Hydrogen Peroxide Bleaching of Canola Straw Chemimechanical Pulp

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Canola straw can be used as a raw material for the production of chemimechanical pulp (CMP). In this work, chemimechanical pulp was first produced from canola straw, and then the bleaching performance of the produced chemimechanical pulp was investigated under different conditions (dosages of NaOH and Na_2SO_3, time, and temperature). The results showed that the yield of chemical pretreatment in the CMP process varied between 57.6% and 64.9%, while the total yield of the process was between 54.1% and 61.9%. Subsequently, the CMP pulp produced via applying 4% NaOH and 8% Na_2SO_3 at 145 °C for 15 min was bleached with H_2O_2. The results showed that pulps bleached via applying one stage of 3% H_2O_2, 3% NaOH, 6% sodium silicate, 0.5% $MgSO_4$, and 0.3% DTPA for 150 min possessed the highest brightness of 60.8% ISO, which is suitable for container board production.

Keywords: Canola straw; CMP; Yield; DTPA; H_2O_2; Bleaching

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INTRODUCTION

The global production of paper products has been continuously increasing, but forest resources as the main supplies for such productions are diminishing. In this respect, the need for preventing fast deforestation in developing regions has prevailed, and environmental regulations have been intensified. As a result, the paper industry is seeking alternative resources. Since the 1970s, non-wood pulping capacities have increased two to three times as fast as wood pulping capacity worldwide (Hedjazi *et al.* 2009). Non-wood fiber organisms grow faster, and their availability is significant in many regions (Shatalav and Pererio 2006). Non-woods have been used as the main raw material for paper productions in many countries including China and India.

Soda pulping is a well-established process with potential to process a vast variety of annual plants, but this process has some disadvantages such as a low yield and difficulty in chemical recovery (Enayeti *et al.* 2009). Mechanical pulping was developed to produce pulp with a higher yield (*i.e*. more efficient use of raw material). However, the strength properties of pulp produced via mechanical pulping are generally inferior to those of pulp produced via soda pulping. Chemimechanical pulping (CMP) was introduced as an option to produce pulp with a relatively high yield and strength (Sian *et al.* 2002; Hosseinpour *et al.* 2010a).

Currently, there are many chemical straw pulping processes that have been implemented at industrial scale. Typically, chemical pulping consumes large quantities of raw materials due to a low yield (typically less than 50%). Therefore, the raw material efficiency is limited and only high capacity installations and capital intensive plants can be erected (Roncero *et al.* 2003; Rousu *et al.* 2002). The chemical pulping of straw requires a high temperature (*e.g.* 175 °C) and a pressurized process (Enayati *et al.* 2009). Alternatively, canola straw chemimechanical pulping has shown promising results (Enayeti *et al.* 2009; Hosseinpour *et al.* 2010a). This work is the continuation of the previous work on the chemimechanical pulping of canola straw with the focus on improving the properties of canola CMP pulp.

Totally chlorine free (TCF) bleaching has mostly been utilized for wood and non-wood based chemical and mechanical pulps (Abrantes *et al.* 2007; Hedjazi *et al.* 2008; Myers and Bagby 1995; Mustajoki *et al.* 2010; Potucek and Wilichovsky 2002; Roncero *et al.* 2003; Shatalov and Pererio 2006; Suss 1996; Tutus 2004; Xu 2001). Hydrogen peroxide bleaching has been commercially applied in wood-based chemi-mechanical pulping processes (Bajpaei *et al.* 2006; Sarwar Jahan *et al.* 2010), and is also used to bleach wood pulp produced via the thermomechanical pulping process. Furthermore, the bleaching of CMP pulp of wheat straw with hydrogen peroxide in a P-P-Paa-P bleaching sequence (Mustajoki *et al.* 2010; Zhao *et al.* 2004), and of cotton (Ali *et al.* 2002) and jute (Xu 2001), both in an alkaline peroxide mechanical pulping (APMP) bleaching sequence, were evaluated. In this work, the hydrogen peroxide bleaching of canola pulp produced in a chemimechanical pulping (CMP) process was investigated for the first time. The main aim of this work was to produce semibleached CMP pulp from canola straw. Such a pulp has potential applications in container and hygienic papers. Usually, the TCF bleaching sequence is comprised of different stages, and it requires expensive equipment and extensive investments. In order to simplify the process, the present work considered one-stage bleaching for canola CMP pulping in order to help implement the proposed process in a small-scale operation.

