The Kinetics of Delignification in Low Sulphidity Kraft-Anthraquinone and Soda-Anthraquinone Pulping of Muli Bamboo (Melocanna Baccifera)

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ABSTRACT: *Kinetic investigations were carried out on alkaline delignification of mill cut chips of muli bamboo, a common species for pulping in Bangladesh. The study was confined to the effect of 0.05% anthraquinone (AQ) in soda and low sulphidity kraft pulping. The results were compared to the kraft control at a sulphidity of 15% and 25%.*

The investigation shows that there are three distinct phases of delignification during pulping of muli bamboo. The delignification rate increases with an increase in sulphidity or an addition of AQ. The delignification rate in the bulk and residual phases are found in the order: $kraft_{15}$ + AQ > soda + AQ > $kraft_{25}$ > $kraft_{15}$. The first and second transition points changes at the lowest lignin yield with AQ in $kraft_{15}$ pulping. Normal $kraft_{15}$ pulping turns at the highest lignin content at the first and second transition points. The transition points during soda + AQ occurrs at almost the same lignin content as in $kraft_{25}$ pulping.

Normally, a bleachable grade pulp from muli bamboo is made at a kappa number of 20. All the pulping processes seem to confine the cooks in the bulk phase to attain at this kappa number. However, addition of AQ either in low sulphidity or soda pulping, or a normal sulphidity pulping may allow the bulk phase to continue to still a lower lignin content.

INTRODUCTION

The kinetics of alkaline pulping has been widely studied (1 - 12). The delignification is considered to proceed in three distinct phases. These are the initial, bulk and residual phases. In all the three phases, the delignification rate appears as a first order reaction with respect to the remaining lignin content of the material used (1,2,4,6,8,11-13).

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In the initial phase, the dissolution of lignin is small with a high consumption of alkali, resulting in a very rapid drop in carbohydrate yield (2,3,6,7,10,12,14). Alkali is consumed by the acidic reaction products which are formed when extractives are dissolved and carbohydrates are split (6,14).

The bulk phase begins at a temperature of about 140°C (2,7,14). The major part of the lignin is removed during this phase and the dissolution of carbohydrates being smaller and the consumption of alkali lower than in other phases (3-7,10,14).

In the residual delignification phase, the removal of lignin slows down. Consequently, the carbohydrate yield rapidly falls and the alkali consumption significantly increases (1-7). The slower rate of delignification in this phase might be due to some inextricably bound lignin (15) or grafting of modified lignin on the cellulose crystallite (11,16). The three phases in pulping are obviously governed by different chemical and physical reactions.

The chemistry of the kinetics of alkaline pulping is very complicated because of many variables that govern the delignification process. However, knowledge of the kinetics is essential for the control of cooking.

The role of anthraquinone (AQ) for reducing the cooking time and stabilizing carbohydrates in alkaline pulping is well documented (17-20). It is reported that soda + AQ and kraft + AQ follow the same kinetics behaviour (21,22). The mechanism of the anthraquinone process is explained on the basis of oxidation - reduction (redox) cycle generated during alkaline pulping (22-24). Since AQ in hot alkaline solution is readily and reversibly reduced by glucose, a carbohydrate-AQ reaction takes places, which accounts for the raducing power in alkaline pulping liquor. The reduced AQ becomes oxidised again by reducing a lignin intermediate produced in a initial reaction. Thus, the reduced AQ prevents the reversal, thereby accelerating the net forward reaction. AQ is effective in catalytic amounts because it enters on oxidation - reduction cycle. The reduction and stabilization of AQ is dependent on 'reducing' end groups in the carbohydrates (24).

It is reported that the effect of AQ on pulping is dependent on species (25-27). Previously most of the works was done on wood species. An extensive survey of the literature shows that no kinetic study in presence of AQ in the kraft and soda pulping of muli bamboo has

been conducted (28,29). In Bangladesh, this bamboo species is the main pulping raw material. Thus, lack of the knowledge of the kinetic behaviour during its pulping restricts efficient control of the cook. An improper cooking design may also bring the cook to the residual phase. Then, there is a danger of a serious loss in pulp yield. This study has, therefore, been undertaken to answer to what extent the cooking is to be continued in alkaline pulping of muli bamboo in presence of AQ for a bleachable grade of pulp. The knowledge of the delignification pattern would make it possible to minimise the attack of alkali on carbohydrates in order to avoid a serious loss in pulp yield and a decrease in pulp strength.

