

A STUDY ON OXYGEN DELIGNIFICATION OF MELOCANNA BACCIFERA (MULI BAMBOO) KRAFT PULP

Roy Thomas,^a Surendra P. Singh,^{a*} and Satyavolu V. Subrahmanyam^b

The response of kraft pulp of *Melocanna baccifera* (Muli bamboo) to different conditions of oxygen delignification and subsequent bleaching using CEHH sequence was studied. Oxygen delignification caused the kappa number of the pulp to drop between 40 and 75% over the range of temperature 70-100 °C, oxygen pressure 2-7 bar, alkali charge 2-4%, and reaction time 5-60 min. The oxygen-delignified pulp could be bleached to a brightness level of 86%. The conditions that favored greater kappa reduction also caused a greater reduction in the pulp viscosity, which called for an economic balance between the environmental benefits and the degradation of the pulp. Reaction temperature during oxygen delignification had an effect on the fiber curl and kink, while other variables such as oxygen pressure, alkali dose, and the reaction time had no significant effect on fiber deformation.

Keywords: *Melocanna baccifera*, *Muli bamboo*, *Oxygen delignification*, *Bleaching*, *Kappa number*, *Curl index*, *Kink index*

Contact information: *a: Department of Paper Technology, Indian Institute of Technology Roorkee, Saharanpur Campus, Saharanpur, India; b: Central Pulp and Paper Research Institute, Saharanpur, India; *Corresponding author: spsptfpt@iitr.ernet.in*

INTRODUCTION

For any bleaching method, environmental and/or economical considerations demand that the lignin content of the pulp should be reduced as much as possible before bleaching. Alkaline oxygen delignification (ODL) allows extended delignification of chemical pulps without a serious loss in pulp yield and with positive environmental impacts (Rodriguez et al. 2007; Gullichsen and Fogelholm 1999; Mukherjee and Bandyopadhyay 1993). ODL fits particularly well with kraft pulping, because oxidized white liquor can be used as an alkali source, and its spent liquor is recoverable with kraft liquor.

An oxygen stage before bleaching reduces the need for bleaching chemicals and the caustic soda required in the first extraction stage, roughly in proportion to the amount of delignification achieved in the oxygen stage. ODL decreases formation of chloroorganics (expressed as AOX) in bleach plant effluents when chlorine-based chemicals are used in subsequent bleaching of the pulp. Regardless of the bleaching chemicals used, ODL decreases BOD, COD, and color of the effluents. Interestingly, the decrease in color is more than expected on the basis of the lignin removed in the oxygen stage.

Although almost all the lignin in kraft pulp can be removed with an oxygen stage, oxygen is less specific at lignin removal than chlorine or other bleaching agents currently in use. Therefore, ODL is used to reduce the value of kappa number of the pulp by only

about 50%, because an attempt at greater reduction in kappa is expected to lead to unacceptable degradation of carbohydrates in the pulp and loss of pulp strength (Biermann 1996). However, oxygen delignification does not affect the pulp yield as negatively as other methods of extended cooking do.

Bamboo is widely used as a non-wood fibrous raw material for production of paper and paperboard in Asia (Atchison 1998). Because of their long fibers (often comparable with softwood), bamboo chemical pulps are used as reinforcing fibers for blending with hardwood and non-wood pulps in many paper products. Bamboo responds well to kraft pulping (Misra 1981). In India, bamboo is often cooked along with hardwoods.

ODL can be applied to bamboo kraft pulps for economical solutions to bleach plant environmental issues (Gomide et al. 1991; Kishore et al. 1995; Singh et al. 1995; Mittal and Maheshwari 1996). Vu et al. (2004) in a study on *bambusa procera acher* found that a pulp suitable for ECF or TCF bleaching could be produced by conventional kraft pulping followed by ODL. They observed that a high sulphidity (35-45%) with lower effective alkali (14-16%) resulted in both high yield and high pulp viscosity compared with low sulphidity (0-15%) with high EA (16-18%) at the same degree of delignification.

Muli bamboo is an important fiber source for the Indian paper industry. Muli bamboo is naturally distributed in Myanmar, Bangladesh, and northeastern states of India, where it represents between 60 and 95% of the region's bamboo resources. Besides northeastern states, Muli bamboo is also found in Orissa, and other lower altitude places of eastern India (Benton 2004). Muli bamboo is a medium-sized ever-green bamboo, 10–20 m tall, having culms of relatively thin walls, 5-12 mm, with 200-500 mm long internodes. The prices of muli bamboo in northeastern states of India are nearly 60% of the prices for other thick walled varieties available in the region. Muli bamboo is a good pulping raw material, except for some difficulties it poses in handling of chips in chip-silos, conveyers, and digester-feed systems because of its thin-walled structure.

