Pilot-Scale Investigation into the Effects of Alkaline Peroxide Pre-Treatments on Low-Consistency Refining of Primary Refined Softwood TMP

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Primary refined coarse softwood thermomechanical pulp was treated with alkaline peroxide prior to low-consistency (LC) refining. The effects of the pre-treatments on pulp quality, refinability, and electrical energy consumption were assessed. Four pre-treatments were conducted with alkali charges of 2.5 and 6% and peroxide charges of 3 and 4%. The pulps were refined to specific energies up to 600 kWh/t by multiple passes through an LC refiner at intensities of 90 and 150 kWh/t. It was found that alkaline peroxide treatments increased tear strength and protected the fibre from cutting, especially during high intensity refining below a specific energy of 300 kWh/t. Treatment with 6% NaOH and 4% or 3% H_2O_2 led to lower brightness gains and scattering coefficients but increased the tensile strength index by 31%, potentially lowering the total electrical energy required to achieve strong pulp. The enhancement of tensile strength caused by the highly alkaline peroxide mostly resulted from increased bonding, which was attributable to acid group generation rather than the promotion of further fibrillation during LC refining.

Keywords: Alkaline peroxide treatment; TMP; Low-consistency refining; Refining intensity; Energy saving; Fibre cutting; Tensile strength; Acid group generation

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INTRODUCTION

Refining at low-consistency (LC), typically at 4%, of thermo-mechanical pulp (TMP) has been reported to be an energy-saving technology, providing more efficient fibre development than refining at a high-consistency (HC) of approximately 40% (Muenster 2005). However, LC refining of TMP is not widely applied in the pulp and paper industry due to fibre cutting (Muenster 2005; Andersson *et al.* 2012). Modern thermo-mechanical pulp processes mostly use HC refining to develop pulp of the desired quality (Tienvieri *et al.* 1999)

A previous laboratory investigation used a Waring blender and a PFI mill to simulate the LC refining of low-freeness TMP. This work demonstrated that highly alkaline hydrogen peroxide increased the tensile strength and made the fibres more flexible and resistant to cutting during the subsequent LC refining (Chang *et al.* 2010). It could be possible to exploit the noted improvements in fibre properties to extend the applicability of energy-efficient LC refining to high-freeness TMP. The extended application of LC refining, along with the tensile strength gain directly obtained *via* alkaline peroxide treatments, provides the potential for significantly lower electrical energy requirements in mechanical pulp production.

The LC refining of the alkaline peroxide-treated TMP in the prior research was conducted at the laboratory-scale under constant refining intensity and specific energy (Chang *et al.* 2010). Before considering this technology for application at the mill scale, it is essential to determine whether similarly enhanced fibre resistance to cutting and tensile gains can be obtained as on the pilot scale over a range of LC refining specific energies and refining intensities. By performing these trials on high-freeness, primary-stage TMP, it was possible to determine whether or not major electrical energy savings could be achieved by using this technology to replace inefficient, secondary-stage, high-consistency refining.

In this paper, the results of a pilot-scale trial of the LC refining of a primary refined hemlock TMP after alkaline peroxide treatments are discussed. The primary TMP, refined at high consistency, was treated with various peroxide and alkali charges prior to LC refining over a range of specific energies at intensities of 90 or 150 kWh/t per pass through the LC refiner. The changes in the fibre and handsheet properties of the resulting pulps were assessed and compared. The effects of the peroxide and alkali charges on the development of the fibre characteristics and pulp properties during LC refining at various refining intensities were reported. The impact of applying alkaline peroxide treatments to high-freeness TMP prior to LC refining on energy savings was assessed. The mechanism for the pulp property changes during treatment was considered.

EXPERIMENTAL

Materials

The pulp, used for peroxide treatments and LC refining, was a primary HC-refined TMP made from whole log hemlock chips using a 36” Andritz 36-1CP disc refiner in the Andritz pilot plant, Springfield, Ohio. The specific energy applied was 685 kWh/t, yielding pulp with freeness 494 mL. The pulp was treated with 0.4% diethylene triamine pentaacetic acid (DTPA) at 60 °C and 4% consistency and was dewatered to approximately 27% consistency using a double wire press prior to alkaline peroxide treatment and LC refining. The primary pulp was not screened.

Alkaline Peroxide Pre-treatment

Alkaline peroxide pre-treatment conditions were based on previous studies at laboratory scale (Chang *et al.* 2010, 2011). Briefly, the dewatered, primary HC-refined pulp was mixed with alkaline peroxide liquor at 20% consistency using an Andritz 401 refiner (USA) with the plates at a constant and wide gap. The specific energy applied in the HC “mixer" was approximately 220 kWh/t, such that the temperature of outlet pulp was increased to 60 °C. The mixed pulp was held in barrels for 1 h before being transferred to a tank, neutralized to pH 7, and diluted to 4% consistency for LC refining. The chemical charges applied were 0.1% magnesium sulfate ($MgSO_4$), 3% sodium silicate (Na_2SiO_3), 3% or 4% hydrogen peroxide (H_2O_2), and 2.5% or 6% sodium hydroxide (NaOH) based on the O.D. pulp mass. The control sample was subjected to the same process but was mixed with water instead of bleaching liquor.