EXPERIMENTAL

Handling of chips

Mill cut muli bamboo chips were used. The chips were air-dried for 15 days. The air-dry chips were screened on a laboratory screen and the fraction passing 32 mm openings but retained on 6 mm opening were the accepts. The screened chips were then hand-sorted to remove decayed bamboo and stored in a cold storage. The dry matter content of the chips was about 87%.

Cooking

The cooks were carried out in 2 litre stainless steel autoclaves in a thermostatically controlled heated air bath. The amount of chips used in each cook was 250 g (oven dry basis). The cooking liquor was prepared with technical grades of NaOH and Na_2S.

Cooking was done by kraft process at a sulphidity of 15% ($kraft_{15}$) and 25% ($kraft_{25}$) to serve as the controls. The effect of AQ was studied in $krafts_{15}$ and soda pulping. The AQ used was 0.05% on od chips. Other cooking parameters were

- Active alkali as NaOH, 18%
- Liquor to bamboo ratio, 4 : 1
- Rise of temperature to 70°C in 15 minutes and from 70°C to 170°C in 90 minutes
- Cooking temperature, 170°C
- Total cooking time (excluding 15 minutes for the rise of temperature to 70°C), varying

Post-cooking treatments

The autoclaves were removed after the specified reaction times and were cooled rapidly with running water. Black liquor samples were then taken and cooked chips were washed overnight in running water. Defibration was then done in a laboratory model Sprout Waldron disc refiner using a plate clearance of 0.25 mm. Chips with cooking time of 0, 20 and 40 minutes were defibrated twice. The rest were defibrated once. After refining, the pulp was screened on a flat vibratory screen with 0.50mm slots. If there was any screening rejects, it was refined in the refiner and mixed with screened pulp. The screened pulp was transferred into a thick cloth bag and pressed to remove excess water and then shreded, mixed and weighed. A portion of it was dried to a constant weight (SCAN-C 3:78) and the yield determined. The pulp was stored in a refrigerator in sealed polythene bags for subsequent analyses.

Analytical work

The black liquor was analysed for residual alkali as per Swedish method (30). The chlorine number of the pulp was determined according to (SCAN-C 29 : 72). It was converted to Klason Lignin + acid soluble lignin by multiplying it by a factor of 0.90 (4), because the chlorine number obtained was less than 24. The lignin yield on oven-dry bamboo was obtained by multiplying the pulp lignin content by the total yield value. The carbohydrate yield was calculated by substracting the lignin yield from the total yield. The pentosan content of pulp was determined as per TAPPI bromination method (T223 ts-63) and the pentosan content on oven dry bamboo was obtained by multiplying the pulp pentosan content by the total yield value.

RESULTS AND DISCUSSION

Details of the experimental data are given in the original study (29), Only the main results are reported and discussed in this article. Conventional linear regression analyses were done with the basic data. The different phases were separated from the index of the best coefficients of correlation of the regression lines. The first transition point was calculated in all the cases by equating two intersecting lines. In the residual phase, it was not possible to obtain systematic lines in all the cases because the data was more scattered. Scattering of the data is unavoidable at the end of cook (6,7). Hence,

it was not possible to draw regression lines in the total yield against chlorine number in the residual phase. In these cases, the lines were drawn by visual estimation and the intersecting point was determined graphically in conformity with the second transition point calculated mathematically (plot of LnL against cooking time and alkali consumption against lignin yield).