It is well known that metals present in pulp decompose hydrogen peroxide and hamper the performance of hydrogen peroxide bleaching. Diethylenetriaminepentaacetic acid (DTPA) and ethylenediaminetetraacetic acid (EDTA) are frequently added prior to hydrogen peroxide treatment to chelate the metal compounds (Yun and He 2013). Alternatively, sodium silicate was used to preserve hydrogen peroxide from decomposition. Another objective of this study was to determine the impact of DTPA and Na_2SiO_3 on the performance of hydrogen peroxide in canola CMP pulp.

EXPERIMENTAL

Materials

Canola straw was received in bales from Roudbar canola field, which is located in northern Iran. The leaves of straw were separated, and the straw was cleaned in the pulping laboratory of Islamic Azad University, Karaj Branch. Subsequently, the cleaned straw was manually depithed and chopped into 2 to 4 cm length. The chopped straw was dried at ambient temperature, and then was stored in plastic bags until used. All chemicals used in this study were purchased from Merck chemical company, Darmstadt, Germany.

Pulping

The experimental conditions for chemimechanical pulping (CMP) of canola straw to reach optimum pulping were set as follows: In each trial, one of the two chemical charges of 4% NaOH and 8% Na_2SO_3 or 8% NaOH and 12% Na_2SO_3 (oven dry weight based on straw) was added to the dried straw, and the liquid to straw ratio was maintained at 7. The treatment was conducted at 145 °C for 15, 30, or 45 min after reaching the pulping temperature.

All chemical treatments were performed in a 4 L rotating digester (Ghomes Wood and Paper Equipment Manufacturing Co., Iran) using 100 g (od) canola straw. At the end of each treatment, the cooked materials were discharged on a 200-mesh screen and then washed using hot water. In each trial, three repetitions were conducted, and the average values were reported. The cooked pulp was then disintegrated using a 25 cm laboratory single disc refiner (Ghomes Wood and Paper Equipment Manufacturing Co., Iran) with a power of 10 kW at 1450 revolutions, and after three passes the CSF freeness reached 300 mL. The consistency of the refining was constant at 15%. Then, the pulp was screened using a set of two screens (14-mesh screen on top of 200-mesh screens). Pulp fibers retained on 14-mesh screen were considered as reject (shives), and the fibers passed through 14-mesh screen and retained on 200-mesh screen were considered as accepted fibers (screened yield).

Handsheets were produced from these fibers and their strength properties were measured following TAPPI's relevant methods.

Bleaching

The CMP pulp produced via pretreating straw with 4% NaOH and 8% Na_2SO_3 for 15 min (pulping trail No. 1) was selected for bleaching experiments via applying hydrogen peroxide. First, the pulp consistency and pH were adjusted at 3% and 5, respectively, and then the pulp samples were chelated by either 0.3 wt.% diethylene-triaminepentaacetic acid (DTPA) or ethylenediaminetetraacetic acid (EDTA) for 30 min at 70 °C in polyethylene plastic bags. After chelation, the pulps were thoroughly washed with deionized water and dewatered by pressing manually. The metal content of raw material (straw), and pulp before and after chelating with either DTPA or EDTA was determined using TAPPI method T266. The bleaching was performed in three steps: 1) the impacts of hydrogen peroxide and NaOH on unchelated CMP pulp were evaluated using four levels of hydrogen peroxide (1, 2, 3, and 4 wt.%) and four levels of sodium hydroxide (1, 2, 3, 4 wt.%). These experiments were conducted using 3 wt.% sodium silicate and 0.5 wt.% $MgSO_4$ at 70 °C for 120 min; 2) the impact of DTPA and sodium silicate dosages was examined via applying two levels of DTPA (0.3 and 0.5 wt.%) and four levels of sodium silicate (0, 3, 6, and 9 wt.% based on unbleached pulp) prior to bleaching with 3 wt.% H_2O_2 and 3% NaOH (*i.e.* optimized dosage of H_2O_2 and NaOH); based on the results, 0.3% DTPA and 6% sodium silicate were selected as optimal chelation conditions for bleaching; 3) canola CMP pulp was bleached using chelated pulp (conducted with 0.3% DTPA, 6% sodium silicate and 0.5% $MgSO_4$) and bleached with 3 wt.% H_2O_2 and 3 wt.% NaOH for various time intervals (90, 120, and 150 min) at 70 °C.

