10 (radial, R) mm were prepared from the same board. Analytical grade glucose (C₆H₁₂O₆; VWR, Sweden), commercial sucrose (C₁₂H₂₂O₁₁, 99% purity), magnesium chloride hexahydrate (MgCl₂·6H₂O; Merck KGaA, Germany), ferrous sulphate heptahydrate (FeSO₄·7H₂O, 99% purity; VWR, Sweden), and 50% stabilized hydrogen peroxide (H₂O₂; AKZO NOBEL, Sweden) were used in this experiment.

Impregnation and Heating

Carbohydrates, ferrous sulphate, and magnesium sulphate were dissolved in 750 mL of water in a plastic vessel. Hydrogen peroxide (16.7%) was added dropwise, using a burette (Table 1). The concentration of the reagent solutions was controlled to 1 M by evaporating water. Three types of reagent solutions (I, II, and III) were used in this trial, as shown in Table 1.

Oxidation of carbohydrates with Fenton's reagent resulted in a decrease in the pH of the impregnating solution (Table 1). Therefore, the reagent solutions were conditioned to the same pH level (2.4) using a diluted alkali solution before impregnation to study the hydrophilic properties of treated specimens.

Table 1. Chemicals and Proportions Used in Reagent Solutions

Reagent solution	Reagent					Reagent solution pH	
	Sugar type	Sugar amount (g)	Fenton's reagent			Before adjustment	After adjustment
			MgCl ₂ ·6H ₂ O (g)	FeSO ₄ ·7H ₂ O (g)	16.7% H ₂ O ₂ (mL)		
I	Glucose	18.0	1.5	0.75	36	1.8	2.4
II	Sucrose	34.4	1.5	0.75	36	1.8	2.4
III (Fenton's reagent)	-	-	1.5	0.75	36	2.4	2.4

Wood samples were dried in an oven at 103 °C for 48 h before impregnation. Five replicates for each three experiments were used; in total, 15 wood samples were used. The heated wood specimens were submersed in the reagent solutions (I, II, and III) under vacuum (50 mbar, 120 min) and left in the solutions for 2 days until the weight of specimens became stable. Specimens were air-dried for 4 days before curing in an oven to remove excess water without severe migration of the water-soluble compounds to the wood surface. The specimens were heated at 103 °C for 48 h.

Dimensional Stability and Water Repellency

A leachability test was used to study whether oxidized sugars could be leached out from wood, as well as the durability of the dimensional stability of the wood. The test consisted of determining the oven-dry volume of the specimen, then submerging the specimen in tap water, and removing entrapped air in the wood using a vacuum desiccator. Vacuum was continued for 30 minutes, released, left for 1 h, re-applied for 30 minutes, and then released. The specimen was left for 24 h at room temperature. The volumetric swelling (S_1) was calculated, and the antismelling efficiency (ASE_1) was studied after soaking in 100 mL water for one day. The pH of the soaking water was measured, and the water was decanted off and replaced by new tap water. The soaking water was changed every day for 7 days total. The specimens were air-dried for 4 days and then dried in an oven at 60 °C for several days until the MC became close to 0%. The weight percent gain (WPG_2) was calculated by comparing the weight before impregnation with the data obtained after 7 days of soaking in water. The soaking test was then repeated for 1 more day as an additional test. The volumetric swelling (S_2) of specimens and ASE_2 after 7 days of soaking were measured by comparing the dry volume of treated samples after 7 days of water soaking with the data obtained after one additional day of

soaking in water. The pH of the soaking water from the additional soaking test was recorded.

The weight percent gain (WPG) after treatment was measured from the oven-dried weights of the specimens before (m_u) and after (m_t) reagent solution treatment or 7 days soaking in water.

$$\text{WPG (\%)} = 100 \times (m_t - m_u) / m_u \quad (1)$$

The bulking coefficient (BC) is useful in analyzing the chemical modification of wood if penetration of the wood cell wall occurs. It is measured from the volume of oven-dried samples before (V_u) and after (V_t) reagent solution treatment.