DELIGNIFICATION

Assuming that the rate of delignification is of the first order with respect to lignin content in the pulp on oven-dry bamboo, then

$$\frac{-dL}{dt} = k.L$$

where,

$$\frac{-dL}{dt} = \text{rate of delignification}$$

L = lignin yield, % on od bamboo

k = delignification rate constant

According to this equation, a straight line is obtained in the plot of the logarithm of lignin yield against cooking time, the slope multiplied by a negative sign being a measure of the delignification rate constant. The bulk and residual phases for $kraft_{15}$, $kraft_{25}$, $kraft_{15}$ + AQ and soda + AQ pulping of muli bamboo are shown in Fig. 1. The initial phase cannot be included because of
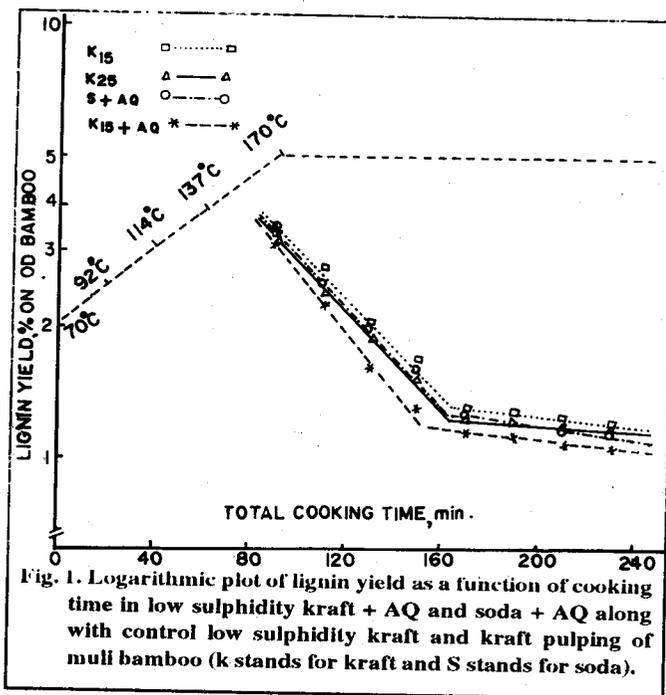
Fig. 1. Logarithmic plot of lignin yield as a function of cooking time in low sulphidity kraft + AQ and soda + AQ along with control low sulphidity kraft and kraft pulping of muli bamboo (k stands for kraft and S stands for soda).

the rise to cooking temperature. The rise of cooking temperature is an integral part of a normal cooking cycle. As such the initial delignification phase can only be included with a specially designed experiment which is out of the scope of this study.

✦ The figure shows that the rate of delignification both in the bulk and residual phases increase with addition of AQ of increase in sulphidity. This is wholly in agreement with literature of wood pulping (5,8,22,23,31,32). In the bulk phase kraft$_{15}$ cook shows the slowest rate. The delignification rate enhanced by about 30% on addition of AQ in low sulphidity pulping. Soda + AQ and kraft$_{25}$ delignifies almost with the same delignification rate. This is however, slightly more rapid than kraft$_{15}$ cook.

In the residual phase the delignification slows down remarkably. The slow rate of delignification in this phase might be due to some inextricably bound lignin (15) or grafting of modified lignin on the cellulose crystallite (11,16). The effect of sulphidity and AQ on the delignification in the residual phase behaves almost similarly as in the bulk phase. The rate constants are given in Table 1.

Table 1.

Delignification rate constants for the bulk (Kb) and residual (kr) phases in alkaline pulping of muli bamboo

pulping processes	$K_b \cdot 10^3$ (Min^{-1})	$K_r \cdot 10^3$ (Min^{-1})
Kraft$_{15}$	11.16	1.12
Kraft$_{25}$	11.21	1.20
Soda + AQ	11.93	1.53
Kraft15 + AQ	14.57	1.72

Soda + AQ, showing almost identical values of rate constants compared with kraft$_{25}$ favours replacement of normal kraft pulping by it. At low sulphidity pulping (sulphidity 15%) addition of AQ seems to be more attractive even to normal karft or soda + AQ as far as the delignification is concerned.

Dissolution of carbohydrates

The carbohydrate yield is plotted as a function of lignin yield in fig.2. The dissolution of carbohydrates with lignin removal in the initial phase is not clear.