LC Refining

The alkaline peroxide-treated pulps (0.65 tonne O.D.) were diluted to 4% consistency in a tank and the pH adjusted to 7. The pulp was refined using a 22-inch

Andritz TwinFlow LC refiner by pumping the pulp through the refiner, from one tank to another, at a specific energy of 90 kWh/t. The pulp was sampled before passing through the refiner again at the same specific energy. The process was repeated seven times to a total specific energy of 630 kWh/t. The same procedure was repeated at higher intensity, using 150 kWh/t per pass through the refiner, to a total specific energy of 1,050 kWh/t. The control pulp was refined in the same manner.

Pulp Freeness

The freeness of the pulps was tested according to TAPPI method T227 (1999).

Handsheet Properties

Handsheets were made according to TAPPI method T205 (2002). Bulk, brightness, tensile strength, and tear resistance were determined according to TAPPI method T220 (2001).

Pulp Fractionation

Bauer-McNett

Bauer-McNett fractionation was conducted according to TAPPI method T233 (1995). The mesh sizes used for fractionation were 14-, 28-, 48-, 100-, and 200-mesh (Tyler standard screen scale).

Britt dynamic drainage jar

Fractionation to obtain long fibres equivalent to a combination of the R28 and R48 Bauer-McNett fractions was conducted using a Britt dynamic drainage jar (DDJ). The pulp suspension for Bauer-McNett fractionation (0.3 g O.D. pulp at 0.3% consistency) was filtered using a 1-L Britt DDJ with a 48-mesh screen on the bottom. During filtration, deionized water was continuously added to the jar to maintain the volume of the suspension, which was stirred using an overhead stirrer at 200 rpm for 20 min. After filtration, the pulp retained in the DDJ was collected and filtered again using the DDJ with a 14-mesh screen following the method described above. The filtrate passing through the 14-mesh screen was collected in another DDJ with a 200-mesh screen.

Acid Group Content

The acid group contents of the whole pulps, prior to LC refining, and the long fibres, obtained by DDJ pulp fractionation, were determined using the method of conductometric titration of pulp described by Beatson (1992).

Surface Charge

The procedure used was based on Sang and Xiao’s method of back-titration using a particle charge detector (Mütek PCD-03, Herrsching, Germany) (Sang and Xiao 2009). Pulp or a fibre suspension at 0.03% consistency (0.05 g O.D.) was added to a beaker containing 2 mL of 1 mM cationic poly-diallyldimethylammonium chloride (poly-DADMAC) and stirred for 2 h. The stirred suspension was then transferred to Falcon tubes and centrifuged at 3000 rpm for 10 min. Approximately 15 g of supernatant was added to the measurement cell of a Mütek Charge Detector and titrated with standard 0.1 mM anionic polyvinyl sulphate, potassium salt (PVSK).

Statistics

The data points in the figures are the means of measurements. The measurements were in duplicate unless otherwise specified. Error bars in the bar graphs refer to the 95% confidence intervals. Statistical analysis was performed manually.

RESULTS AND DISCUSSION

Chemical Treatments and LC Refining

HemlockHC-refined primary TMP with a freeness of 494 mL was treated with three different combinations of sodium hydroxide (NaOH) and hydrogen peroxide (H_2O_2): 4% H_2O_2 and 2.5% NaOH; 3% H_2O_2 and 6% NaOH; and 4% H_2O_2 and 6% NaOH. The treatment with 4% H_2O_2 and 2.5% NaOH used an alkali-to-peroxide ratio typically for pulp brightening (Presley and Hill 1996) and succeeded in increasing the brightness of the primary pulp from 41.2% to 70.7% ISO. On the other hand, the treatment at higher alkalinity (4% H_2O_2 and 6% NaOH) primarily served to increase the tensile strength (Chang *et al.* 2010), and the brightness gain was limited to 14% ISO. The response of these treated pulps to pilot-scale LC refining served to evaluate the impact of the alkalinity and peroxide on the development of pulp and fibre properties during LC refining.

During the mixing of the primary TMP with the treatment liquors (using the HC refiner with the plates at a constant and wide gap size), the pulp freeness dropped from 494 to 385 mL and a temperature of 60 °C was achieved at the refiner discharge. During the 1-h retention of the pulp/chemical mixtures in barrels, the pH values dropped from 11 to 8.5, 12.1 to 11, and 12.5 to 11.6 when charged with 4% H_2O_2 and 2.5% NaOH, 4% H_2O_2 and 6% NaOH, and 3% H_2O_2 and 6% NaOH, respectively.

The pulps treated with 4% H_2O_2 and 2.5% or 6% NaOH were refined in seven stages in the LC refiner at a specific energy of 90 kWh/t per stage; the pulps treated with 3% H_2O_2 and 6% NaOH or 4% H_2O_2 and 6% NaOH were refined in a similar manner, but at higher intensity, 150 kWh/t per stage. The effects of the alkaline peroxide treatments on LC refining were evaluated by comparing the properties of the pulps to those of a non-treated control after each stage of refining.

