

## Study on the modification of bleached eucalyptus kraft pulp using birch xylan

Wenjia Han<sup>a,b,c</sup>, Chuanshan Zhao<sup>a</sup>, Thomas Elder<sup>d</sup>, Kefu Chen<sup>c</sup>, Rendang Yang<sup>c</sup>, Dongho Kim<sup>b</sup>, Yunqiao Pu<sup>b</sup>, Jeffery Hsieh<sup>b</sup>, Arthur J. Ragauskas<sup>b,\*</sup>

<sup>a</sup> Key Lab of Paper Science and Technology of Ministry of Education, Shandong Institute of Light Industry, Ji'nan 250353, China

<sup>b</sup> Institute of Paper Science and Technology, Georgia Institute of Technology, 500 10th Street, N.W. Atlanta, GA 30332-0620, USA

<sup>c</sup> State Key Lab of Pulp and Paper Engineering, South China University of Technology, Guangdong Public Laboratory of Paper Technology and Equipment, Guangzhou 510640, China

<sup>d</sup> USDA-Forest Service, Southern Research Station, 2500 Shreveport Highway, Pineville, LA 71360, USA

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### ABSTRACT

In this study, birch xylan was deposited onto elementally chlorine free (ECF) bleached eucalyptus kraft pulp, and the corresponding changes in physical properties were determined. An aqueous 5% birch xylan solution at pH 9 was added to 5 wt% slurry of bleached kraft eucalyptus fibers, with stirring at 70 °C for 15 min after which the pH was adjusted to 5–6. The xylan enriched fibers were isolated by filtration and used for physical testing. A 1.15 wt% adsorption of birch xylan on the kraft fibers at 8% xylan addition increased the tensile index, strain and tensile energy absorption values by ~10%, while the burst index increased by 20.15%. The tear index increased by 2.55% with the adsorption of 0.87 wt% birch xylan on the eucalyptus kraft pulp at 3% xylan addition. The pulp beatability was also improved by adding birch xylan. The surface morphology of the unmodified and modified pulp samples were analyzed using atomic force microscopy (AFM) in the tapping mode. The analysis revealed the differences in the fine structure of fibers which showed micrometer-sized xylan structures spreading over the fiber surfaces.

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### 1. Introduction

Hemicellulose is one of the important carbohydrate components in wood. One of the primary hemicelluloses in softwoods is arabino-4-O-methyl glucuronoxylan, at about 20 w/w%, while 4-O-methyl glucuronoxylan, at 15–30 w/w%, dominates in hardwoods (Ban & Van Heiningen, 2011; Leopold & McIntosh, 1961). In terrestrial plants xylans are heteropolymers possessing a  $\beta$ -(1→4)-D-xylopyranose backbone, which is branched by short carbohydrate chains. The branching sugars include D-glucuronic acid or its 4-O-methyl ether, L-arabinose and/or various oligosaccharides, composed of D-xylose, L-arabinose, D- or L-galactose and D-glucose. Woody based xylans are polymeric sugars with a  $\beta$ -(1-4)-D-xylopyranosyl backbone to which arabinopyranosyl, glucuronic acid, and acetyl substituents are attached (Henriksson & Gatenholm, 2002). Recent studies have demonstrated that xylans from wood and other plant resources can be used as biodegradable and renewable cellulose fiber modifying agents due to their inherent affinity to cellulose (Bhaduri, Ghosh & Deb Sarkar, 1995; Henriksson & Gatenholm, 2002; Schonberg, Oksanen, Suurnakki, Kettunen, & Buchert, 2005). Due to the harsh alkaline conditions of kraft pulping most of the hemicelluloses in wood are lost