In the present work, we have studied the response of kraft pulp of *Melocanna baccifera* (Muli bamboo) to different conditions of oxygen delignification and subsequent bleaching. A study on response of muli bamboo to ODL is of interest to the pulp mills whether they are using the conventional or a modern bleaching sequence. A CEHH (Chlorination-Extraction-Hypochlorite-Hypochlorite) sequence for bleaching of the ODL pulp was used in the present study, since elemental chlorine and hypochlorite are still widely used for bleaching of bamboo pulps in India and other Asian countries. It will take a few years before pulp bleaching will be completely ECF or TCF in this region. Many existing mills have, however, moved towards partial substitution of chlorine by chlorine dioxide in chlorination stage and using oxygen-based chemicals in extraction stages.

EXPERIMENTAL METHODS

Pulping and Bleaching

Samples of muli bamboo were obtained from an integrated pulp and paper mill located in Assam, India. The supply of muli bamboo to the mill was from the forests of North-Eastern States of India. One sample of bamboo freshly arrived from the forests and

another sample of bamboo stored for about six months in the yard of the mill were chipped separately. A 50:50 mixture of the two types of chips was used for further study.

The bamboo chips were cooked by kraft pulping process in a laboratory rotary digester consisting of six bombs rotating in an electrically heated polyethylene glycol bath. Each bomb was charged with 300 g chips and an appropriate amount of white liquor of 20% sulphidity. The schedule of digester heating consisted of 30 min for heating from ambient temperature to 100 °C, 90 min for heating from 100°C to 165°C, and 120 minutes at 165°C. The pulp obtained was washed with warm demineralized water and screened in a laboratory vibratory screen having 0.2-mm slots.

Oxygen delignification (ODL) of the pulp was carried out in a 3-L, Mark V, Quantum reactor at 10% consistency, using 250 g (o.d.) pulp for each run. The pulps after oxygen delignification were bleached using a CEHH sequence under the conditions given in Table 1.

Table 1. Bleaching Conditions

	C-stage	E-stage	H1-stage	H2-stage
Consistency, %	3	10	10	10
Retention time, min	30	60	120	120
Temperature, °C	25	65	45	40 - 45
Chlorine dose (as active chlorine), % of o.d. pulp	0.25 to 0.27 times kappa number		1.2	1.2
NaOH, % o.d. pulp		1.5 – 2.0%		
NaOH as buffer, % of calcium hypochlorite used			20	20

Evaluation of the Pulp

Kappa number and brightness of the pulps were determined using TAPPI standard procedures T 236 and T 525 respectively. Pulp viscosity in CED (cupriethylene diamine) solution was determined using SCAN-C15:62 method and expressed as intrinsic viscosity (ml/g). Fiber dimensions and fiber deformations were determined using OpTest (OpTest 2000) laboratory fiber quality analyzer (FQA).

RESULTS AND DISCUSSION

In the current industrial practice, the kappa number for bleachable grade bamboo kraft pulp was kept at some value between 16 and 18. During this study, several cooks were prepared in the laboratory by varying the active alkali charge and the maximum temperature of cooking. It was targeted to obtain a pulp of kappa number 20 so that after the oxygen delignification, a pulp of kappa number of about 10 could be obtained. Some conditions were screened out because the kappa number, pulp yield, and the residual active alkali content in black liquor were outside the acceptable ranges for this study. Four pulps, as shown in Table 2, were used to further study the response of these pulps to oxygen delignification and bleaching.

Table 2. Response to Pulping, ODL, and Bleaching

	Pulp 1	Pulp 2	Pulp 3	Pulp 4
<i>Kraft pulping, sulfidity 20%, raw material to liquor ratio 3:1, Maximum temperature 165 °C</i>				
Active alkali charge (as Na ₂ O), %	13	14	15	16
Kappa number	32.3	22.7	20.2	16.4
Unscreened pulp yield	56.0	55.1	53.2	52.2
Screened pulp yield, %	51.3	52.9	52.4	47.7
pH of black liquor	11.0	11.2	11.9	12.1
Residual active alkali in black liquor (as Na ₂ O), g/L	4.8	6.9	8.6	10.0
CED viscosity, ml/g			1066	
<i>Oxygen delignification (ODL): O₂ pressure 5 bar, Temperature 90 °C, Alkali charge 2%, Consistency 10%, Residence time 60 min</i>				
Kappa number	15.1	10.5	9.3	7.7
ODL pulp yield, %	48.9	51.9	51.7	46.2
CED viscosity, ml/g	917	988	910	837
<i>Bleaching sequence CEHH, Chlorine charge 0.25*Kappa number at C-stage, 1.2 % at H1-stage and 1.2% at H2-stage, 1.5% NaOH at E-stage</i>				
Bleached pulp yield, %	45.1	49.4	49.5	43.7
Brightness (ISO), %	86.0	86.9	86.2	86.4

The pulp produced using 15% active alkali charge was chosen for oxygen delignification as it gave the desired kappa number and adequate residual active alkali content of the black liquor. The CED viscosity of this pulp was found to be 1066 ml/g. This pulp was used for oxygen delignification under different conditions of oxygen pressure, temperature, alkali charge, and residence time in the oxygen reactor.