Bleaching experiments were conducted in polyethylene bags and a hot water bath. During the bleaching trials, the samples were hand kneaded. At the end of each bleaching trial, the spent liquor of bleaching trails was separated from the pulp, and its residual chemicals (H_2O_2 and NaOH) were determined using the procedure provided by Strunk (1987). After bleaching, the pulp was thoroughly washed with tap water. Finally the pulp was dewatered and handsheets were prepared for further testing. Bleaching trails were conducted in three replicates.

The following TAPPI test methods were used for analysis in this research: straw powder preparation with T257; ash in straw and pulp with T211; extractive free powder, with T264; lignin in straw and pulp with T225; holocellulose with T9, pentosans with T223; pulp viscosity with T230; beating with T248; freeness with T227; handsheet preparation with T205; brightness with T452; opacity with T425; tear strength with T414; tensile strength with T494; burst strength with T403; and metal elements with T266. ASTM D1103 test method was used for alpha-cellulose measurements, and Kurschner-Hoffer method was used for cellulose analysis (Hosseinpour *et al.* 2010b).

RESULTS AND DISCUSSION

Pulping

To optimize the conditions of canola straw CMP pulping, different chemical charges and pulping times were selected, and the results of this analysis are listed in Table 1. As can be seen, the total yield after defibration varied between 57.6% (P6) and 64.9% (P1). This yield is marginally lower than that of wheat straw chemimechanical pulp reported earlier (Petit-Conil *et al.* 2001). The holocellulose content of straw was 77.1%, which included 17% pentoses. The chemical compositions and viscosity of the CMP pulps produced by applying different chemical treatments are summarized in Table 1. Generally, under milder conditions (*i.e.* lower chemical charges and shorter treatment times), more cellulose than lignin was removed from straw. By increasing the chemical charges or time of treatment, the lignin removal was slightly increased, while cellulose removal dropped. The viscosity results also showed that, by increasing the dosages of chemicals and/or extending the pretreatment time, the viscosity of pulp was generally reduced. Furthermore, it is apparent in Table 1 that the screened yield of the CMP process was lower than the total yield, as a part of fiber was lost in the mechanical disintegration of fibers. However, the actual defibration yield in continuous pulping operation is usually a few percent higher than laboratory pulping due to process water circulation and consequent saving of fines.

Table 1. Chemical Composition of Canola Straw and the Produced Chemi-mechanical Pulp

Pulping trail no.	NaOH (%)	Na_2SO_3 (%)	Time (min)	Yield[2] (%)	Yield[1] (%)	α-Cellulose (%)	Lignin (%)	Viscosity (mPa.s)	Ash (%)
Canola Straw	-	-	-	-	-	39.6	20	N/A	6.11
P1	4	8	15	60.7	64.9	34.2	19.8	2.94	3
P2	4	8	30	61.9	64	35.7	19.3	2.61	3
P3	4	8	45	54.8	58.6	36	19	2.57	2
P4	8	12	15	60.7	63.8	35	19.6	2.84	3
P5	8	12	30	54.7	58.0	35.2	19.1	2.63	2
P6	8	12	45	54.1	57.6	36	18.9	2.53	2

[1]chemical pretreatment yield; [2]overall yield

The results of pulp optical and strength measurements are given in Table 2. The data in Table 2 show that the brightness of CMP pulp produced in this research was generally higher than that reported for other chemical and mechanical straw pulps.

