

EVALUATION OF PRE-HYDROLYZED SODA-AQ DISSOLVING PULP FROM *POPULUS DELTOIDES* USING AN ODED BLEACHING SEQUENCE

Mostafa Nikkhah Dafchahi ^{*a} and Hossein Resalati ^b

In the present work, the production of dissolving pulp from fast growing 30-year-old local plantation aspen was investigated. The mill-made chips of aspen (*Populus deltoides*) after being pre-hydrolyzed with water at 170 °C, were pulped with a soda-AQ pulping process at a kappa number of 15. It was further delignified to a kappa number of 9.2 using a single stage oxygen delignification and bleached with a D₀ED₁ bleaching sequence at different kappa factor. The pulp viscosity was increased at higher brightness and similar α -cellulose when the kappa factor was increased from 0.30 to 0.45 in the D₀ stage of the D₀ED₁ bleaching sequence, due to higher lignin dissolution and more fiber purification. The results showed that pre-hydrolyzed soda-AQ dissolving pulp can be made from *Populus deltoides* at a high level of alpha-cellulose content and acceptable levels of brightness and viscosity by means of proper control and optimization of pre-hydrolysis, pulping, and oxygen pretreatment conditions, and an optimized DED bleaching sequence.

Key words: α -cellulose; Pre-hydrolysis; Soda-AQ; Viscosity; *Populus deltoides*; Dissolving pulp

Contact information: a: Corresponding author, M.S. Student of Pulp and Paper Industries, Gorgan University of Agricultural Sciences and Natural Resources, Iran; b: Prof. Dept. of Pulp and Paper Sciences and Technology, Gorgan University of Agricultural Sciences and Natural Resources, Gorgan, Iran; *Corresponding author: Nikkhah.Mostafa@Yahoo.Com

INTRODUCTION

Dissolving pulps are high quality and purity pulps with high α -cellulose content, high brightness, low hemicelluloses, and low lignin that are used as raw materials for producing various cellulosic derivatives such as: cellophane, rayon, acetates, nitrates, and carboxymethylcellulose (Sjöström 1981). Therefore, all of the wood components except cellulose, such as hemicelluloses, lignin, extractives, and ash are regarded as trouble-makers, and efforts are made to remove them from the production processes. Dissolving pulps should have distinct characteristics such as high purity, a uniform molecular weight distribution, suitable reactivity, and accessibility of the cellulose to chemicals (Krassig 1993). Kraft and soda processes, by employing a pre-hydrolysis stage before pulping, can be used for the production of dissolving pulp. A pre-hydrolysis stage can be used to degrade and remove a large number of hemicelluloses from wood (Patt *et al.* 1994). However, the efficiency of pre-hydrolysis for hardwoods is higher than softwoods (Gause 1994). The liquor of a pre-hydrolysis stage is a source of sugars and could be used in producing chemicals such as ethanol (Niu 2003).

The acid sulfite and pre-hydrolysis kraft processes are the most globally used methods for dissolving pulp production from wood (Hinck *et al.* 1985; Biermann 1993; Sixta and Borgards 1999). Innovative processes for dissolving pulp production such as pre-hydrolysis-alkaline sulfite, ethanol-water, soda-ethylenediamine, and formic acid processes have also been investigated (Kircl and Akgul 2002; Kordsachia *et al.* 2004 and Sarwar Jahan *et al.* 2008). The pre-hydrolysis-soda/AQ process, due to the absence of sulfur, produces less environmental pollution in comparison to the pre-hydrolysis-kraft process. The soda-AQ process with a pre-hydrolysis stage could be used for dissolving pulp production from hardwoods (Kordsachia *et al.* 2004).

The quality level of dissolving pulp is important for some products such as viscose, carboxymethyl cellulose, and sausage skin. In such applications, the quality is related to both raw materials and the production process (Sarwar Jahan *et al.* 2008). Therefore, if high quality dissolving pulp is needed, cotton linter should be used, since it leads to a dissolving pulp with higher α -cellulose and quality than wood-derived pulps. The ability of chlorine dioxide as a bleaching agent in achieving higher levels of brightness, brightness stability, and lower viscosity loss, have been the main reasons for its employment for this study. Chlorine dioxide more selectively attacks the phenolic groups of lignin, leading to degradation of the lignin molecules and consequently preserving the quality of the pulp (Szilard 1973 and Svenson *et al.* 2006). By contrast, the use of elemental chlorine as a bleaching agent results in the production of toxic compounds such as AOX, dioxins, and furans in the effluent (Brunner and Pulliam 1993; Mishra *et al.* 2001), which can be reduced or eliminated by employment of chlorine dioxide in place of elemental chlorine (Huggett 1994).