during pulping (Danielsson & Lindstrom, 2005; Janzon, Saake, & Puls, 2006; Kettunen, Laine, Yrjälä, & Virkola, 1982). As a consequence, the addition of hemicelluloses to a kraft pulp can be viewed as a potential way of modifying the physical properties of paper. Indeed, the deposition of xylan onto bleach grade kraft fibers in order to improve the paper strength is a growing field of research in fiber modification. The chemistry of xylan adsorption on cellulosic fibers and its impact on physical properties such as tensile strength, wettability, beatability and resistance to hornification has been a research subject of interest (Hansson & Hartler, 1969; Henriksson & Gatenholm, 2002; Linder, Bergman, Bodin, & Gatenholm, 2003; Kabel, Vincken, Voragen, & Schols, 2007; Köhnke, Lund, Brelid, & Westman, 2010; Paananen et al., 2004; Schonberg et al., 2005). A recent study has reported on never dried and once dried Scandinavian softwood kraft pulp modified with the addition of 40, 80, 160 mg/o.d.g fiber of 4-O-methylglucuronoxylan which was acquired from birch wood (Köhnke et al., 2010). The specified amount of 4-O-methylglucuronoxylan was dissolved in water and then added to a pulp fiber suspension under stirring. The suspension was placed in a steel autoclave and rotated in a preheated polyethylene glycol bath at 120 °C for 180 min. After treatment, the pulp was washed with deionized water until a solution conductivity of less than 5  $\mu$ S/cm was reached. The result showed that the degree of hornification under the optimum condition was reduced by 50% compared to unmodified fiber. Lima, Chaves, and Buckeridge (2003) reported that the addition of 1% (w/w) of seed storage

\* Corresponding author. Tel.: +1 404 894 9701; fax: +1 404 894 4778.

E-mail address: [arthur.ragauskas@ipst.gatech.edu](mailto:arthur.ragauskas@ipst.gatech.edu) (A.J. Ragauskas).

xyloglucans to eucalyptus unbleached kraft pulp at the wet-end increased the mechanical properties such as, burst and tear indexes, by approximately 30% (Lima et al., 2003). As a novel alternative approach, Muguet, Pedrazzi, and Colodette (2011) have shown that the xylan content of unbleached kraft eucalyptus pulp can be elevated through deposition of eucalyptus xylan in the course of the oxygen delignification stage. The deposited xylan improved the beatability and mechanical properties of the pulp. The benefits of reinforcing a fully bleached OD(EPO)DP eucalyptus kraft pulp was also recently reported using methylglucuronic acid xylan, an enriched hexenuronic acid xylan and low uronic acid xylan (Silva et al., 2011). The absorption properties of these xylans were found to be dependent on the structure of the starting xylan and process conditions used. Water retention values and beatability of the xylan rich fibers was found to improve while hornification properties decreased which are well known favorable trends for kraft pulps.

Several researchers have reported on surface modification of cellulose fibers by the assembly of birch glucuronoxylans from solution under pulping-like conditions (Henriksson & Gatenholm, 2001). In earlier model studies, they detected the agglomeration of hardwood xylan and surface deposition on bacterial cellulose. The proposed mechanism for xylan retention is that the xylan can associate with cellulose through interactions between the unsubstituted, linear regions of the chains (Linder et al., 2003; Linder & Gatenholm, 2004). A study by Shin and Stromberg (2007) provides a cautionary note at using kraft pulping conditions to modify xylan content and physical strength properties of kraft Eucalyptus pulps. By using a series of alkali-profiled continuous kraft cooking technologies the authors provided compelling evidence that the “total” xylan content of eucalyptus kraft pulps did not correlate with tensile index under the conditions reported. The issue of changes in structure and location of pulp xylans formed from these different cooking approaches needs further examination (Shin & Stromberg, 2007).

In this study, we investigated the deposition of birch xylan onto eucalyptus bleached pulp under mild conditions. As global demand for the eucalyptus kraft pulp has grown and displaced other hardwood furnishes this provides an opportunity to use birch-xylan as a value added additive to enhance the performance of this renewable material. Our main interest was to study the effect of xylan deposition on the physical properties (tensile, tear, burst and water retention value (WRV)) of paper test sheets made from elementally chlorine free (ECF) bleached eucalyptus kraft pulp. In addition, the surface morphology of xylan deposited cellulose fiber was studied by AFM.

## 2. Experimental

### 2.1. Materials

A never dried elementally chlorine free bleached eucalyptus kraft pulp was acquired from a commercial pulp manufacturer in South America. All chemicals and reagents were purchased from Sigma–Aldrich Corporation, USA. Likewise, birch xylan was acquired from Sigma–Aldrich Corporation, USA.

### 2.2. Xylan solution

A birch xylan (6.00 g) with a reported MW of ~9600 g/mol (Köhnke & Gatenholm, 2007) was added to 120.00 mL deionized water. This solution was then adjusted to pH 11.30 using 2.50 N NaOH and then vigorously mixed at 80 °C until the xylan was dissolved.