Table 2. Results of Canola Straw CMP Pulping and the Strength Properties of the Produced Pulps

Pulping trail no.	NaOH (%)	Na_2SO_3 (%)	Time (min.)	Yield[2] (%)	Yield[1] (%)	Tensile index (N.m/g)	Burst index (kPa.m^2/g)	Tear strength index (mN.m^2/g)	Brightness (%ISO)	Opacity (%ISO)	Reference
P1	4	8	15	60.7	64.9	27.3	1.5	4.3	40.0	99.0	PS[3]
P2	4	8	30	61.9	64	28.3	1.5	3.8	33.0	98.2	PS
P3	4	8	45	54.8	58.6	35.2	1.7	4.7	32.0	99.8	PS
P4	8	12	15	60.7	63.8	28.3	1.5	4.3	35.7	99.0	PS
P5	8	12	30	54.7	58.0	35.0	1.4	4.8	34.0	99.0	PS
P6	8	12	45	54.1	57.6	38.4	2.0	4.9	31.0	99.1	PS
Soda/AQ	16			48.1		84			24.9		Jahan and Hadjazi 2006
Soda	16			58.4		86			25		Hadjazi *et al.* 2009
Kraft/AQ	16			44.7		89.2		7.74			Ates *et al*. 2008
Allcel (50% ethanol)				47.4		53.2		5.97			Ates *et al.* 2008
CMP	6.6			66		56.10[4]		3.5			Petit-Conil *et al.* 2001

[1] chemical pretreatment yield; [2] overall yield; [3]Present work; [4]breaking length (m)

The strength properties of the canola CMP pulps were evaluated and are included in Table 2. Generally, the tensile strength of canola pulp was marginally lower than that of chemical and CMP pulps produced from wheat straw (Ates *et al.* 2008; Hedjazi *et al.* 2008; Jahan Latibari and Hedjazi 2006; Petit-Conil *et al.* 2001).

The results indicated that, by increasing the dosage of chemicals in the pretreatment stage, the chemical treatment and overall yields were reduced to some extent, which can be attributed to the dissolution of lignocelluloses and softening of straw under stronger chemical treatment conditions (Fatehi *et al.* 2011), but the strength properties and opacity of papers were not significantly changed. However, brightness was somehow reduced by increasing the dosage of chemicals in the pretreatment stage, which can be ascribed to lignin condensation. This phenomenon has been observed in the chemical treatment of non-wood species (including canola) in the past (He and Ni 2008; Yun and He 2013). Also, by extending the time of the pretreatment stage, the chemical pretreatment and overall yields of the pulp were reduced, while strength properties were improved. As is well known, by extending the time of chemical pretreatment, more lignocelluloses will generally be removed from straw, which will reduce the pulping yield and improve the paper strength (Hosseinpour *et al.* 2010a). However, the brightness of paper was reduced, as lignin condensation would occur at a prolonged pretreatment time. A similar behavior was observed in another study on straw (He and Ni 2008; Yun and He 2013). Therefore, with consideration of the pulping yield and pulp strength (Table 1), pulp produced via 4% NaOH and 8%Na_2SO_3 for 15 min pretreatment time generated the best CMP pulp, and therefore was selected for TCF bleaching analysis.

DTPA vs EDTA Chelation

Table 2 lists the metal content of unbleached canola CMP before and after chelation with DTPA or EDTA. As can be seen, canola CMP pulp had more Ca and Fe than Na, Cu, and Mn. Also, DTPA treatment resulted in chelated pulp with lower metal content than EDTA did. Therefore, DTPA was selected as the chelating agent for further analysis.