Tensile Strength and Specific Energy

As shown in Fig. 1, regardless of the peroxide charge, the highly alkaline peroxide-treated pulps (6% NaOH) required considerably less specific energy to obtain a desired tensile strength than the pulps treated at a lower alkali charge or those with no alkaline peroxide treatment.

At LC refining intensites of both 90 and 150 kWh/t, the highly alkaline treatments saved approxmately 300 kWh/t, or 25% of the total electrical energy used in achieving a handsheet tensile strength of 42 Nm/g. The treatments of the primary pulp with peroxide at higher alkalinity increased the initial pulp tensile strength by 5 Nm/g or 20%. Subsequent LC refining of these pulps gradually led to an additional tensile strength gain of up to 5 Nm/g above that of the control pulp. Beyond the specific energy required to obtain 5 Nm/g of additional tensile strength (approximately 200 kWh/t), the changes in the tensile strength of the alkaline peroxide-treated pulps with the specific energy applied during refining were the same as in the control. However, the control and the treated pulps exhibited different tensile development trends at the different refining intensities.

At low intensity (90 kWh/t), the tensile strength of the control increased at a constant rate with applied specific energy. At high intensity (150 kWh/t), the rate of tensile strength increase of the control was initially high, then levelled off, after approximately 200 kWh/t specific energy was applied.

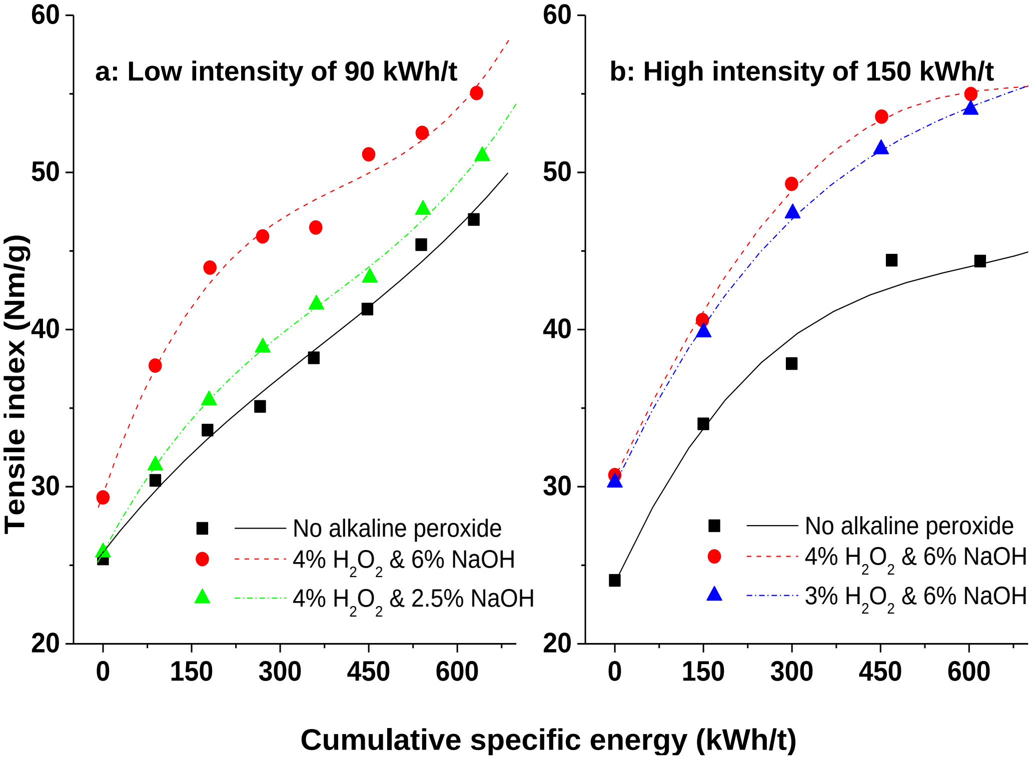

Fig. 1. Highly alkaline peroxide treatments increased the tensile strength achievable through LC refining

The different responses of the pulp tensile strength to LC refining at high and low refining intensities are consistent with the observations of Luukkonen *et al.* (2009). The observation that the alkaline peroxide treatments did not change the rate of pulp tensile strength development with specific energy at either refining intensities, especially after the primary pulps were refined to a freeness of 160 mL with approximately 200 kWh/t specific energy (Fig. 2), suggests that alkaline peroxide treatments did not promote further fibrillation of well-developed pulp during LC refining. This is consistent with the results of a previous study using a pulp with low freeness of 137 mL (Chang *et al.* 2010).

However, compared with treatments on pulp with low freeness using highly alkaline peroxide, the same treatments on primary pulp with high freeness resulted in 50% lower initial tensile strength gain, and the other 50% tensile strength gain was obtained gradually *via* LC refining. As discussed in a later section, the gain in tensile strength following the LC refining of the primary pulp is an indication of surface charge enhancement rather than promotion of fibrillation by the highly alkaline peroxide treatments.