$$\text{BC (\%)} = 100 \times (V_t - V_u) / V_u \quad (2)$$

The stabilization effect can be described by antiswelling efficiency (ASE) values (Rowell and Banks 1985). ASEs of impregnated samples were calculated from volumetric swelling values of impregnated (S_T) and control (S_R) samples after soaking in water. Volumetric swelling was calculated from the volume differences divided by oven-dried volume.

$$\text{ASE (\%)} = 100 \times (S_R - S_T) / S_R \quad (3)$$

Water repellent effectiveness (WRE) was estimated based on the water absorption (WA: absorbed water divided by oven-dry mass) of the control (WA_c) and treated (WA_t) samples after the 7-day soaking in water test:

$$\text{WRE (\%)} = 100 \times (WA_c - WA_t) / WA_c \quad (4)$$

The dimensions of the specimens were determined by immersing the wood samples in water and measuring the weight when the samples were immersed and suspended in water. This was quickly performed without observing any air bubbles released from the wood pieces when suspended in water. The Archimedean principle was used; the volume of wood sample is the same as the sample mass because the water density is 1.00 g/cm^3 (Wei *et al.* 2003).

HPLC Analysis

High performance liquid chromatography (HPLC) was used to investigate the generation of furfural after heating. The soaking solutions used on the first day of testing were collected, filtered ($50 \mu\text{m}$), and analyzed by HPLC. A water Hi-plex Pb-column ($8 \mu\text{m}$ and $250 \times 4 \text{ mm}$) heated to $60 \text{ }^\circ\text{C}$ was used. The flow of water eluent was 0.3 mL/min and the elution time was 35 min. 5-hydroxymethylfurfural (HMF) and furfural were detected as separated peaks with an ultra violet (UV) detector operating at approximately 280 nm, and glucose was detected with a refractive index (RI) detector.

Scanning Electron Microscope (SEM)

SEM specimens measuring *ca.* $5 \text{ (R)} \times 5 \text{ (T)} \times 5 \text{ (L)} \text{ mm}$ were prepared from control and impregnated samples. Samples were sputter-coated for 2 min with gold, using a sputter apparatus (Desk II Cold Sputter, Denton Vacuum Inc., Moorestown, USA) and

were examined under JSM-5200 (JEOL, Akishima, Japan) SEM at an accelerated voltage of 15 to 20 kV.

Four images of each sample were then used to determine if cell wall bulking occurred before and after the 7-day soaking in water test. In each image, 10 measurements were performed to obtain the 2 fiber wall thicknesses in the tangential direction.

RESULTS AND DISCUSSION

Dimensional Stability and Water Repellency of Treated Wood After the First Day of Soaking in Water

Small pieces of wood were impregnated with carbohydrates (both glucose and sucrose), oxidized with Fenton's reagent at pH 2.4, heated to 103 °C, and leached with water for one day. It was observed that the impregnated material leached from the wood samples during soaking in water, by a change to a somewhat darker color and a decrease in the pH of the soaking water. The volumetric swelling (S) and antismelling efficiency (ASE) of the treated wood were calculated and are shown in Table 2. Wood impregnated with oxidized carbohydrates (both glucose and sucrose) displayed better dimensional stability than untreated samples after the first day of soaking. Wood treated with oxidized glucose had an ASE of about 45% and the wood treated with oxidized sucrose had an ASE of nearly 35% after the first day of soaking (Table 2).

The dimensional stability of wood is dependent on the impregnation of suitable chemicals that can react with cell wall components or the use of water-repellent chemicals (Ahmed and Morén 2012; Feist *et al.* 1991). The improvement in the dimensional stability of wood treated with Fenton's reagent was less than in presence of the oxidized carbohydrates. The ASE was around 10% (Table 2).

The reason for the improvement in dimensional stability of wood only treated with Fenton's reagent may be that hydrophilic wood components, such as hemicelluloses, were degraded. Similar degradation of hydrophilic wood components induced by Fenton's reagent was reported by Halliwell (1965). During heat treatment, the wood pieces became darker, and colored material subsequently found in the soaking water could be related to degradation products from the wood (Table 2).

