

MODIFICATION OF JACK PINE TMP LONG FIBERS BY ALKALINE PEROXIDE – PART 1. CHEMICAL CHARACTERISTICS OF FIBERS AND SPENT LIQUOR

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The purpose of this work was to improve the quality of jack pine TMP long fibers, particularly with respect to the strength properties, by alkaline peroxide treatment. This paper reports the chemical characteristics of the treated long fibers and the spent liquors originating from various treatments. It was observed that, in comparison with hydrogen peroxide, the alkalinity of the treatment solutions had a greater influence on most fibre characteristics and spent liquor properties.

Keywords: *Pinus banksiana*; Sodium hydroxide; Hydrogen peroxide; Thermomechanical pulp; Carboxyl group; X-ray photoelectron spectroscopy; Cationic demand; Total organic carbon

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INTRODUCTION

The expected shortfall in supply of traditional softwoods (e.g. spruces and balsam fir) forces pulp producers in Quebec (Anon. 2004) to increase the use of less-desirable softwood species such as jack pine (*Pinus banksiana*) and larches (*Larix*) or hardwood species such as white birch (*Betula papyrifera*) and maples (e.g. *Acer saccharum* and *A. rubrum*). Presently, the incorporation rate of jack pine in mechanical pulping is relatively low, at about 20% (Law and Valade 1994). The poor papermaking properties of jack pine mechanical pulp have long been acknowledged (Barbe and Macdonald 1986; Jossart et al. 1988; Barbe et al. 1989a, 1989b; Gagné et al. 1990; Lanouette et al. 1997, 1998; Tyrvaäinen et al. 1997a, 1997b; Law et al. 1998; Miles and Omholt 2007). Relatively high extractives content and high proportion of thick-walled latewood fibers have rendered jack pine mechanical pulp to be undesirable.

Various efforts had been made to use alkaline peroxide as an enhancing solution in thermomechanical pulping or rejects treatment (Sferrazza et al. 1988; Strunk et al. 1988, 1989, 1990; Tyrvaäinen et al. 1997a, 1997b; Lanouette et al. 1997, 1998; Bian et al. 2007). Some improvements in physical and optical properties of handsheets were reported in these works.

Alkaline peroxide treatment on other species has proved to be a good technique to improve both the strength and brightness of mechanical pulp (Allison 1983; Moldenius 1984; Haikkala et al. 1989; Engstrand et al. 1991; Zhang et al. 1994; Korpela 2002; Pan and Yuan 2004; Pan 2004; Konas et al. 2006). Besides the elimination chromophores as a result of hydrogen peroxide treatment, carboxylic acid groups are also introduced on the

fiber surface (e.g. Zhang et al. 1994; Pan and Yuan 2004; Pan 2004). It is well documented that the amounts of carboxylic acid groups has strong influence on inter-fiber bonding (Katz et al.1981; Engstrand et al.1991; Barzyk et al.1997). The increase in fiber bonding potential is also related to the dissolution of wood components from fiber, for example, lignin, hemicelluloses, and extractives (Holmbom et al. 1991; Zhang et al. 1994; Korpela 2002; Pan and Yuan 2004; Pan 2004; Konas et al. 2006). The partial removal of lignin increases fiber flexibility and conformability in sheet consolidation, while the removal of extractives enhances the hydrophilicity of fiber surfaces, augmenting inter-fiber bonding capability.

This report on the use of alkaline peroxide constitutes only part of a larger project aiming at improving the mechanical characteristics of jack pine TMP. It is learned that the long fibers in TMP are of poor physical properties in comparison to the short fiber fragments and fines (Law 2000). The short elements and fines have comparatively much larger specific surface and yield relatively good inter-fiber bonding. Additionally, due to their high specific surface, these fine components would react easily with treatment chemicals when a whole pulp is used. To minimize chemical usage and energy we had chosen to only treat the long fiber fraction. During scale-up of this technology it is assumed that the shorter elements will be reintegrated in the furnish of treated fibers. This paper reports the influence of hydrogen peroxide and sodium hydroxide on the chemical nature of fibers and the spent liquor from alkaline peroxide treatment of jack pine (*Pinus banksiana*) TMP long fibers.

EXPERIMENTAL

Material

The jack pine TMP used in this work was produced using our Metso CD-300 pilot-scale unit. The refining was performed in two stages. The primary stage was carried out at 126 °C with a discharge consistency of about 24 %. The primary pulp, having a freeness of 320 mL, was further refined under atmospheric conditions at about 13% consistency, yielding a final freeness of 225 mL. The second-stage pulp was fractionated using a Bauer McNett classifier to retain the fibers on the 14-, 28-, and 48-mesh screens (R14+R28+R48) and used for chemical treatment. These long fibers represented 58 % by weight of the whole pulp.