### 2.3. Adsorption of xylan on the kraft pulp

A never-dried bleached eucalyptus kraft pulp was diluted to 5% consistency and pH adjusted to 9.00 with 2.50 N sodium hydroxide. The xylan solution was added to pulp and the mixture was stirred at 70 °C for 15 min. The pH of the xylan–pulp slurry was adjusted to 5 with the addition of 2.00 N H<sub>2</sub>SO<sub>4</sub>. After cooling the pulp slurry to room temperature, the xylan modified pulp was filtered and washed with deionized water. The control pulp was treated in the same manner except no xylan was added.

### 2.4. Preparation of handsheets

Test sheets were made from pulp by dewatering on a screen. These sheets were pressed at 0.35 MPa for 5 min and then dried on a plate dryer at 105 °C for 10 min. Eight test sheets were prepared for each treatment. The test sheet grammage (weight per unit area) was 160 g/m<sup>2</sup> and all handsheets were conditioned according to TAPPI standard conditions before testing (TAPPI Test Method T402, 2003).

### 2.5. Absorption amount of xylan

The xylan fiber content was determined by sugar analysis of the test sheets. This was accomplished using approximately 0.175 g of the pulp weighed into a digestion tube to which 1.50 mL of 72% sulfuric acid was added with stirring until wetted. The samples were then placed into a Digibloc<sup>®</sup> set at 30 °C and treated for 1 h followed by cooling to room temperature and dilution with 42.00 mL of water to 3% sulfuric acid concentration (Hu & Ragauskas, 2011; Köhnke, Breliid, & Westman, 2009). The samples were then autoclaved for 1 h at 121 °C. A 1.00 mL aliquot of the autoclaved liquid was diluted to 25.00 mL and then it was detected by HPLC. The samples were assayed using an Agilent 1200 HPLC series system, equipped with an AminexHPX–42C column (300–7.8 mm) and a refractive index detector. Samples (10.0 μL) were filtered using a 0.45 μm polytetrafluoroethylene syringe filter and eluted at 0.60 mL/min with nitric acid (10.0 mM). The temperatures used for the column and refractive index detector were 65.0 and 45.0 °C, respectively.

### 2.6. Physical testing

Tensile, tear and burst values were determined according to the TAPPI Test Methods (TAPPI Test Method T494, 2001; TAPPI Test Method T414, 2004; TAPPI Test Method T403, 2010). Tensile Index, Tensile Energy Absorption (TEA) and strain were performed automatically using a QC 1000 tensile tester from Thwing–Albert Instrument Co., Philadelphia, PA. Elmendorf tearing tester was also made by Thwing–Albert Instrument Co. Under TAPPI standard all tests were carried out at 23 °C ± 1 °C and 50 ± 2% relative humidity (TAPPI Test Method T402, 2003). These physical strength tests provide an evaluation of key paper properties. In brief, tensile strength is influenced by fiber strength, fiber bonding and fiber length as it measures the force required to produce a rupture in a strip of paper. The extent of interfiber bonding is considered the most important factor contributing to tensile strength properties. Tear test, measures the internal tearing resistance of paper which is measured by the force perpendicular to the plane of the paper necessary to tear a sheet through a specified distance after the tear has already been started. Burst involves clamping a sheet of paper firmly between two plates equipped with an opening and a rubber diaphragm. As the chamber is pressurized the bulging diaphragm causes the paper to rupture and this provides a measure of the bursting pressure needed to rupture paper.

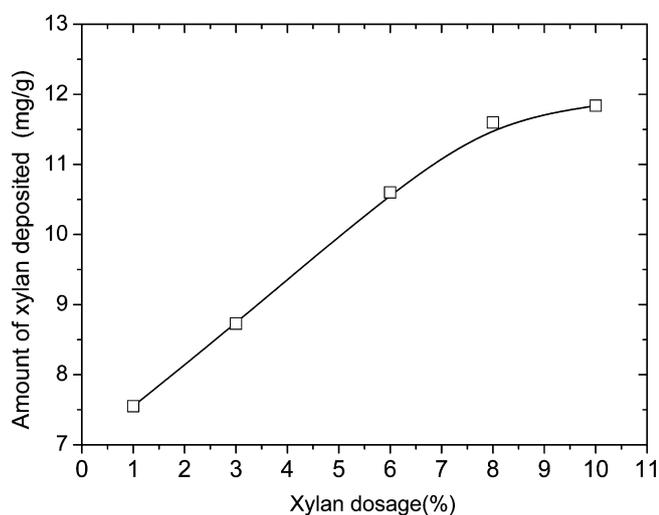


Fig. 1. Deposited amounts of xylan on the cellulose fiber.