Table 3. Elemental Analysis (in ppm) of Pulp Chelated with DTPA and EDTA

Pulp sample	Na	Ca	Cu	Fe	Mn
Canola straw	18.35	74.33	0.06	0.71	0.92
Unchelated	16.30	92.20	3.81	86.56	5.85
DTPA chelated	7.420	75.45	0.30	30.22	0.22
EDTA chelated	8.94	78.96	0.40	40.17	0.34

Bleaching

In this study, one stage hydrogen peroxide bleaching was applied to produce CMP pulp. At first, the dosage of hydrogen peroxide and sodium peroxide was optimized. Figures 1 and 2 depict the effect of H_2O_2 and NaOH dosages on the brightness and opacity of the CMP pulp (without chelation treatment), respectively. As was expected, the higher dosages of hydrogen peroxide improved the brightness of the bleached pulps, and the highest brightness was reached using 3% hydrogen peroxide and 3% sodium hydroxide; thus, these were selected as optimal dosages. The increase in the dosage of NaOH to 4% or H_2O_2 to 4% tended to reduce the pulp brightness, which is due to the alkali darkening of the pulp. This phenomenon has been observed in previous studies of the hydrogen peroxide bleaching of mechanical pulp (He and Ni 2008; Yun and He 2013).

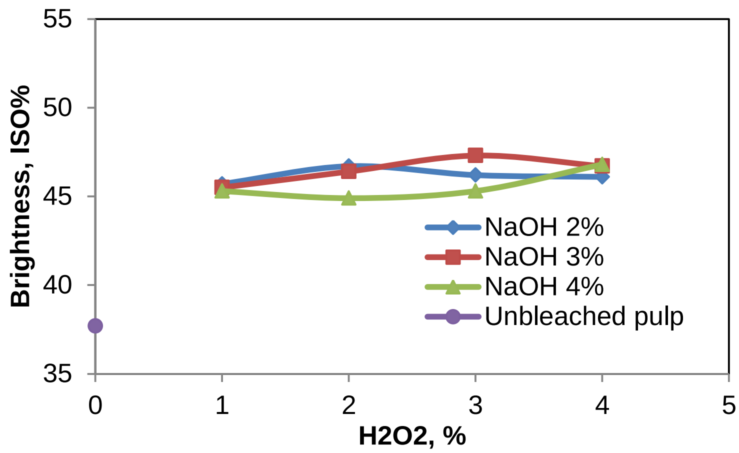

Fig. 1. Impact of H_2O_2 and NaOH charges on brightness of CMP pulp

As can be seen in Fig. 2, the opacity of pulp was generally reduced by treating with H_2O_2 and NaOH. As pulp was bleached in the hydrogen peroxide stage, its lignin was reduced and thus fibers became softer, which resulted in papers with a lower opacity. A part of the opacity reduction may be due to the removal of chromophores from cellulose fibers as a result of hydrogen peroxide bleaching.