The ODL pulps obtained under different conditions were subsequently bleached using CEHH sequence. Figures 1 to 3 show the effect of temperature, oxygen pressure, alkali charge, and the residence time on the kappa number of the ODL pulp. The ODL caused the kappa number of the pulp to drop between 40 and 75% over the conditions used in these experiments. The kappa number reduction during ODL was greater at higher reaction temperature, higher oxygen pressure, higher alkali dose, and longer residence time. The oxygen reaction was very fast in the beginning and only five minutes were sufficient to cause about 50% drop in kappa number.

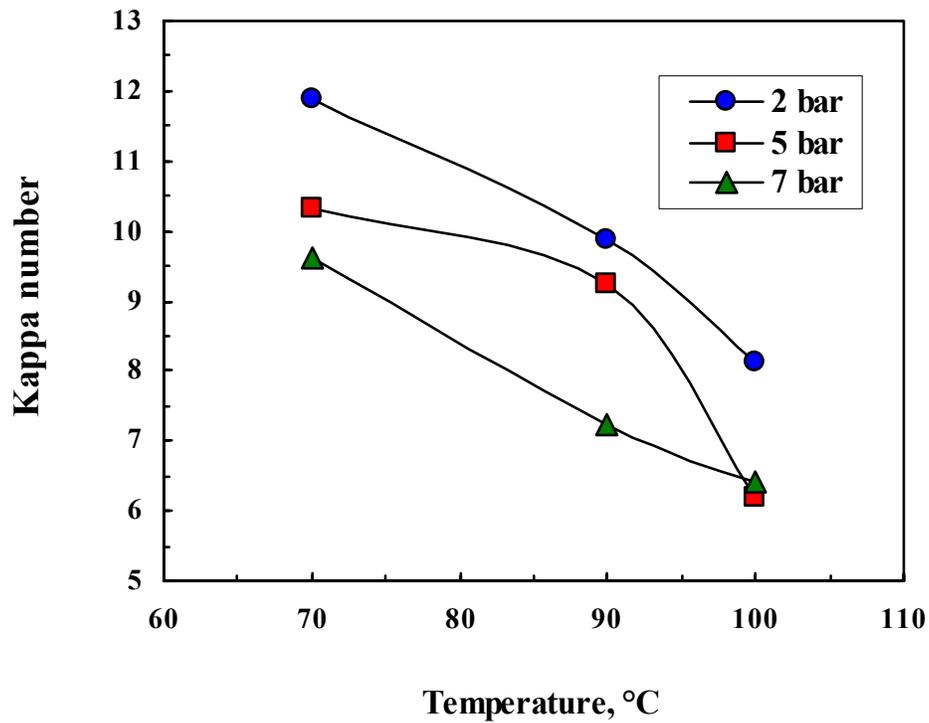


Fig. 1. Effect of oxygen pressure and temperature during ODL on kappa number. (Kappa number before ODL 20.2, Alkali charge 2%, Residence time 60 min)

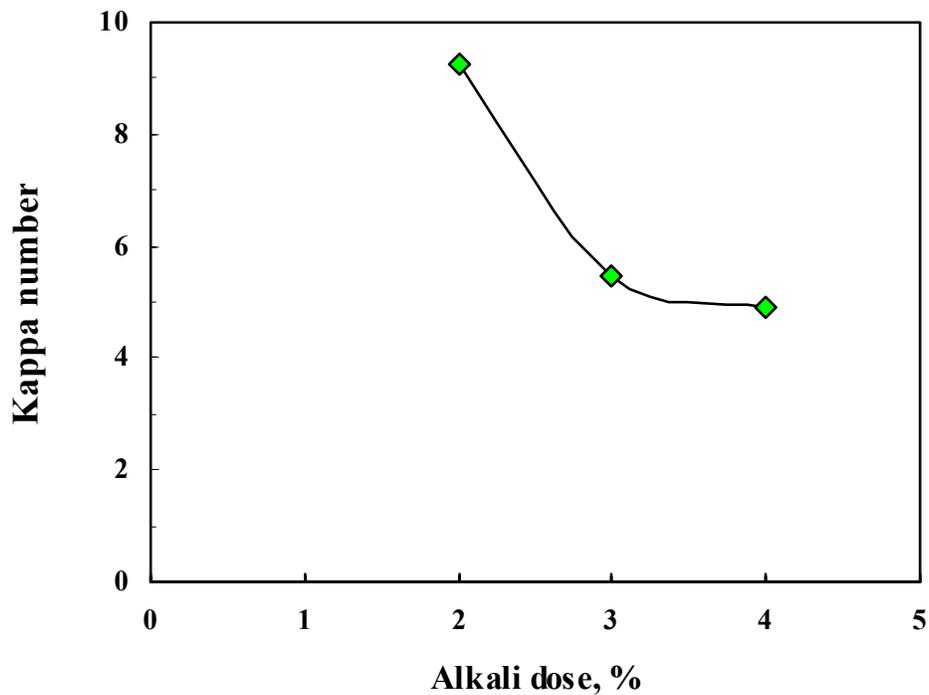


Fig. 2. Effect of alkali charge on kappa number of ODL pulp. (Kappa number before ODL 20.2, Oxygen pressure 5 bar, Temperature 90 °C, Residence time 60 min)