### 2.7. Water retention value (WRV)

Water retention value (WRV) was determined by a centrifuging 1.50 g samples (o.d.) at 3000 r/min for 15 min. The pulp weight after centrifugation and dry weight after drying are used to calculate the WRV as:

$$WRV = \frac{M_1 - M_2}{M_2} \quad (1)$$

Where  $M_1$  is the weight of wet pulp after centrifugation,  $M_2$  is the weight of dry pulp (Chen, Wan, Ma, & Lv, 2010).

### 2.8. Atomic force microscopy analysis

The AFM images were obtained by a Nanoscope Dimension 3100 SPM, with a Nanoscope IIIa controller. Images were collected in tapping mode in air, with a phosphorus doped silicon tip with a nominal frequency of 150 kHz.

## 3. Results and discussion

### 3.1. The adsorption content of xylan on the fiber

The amount of xylan deposited (mg xylan per gram of pulp) as a function of xylan dosage is shown in Fig. 1.

Fig. 1 depicts that the amount of xylan deposited on the eucalyptus fibers increased with increasing xylan dosage. When the xylan content is 8%, the amount of xylan deposited reaches 1.15 wt% cellulose fiber and above this point the absolute amount of xylan deposited began to decrease.

### 3.2. Mechanical properties of never dried modified pulp

The results of tensile, tear and burst index analyses are summarized in Figs. 2 and 3.

As shown in Fig. 2, the tensile properties including tensile index, strain and tensile energy absorption (TEA) all increased by adding xylan and reached the maximum at 8% of xylan dosage.

The maximum increase of tensile index, strain and TEA were 7.80%, 9.60% and 12.50%, respectively compared to the control samples.

Tear index measurements (Fig. 3A) shows a relatively small degree of improvement (at the level of xylan addition below 3%) and declines as the xylan dosage increases beyond 3% (Fig. 3A). The burst index values were affected by addition of xylan (Fig. 3B),

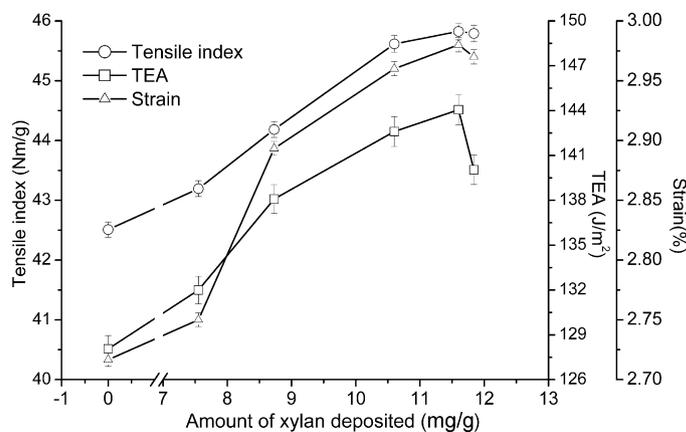


Fig. 2. Effect of xylan addition on tensile index.

exhibiting maxima at 2.98 kPa m<sup>2</sup>/g with the addition of 8% and 3% xylan. There was a negative effect on burst index at additions above 8%.

Figs. 1–3 show that a 1.15 wt% adsorption of birch xylan on the kraft fibers at 8% xylan addition increased the tensile index, strain and tensile energy absorption values by ~10%, while the burst index increased by 20.15%. The tear index increased by 2.55%, with the adsorption of 0.87 wt% birch xylan on the eucalyptus kraft pulp at 3% xylan addition. The improvements in mechanical properties were attributed to increasing inter-fiber bonding strength due to the addition of xylan, as evidenced by Fig. 3C showing that xylan-containing pulps have a higher capacity to retain water. This may be due to presence of a significant amount of hydroxyl groups introduced with xylan and the inherent hygroscopicity of hemicelluloses (Buckeridge, Pessoa dos Santos, & Tine, 2000). The addition of xylan to paper can also strengthen the interaction with cellulose fibers due to a higher degree of hydrogen bond formation between the polysaccharides. While mechanical properties are highly influenced by the interfiber bonding strength (Muguet et al., 2011; Lima et al., 2003).