The minor effect of the treatment with 4% H_2O_2 and 2.5% NaOH on the tensile strength development of the primary pulp during LC refining was consistent with previous laboratory-based work and confirms the requirement of high alkalinity for major pulp tensile strength enhancement in alkaline peroxide treatments (Chang *et al.* 2010).

Freeness

The significant increases in tensile strength were not accompanied by a corresponding drop in freeness (Fig. 2).

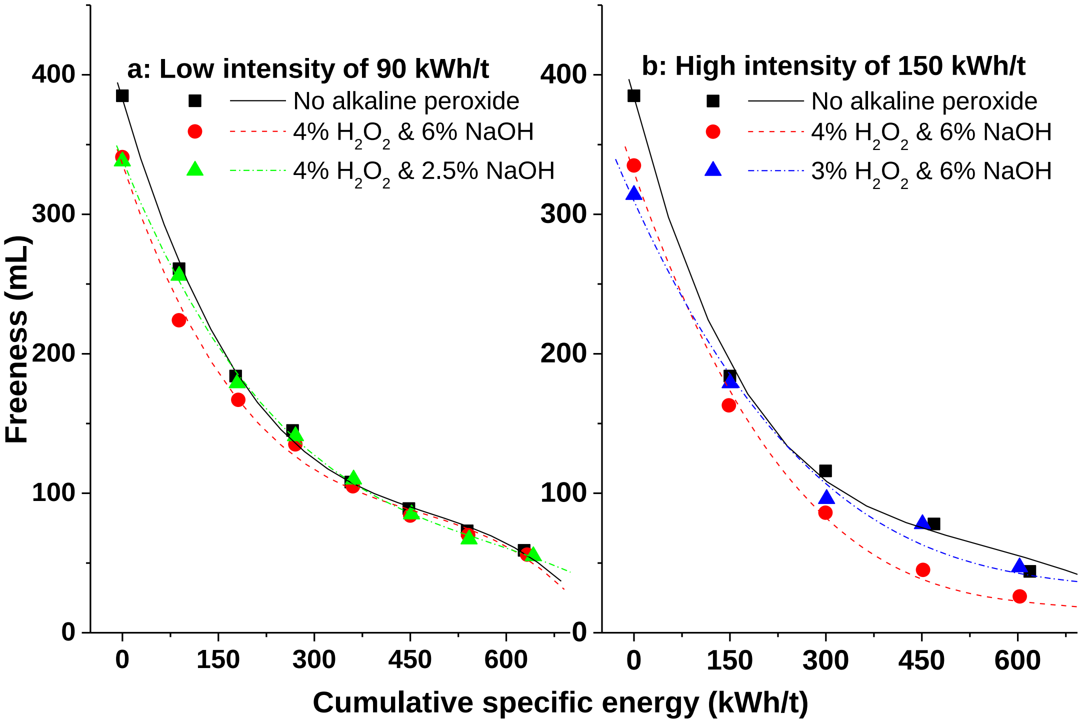

Fig. 2. The development of freeness during LC refining was affected little by chemical treatment

The freeness level at a given specific energy, especially above 150 kWh/t, was similar for all pulps, except for the pulp treated with 4% peroxide and 6% caustic and refined at high intensity. In this pulp, the freeness was about 30 mL lower than that of the control, likely related to the susceptibility of this pulp to fibre damage, as reflected by the low tear values (Fig. 3) and high fines content (Fig. 4) observed at higher specific energies.

Tear Strength

The tear strength of all pulps decreased with tensile strength gain during LC refining (Fig. 3). A decrease in tear strength with tensile strength is common for mechanical pulps and is related to increased bonding and is not necessarily an indication of lower fibre length (Shallhorn and Karnis 1979). However, during high intensity refining, the tear strength of the control pulp dropped more quickly with increasing tensile strength than during low intensity refining, resulting in much lower tear strength at a given tensile strength or bonding level.

This is consistent with greater fibre cutting in the control pulp during high intensity refining, as can be seen from the changes in the fibre distribution analyzed by Bauer McNett fractionation (Fig. 4). At the same tensile strength, the tear strength of the alkaline peroxide-treated pulps was higher than that of the control pulp with no alkaline peroxide treatments.