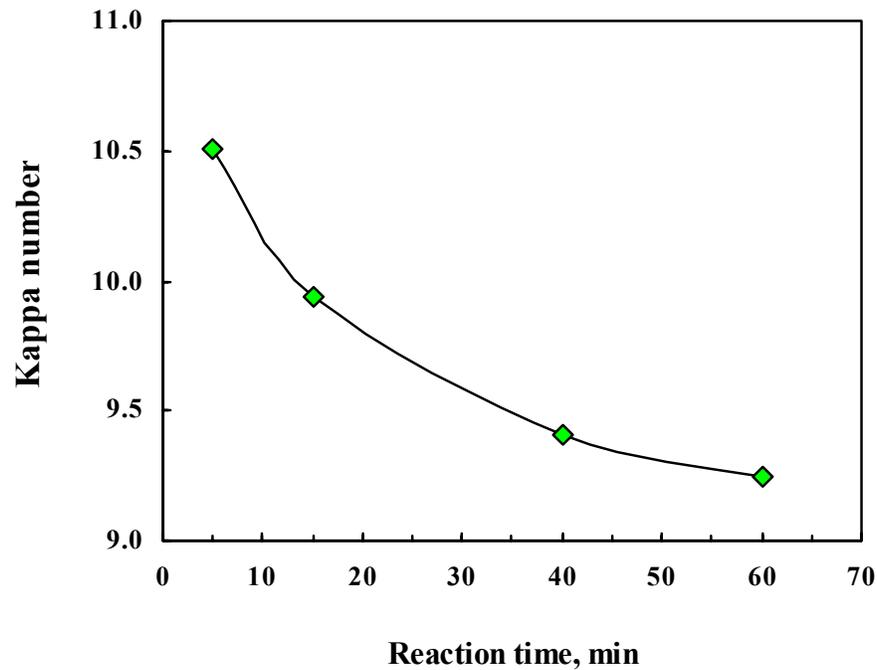


Fig. 3. Effect of ODL reaction time on kappa number of ODL pulp. (Kappa number before ODL 20.2, Oxygen pressure 5 bar, Temperature 90 °C, Alkali charge 2%)

Figures 4 to 6 show the effect of ODL conditions on the pulp viscosity after the ODL and after subsequent bleaching.

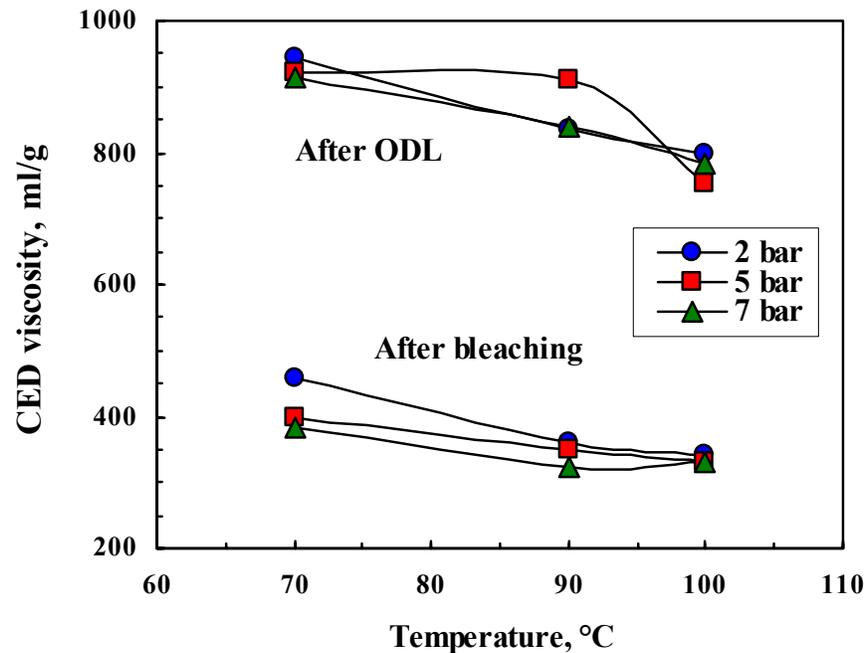


Fig. 4. Effect of oxygen pressure and temperature during ODL on pulp viscosity after ODL and after final bleaching. (Viscosity before ODL 1066 ml/g, Alkali charge 2%, Residence time 60 min)