### 3.3. Physical properties of modified pulp after drying

The effect of drying process on the properties of hardwood kraft pulp modified with xylan is of clear practical interest (Köhnke et al., 2010) and is examined in Fig. 4A–D. Similar to the never dried pulp, the tensile (Fig. 4A), tear (Fig. 4B) and burst (Fig. 4C) of the dried handsheets were analyzed, and trends in retention of tensile index compared. The retention of tensile index after drying is calculated as:

$$RTI = \frac{T_2}{T_1} \quad (2)$$

Where  $T_1$  is the tensile index of testsheet made from never dried pulp,  $T_2$  is the tensile index of testsheet made from pulp after drying.

From these analyses, a decrease in tensile, tear, and burst indices of the handsheets compared to undried pulp was observed (Fig. 4A–C). However, the retention of tensile index increased with the increase of xylan dosage, with values about 50% lower than those of undried pulps. The retention of tensile index reached approximately 65% at 10% xylan addition, with the tear and burst indices showing the same trend in relation to retention. The results show that the drying treatment could negatively affect most mechanical properties due to hornification of fibers. The improvement was made more significant by xylan addition. The increase was from 28.5 to 42.0% while the dosage of xylan was varied from 0 to 10%.

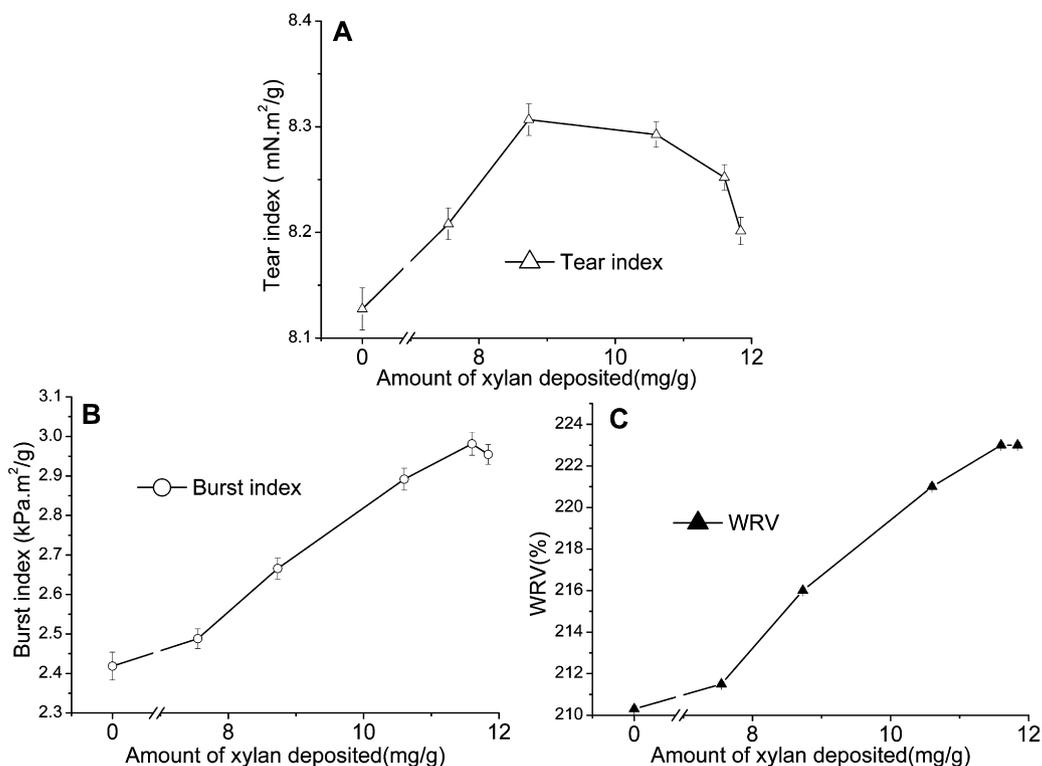


Fig. 3. Effect of xylan addition on tear (A), burst (B), and WRV (C).