Chemical Treatment

The long fibers were treated under two sets of experimental conditions to study the influence of H₂O₂ at constant NaOH (4%) and that of NaOH at a given H₂O₂ charge (2%). In the former, the H₂O₂ charges were 1.5, 1.7, 1.9, and 2.1%, while in the latter the NaOH doses were 1, 2, 3, and 4 %. Other conditions were 0.05% MgSO₄, 3% NaSiO₃, 0.15% DTPA, 75 °C, 120 min, and 15% consistency. These conditions were chosen according to our previous experiences dealing with jack pine pulp. All treatments were conducted in duplicate.

Predetermined amounts of pulp samples were placed in polyethylene bags and heated to the desired temperature before being mixed with the chemical solution. The bags containing the experimental pulps were submerged in a water bath at 75 °C. Inter-

mittent manual kneading was performed during the course of reaction. At the end of the predetermined retention time, the spent liquor was separated from the pulp by filtration, and its pH, TOC, residual H₂O₂, and cationic demands were measured. The treated pulps were acidified to about pH 5 using SO₂, then thoroughly washed with de-ionized water and filtered prior to the characterization of their chemical properties and the formation of handsheets.

Characterization of Pulp

Klason lignin, extractives, and carbohydrate contents were determined according to TAPPI test methods T222om-98, T204, and T249cm-85-liquid chromatography, respectively. An HPLC device, supplied by WatersTM, consisting of three main components: 717 *plus* Autosampler, 600S Controller, and 432 Conductivity Detector, was used for determining the carbohydrate content.

Determination of Carboxylic Acid

Carboxylic acid content of pulp samples was determined using a conductometric titration technique described by Katz et al. (1984). The instrument used was a Metrohm (Brinkmann) titrator equipped with a conductivity meter (Thermo Orion, model 150). The technique includes converting the sample pulp (about 3 g o.d.) to hydrogen form by soaking it twice in 200 mL 0.1 M HCl for 45 min, then washing with de-ionized water to a constant conductance. The filtered sample was suspended in 450 mL 0.001 M NaCl for the titration process, which was carried out with 0.1M sodium hydroxide. The suspension was continuously stirred under a nitrogen atmosphere. The measurements of carboxyl content were repeated at least twice and processed by a computer.

Determination of Cationic Demand

Cationic demand of the spent liquors was determined by polyelectrolyte titration by means of a Mütek PCD 03 charge analyzer. This technique involved the use of a cationic titrant, polydiallyldimethylammonium chloride (P-DADMAC).

Measurement of Total Organic Carbon (TOC)

TOC of spent liquors was estimated using the filtrates from alkaline peroxide treatment stages. The analysis was performed using a Dorhmann TOC DC-190.

X-ray Photoelectron Spectroscopy (XPS) Analysis

Standard handsheets (1.2 g) were prepared with circulation of white water to retain the fines. To minimize any possible contamination, we made three identical sheets. The sheet destined to XPS study was sandwiched between two other sheets. XPS measurements were taken on the smooth side of the middle sheet at 3 to 4 locations. The measured area at each point was 1x1 mm. A Kratos Axis Ultra (HIS 165) spectrometer operating with high energy silver X-rays was used for the analyses. Pass energy of 160 eV was employed with two sweeps. Total acquisition time was 242 s. Data acquisition and processing were performed by a SUN Sparc Station IPX computer (Vision 2).

Thermal and Light-Induced Yellowing

Two sample sheets were prepared using a Büchner funnel according to TAPPI test method T218 sp-97. After the measurement of their initial brightness, one of the sheets was placed in an oven at 105°C, for 3 hours to study the thermal-induced yellowing effect, while the other one was placed under UV lamps of 350 nm wavelength for 10 to 70 min to evaluate the influence of UV-visible light on yellowing. Both thermal and light-induced yellowing can be measured by the decrease in brightness. The yellowing can also be expressed by a post colour (p.c.) number, which is calculated according to the following equation (Heitner 1996). The coefficient k and s were measured at a wavelength of 457 nm.

$$\text{P.C. number} = 100[(k/s) - (k_0/s_0)] \quad (1)$$

where

- k : specific light absorption after aging
- k_0 : specific light absorption before aging
- s : specific light scattering after aging
- s_0 : specific light scattering before aging

RESULTS AND DISCUSSION

Properties of Fibers

Table 1 presents the influence of H_2O_2 at a constant NaOH charge of 4%, while Table 2 shows that of NaOH at a fixed dose of H_2O_2 at 2%.