Oxygen delignification is a modern and environmentally friendly process that usually is used before the ECF and TCF bleaching sequences for continuance of delignification and it reduces the lignin of pulp by about 35 to 55% prior to the bleaching stage (Samuelson 1994). Magnesium is one of the additives that is used in oxygen delignification to increase the selectivity of delignification due to an ion exchange in the magnesium hydroxide precipitate formed under alkaline conditions (Liden and Ohman 1997). Elevated temperatures and alkali charge have an inverse effect on selectivity of oxygen delignification, particularly when the process is carried out to lower kappa numbers (McDonough 1986; Irabarne and Schroeder 1997). Selectivity of oxygen delignification can be increased by the removal of metals (Sjogren and Hook 2000). Oxygen pre-treatment prior to bleaching sequence causes a reduction in bleaching chemicals, as well as a reduction in environmental pollutants such as AOX, BOD, COD, and color in bleaching effluent (Parthasarathy 1992), due to its ability to reduce the kappa number. The advantage of oxygen delignification is the fact that effluents of the oxygen stage, in combination with weak black liquor, can be sent to the kraft recovery system for chemical and energy recovery. Also, there is a lower cost for oxygen delignification in comparison to other bleaching chemicals such as chlorine dioxide and hydrogen peroxide (Dence and Reeve 1996). Employing fast-growing non-forest wood species in pulp production is very critical for the countries with low natural forest land such as Iran. *Populus deltoides* is a fast growing non-forest species that is very adaptable to the climate conditions of Iran (especially in humid and semi-arid regions). This species was used in this investigation for production of high quality dissolving pulp using environmentally

friendly processes, such as soda-AQ pulping, oxygen delignification, and an ECF bleaching sequence. Since the selectivity of ECF bleaching is usually higher than TCF and, as a result, the DP and viscosity of ECF bleached dissolving pulp is higher than TCF pulp, the ECF bleaching sequence was preferred to TCF for the present work.

EXPERIMENTAL

Materials

Five selected 30-year-old plantation trees of *Populus deltoides* were cut and, for more final pulp purification, were debarked by hand in order to avoid mixing them with other species in an industrial debarker. The debarked logs were converted to chips in the MWPI (Mazandaran pulp and paper mill in Iran) clean chipper with average sizes of length: 3 to 5 cm; width: 2.5 to 3 cm; and thickness: 0.7 to 1cm. The chips were screened in the wood preparation plant at MWPI, air dried, and their moisture content was determined according to the TAPPI Standard method (T258 om-11). The chemical compositions of *Populus deltoides* wood were determined according to the TAPPI standard test methods as T264 cm-97 for Sample preparation, T13 os-54 for lignin, T204 cm-97 for extractives, and T15 os-58 for ash content. Cellulose was measured using the Kurschner-Hoffer method (Browning 1967). Hemicelluloses content was estimated by subtracting total content of lignin and cellulose from extracted-free wood meal. All measurements were done with 3 replicates.

Pre-hydrolysis

By using an electrically heated digester containing 6 bombs, each with 2.5 liter capacity rotating at 10 rpm, the air dried *Populus* chips were subjected to pre-hydrolysis by water at 170 °C, and liquor to wood ratio of 6:1 for 10, 20, and 30 minutes to achieve a predetermined target weight loss, based on previous studies (Hinck *et al.* 1985 and Kordsachia *et al.* 2004). After the pre-hydrolysis stage, the pre-extracted chips were washed thoroughly with hot water, air-dried, and the weight loss was determined gravimetrically.

Pulping Stage

The conditions used for the pulping stage were selected based on previous studies (Kordsachia *et al.* 2004; Sarwar Jahan *et al.* 2008). Pre-hydrolyzed chips of *Populus* were pulped via soda-AQ process at 170 °C, with a liquor to wood ratio of 6:1, an AQ charge 0.1 %, and a NaOH charge of 20%, based on OD chips, for various cooking times of 60, 90, and 120 minutes, using the same digester that was used in the pre-hydrolysis stage. The pulps were washed thoroughly with water and screened by Somerville screen to remove the rejects. The screened yields and kappa number of pulps were determined gravimetrically and by the TAPPI standard of T236 om-99, respectively.