However, it is known that the dissolution of carbohydrates in this phase is independent of the sulphidity (4). The figure shows a milder dissolution of carbohydrates on addition of AQ in low sulphidity kraft pulping. The behaviour of the carbohydrates at the transition point between the initial and bulk phases is clear, resulting in highest carbohydrate at the transition point is almost the same in soda + AQ and karaft$_{25}$.
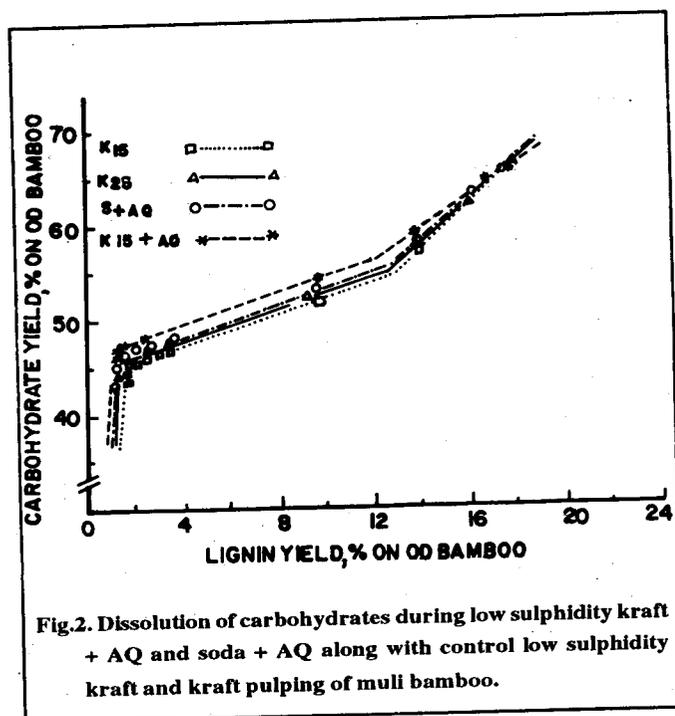
Fig.2. Dissolution of carbohydrates during low sulphidity kraft + AQ and soda + AQ along with control low sulphidity kraft and kraft pulping of muli bamboo.

Fig. 2 also shows that in the bulk phase the dissolution of carbohydrates is smallest with kraft$_{15}$ + AQ cook. Soda + AQ and karft$_{25}$ shows almost similar trends in carbohydrate dissolution. Kraft$_{15}$ cook had the highest dissolution of carbohydrates. The higher carbohydrate yield with kraft$_{25}$ is due to the more selective action of hydrosulphide ion thereby increasing the rate of delignification. The observed increase in carbohydrate yield at a given lignin content in kraft$_{15}$ + AQ and soda + AQ is a combined effect of catalized delignification and oxidative stabilization of the end groups in the carbohydrates (19,20). Soda + AQ pulping shows almost the same response as in kraft$_{25}$. Thus, soda + AQ pulping of muli bamboo is a preferable alternative to the kraft process. In the residual phase the carbohydrate yield with kraft$_{15}$ + AQ is higher than with kraft$_{15}$ cook. The yield with soda + AQ and karft$_{25}$ approaches each other. The carbohydrate yield drops rapidly in this phase. Consequently, commercial alkaline pulping of muli bamboo should be confined in the bulk phase.