Table 1 shows that the extractives content in the untreated long fibers (RL) was substantially lower than that for the whole pulp (WP), indicating that most of the extractives were present in the fines. Higher extractives content on mechanical pulp fines has been reported by other workers (Luukko et al. 1999; Rundlof et al. 2000; Kleen et al. 2003). Luukko et al. (1999) suggested that extractives are reprecipitated onto the surface of fines during different process stages. It is noted here that the extractives content of pulps was substantially reduced as a result of alkaline peroxide treatment, from 0.47% down to about 0.10%. After the initial drop, the decreases were rather small as the H_2O_2 or the NaOH increased.

The untreated long fiber had lower lignin content when compared to the whole pulp, 25.2 vs. 28.4 %. It is conceivable that the compound middle lamella, which is rich in lignin, is detached from the secondary cell wall and ends up in the fines during the process of refining. As a result, the long fiber has less lignin. The changes in lignin and carbohydrate contents were not significant, which was attributed to the relatively mild alkaline condition and low temperature (75 °C) used in the experiments.

Alkaline peroxide treatment was effective in generating carboxyl acid on fiber surfaces. Pan (2004) observed a strong direct relationship between alkali charge, the dissolution of pulp components, and the generation of carboxylic acid groups in bleaching of aspen CTMP. For a given charge of NaOH at 4%, the increases in H_2O_2 from 1.5 to 2.1% resulted in little change in carboxylic acid. However, the changes were

much more noticeable when a higher dose of H₂O₂ at 2% was used. This implies that H₂O₂ also plays an important role in the creation of carboxylic acid on fiber.

There were small increases in water retention value (WRV) of fiber as a result of the alkaline peroxide treatment, which is associated with the dissolution of fiber materials and subsequent swelling effect of NaOH. However, the swelling of fiber had no influence on the freeness of pulps studied. In fact, all the freeness values of long fiber were well above the maximum valid value of 700 mL, in accordance with the method of measurement. On the other hand, the freeness of mechanical fiber is known to be mainly dependent on the quantity of fines generated during refining (Law 2005); the fines were absent in the long fiber fraction.

In comparison to the whole pulp, the long fiber had a higher atomic O/C ratio. This is because the latter, which lacked fines, had less lignin and extractives, as discussed earlier. The treatment conditions used resulted in very little increase in the atomic oxygen/carbon (O/C) ratio of the fiber surface. As a result, alkaline peroxide treatment was found not to be effective in increasing the O/C ratio, despite the fact that this treatment noticeably reduced the extractives content, as Table 1 shows.

There was a substantial gain of nearly 10%-points in brightness as a result of alkaline peroxide treatment. An additional gain by 2-6%-points was achieved by increasing the proportion of peroxide or sodium hydroxide in the treatment solution, as shown in Table 1. It is noteworthy that the peroxide consumed in actual removal of chromophores may not be a significant part of the total consumption according to the finding of Xu (2002).

Table 1. Properties of Alkaline Peroxide Treated Jack Pine Long Fibers

Pulps	WP*	RL**	RLA	RLB	RLC	RLD	RLE	RLF	RLG	RLH
H ₂ O ₂ , %			1.5	1.7	1.9	2.1	2	2	2	2
NaOH, %			4	4	4	4	1	2	3	4
Extractives %	3.03	0.47	0.14	0.13	0.12	0.12	0.14	0.13	0.11	0.09
Lignin, %	28.4	25.2	25.0	25.0	24.2	24.8	25.0	24.9	24.8	24.8
Carbohydrate %	78.9	77.9	77.0	76.7	76.5	76.2	77.8	77.0	76.3	75.8
Carboxyl, mmol/kg	86	71	181	182	183	185	173	190	265	268
WRV, %	---	108	122	121	128	126	112	116	118	120
CSF, mL	225	750	750	750	750	750	730	740	740	750
O/C ratio	0.39	0.49	0.50	0.50	0.50	0.51	0.48	0.48	0.49	0.49
Brightness, ISO %	46.9	44.5	55.7	58.1	59.0	59.4	58.3	61.5	61.3	58.1
Thermal yellowing, p.c,		1.00	1.56	1.48	1.35	1.22	0.96	1.10	1.18	1.26

Note: * whole pulp; ** untreated long fibre; results of photoyellowing are presented separately in the form of a figure.