Furfural can be formed via the dehydration of pentose-containing carbohydrates such as xylose and xylan, whereas 5-(hydroxymethyl) furfural (HMF) can be formed from carbohydrates that contain hexose, such as glucose (Taherzadeh and Karimi 2007; Karlsson *et al.* 2012). Upon standing, such material will form a dark resin (Gandini and Belgacem 1997). The HPLC chromatogram showed that the concentration of HMF (1.761 g/L) was much lower than furfural (27.197 g/L) in the soaking water from the samples only treated with Fenton's reagent after the first day.

Since xylan is the dominant hemicellulose in aspen, the favored formation of furfural implies that hemicelluloses were degraded during heat treatment at 103 °C. This is probably due to the fact that the pH of the reagent solution was fairly low (pH 2.4). When spruce boards were dried and heated at 110 °C and 130 °C at a nearly neutral pH, degradation of mono- and disaccharides was the main occurrence (Karlsson *et al.* 2012). Thus, the degradation of hydrophilic hemicelluloses might influence the dimensional stability of wood in the other treated wood samples.

Table 2. Data of Wood Samples Treated with Different Reagent Solutions (pH 2.4) After the First Day of Soaking in Water

Sample group	Reagent solution	Before soaking in water		After the first day of soaking in water	
		WPG ₁ (\pm SD) after heating	BC ₁ % (\pm SD)	S ₁ % (\pm SD)	ASE ₁ %
1 st group (control)	-	-	-	13.60 \pm 0.51	-
2 nd group	I	24.03 \pm 0.39	10.85 \pm 1.95	6.41 \pm 1.40	45.13
3 rd group	II	57.71 \pm 1.99	8.56 \pm 2.72	7.81 \pm 1.27	34.28
4 th group	(III) Fenton's reagent	0.36 \pm 0.17	2.35 \pm 3.34	12.20 \pm 2.59	10.31

WPG, weight percent gain; BC, cell wall bulking; S, volumetric swelling; ASE, antiswelling efficiency; SD, standard deviation

From the data, it can be hypothesized that products from the oxidation of carbohydrates helped to improve the dimensional stability of aspen wood. Wood treated with oxidized glucose had better dimensional stability, in terms of higher ASE, than with oxidized sucrose. However, the average weight percent gain (WPG) of the specimens treated with oxidized glucose was smaller than the specimens impregnated with oxidized sucrose (Table 2).

Figure 1 indicates that the relation between cell wall bulking and ASE among wood modified in the presence of the two different oxidized carbohydrates and control samples was almost linear after the first day of the soaking test. From the results, it is clear that the bulking coefficient (BC) was the most important factor influencing ASE after the first day of the soaking test. This finding is in accordance with previous results mentioned in Li *et al.* (2000). The pH of the soaking liquid containing the treated samples after the first day was approximately 3.5.

A large amount of material from the reagent solution used to impregnate the wood as well as liberated wood components was washed away by water during the first day of the soaking test. This indicates that most of the material in the reagent solution was not strongly attached to the wood by stable chemical bonds. Stamm (1937) reported that invert sugar could be a good anti-shrinking agent in conditions that would not involve water leaching of sugar from the wood. The influence of non-bonded compounds in reagent solutions on BC and ASE should be taken into consideration, as well as whether or not cross-linking between compounds in the reagent solutions and wood cell walls occurred.

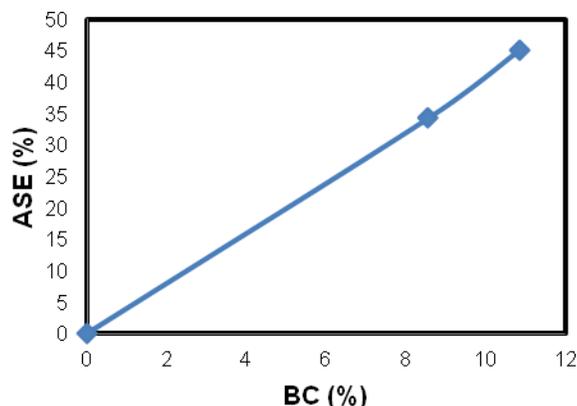


Fig. 1. The relationship between bulking coefficient (BC) and antismelling efficiency (ASE) of wood treated with oxidized carbohydrates and the control sample (origin point)

Dimensional Stability and Water Repellency of Treated Wood After Further Water Soaking

After 7 days of soaking in water, the pH of the soaking water changed to neutral, and a disappearance of color was observed. This pointed to the fact that most of the reagent solution had been leached out during soaking with water. Comparison of Tables 2 and 3 shows that WPG of wood treated with oxidized carbohydrates decreased to a great extent after repeated soaking in water.