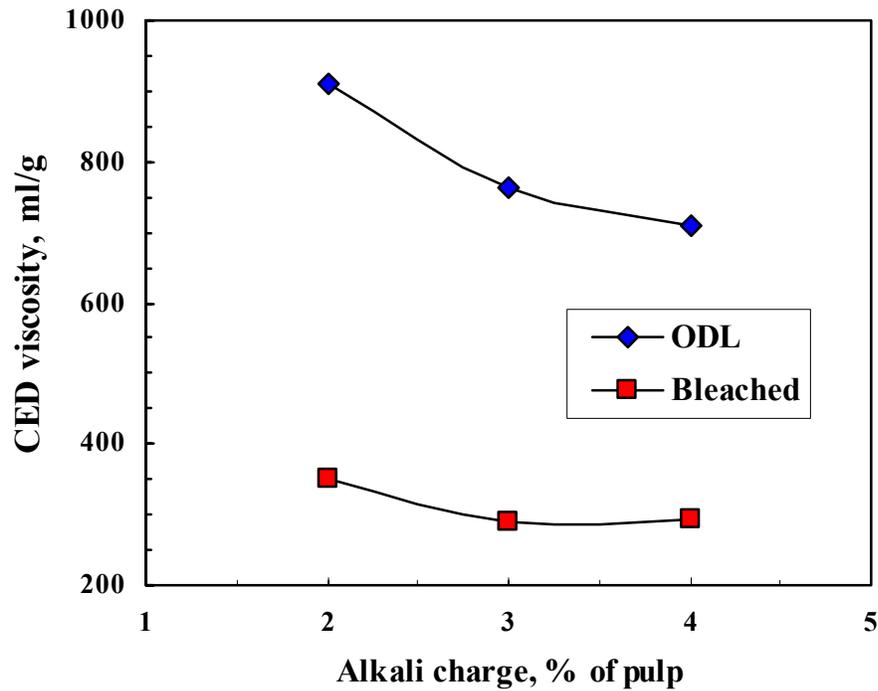


Fig. 5. Effect of alkali charge on viscosity of ODL pulp. (Viscosity before ODL 1066 ml/g, Oxygen pressure 5 bar, Temperature 90 °C, Residence time 60 min)

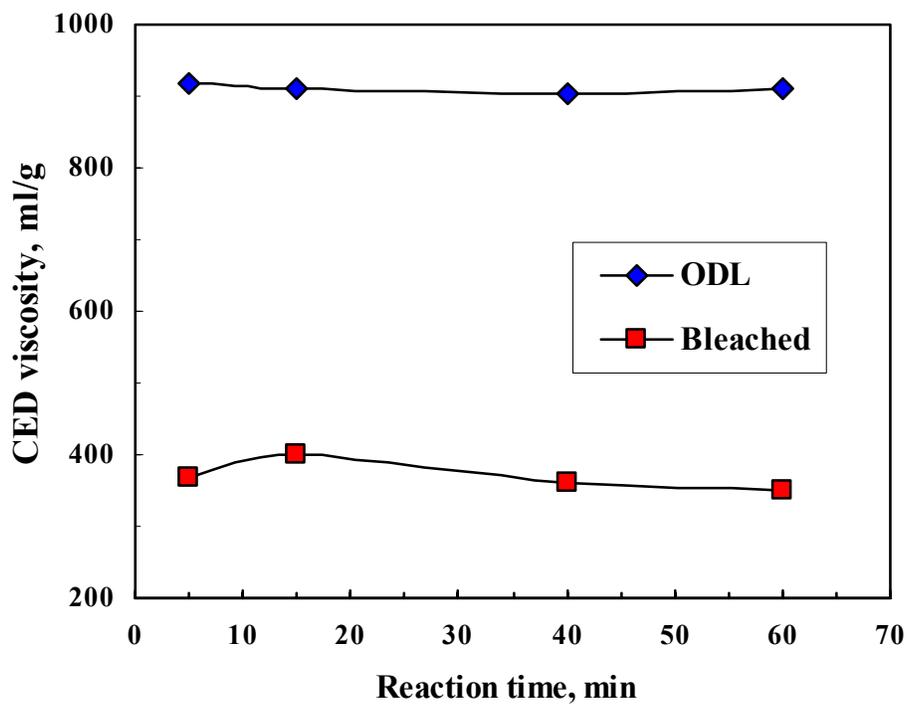


Fig. 6. Effect of ODL reaction time on viscosity of ODL pulp. (Viscosity before ODL 1066 ml/g, Oxygen pressure 5 bar, Temperature 90 °C, Alkali charge 2%)

As shown, the conditions that favored greater kappa reduction also caused a greater reduction in the pulp viscosity. Thus, the ODL conditions need to be chosen to provide the economic balance between the environmental benefits and the degradation of the pulp. Oxygen pressure had a relatively minor effect on the pulp viscosity.

Figure 7 shows the effect of ODL condition on the brightness of the finally bleached pulp. The maximum brightness attainable was 86.2% when the temperature during ODL was kept at 90 °C and the oxygen pressure was maintained at 5 bar.