Any lignin-retaining bleaching (reductive bleaching with sodium dithionite or oxidative bleaching with alkaline peroxide) is expected to have a significant effect on the yellowing associated with the thermal aging of lignin-containing mechanical pulps such as CMP and TMP. These bleaching techniques increase the extent of yellowing during aging in the dark (Janson and Lorås 1968; Abadie-Maumert and Lorås 1977; Lee

et al. 1989). Our work on alkaline peroxide treatment increased the thermal-induced yellowing of pulps when compared to the untreated counterpart (Table 1). However, the p.c. number decreased gradually with increasing H_2O_2 at a constant charge of 4% NaOH. Interestingly, at a higher dose of H_2O_2 (2%) a slightly smaller p.c. number of 0.96 was obtained at 1% NaOH. But it increased slightly with increasing NaOH addition.

The responses of untreated and treated long fibers to exposure of UV light are presented in Figs. 1 and 2, which show that all the alkaline peroxide treated samples exhibited similar trends in brightness reduction as a function of exposure time.

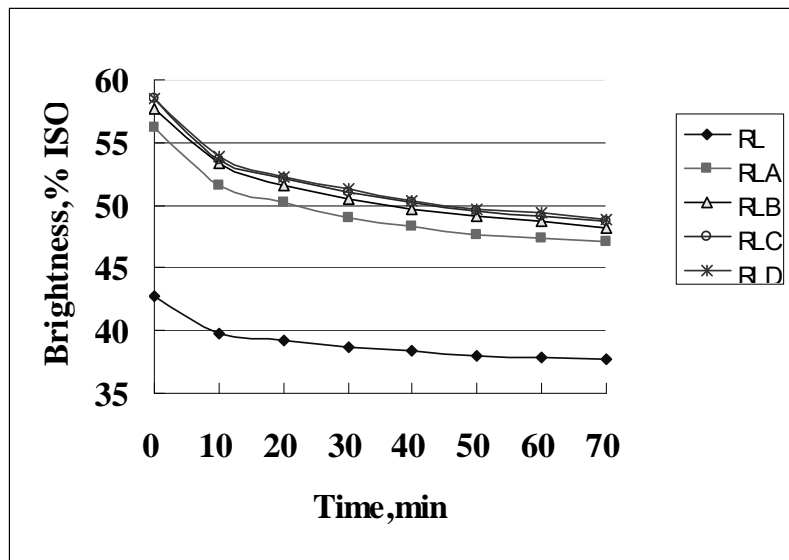


Fig. 1. Effect of exposure time on brightness (4% NaOH), RL: untreated, RLA: 1.5% H_2O_2 , RLB: 1.7% H_2O_2 , RLC: 1.9% H_2O_2 and RLD: 2.1% H_2O_2

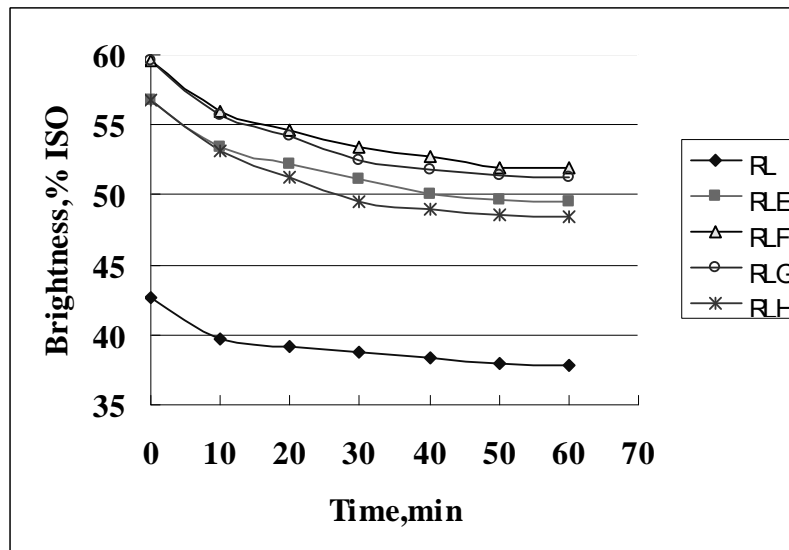


Fig. 2. Effect of exposure time on brightness (2% H_2O_2), RL: untreated, RLE: 1% NaOH, RLF: 2% NaOH, RLG: 3% NaOH and RLH: 4% NaOH

The most significant drop was observed in the first 10 min of exposure, after which the decreases in brightness gradually leveled off. Further, the reduction rate in brightness for the treated fiber was similar to that for the untreated counterpart, which might be due to the sheet thickness. As shown by Schmidt and Heitner (1993), during UV light exposure the formation of chromophores was greatest at and near the sheet surface and decreased with increasing depth. In general there was no significant difference between the treated fibers in terms of brightness loss; the magnitude of difference in brightness between them was dependent on the treatment conditions.