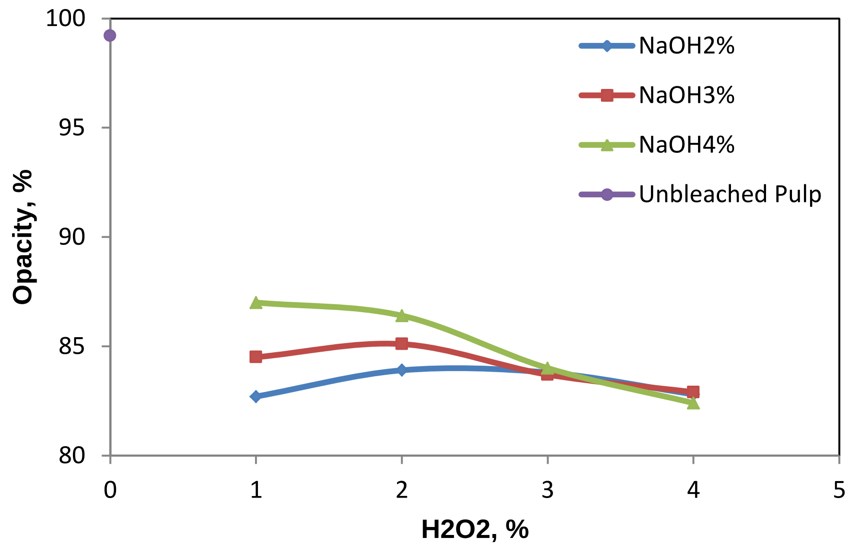

Fig. 2. Impact of H_2O_2 and NaOH dosages on the opacity of canola CMP pulp

Impact of DTPA

Figure 3 shows the impact of DTPA and sodium silicate on the performance of hydrogen peroxide treatment. In this set of experiments, pulps were treated with various dosages of DTPA and sodium silicate, and then bleached with 3% hydrogen peroxide and 3% sodium hydroxide. The results in Fig. 3 indicate that the treatment with 0.3% DTPA and 6% sodium silicate generated the highest brightness of 51%ISO. Higher levels of DTPA or sodium silicate did not improve the brightness of pulp. The improvement in brightness of pulp as a result of DTPA chelation prior to peroxide treatment was reported in the past (Colodette *et al.* 1989; Ghasemi *et al.* 2010; Kangas *et al.* 2002; Ni *et al.* 2000; Prasakis *et al.* 1996; Wekesa and Ni 2003).

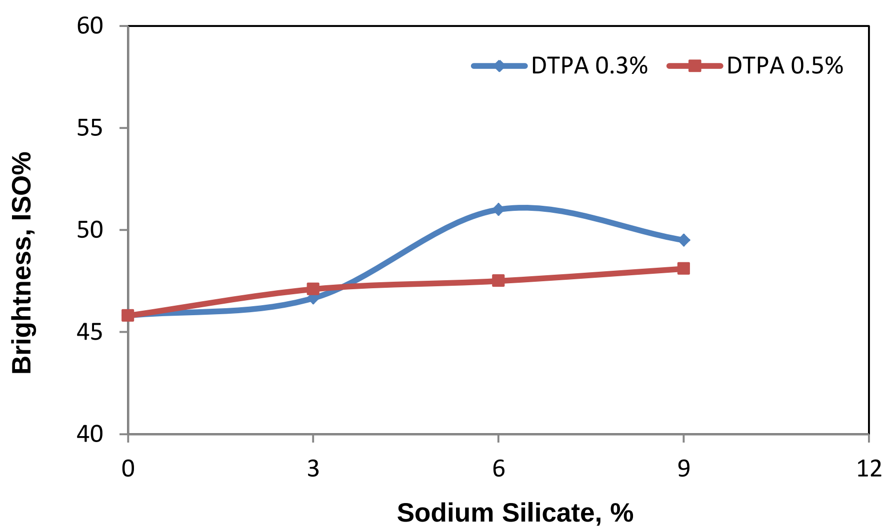

Fig. 3. Impact of sodium silicate and DTPA on brightness of pulp bleached with 3% H_2O_2 and 3% NaOH

Figure 4 shows the opacity of pulp pretreated with DTPA and sodium silicate prior to bleaching with 3% hydrogen peroxide and 3% sodium hydroxide. Evidently, the opacity of paper was insignificantly affected by the sodium silicate and H_2O_2, but when the dosages of DTPA and sodium silicate were 0.3 % and 6%, respectively, the opacity of pulp was 83%. The results in Figs. 3 and 4 indicate that when the dosages of DTPA and sodium silicate were 0.3 % and 6%, respectively, the peroxide bleaching generated the highest brightness and acceptable opacity. Thus, those conditions were selected for bleaching of canola CMP pulp.