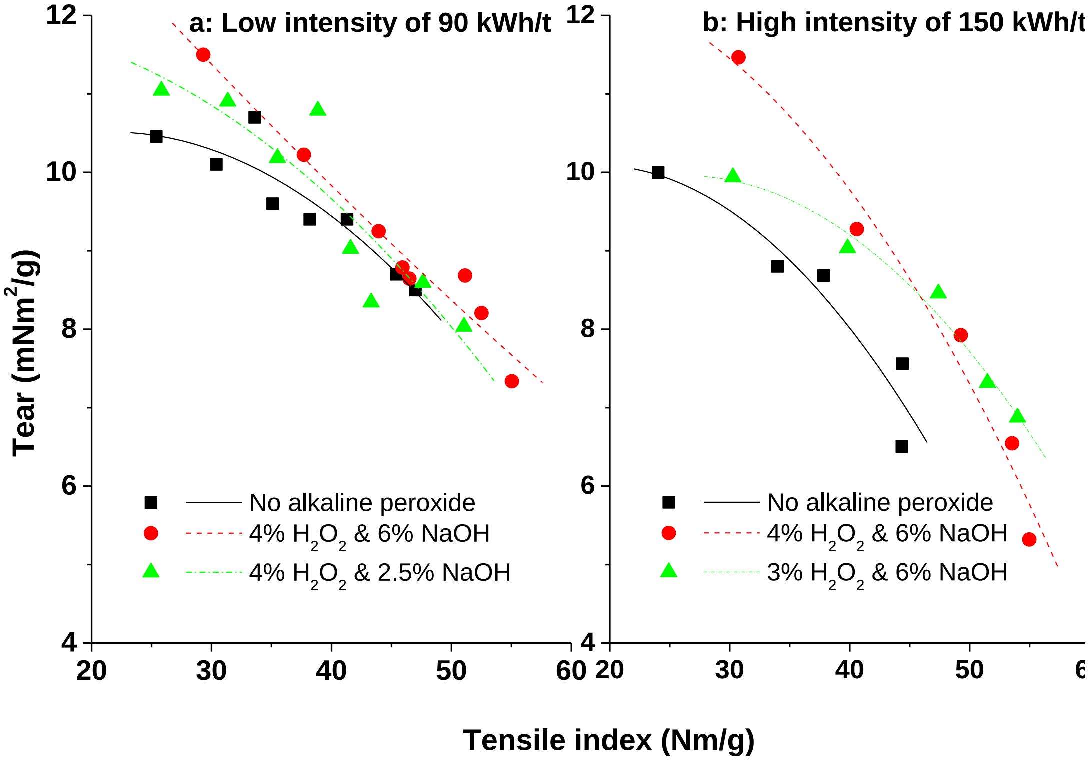

Fig. 3. Alkaline peroxide treatment enhanced tear strength, especially during high intensity LC refining

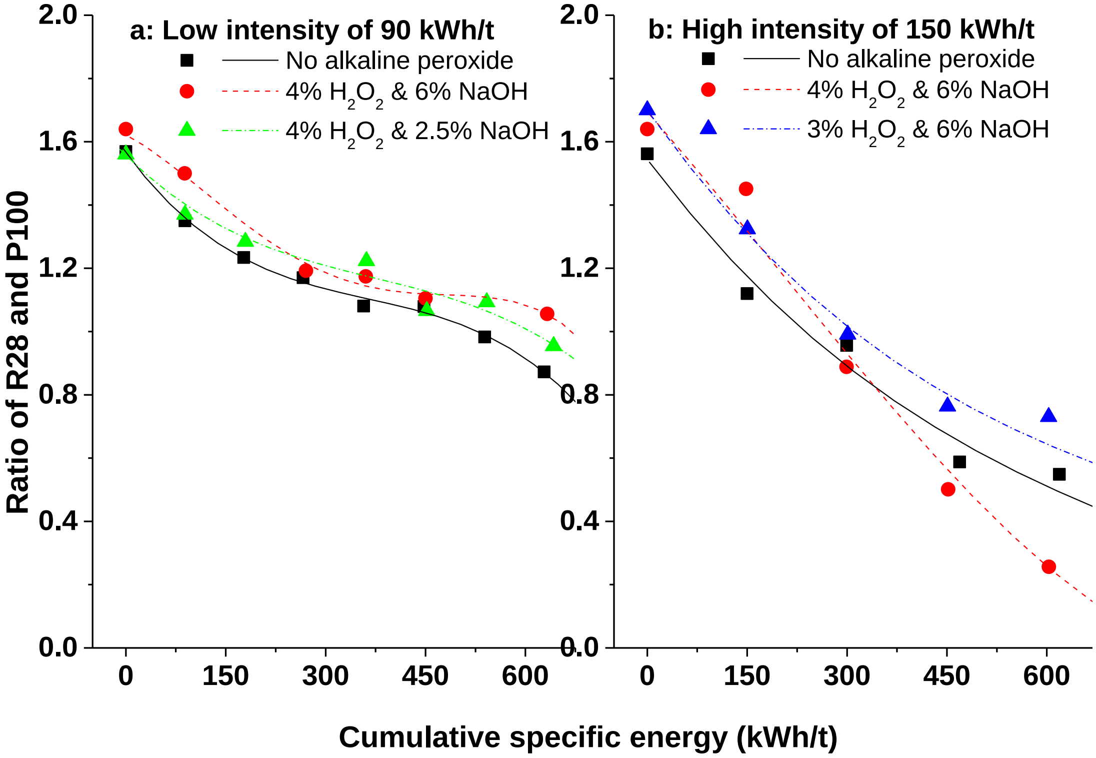

Fig. 4. Alkaline peroxide treatments protected fibre length during LC refining, except at high intensity and high specific energy

During low intensity LC refining, the tear strength differences between the alkaline peroxide-treated pulps and the control were initially large and then gradually decreased with increasing specific energy. During high intensity LC refining, the alkaline peroxide-treated pulps maintained significantly higher tear strength than the control at a given tensile strength or bonding level. This is consistent with the previous observation that alkaline-peroxide treatment protects fibres from cutting. This protection is expected to be more effective at higher refining intensities. Indeed, as shown in Fig. 4, the treated pulps maintained higher fibre length during high intensity refining. The loss in tear strength at the high tensile for the pulp treated with 4% peroxide and 6% alkali (Fig. 3) corresponded to a drop in fibre length (Fig. 4) and freeness (Fig. 2) at high refining energy and refining intensity.