Fig. 4A–C show that the mechanical properties of handsheets made from dried, modified pulps decreased compared to the handsheets made of undried pulp by 45–65%. This was readily attributed to the hornification of cellulose fibers. The extent of pulp

hornification has been evaluated by determining water retention values (WRV) pulp after drying (Nazhad & Paszner, 1994). Drying generates intra-fibrillar bonding, which closes pore spaces and decreases the elasticity of the fiber wall, both resulting in lower

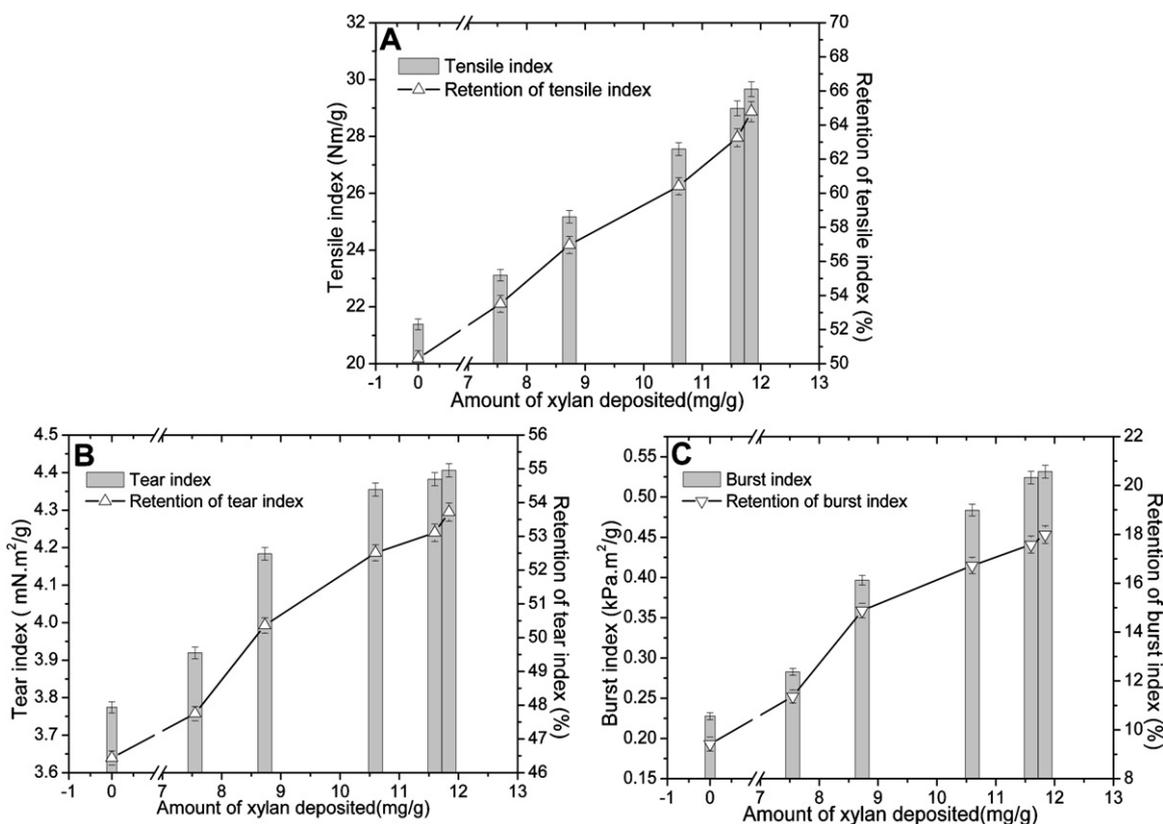


Fig. 4. Effect of xylan addition on tensile (A), tear burst (B), and burst (C) after drying.

fiber swelling. When the cellulose fiber is rewetted, the elasticity of the fiber wall cannot be fully recovered. As a result, the water retention, flexibility and bonding potential of kraft pulp are lower, explaining why the values of tensile, tear and burst indexes all decreased after drying (Köhnke, Pujolras, Roubroeks, & Gatenholm, 2008; Zhang, Hubbe, Venditti, & Heitmann, 2002).

It has been suggested that this effect diminished the hornification of cellulose fibers (Fernandes Diniz, Gil, & Castro, 2004; Köhnke et al., 2010), and that the effect of hornification was reduced by adding xylan. Xylan treatment of pulp fibers is performed before they are dried in order to inhibit the effects associated with drying.

#### 3.4. The influence of xylan on refining performance of pulp

The eucalyptus pulp modified with 8% xylan addition was mechanically refined using a PFI mill refiner at 0, 500, 1000 and 2000 revolutions. The Canadian standard freeness (CSF) was tested and the handsheets were prepared for physical testing.

As shown in Fig. 5, under the same number of beating revolutions the CSF of modified pulp decreased quicker than in control sample, demonstrating that xylan addition can improve the beating performance of pulp. This phenomenon may be due to the fact that xylan swells more in water than the cellulose fibers as has been shown in Fig. 3C.