Oxygen Delignification

For continuance of delignification, the pre-hydrolyzed soda-AQ pulps were subjected to oxygen delignification. Optimization of oxygen delignification stage was

done based on the previous literature (Samuelson 1994; Sjogren and Hook 2000). To optimize the oxygen delignification stage, the pulps were pre-treated at various conditions with oxygen (Table 1).

Table 1. Conditions of Oxygen Prebleaching

Time of reaction (min.)	MgSO ₄ (%)	Temp. of reaction (°C)	Alkali charge (%)	Oxygen Pressure (Kg/Cm ²)
90	0.6	100	2.0	6
60	0.6	100	1.0	5
45	0.6	100	0.75	4

At the end of oxygen delignification, the pulps were fully washed with water, and their kappa number was determined according to the TAPPI Standard method (T 236 om-99).

Bleaching Sequence and Conditions

An ECF bleaching system was implemented using a D₀ED₁ sequence. Three kappa factors of 0.30, 0.40, and 0.45 were used in D₀ stage of the D₀ED₁ bleaching sequence. Kappa factor is defined as percent total equivalent chlorine applied divided by unbleached kappa number. The reaction time and temperature at D₀ stage were 60 minutes and 70 °C, respectively. The alkali extraction (E) stage was done with hot and cold alkali extraction under conditions of 100 °C and 8% alkali charge for the hot alkali extraction and 30 °C and 10% alkali charge for the cold alkali extraction stage, and the cold alkali extraction were carried out immediately after the hot alkali extraction. So, two different alkali extraction steps were used in the bleaching sequence. The amount of ClO₂ charge at D₁ stage was about 50% of ClO₂ used at D₀ stage. The reaction time and temperature of D₁ stage were 120 minutes and 70 °C, respectively.

Evaluation of Dissolving Pulp

The dissolving pulps were evaluated according to the TAPPI (Technical Association of the Pulp and Paper Industry) standard methods: Brightness: T 452 om-02, α-cellulose: T 203 cm-99, viscosity: T 230 om-04, extractive: T204 cm-97, R₁₀ and R₁₈: T235 cm-00, Ash: T211 om-02, and Copper number: T430 cm-09.

RESULTS AND DISCUSSION

Pre-hydrolysis

The average chemical compositions of *Populus deltoides* wood were determined as: cellulose, 51.3%; hemicelluloses, 24.8%; lignin, 20.1%; extractives, 2.5%; and ash, 1.1%. By increasing the pre-hydrolysis time from 10 to 30 minutes, the weight loss of wood was increased from 8.1 to 14.7%, due to acid hydrolysis. The acetic acid required in the pre-hydrolysis stage was generated through the cleavage of acetyl groups in the hemicelluloses molecules of the *Populus* wood (Kircl and Akgul 2002). The end pH of the pre-hydrolysis liquor was decreased by increasing the reaction time, and it was

recorded as 3.8, 3.5, and 3.5 after 10, 20, and 30 minutes of pre-hydrolysis times, respectively. The reaction time of 30 minutes, which leads to 14.7% weight loss, was selected to be an acceptable pre-hydrolysis treatment for producing dissolving pulp from *Populus* wood, based on the previous studies (Hinck *et al.* 1985; Kordsachia *et al.* 2004).

Soda- AQ pulping

The effects of soda-AQ pulping time on screened pulp yield and kappa number are shown in Fig. 1. It is clearly shown that under the constant cooking condition, the kappa number of pulp reached about 15 at a pulp yield of 41.1% for a cooking time of 60 minutes, while by increasing the cooking time up to 120 minutes, the kappa number was only dropped to 14 at a pulp yield of 38.4%. Therefore, 60 minutes soda-AQ cooking time was selected for production of the bleachable pulp, due to higher screened yield at similar kappa number and equivalent low reject content.