This drop in the tear strength, fibre length, and freeness could be related to weakening of fibre wall caused by the chemical treatment. A similar phenomenon was previously observed in ozone-treated pulps (Chang *et al.* 2011). The protection of fibres by highly alkaline peroxide is apparently limited to a certain cumulative specific energy during high intensity LC refining. Beyond this limit, the fibre wall flaws created by highly alkaline peroxide treatments lead to enhanced fibre cutting and a rapid drop in tear strength and freeness.

Bulk

For all pulps, higher tensile strength corresponded to decreased bulk (Fig. 5). The bulk/tensile relationship was mostly independent of the chemical treatment and refining conditions. Thus, it is possible, through chemical treatment, to produce a pulp with a given bulk and tensile strength using reduced specific energy (Fig. 1).

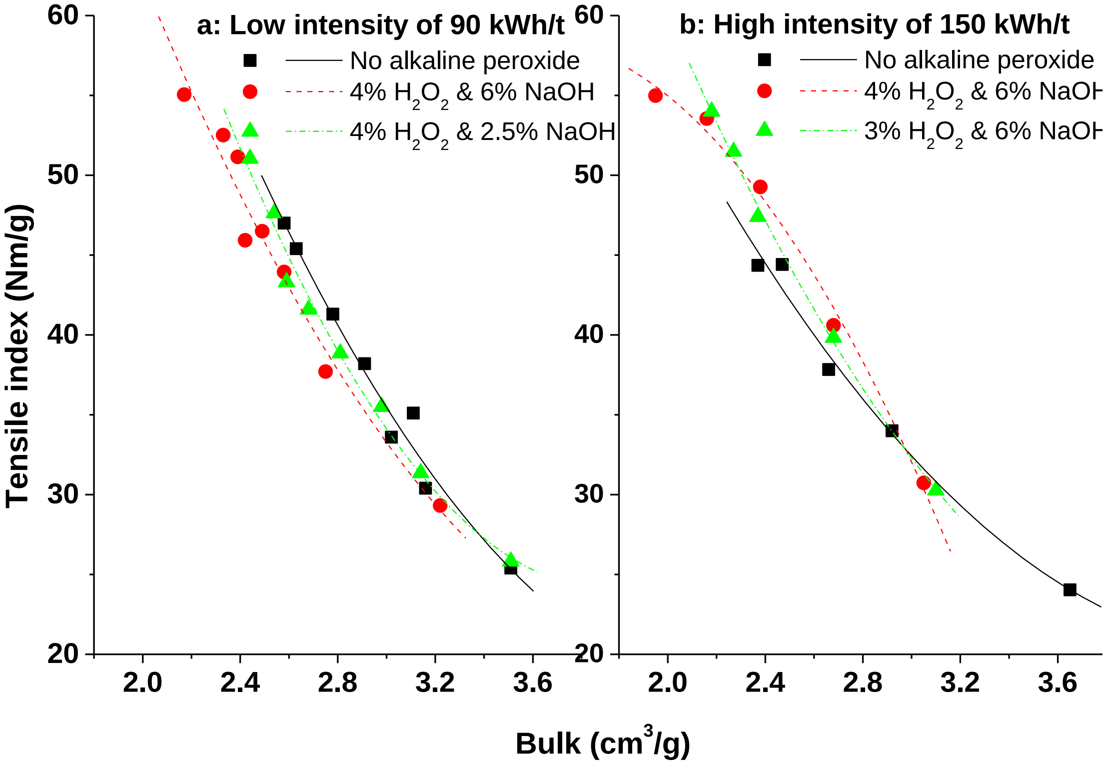

Fig. 5. The tensile/bulk relationship was affected little by chemical treatment

Chemical treatments at high alkalinity yielded pulp with tensile strengths beyond those achievable by LC refining alone, but these tensile strength increases were accompanied by further bulk losses.

Scattering Coefficient

As refining progressed, both the tensile strength and the scattering coefficient increased, as is typical for mechanical pulps (Fig. 6). It was not possible to achieve a high scattering coefficient in pulps treated at high alkalinity. However, at a given scattering coefficient below 50 m^2/kg, the tensile strength was significantly higher for the treated pulps. This could indicate increased inter-fibre bond strength, as discussed in a previous work (Chang *et al.* 2011). For peroxide treatment at low alkalinity, it was possible to obtain pulps with higher scattering coefficients at the same tensile strength as that of the control. It is possible that this treatment promotes the creation of non-bonding fines or fibrils during the early stages of refining, whereas at high alkalinity, such fines are more highly bonding and collapsible.