The water repellence effectiveness (WRE) after 7 days of the soaking test for samples treated with Fenton's reagent or treated with a reagent solution of oxidized glucose or sucrose is presented in Fig. 2. Samples treated with oxidized sucrose displayed the highest WRE, which was over 50%. Samples treated with the oxidized glucose reagent solution also showed a considerable increase in WRE. Treatment with Fenton's reagent only resulted in a WRE of about 5% (Fig. 2).

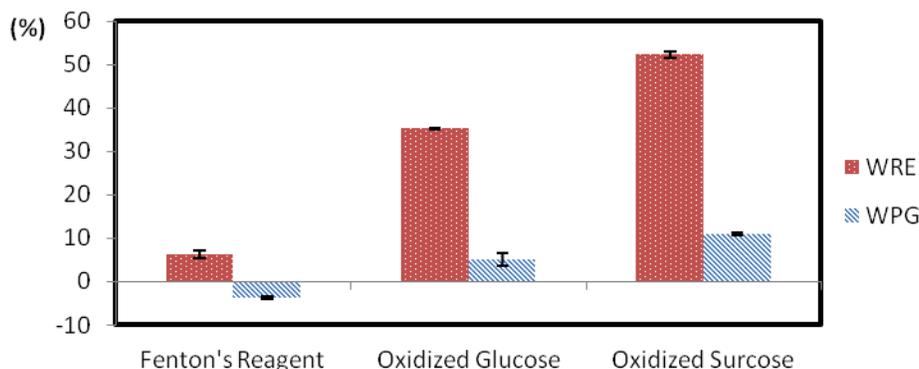


Fig. 2. Water repellence effectiveness (WRE) and weight percent gain (WPG) of wood treated with reagent solutions after 7 days of the soaking test

By comparing WRE data in Figure 2 and WPG data in Table 3, it can be concluded that the higher the WPG of samples after impregnation and heating, the higher the WRE of samples after the soaking test. Thus, the WPG seems to influence the water repellency of wood treated with oxidized carbohydrates. The reason for the improved WRE is complex, involving physical bulking of cell wall and lumen, cross-linking formed by oxidized glucose inside wood, and hemicelluloses degradation.

Table 3. Dimensional Stability of Wood Samples Treated with Reagent Solutions After 7 Days of Soaking in Water

Sample Group	Reagent solution	Before additional 1 day of soaking in water	After additional 1 day of soaking in water			
		WPG ₂ % (±SD)	Two cell wall thickness, μm (±SD)	S ₂ % (±SD)	ASE ₂ %	pH of soaking liquid
1 st group (control)	-	-0.65±0.06	6.05±0.54	17.58±0.86	-	7.33
2 nd group	I	5.02±0.30	5.66±0.91	14.54±0.86	22.45	6.15
3 rd group	II	10.94±1.44	5.53±0.4	15.64±0.98	16.58	5.89
4 th group	III Fenton's reagent	-3.6±0.23	4.59±0.89	18.75±1.48	4.34	7.07

WPG, weight percent gain; S, volumetric swelling; ASE, antismelling efficiency; SD, standard deviation

Cell Wall Thickness

Studying the cell wall thickness of wood samples using microscopy can even better describe the extent of cell wall bulking than changes in the outer dimensions of the wood samples (Wallström and Lindberg 1999). In Fig. 3 it can be observed that the average cell wall thickness was larger in the samples treated with oxidized glucose than the untreated samples before repeated soaking cycles (Fig. 3a, 3b). The average cell wall