As shown in Fig. 6, mechanical properties including tensile index (A), tear index (B) and burst index (C) are all improved with increasing refining revolutions. Comparison of modified and unmodified with xylan pulps suggests that mechanical properties of handsheets made from xylan modified pulps exhibited enhanced physical strength properties over the control pulps with PFI refining.

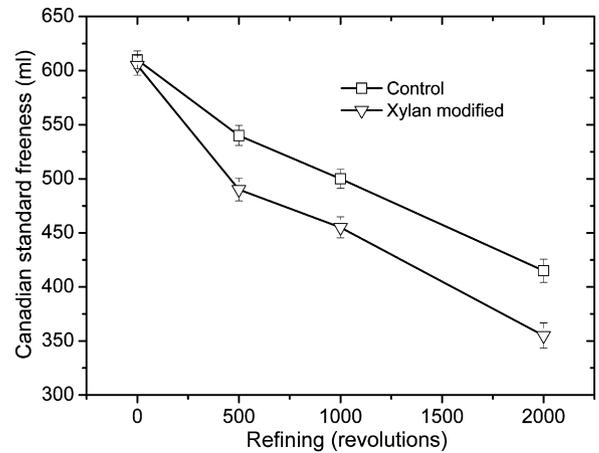


Fig. 5. Effect of xylan on the PFI refining.

Refining of pulp is a mechanical process that increases the surface area of fibers and exposes more hydroxyl groups on the surface of the fiber. As such, it is a very important treatment for the improvement of the mechanical properties of handsheets that increases the number of bond-forming sites on the fiber surface. Xylan is much more susceptible to swelling in water than cellulose fibers so the pulps modified with xylan can be readily refined. The effect of xylan addition on pulp after refining could be explained by the higher capacity for binding to those cellulose fibers, resulting in paper sheets with higher values of mechanical properties.