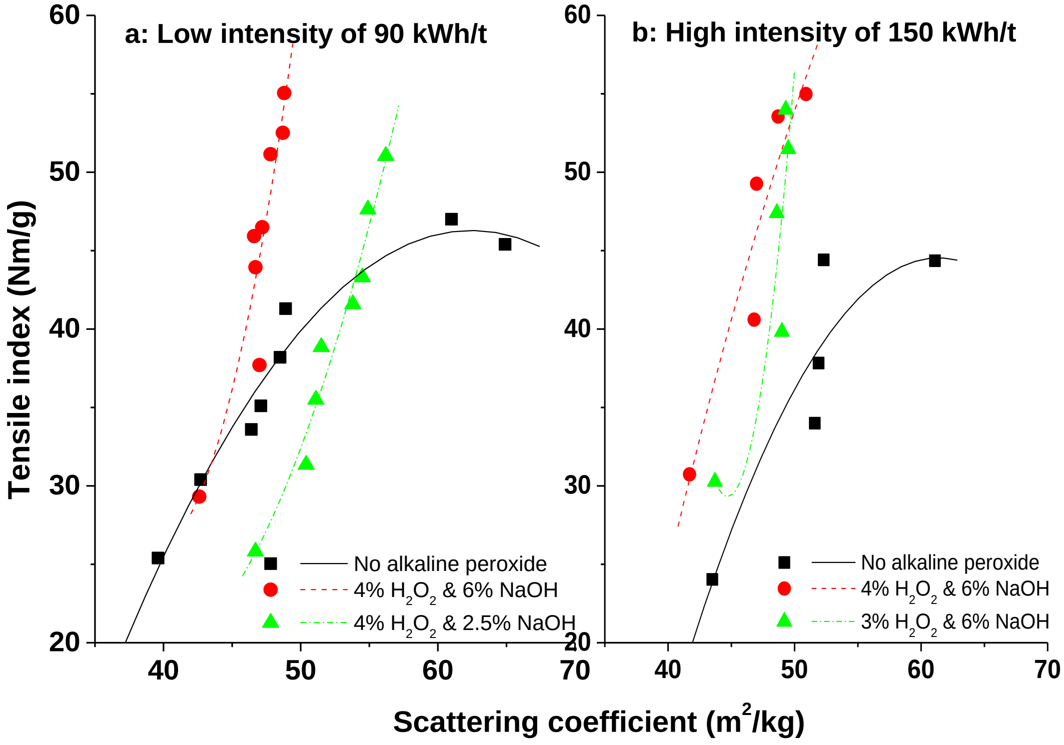

Fig. 6. Highly alkaline peroxide treatments lowered the scattering coefficient

Chemical Changes in the Pulps behind the Effects of the Alkaline Peroxide Treatments

The acid group content and surface charge were measured on the long fibres and the whole pulp of the coarse TMP after treatment with various alkaline peroxide charges in an attempt to determine the mechanism behind the effects of the chemicals on the pulp properties. Figure 7 shows that the alkaline peroxide treatments increased the acid group contents in both the long fibres and the whole pulp. The increase in the acid group content in whole pulp correlated well with the enhancement of the tensile strength of the pulps (Figs. 1 and 7). This is consistent with a previous study and shows the significance of acid group generation for increasing pulp strength (Chang *et al.* 2010).

The pulp treated with 4% H_2O_2 and 6% NaOH contained more acid groups than the pulp treated with 4% H_2O_2 and 2.5% NaOH. During alkaline peroxide treatments, acid groups were generated by reactions between lignin and the hydroperoxide anion formed from the alkali and hydrogen peroxide, brightening the pulp (Presley and Hill 1996). Acid groups could also be generated from the alkaline hydrolysis of the ester groups of pectins and xylans. Pranovich *et al.* (2003) found that, during alkaline peroxide treatments at 60 °C, the release of acid groups from pectins required a pH over 9. The treatment with 4% H_2O_2 and 6% NaOH had an initial pH of 12.1 and a final pH of 11, while the treatment with 4% H_2O_2 and 2.5% NaOH started at pH 11 and ended at pH 8.5. This may explain why the former had higher acid group content and pulp tensile strength, despite having lower brightness. These results are consistent with Holmbom's (1997) conclusion that high alkali is the dominant factor determining the amount of acid groups generated during alkaline peroxide treatments. They are also consistent with Korpela's summary that high alkalinity is critical to the development of pulp tensile strength during such treatments (Korpela 2002).

Figure 7 shows that the differences in the acid group contents of the whole pulp and long fibres obtained using a Britt dynamic jar were significantly higher following treatments at higher alkalinity. It is apparent that the extra acid groups generated at higher alkalinity were concentrated in the fines. Thus, it appears that the acid content of the fines, rather than that of the fibres, is critical to the development of tensile strength. This observation is consistent with the previous suggestion that tensile strength gains mainly result from acid group generation outside the fibre wall, which increase the interfibre bonding strength (Chang *et al.* 2010).