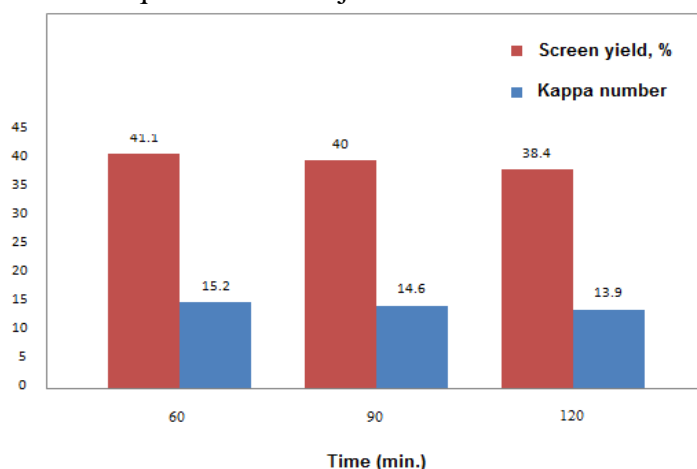


Fig. 1. Comparison of yields and kappa numbers of unbleached PH-Soda/AQ pulps

Oxygen Delignification

The effects of reaction conditions in oxygen delignification stage on the rate of kappa reduction are shown in Table 2. It is clear that more delignification can be obtained at higher alkali charge, oxygen pressure, and reaction time. However, it was shown that at a higher rate of delignification, the degradation of cellulose will be accelerated due to reduction of delignification selectivity. Based on these considerations, a 30 to 40% kappa reduction, in a single stage oxygen delignification in case of hardwood kraft pulp, was shown to be an acceptable range of delignification (Samuelson 1994; Ulrich 1998).

Table 2. The Effects of Oxygen Delignification Conditions on Kappa Reduction

Time of reaction (min.)	Alkali charge (%)	Oxygen Pressure (Kg/Cm ²)	Final kappa number	Δ Kappa number	Kappa reduction (%)
90	2.0	6	4.1	11.1	72.9
60	1.0	5	6.1	9.2	60.1
45	0.75	4	9.2	6.1	39.8

The oxygen delignification process condition in which 39.8% kappa reduction was obtained (final kappa number of 9.2) was selected as the optimum condition for oxygen pre-treatment.

DED Bleaching

An ECF bleaching system using a D_0ED_1 sequence was used for bleaching dissolving pulps. Based on the preliminary experiments, it was found that the final bleached pulp brightness was relatively low (83.4%) even at a kappa factor of 0.30 at D_0 stage. Therefore, to obtain a final pulp brightness of at least 86%, kappa factors of 0.40 and 0.45 were also used at the D_0 stage in the D_0ED_1 bleaching sequence.

Table 3. Properties of Bleached Dissolving Pulps using D_0ED_1 Sequence at Different Kappa Factors

Kappa factor in D_0	α -cellulose (%)	Viscosity (mPa.s)	Final yield (%)	DP	Brightness (%)	DCM Extractive (%)	Ash (%)	R_{10} (%)	R_{18} (%)
0.3	95.74	6.50	33.9	1348	83.4	0.11	0.04	98.1	98.4
0.4	95.92	6.89	33.5	1750	86.6	0.11	0.04	97.5	97.8
0.45	96.09	7.68	33.0	1800	87.3	0.10	0.03	97.0	97.7

By increasing the kappa factor from 0.30 to 0.45 in D_0 stage of a D_0ED_1 bleaching sequence of dissolving pulp, the pulp viscosity was increased at similar α -cellulose but at higher brightness, due to higher lignin dissolution and more fiber purification (Table 3). No detectable residual lignin was observed in the final bleached pulp, based on kappa number determination. Thus, almost complete delignification of the bleached pulp at higher kappa factor led to increased DP and viscosity. The final yields of dissolving pulps decreased from 33.9 to 33.0 %, by increasing kappa factor from 0.30 to 0.45 in D_0 stage (based on initial OD chips). The amounts of DCM insoluble extractives and ash content in the dissolving pulps remained pretty much unchanged by pulp samples, due to the use of distilled water in the process of bleaching and bleached pulp washing. However, the values of R_{10} and R_{18} were slightly reduced through the increasing kappa factor due to higher chemical reactions and, as a result, higher dissolution in 10 and 18% sodium hydroxide solutions. The higher viscosity at higher brightness level and at comparable values for other measured properties for dissolving pulp indicated that the kappa factor of 0.45 in D_0 stage could be an optimum chlorine dioxide charge in the present work. The properties of the dissolving pulps produced from *Populus deltoides* in the present work are compared with those of other dissolving pulp samples in Table 4.

The data in Table 4 clearly indicate that the values of viscosity, α -cellulose, and ash content in dissolving pulp produced from *Populus deltoides* in this study were quite comparable with the related published results for dissolving pulp, at a similar level of final pulp brightness. The solubility of dissolving pulp in 10% sodium hydroxide solution is higher than 18% ($S_{10} > S_{18}$); thus the values of R_{10} is usually lower than R_{18} ($R_{10} < R_{18}$).