The OCEHH bleaching has distinct advantages over the conventional CEHH bleaching sequence in terms of reduced chlorine consumption, increased pulp yield, and improved pulp brightness. For conventionally bleached pulp of muli bamboo, the data available in our laboratory suggest that the optimized conditions would be to obtain an unbleached kraft pulp with kappa number 16-17 and to bleach it to brightness 82-84%. These conditions correspond to 16% active alkali charge in pulping, 6-7% total active chlorine charge in bleaching, and the viscosity of the resulting pulp 450-500 ml/g.

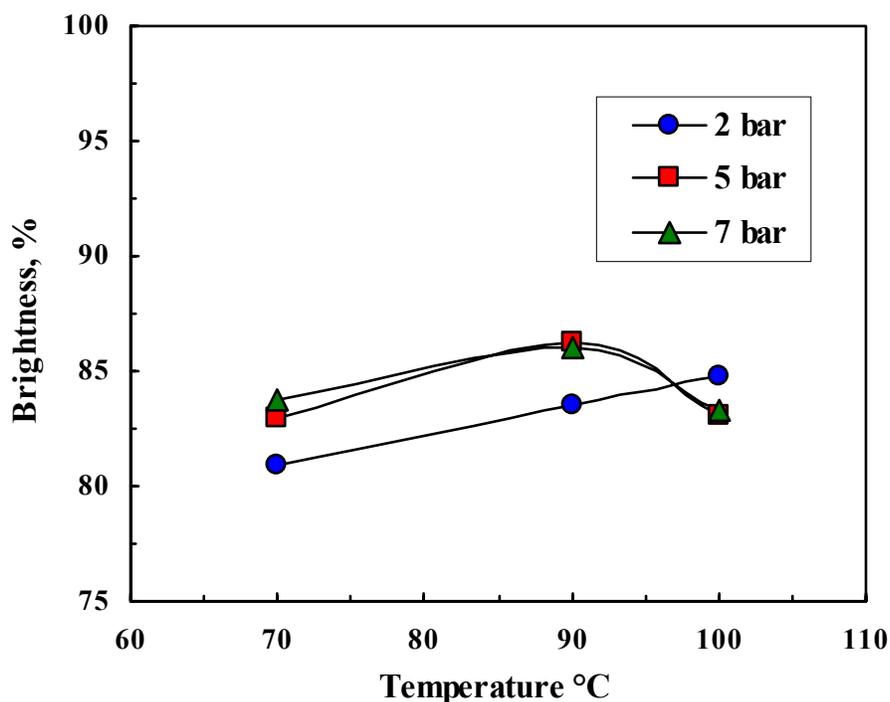


Fig. 7. Brightness of bleached pulp as function of oxygen pressure and temperature during ODL. (Alkali charge 2%, Residence time 60 min)

Fiber Quality Analysis of the ODL Pulp

The bleached pulp was analyzed for such fiber characteristics as fiber length, curl index, and kink index using OpTest laboratory fiber quality analyzer. The FQA fiber length measurements give similar results as given by the Kajaani FS-200 fiber length analyzer (Robertson et al. 1999). The mean fiber length and width were measured for different conditions of ODL. It was observed that the fiber dimensions were not affected

by the oxygen delignification process. The mean values and the standard deviations of the fiber dimensions of the pulps obtained under different conditions of ODL are given in Table 3.

Table 3. Mean Dimensions of the Fibers

Weight weighted mean fiber length, mm	2.04 (Standard deviation = 0.043)
Length weighted mean fiber length, mm	1.56 (Standard deviation = 0.034)
Arithmetic mean fiber width, μm	17.43 (Standard deviation = 0.166)

In industrial applications, fiber deformation is very important fiber characteristic. Fibers in the plant stem are straight, but they become curly during pulping, mixing, and refining operations because of being subjected to bending and axial compressive stresses, particularly at medium and high consistencies (Robertson et al. 1999). The inclusion of fiber deformation with other fiber characteristics in pulp evaluation could greatly improve the understanding of the relationship among fiber properties, pulping and papermaking processes, and paper quality (Mohlin et al. 1996). In general, an increase in fiber deformation results in a decrease in tensile index and an increase in stretch-to-break of the paper.

The FQA device allows measurement of fiber deformation in terms of curl index, number of kinks per fiber, kink angles, and kink index. It was observed that these measurements of fiber deformation correlated highly with each other for the ODL pulps obtained under different conditions. Values of curl index and kink index only have been included in this discussion.