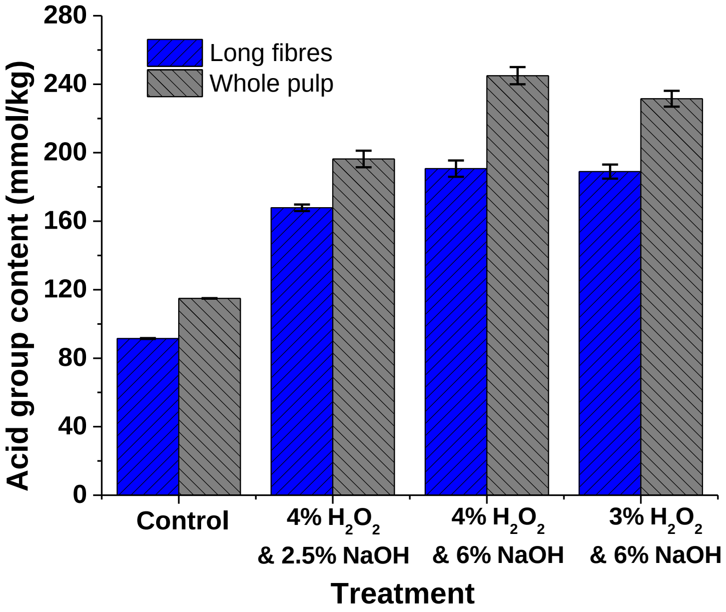

Fig. 7. The acid group content in long fibres and whole pulp increased with alkaline peroxide treatment

The high amount of acid groups in the fines generated during highly alkaline peroxide treatments was, perhaps, due to the high concentration of pectins located in the primary wall and middle lamella (Westermark *et al.* 1986). The pectin-rich middle lamella and primary wall were peeled off during refining and concentrated in the fines. Subsequent demethylation of pectins in the fines under alkaline conditions would generate the observed acid groups.

The surface charge of whole pulp was the highest for high alkalinity treatments, corresponding to the highest acid group contents and tensile strengths (Fig. 8). However, the surface charges in different alkaline peroxide-treated fibres were similar, exhibiting a different pattern than that of their acid group contents. This was probably due to low surface area per unit weight of long fibres. As pointed out by Kleen *et al.* (2003), the fibre surface and fines have similar compositions, including pectin and xylan. The acid group content generated from pectin and xylan on the fibre surface during alkaline peroxide treatments are peeled off and added to the fines fraction during LC refining. The accumulating acid group content in the fines could explain why the alkaline peroxide-treated pulps gained an additional 5 Nm/g of tensile strength upon LC refining at up to 200 kWh/t (Fig. 1).

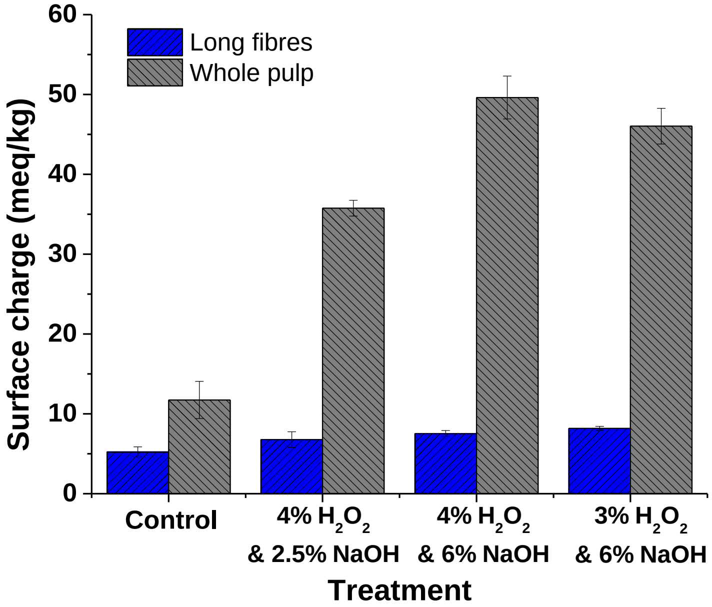

Fig. 8. The surface charge increased with alkaline peroxide treatment, especially for fines

CONCLUSIONS

1. Highly alkaline peroxide treatments of coarse primary softwood TMP increased the tensile strength index by 31%, after subsequent LC refining, saving approxmately 25% of the total electrical energy required to obtain a tensile strength of 42 Nm/g.
2. The relationship between the bulk and tensile strength was affected little by the treatments, whereas the scattering coefficient was lowered by peroxide treatment at high alkalinity. However, at a given scattering coefficient below 50 m^2/kg, the tensile strength of the pulp was significantly increased by the highly alkaline peroxide treatments.

3. The tensile strength ehancement by alkaline peroxide treatments was mostly a result of the generation of acid groups in the fines or on the fibre surface, rather than through the promotion of further fibrillation during LC refining.
4. Alkaline peroxide treatments enhanced the tear strength and increased the resistance to cutting during low intensity LC refining over the whole range of specific energies studied, up to 630 kWh/t. However, for the highly alkaline peroxide treatment, the protection of the fibre from cutting during the high intensity LC refining was limited to specific energies below 300 kWh/t.

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