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Pulp yield

The plot of pulp yield against chlorine number is given in Fig. 3 which shows no clear picture in the initial phase. But it is seen in the figure that the pulp yield is
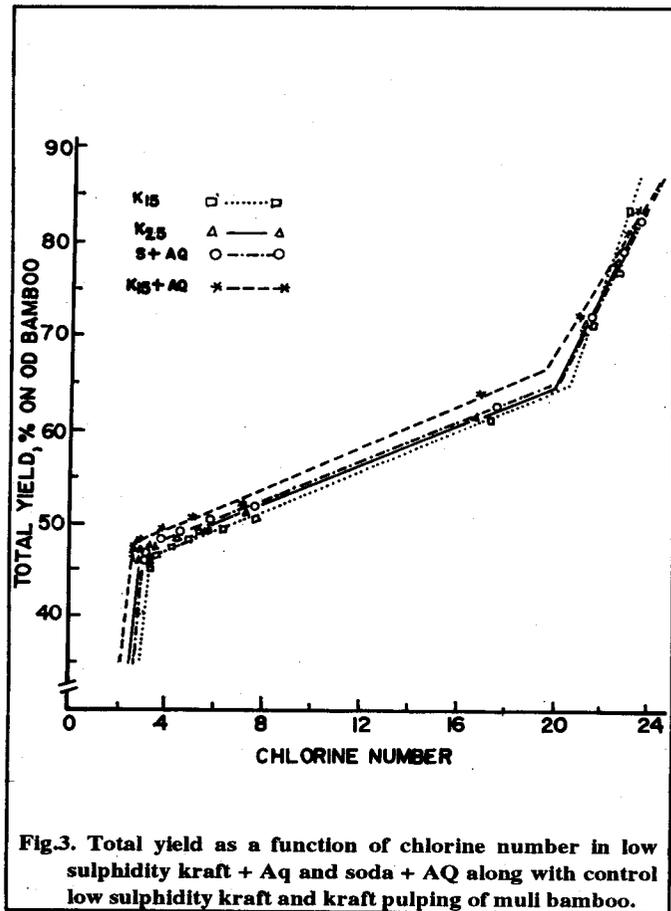
Fig.3. Total yield as a function of chlorine number in low sulphidity kraft + Aq and soda + AQ along with control low sulphidity kraft and kraft pulping of muli bamboo.

maximum in kraft$_{15}$ + AQ pulping. In the bulk phase the pulp yield is in the order kraft$_{15}$ + AQ > soda + AQ > kraft$_{25}$ > kraft$_{15}$. This is obviously because AQ accelerates the dissolution of lignin and suppresses the endwise peeling degradation of carbohydrates (20). It is seen that about 2.5% higher total yield (on od bamboo) is obtained in kraft$_{15}$ + AQ pulping at a chlorine number of 4 compared to that obtained in kraft15, and about 1% higher than from kraft$_{25}$ or soda + AQ cook. It is also known that the pulp yield increases with the increase of sulphidity (3). Thus, the total pulp yield in kraft$_{25}$ is higher than kraft$_{15}$. The gain in yield amounts to about 0.8% on od bamboo at a chlorine number of 4. The figure also shows that the yield decreases more rapidly towards the end of cook. The turning of the yield decline occurs at a higher chlorine number when the sulphidity is lower or no AQ is used.

Pentosans dissolution

Fig. 4. illustrates how sulphidity and AQ affect the dissolution of pentosans. It is observed that the pentosans content when plotted against residual lignin in bamboo exhibits similar trends as with carbohydrate dissolution (Fig. 2). In the initial phase the dissolution of pentosans is not clear. In the bulk phase the dissolution of pentosans is in order kraft$_{15}$ + AQ < soda + AQ < kraft$_{25}$ < kraft$_{15}$.
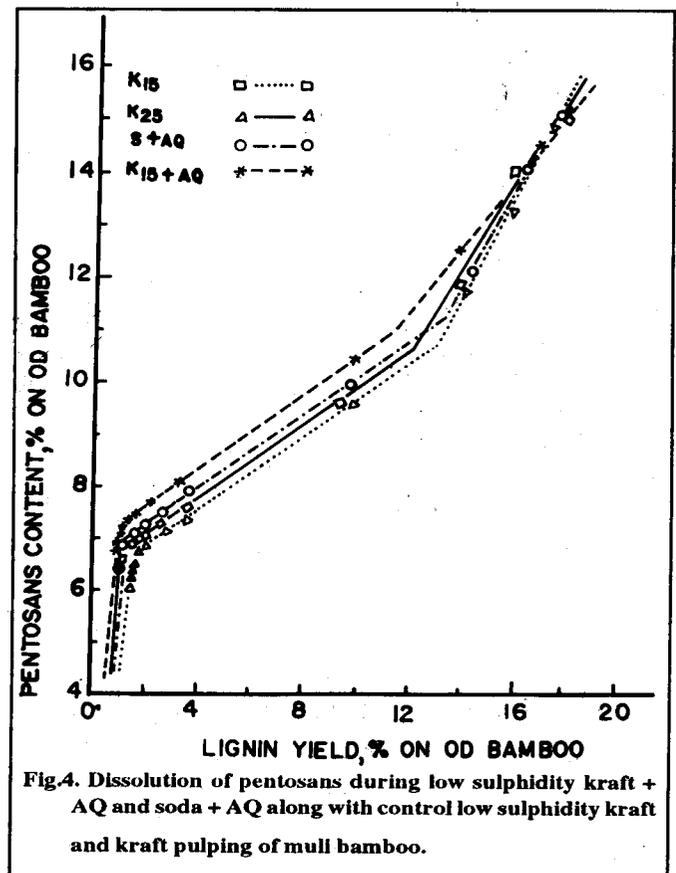
Fig.4. Dissolution of pentosans during low sulphidity kraft + AQ and soda + AQ along with control low sulphidity kraft and kraft pulping of muli bamboo.