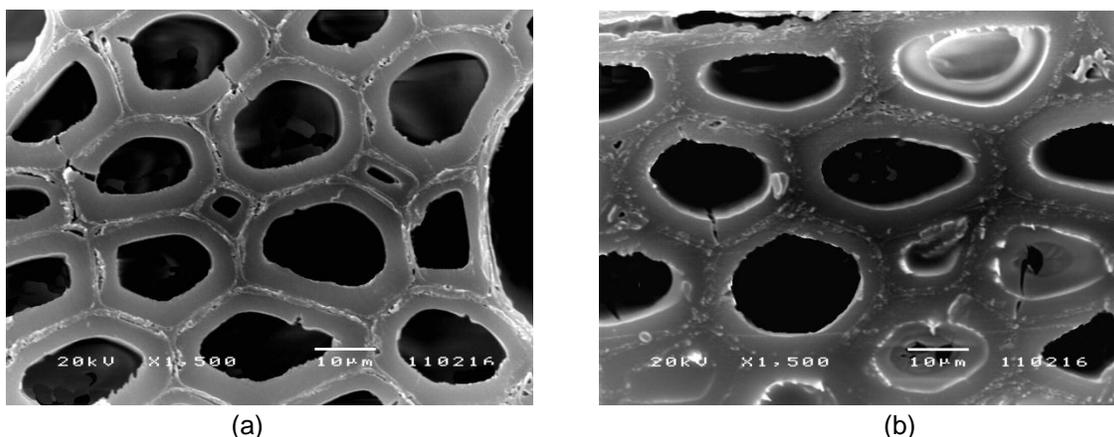


Fig. 3. (a) Measurement of two cell wall thicknesses of the control sample, (b) measurement of two cell wall thicknesses of the sample treated with oxidized glucose

thickness increased about 10% after treatment with oxidized glucose. From the observed increase in cell wall thickness and the data presented in Tables 2 and 3, it is suggested that oxidized glucose penetrates the cell wall and prevents water from entering the cell wall during the soaking experiment.

SEM images show that oxidized sugars in reagent solutions can easily penetrate the wood during treatment; a filled lumen can be seen in Fig. 3b. However, results from analyzing WPG (Table 3) after 7 days of soaking in water indicated that the bonding of substances in the lumen and cell wall is questionable. The thickness of wood cell walls (at 0% MC) treated with oxidized carbohydrates and soaked in water for 7 days were measured, and seemed to be smaller than control samples (Table 3). Wood treated with Fenton's reagent had the smallest average cell wall thickness (Table 3 and Fig. 4d).