Characteristics of Spent Liquor

Albeit alkaline peroxide treatment is a lignin-retaining bleaching process, dissolution of some pulp materials is unavoidable ((Holmbom et al. 1991; Zhang et al. 1994; Korpela 2002; Pan and Yuan 2004; Pan 2004; Konas et al. 2006). Characterizing the spent liquor provides us with useful information regarding the impact of the treatment conditions on pulp yield, for example (Pan 2003).

Spent liquor at 4% NaOH

Since the charges of NaOH and Na₂SiO₃ were kept constant, the initial pH remained stable at 12.8 with the addition of peroxide ranging from 1.5 to 2.1%. However, the pH dropped to about 10.6 at the end of 120 min reaction time due to the formation of perhydroxyl groups.

The residual peroxide increased from 10.1 to 18.6% when the peroxide charge rose from 1.5 to 2.1%. Higher charge of peroxide gave better brightness, as Table 2 reveals.

The total organic carbon (TOC) increased with increasing peroxide charge, which is the result of a slight drop in pulp yield. The TOC only measures the total amount of dissolved organic substances but it does not differentiate between the dissolved components (e.g. lignin, hemicelluloses and extractives). The yield loss was 1.1% when the peroxide charge increased from 1.5 to 2.1%. The cationic demand remained, however, relatively stable at about 6.17×10^{-3} eq/L. Pan and Yuan (2004) noted that TOC has a first order linear relationship with pulp yield and the cationic demand has a first order linear relationship with pulp carboxylic group in peroxide bleaching of aspen CTMP. However, they cautioned that these interrelationships could be wood species-dependent.

Table 2. Properties of Spent Liquor at 4% NaOH

Pulp	H ₂ O ₂ %	NaOH %	Initial pH	End pH	Residual H ₂ O ₂ %	TOC mg/L	Cationic Demand eq/Lx 10 ⁻³	Yield %
RLA	1.5	4	12.8	10.7	10.1	3373	6.19	98.8
RLB	1.7	4	12.8	10.6	13.7	3426	6.17	98.6
RLC	1.9	4	12.8	10.5	16.5	3503	6.15	98.0
RLD	2.1	4	12.7	10.5	18.6	3536	6.15	97.7

Other conditions: 0.05% MgSO₄, 3% NaSiO₃, 0.15% DTPA, 75 °C, 120 min, 15% consistency

Spent liquor at constant 2% H₂O₂

At constant charges of peroxide (2%) and sodium silicate (3%) the initial pH of the spent liquor increased gradually from 11.2 to 12.6 as the dosage of NaOH was raised from 1 to 4%. As a result, the end pH rose from a near-neutral 7.8 to 10.3, Table 3 shows.

As a consequence of the decrease in H₂O₂/NaOH ratio or an increase in alkalinity the amount of residual peroxide fell from 55.8 to 16.2%. Meanwhile, the TOC increased considerably from 1360 to 3657 mg/L, and the cationic demand moved up significantly from 1.48 to 6.16 x10⁻³ eq/L. These observations are associated with the drop in pulp yield from 99.9 to 97.5% (2.4% loss) as the NaOH was increased from 1 to 4%.

Table 3. Properties of Spent Liquor at 2% H₂O₂

Pulp	NaOH	H ₂ O ₂	Initial	End	Residual	TOC	Cationic Demand	Yield
	%	%	pH	pH	H ₂ O ₂ ,%	mg/L	eq/Lx 10 ⁻³	%
RLE	1	2	11.2	7.8	55.8	1360	1.48	99.9
RLF	2	2	11.8	8.7	42.4	2178	2.54	98.6
RLG	3	2	12.3	9.8	28.8	2908	4.39	98.1
RLH	4	2	12.6	10.3	16.2	3657	6.16	97.5

Other conditions: 0.05% MgSO₄, 3% NaSiO₃, 0.15% DTPA, 75 °C, 120 min, 15% consistency

CONCLUSIONS

This study on alkaline peroxide treatment of jack pine long fiber reveals that:

1. Alkaline peroxide (AP) treatment substantially reduces the extractives content of fiber.
2. Lignin and carbohydrate suffer relatively small dissolution in AP treatment within the limit of this investigation.
3. Sodium hydroxide is more effective than hydrogen peroxide in generating carboxylic acid.
4. AP treatment increases slightly the water retention value (WRV) of pulp.
5. AP treatment increases pulp brightness and the thermal yellowing effect, but it has no significant influence on light-induced yellowing, probably due to sample thickness.
6. The TOC and cationic demand of spent liquors increase with increasing alkalinity, whilst the pulp yield falls correspondingly.

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