Alkali consumption

The consumption of active alkali as a function of residual lignin in bamboo pulping is shown in Fig. 5. The figure again indicates that the initial phase does not show a clear picture on the consumption of alkali. However, kraft$_{15}$ consumes more alkli. The alkali consumption is in conformity with dissolution of carbohydrates as shown in Fig. 2.

In the bulk phase, the alkali consumption at a particular lignin yield decrease with an increase in

sulphidity or addition of AQ. This is due to the lower degree of degradation of carbohydrates. The figure shows that the consumption of alkali for soda + AQ cook is almost the same as that of kraft$_{25}$ cook. Kraft$_{15}$ consumes the highest alkali. The alkali consumption rapidly increases in the residual phase when the removal of lignin is very poor. This means that in the residual phase most of the alkali is consumed by carbohydrate reactions. But the rate of alkali consumption decreases with an increase in the sulphidity or AQ addition. Thus, kraft$_{15}$ + AQ pulping shows lower consumption of alkali than kraft$_{25}$ or kraft$_{15}$.
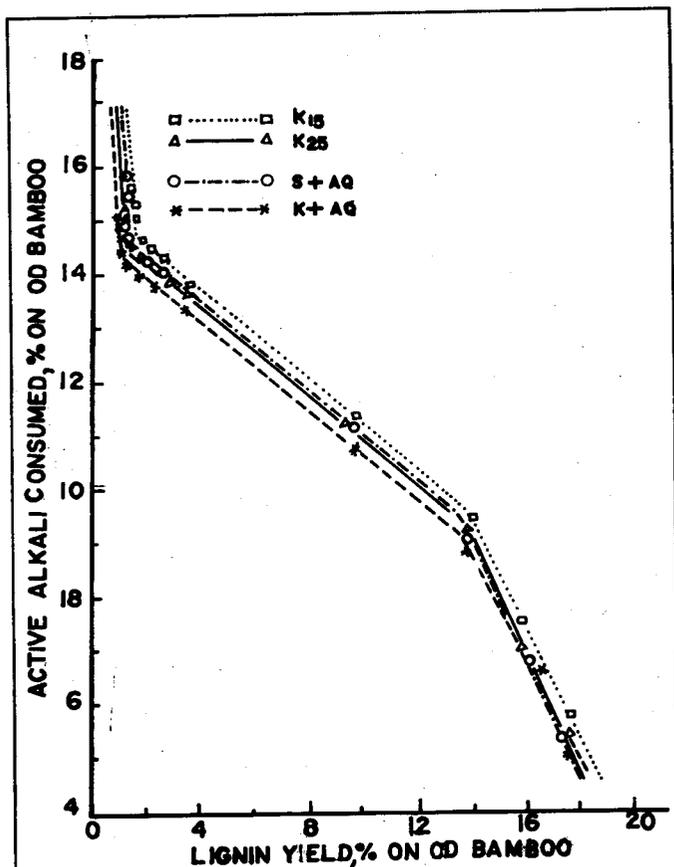
Fig.5. Consumption of active alkali during delignification in low sulphidity kraft + AQ and soda + AQ along with control low sulphidity kraft and kraft pulping of muli bamboo.