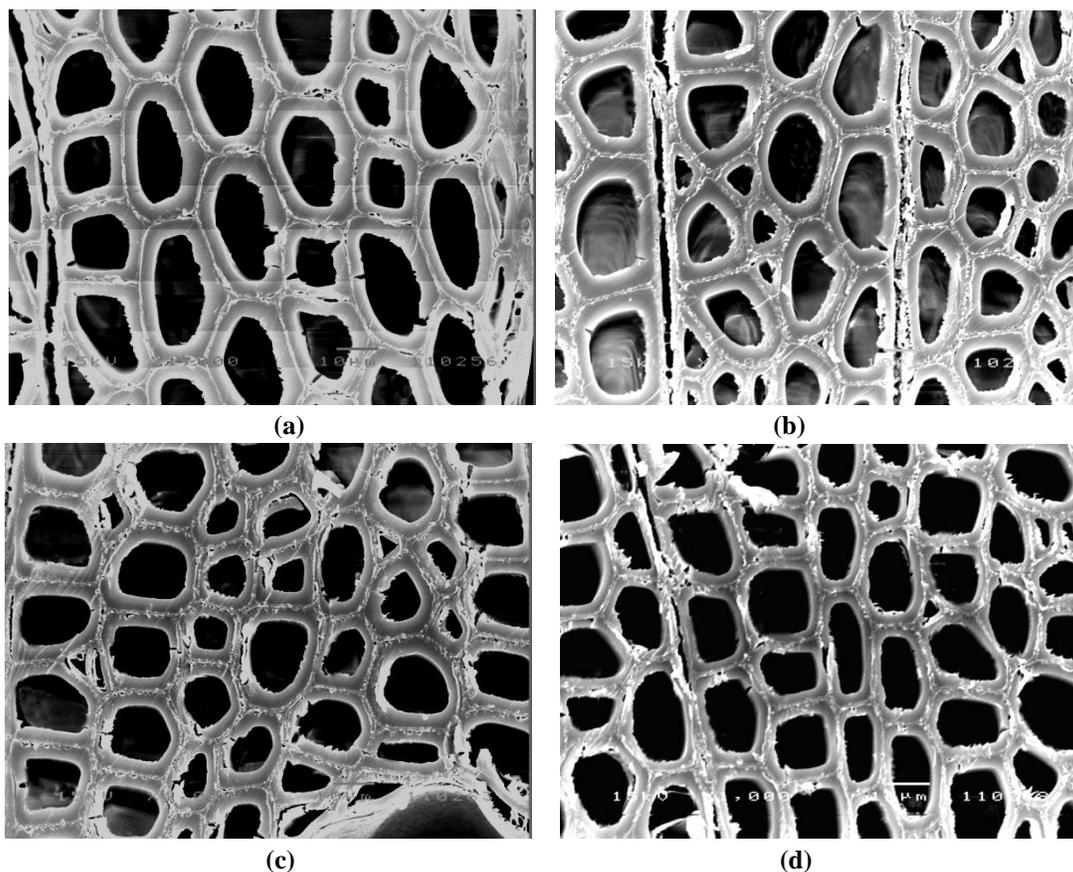


Fig. 4. Measurement of two cell wall thicknesses of samples: (a) aspen control sample, (b) aspen sample treated with oxidized glucose, (c) aspen sample treated with oxidized sucrose, and (d) aspen sample treated with Fenton's reagent

The cell wall structure of treated aspen wood might be destroyed during heating, since the pH of reagent solutions was low (pH 2.4). In this regard, it has been reported that sugi heartwood (*Cryptomeria japonica*) and tochinoki (*Aesculus turbinata*) treated in acetic acid buffer solution and Fenton's reagent both displayed a lower content of holocellulose and Klason lignin (Kohdzuma *et al.* 1991). No solid material from oxidized carbohydrate solutions that filled the inner side of the lumen after impregnation can be seen in the SEM images (Fig. 4) after 7 days of soaking. However, WPGs were higher than in the samples treated with Fenton's reagent, indicating that compounds from the reagent solutions still resided inside the oxidized carbohydrates-treated wood and that

these compounds might be bonded with the cell wall (Table 3). Since wood samples treated with oxidized glucose and oxidized sucrose exhibited a smaller reduction in cell wall thickness compared with the wood treated with Fenton's reagent (Table 3), the remaining stable, oxidized carbohydrates inside the cell wall may compensate for the loss of cell wall structure due to attack from the acidic Fenton's reagent.

To study the effect of the non-bonded reagent solution on ASE after 7 days of soaking in water, one additional cycle of soaking followed by oven drying was performed. ASE was reduced from 45% to 22%, while the WPG was reduced from 24% to around 5%, for samples treated with oxidized glucose (Tables 2 and 3). Wood treated with Fenton's reagent showed only a small positive effect on the dimensional stability of wood (Table 3). Thus, the contribution of Fenton's reagent to the observed dimensional stability in wood treated with oxidized carbohydrates seems to be low, especially because the pH was adjusted to the same level in the experiments. Furthermore, although it seems that a part of ASE in wood treated with oxidized carbohydrates was affected by non-bonded compounds in the reagent solution, there was still some effect on the dimensional stability from oxidized carbohydrates that could not be easily leached away from the treated wood. Although only two types of soluble sugars were used in this study, we believe that also other types of oxidized and soluble carbohydrates can be active in such a process to improve dimensional stability of wood.

CONCLUSIONS

1. The results presented in this work showed that it is possible to significantly reduce wood swelling by impregnation with glucose that was activated by Fenton's reagent, followed by heating. Furthermore, absorption of liquid water was also lower for the treated samples, which should be advantageous for the purpose of reducing swelling.
2. The method shown in this paper is to a great degree based on oxidation of renewable carbohydrates and has the potential to become an environmentally friendly way to improve dimensional stability of solid wood. It is well-known that Fenton's reagent is not a particularly selective oxidant and may produce many oxidation products but also that more efficient ways of oxidation exist such as the photo-Fenton method. Most of the reagent solution was also not bonded to the wood, which suggests that some specific oxidized components are active in the modification process. The process has the potential to be greatly improved by a modified and more selective oxidation procedure.

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