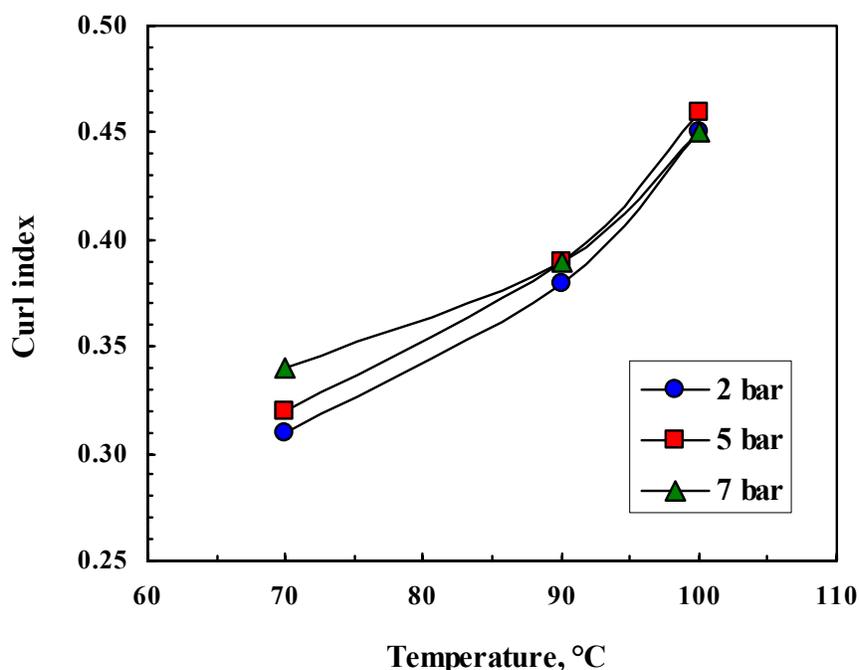


Fig. 8. Curl index of ODL pulp as function of oxygen pressure and temperature during ODL. (Alkali charge 2%, Residence time 60 min)

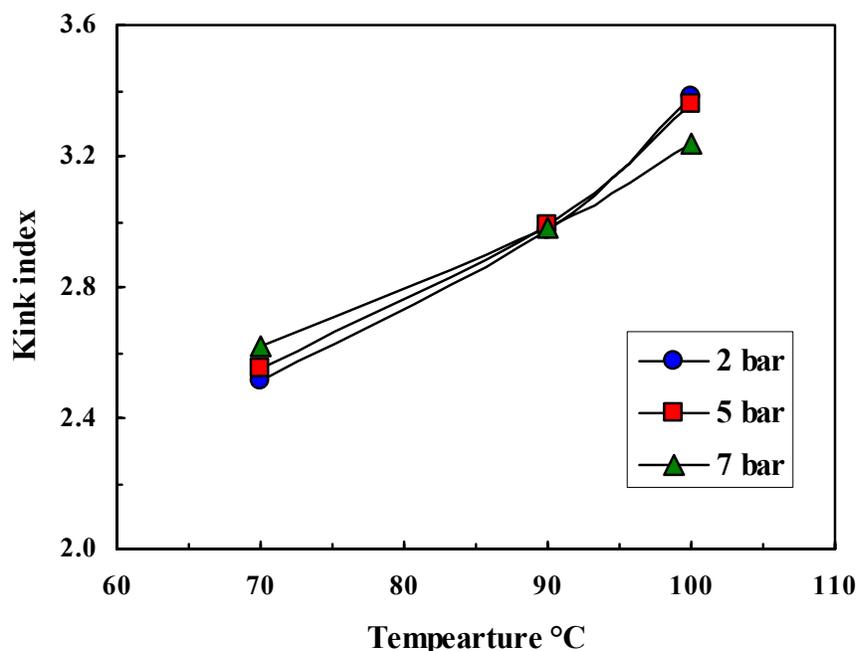


Fig. 9. Kink index of ODL pulp as function of oxygen pressure and temperature during ODL. (Alkali charge 2%, Residence time 60 min)

Fiber curl describes the deviation from straightness of the fiber axis. In FQA, the curl index is defined as the relative increase in the length of a fiber when it is straightened but not stretched (Page et al. 1985). Kink refers to an abrupt change in fiber curvature. The FQA uses a modified Kibblewhite equation for calculating kink index (OpTest 2000). As evident from Figures 8 and 9, the fiber curl and kink increased with increase in temperature. The oxygen pressure, alkali dose, and the retention time in oxygen reactor did not significantly affect the curl or kink index.

CONCLUSIONS

1. For muli bamboo, a kraft pulp of kappa number of 20 could be produced using an active alkali charge of 15% (as Na_2O) at the usual cooking conditions (sulphidity 20%, maximum temperature 165 °C). These conditions produced a pulp of high yield, high bleached brightness, low pulp degradation, and adequate residual active alkali content of the black liquor.
2. The ODL caused the kappa number of the pulp to drop between 60 and 30% over the range of temperature 70-100 °C, oxygen pressure 2-7 bar, alkali charge 2-4%, and reaction time 5-60 min. The kappa number reduction during ODL was greater at higher reaction temperature, higher oxygen pressure, higher alkali dose, and longer residence time. The oxygen reaction was very fast in the beginning and only five minutes were sufficient to cause about 50% drop in kappa number. This should be

economically advantageous, as a shorter retention time in an ODL reactor could be required.