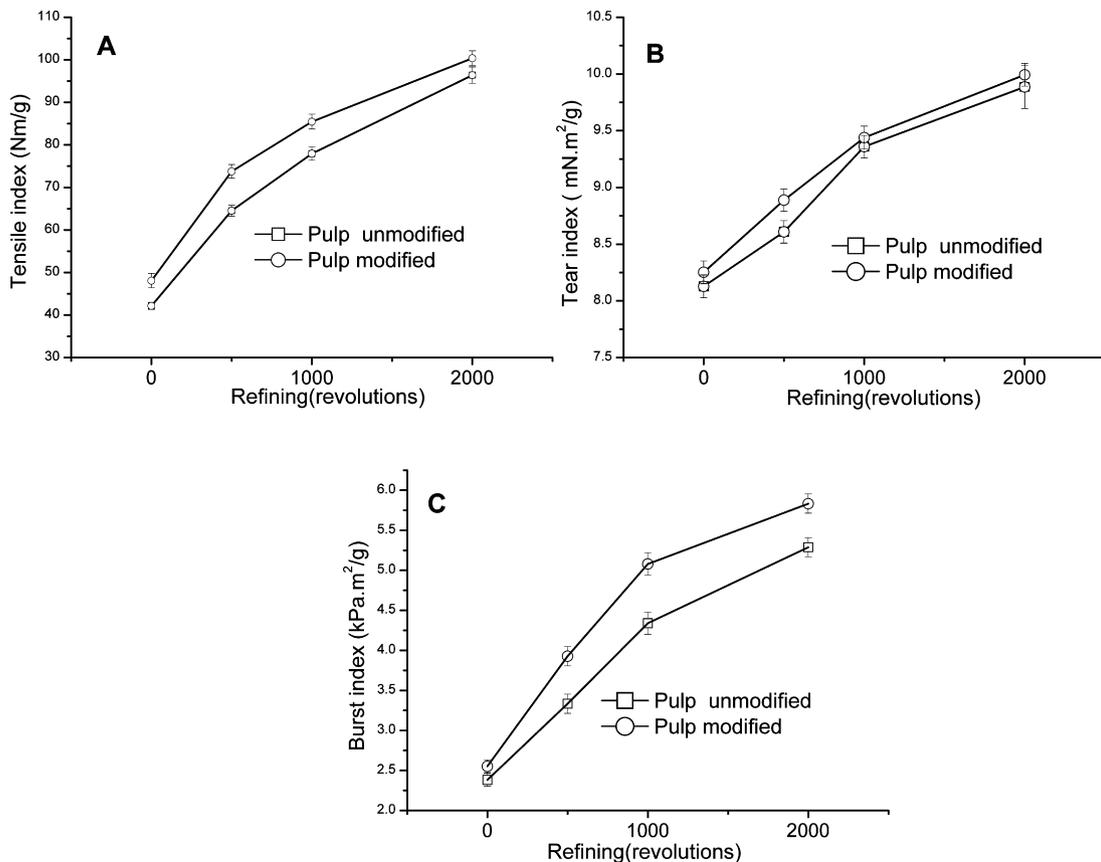


Fig. 6. Effect of refining on the mechanical properties.

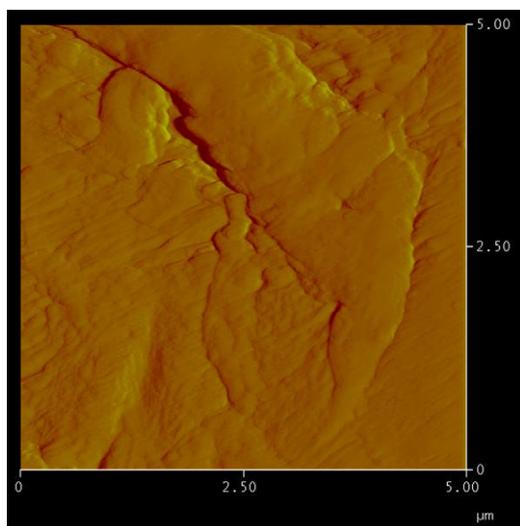


Fig. 7. Atomic force microscopy (AFM) images of control cellulose fiber.

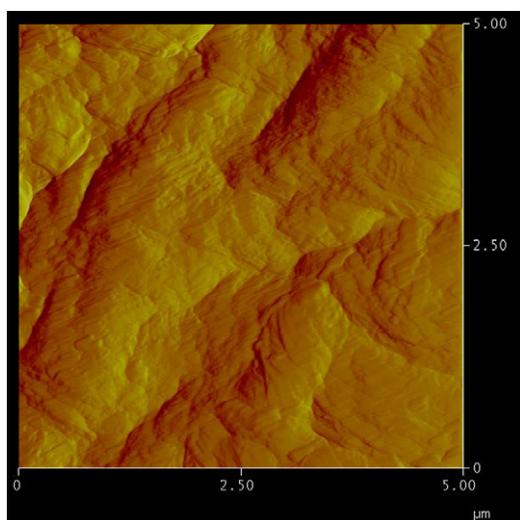


Fig. 8. Atomic force microscopy (AFM) images of modified cellulose fiber by xylan.

### 3.5. Surface morphology of cellulose fiber

AFM analysis provides a means of exploring the xylan distribution on the cellulosic eucalyptus fibers used in this study. The amplitude images obtained from the control sample and sample modified by 10% xylan are shown in Figs. 7 and 8.

The images show obvious difference between surface of modified and unmodified cellulose fibers. The unmodified cellulose fibers in Fig. 7 have a relatively smooth surface. The fibers with adsorbed xylan (Fig. 8) appear to have a higher surface roughness with more pronounced 'hills and valleys'. As is the case of adsorbed xylan in comparison to the surfaces of unmodified fibers, it is believed that these submicron structures are composed of xylan molecules adsorbed from solution and assembled on the cellulose fibers (Henriksson & Gatenholm, 2002). This effect is caused by the xylan depositing on the cellulose fiber. The distribution of xylan on the surface of fibers changes the topography of fiber.

## 4. Conclusion

In this study, xylan was absorbed onto the cellulose fiber under relatively mild conditions, by mixing with cellulose fibers for

15 min at 70 °C, after which the pH was adjusted from 9.00 to 6.00. Adsorbing xylan onto cellulose fibers can improve some mechanical properties of paper, namely: tensile and burst index, while there was little change in the tear index. The retention properties of paper sheets made of dried pulp are increased with the increasing content of xylan due to the higher resistance of cellulose fibers toward hornification and better combination performance with fiber. The characteristics of beating were also improved by adding xylan. The amount of xylan deposited in handsheets was detected by HPLC which appeared to have increased with increasing xylan content. The atomic force microscopy (AFM) analysis shows that samples with xylan deposited on them have surfaces of fibers with higher degree of roughness than the unmodified fibers.

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