Table 4. Comparison of Dissolving Pulp Properties of this Work with Other Dissolving Pulp Samples

Species	Process	α -Cellulose (%)	Viscosity	Brightness (%)	R ₁₀ (%)	R ₁₈ (%)	Ash (%)	References
Beech	PHK	97	475 (cm ³ /g)	88.3	-	-	-	Kerr and Harwood (1976)
Cotton linter	Kraft	98	-	-	-	-	0.05	Patt <i>et al.</i> (1994)
<i>Populus euroamericana</i>	Ethanol-Water	95.8	677 (cm ³ /g)	83	-	-	0.14	Kircl and Akgul (2002)
Jute	PHK	91.8	6.3 (mPa.s)	83.3	89.7	92	-	Sarwar Jahan <i>et al.</i> (2007)
Corn stalk	PHK	94.7	269 (cm ³ /g)	-	-	-	1	Behin <i>et al.</i> (2008)
<i>Trema orientalis</i>	PHK, PH-Soda/E DA	92.9, 93.2	8.2, 11.7 (mPa.s)	84.1, 79.6	94.3, 95.2	90.5, 93.3	0.12, 0.11	Sarwar Jahan <i>et al.</i> (2008)
<i>Populus deltoides</i>	PH-Soda/A Q	95.74	6.50 (mPa.s)	83.4	98.1	98.4	0.04	Present work
<i>Populus deltoides</i>	PH-Soda/A Q	96.09	7.68 (mPa.s)	87.3	97.0	97.7	0.03	Present work

PHK: Pre-hydrolysis-kraft pulping

Low molecular weight cellulose and hemicelluloses can be dissolved in 10% sodium hydroxide solution (S₁₀) (Wilson *et al.* 1952; Ohlsson 1952 and Sarwar Jahan *et al.* 2008). In other words, R₁₀ and R₁₈ are an indication of relative resistance to solubility in 10 and 18% sodium hydroxide solutions, respectively. Although R₁₀ was lower than R₁₈ in the present work, their values were surprisingly high, as compared to the published results in the literature. In addition, the α -cellulose content of dissolving pulp, which is the insoluble portion of dissolving pulp in 17.5 to 18% sodium hydroxide solution and should have an equivalent value as R₁₈, was lower than R₁₀ and R₁₈ in the present work, for some unknown reasons.

CONCLUSIONS

A reaction time of 30 minutes was selected for the pre-hydrolysis stage at a weight loss of 14.7%. Soda-AQ bleachable pulp was produced at a screened pulp yield of 41.1% and kappa number of about 15, at 60 minutes cooking time. Since 30 to 40% kappa reduction in a single stage oxygen delignification of hardwood kraft pulp are reported to be an acceptable range of delignification, the oxygen delignification process condition to achieve about 40% kappa reduction with final kappa number of 9.2, was selected as the optimum condition for oxygen pre-treatment stage. Since the final

bleached pulp brightness was relatively low (83.4%) even at a kappa factor of 0.30 at D₀ stage, kappa factors of 0.40 and 0.45 were also used at D₀ stage to obtain a final pulp brightness of around 86%. The pulp viscosity was increased at similar α -cellulose but at higher brightness when kappa factor was increased from 0.30 to 0.45 in the D₀ stage of D₀ED₁ bleaching sequence, due to higher lignin dissolution and more fiber purification. The final yields of dissolving pulps decreased from 33.9 to 33.0 %, by increasing kappa factor from 0.30 to 0.45 in D₀ stage (based on initial OD chips). However, the amounts of DCM-insoluble extractives and ash content remained pretty much unchanged, and the values of R₁₀ and R₁₈ were slightly reduced by increasing kappa factor due to higher chemical reactions and higher dissolution in 10 and 18% sodium hydroxide solutions.

The values of viscosity and α -cellulose in *Populus deltoides* dissolving pulp produced in this study were quite comparable with the related published results at similar level of final pulp brightness. However, the values of R₁₀ and R₁₈ were surprisingly high, as compared to the other published results, and the value of α -cellulose was lower than R₁₈ for some unknown reasons. The observations in the present study indicated that pre-hydrolyzed soda-AQ dissolving pulp can be made from *Populus deltoides* at a high level of alpha-cellulose content, and acceptable levels of final yield, brightness, and viscosity by means of proper control and optimization of pre-hydrolysis, pulping, and oxygen pretreatment conditions, and an optimized DED bleaching sequence.

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