3. The conditions that favor greater kappa reduction also caused a greater reduction in the pulp viscosity. However, oxygen pressure had a relatively less effect on the pulp viscosity. The ODL conditions should be chosen to provide the economic balance between the environmental benefits and the degradation of the pulp.
4. The maximum brightness attainable after bleaching was 86.2% when the temperature during ODL was kept at 90 °C and the oxygen pressure was maintained at 5 bar.
5. Fiber curl and kink increased with increase in temperature during ODL. The oxygen pressure, alkali dose, and the retention time in oxygen reactor did not significantly affect the curl index or kink index of the fibers. The increase in fiber curl and kink should help in increasing tensile energy absorption of the paper.

ACKNOWLEDGEMENTS

The authors are grateful for the support of the Director, Central Pulp and Paper Research Institute, Saharanpur in carrying out this work.

REFERENCES CITED

- Atchison, J. E. (1998). "Update on global use of non-wood plant fibers and some prospects of their greater use in the United States," In: *Proceedings of the 1998 Tappi North American Non-Wood Fiber Symposium*, 17-18 Feb. 1998, Atlanta, GA, United States, 13-42.
- Benton, A. (2004). *Fact sheet: International Network for Bamboo and Rattan*, <http://www.inbar.int>
- Biermann, C. J. (1996). *Handbook of Pulping and Papermaking*, 2nd Ed., Academic Press.
- Gomide, J. L., Colodette, J. L., and Campos, A. S. (1991). "Kraft pulping and oxygen delignification of bamboo," In: *Proceedings of the 1991 Pulping Conference* 3-7 Nov 1991, Orlando, Fl, Book 1, pp 419-426.
- Gullichsen, J., and Fogelholm, C. J. (eds). (1999). *Papermaking Science and Technology - Book 6A - Chemical Pulping*, FAPET OY, Helsinki, Finland.
- Kishore, H., Chand, S., Rawat, N., Gupta, M. K., Janbade V. T., Bist, V., Roy, T. K., Pant, R., and Jauhari, M. B. (1995). "Oxygen treatment followed by CEH bleaching is economic and quick solution to the pollution problems to Indian paper mills," In: *Proceedings of the 2nd International Seminar on Pulp and Paper Industry*, Paprex-1995, New Delhi, India, 9-11 Dec, pp 69-83.
- Misra, D. K. (1981). "Pulping and bleaching of non-wood fibers," In: Casey J. P. (ed), *Pulp and Paper - Chemistry and Chemical Technology*, Vol. 1, 3rd Ed., Wiley, New York.

- Mittal, S. K., and Maheshwari, S. (1996). "Mill experience on manufacture of ECF bamboo market pulp," In: *Proceedings of the 3rd International Non-Wood Fiber Pulping and Papermaking Conference*, Beijing, China, Vol. 1, 378-382.
- Mohlin, U. B., Dahlbom, J., and Hornatowska, J. (1996). "Fiber deformation and sheet strength," *Tappi J.* 79(6), 105-111.
- Mukherjee, D., and Bandyopadhyay, N. (June 1993). "Oxygen delignification technology for improved product quality and pollution abatement in pulp and paper industry," *IPPTA Journal* 5(2), 27-36.
- OpTest Fiber Quality Manual (2000).
- Page, D. H., Seth, R. S., Jordan, B. D., and Barbe, M. C. (1985). "Curl, crimps, kinks and microcompressions in pulp fibers- their origin, measurements and significance", In *Paper Making Raw Materials: Their Interaction with the Production Process and their Effect on Paper Properties. Transactions of the 8th Fundamental Research Symposium held in Oxford September 1985*, V. Punton (ed.) Vol. 1, Mechanical Engineering Publications Limited. London, pp. 183-227.
- Robertson, C., Olson, J., Allen, P., Chan, B., and Seth, R. (1999). "Measurement of fiber length, coarseness, and shape with the FQA," *Tappi J.* 82(10), 93-98.
- Rodriguez, A., Jimenez, L., and Ferrer, J. L. (2007). "Use of oxygen in delignification and bleaching of pulps," *Appita J.* 60(1), 17-22.
- Singh, S. V., Datta, S. K., and Rai, A. K. (Sept. 1995). "Delignification of kraft pulp with oxygen chlorine combinations," *IPPTA Journal*, 7(3), 57-63.
- Vu, T. H. M., Pakkanen, H., and Alen, R. (Jan 2004). "Delignification of bamboo (*Bambusa procera acher*) - Part 1. Kraft pulping and the subsequent oxygen delignification to pulp with a low kappa number," *Industrial Crops and Products*, Elsevier Science, 19(1), 49-57.

Article submitted: May 25, 2007; First round of reviewing completed: July 13, 2007; Revised version received: July 30, 2007; Accepted: July 30, 2007; Published: August 1, 2007