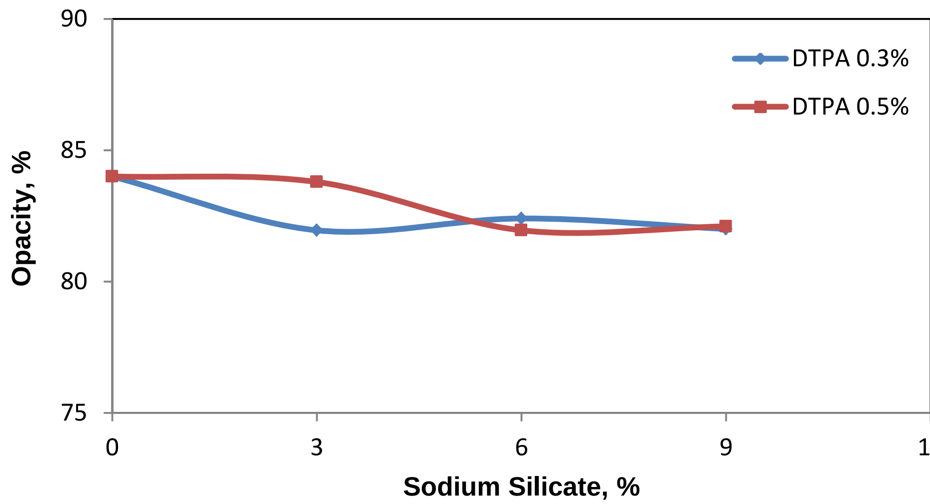

Fig. 4. Effect of sodium silicate and DTPA on the opacity of pulp bleached with 3% H_2O_2 and 3% NaOH for 30 min at 70 °C

Table 4 lists the results of one-stage hydrogen peroxide bleaching trials after pretreating with 0.3% DTPA and 6% sodium silicate. It is apparent that, by increasing the time of treatment, the brightness of pulp was significantly improved, while the opacity slightly dropped. The highest brightness (60.8%ISO) was obtained when the treatment time was 150 min. The results also showed that about 10 to 15% of H_2O_2 and 30% of NaOH remained in the solution after bleaching.

Table 4. H_2O_2 Treatment of Canola CMP Pulp Pretreated with 0.3% of DTPA and 6% of Sodium Silicate under Different H_2O_2 Dosages and Time

Bleaching trail no.	H_2O_2 (%)	Bleaching Time (min)	Residual NaOH (%)	Residual H_2O_2 (%)	Brightness (%)	Opacity (%)	Viscosity (mPa.s)	Lignin (%)	α-Cellulose (%)	Tensile index (N.m/g)	Tear index (mN.m^2/g)
B1	3	90	1.0	0.4	49.3	83.0	3.02	19.74	34.26	27.33	4.01
B2	3	120	1.1	0.5	51.4	82.1	2.77	19.52	34.19	27.14	3.84
B3	3	150	1.3	0.5	60.8	81.1	2.69	19.46	34.13	27.01	3.79

The previous reports stated that 80%ISO brightness was difficult to be achieved by mechanical and chemimechanical pulping of nonwood species (Pan and Leary 2000;

Xu 2001). Zhao *et al*. (2004) produced APMP pulp via enzyme treatment of wheat straw by applying 8% NaOH and 4% H_2O_2, achieving 49.7%ISO brightness. Further TCF bleaching of the APMP pulp with an additional 4% H_2O_2 on pulp an ISO brightness of 70.5% ISO could be reached. These results indicated that one-stage bleaching of canola CMP can generate pulp with a moderate brightness, which is sufficient for container-board production (Hosseinpour *et al.* 2010a; Fatehi *et al.* 2011). Otherwise, another step of bleaching is required to increase the brightness of the pulp.

CONCLUSIONS

1. CMP pulps could be produced from canola straw via pretreating the canola straw with 4% NaOH and 8% Na_2SO_3 at 145 °C for 15 min. This combination resulted in pulp with an overall yield of 64.9%.
2. DTPA was more effective than EDTA in chelating and effectively removing the metal content of canola CMP pulp.
3. Also, 0.3% DTPA and 6% sodium silicate were optimal dosages for pretreating the canola CMP pulp prior to H_2O_2 treatment.
4. Bleaching of the pretreated CMP pulp with 3% H_2O_2 and 3% NaOH for 150 min resulted in 60.79% ISO brightness and 81.10% opacity.

ACKNOWLEDGMENTS

The authors would like to thank Islamic Azad University, Karaj Branch, and NSERC Canada for supporting this research.

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Article submitted: November 8, 2013; Peer review completed: January 4, 2014; Revised version received and accepted: January 12, 2014; Published: January 14, 2014.