Transition point

The result in Figs. 1,2,4 and 5 shows that the first transition point between the initial and bulk phases, and the second transition point between the bulk and residual phases tend to change with increase in sulphidity or

addition of AQ. The behaviour of these pionts are summerised in Tables 2 and 3.

Table 2.

Behaviour of the first transition point with addition of AQ or increase in sulphidity in alkaline pulping of muli bamboo

Pulping Process	Lignin yield* %	Carbohydrate Yield* %	Active alkali consumption* as NaOH %	Pentosan content* %
Kraft$_{15}$	13.2	54.9	9.60	10.6
Kraft$_{25}$	12.9	55.5	9.56	10.4
Soda + AQ	13.0	55.6	9.50	11.0
Kraft$_{15}$+AQ	12.6	56.3	9.33	11.2

% on oven dry bamboo
* Average values from the plot of carbohydrate yield and alkali consumption against lignin yield

The first transition point occurs at the highest lignin yield, lowest carbohydrate yield with the highest alkali consumption in kraft$_{15}$. The lignin yield and alkali consumption at the transition point decreases and the carbohydrate yield increases with an increase in sulphidity or addition of AQ. Soda + AQ and kraft$_{25}$ retains almost the same amount of lignin and carbohydrates and consumes almost the same quantity of active alkali at the transition point. The transition point occurs at the lowest lignin yield, the highest amount of carbohydrates and the lowest alkali consumption in kraft$_{15}$ + AQ cook. The pentosans content at the transition point is the highest in the kraft$_{15}$ + AQ and soda + AQ cooks. This is because AQ promotes delignification and leads to oxidative stabilization of the end groups of carbohydrates.

Table 3.

Behaviour of the second transition point addition of AQ or increase in sulphidity in alkaline pulping of muli bamboo

Pulping Process	Lignin yield* %	Carbohydrate Yield* %	Active alkali consumption* as NaOH, %	Pentosan content* %
Kraft$_{15}$	1.40	45.4	15.0	6.7
Kraft$_{25}$	1.35	46.0	14.4	6.8
Soda + AQ	1.34	46.2	14.7	7.0
Kraft$_{15}$+AQ	1.29	47.2	14.2	7.3

% on od bamboo
* Average values from the plot of LnL against cooking time and alkali consumption against lignin yield

Table 3. shows the location of the second transition point. It shifts to different lignin contents with AQ addition or increase in sulphidity. With $kraft_{15}$ + AQ the residual phase turns at the lowest lignin yield. With soda + AQ the transition point shifts to a higher value of lignin than with $kraft_{15}$ + AQ. However, with soda + AQ, the transition point occurs at almost the same lignin yield as with $kraft_{25}$. $kraft_{15}$ cook reaches to the residual phase from the highest lignin content.

The study shows that the second transition point shifts to a lower lignin content on addition of AQ or increase in sulphidity. The effect of sulphidity is in conformity with the literature (31,33). The effect of AQ also is in confirmity with the hypothesis that any agent which accelerates the rate of delignification shifts the second transition point to a lower lignin content (34). It is seen that the second transition point occurs at the highest carbohydrate yield, the highest pentosans yield and the lowest alkali consumption with $kraft_{15}$ + AQ. On the contrary, it occurs at the lowest carbohydrate yield, the lowest pentosans content and the highest alkali consumption with $kraft_{15}$ cook. With soda + AQ, the properties of the transition point with regard to carbohydrate yield, alkali consumption and pentosans content is almost similar to $kraft_{25}$.

A low sulphidity pulping i.e., a 15% sulphidity may bring the delignification to the residual phase if the targetted lignin yield is lower than 1.4% on od bamboo or a chlorine number of 3.39 or a kappa number of about 20. Then, there is a danger of degradation of the carbohydrates resulting in a serious loss in pulp yield which can be minimised by increasing sulphidity or addition of AQ. With 25% sulphidity, the second transition point occurs at a lignin yield of 1.35, i.e., a chlorine number of 3.22 or a kappa number of about 19. With $kraft_{15}$ + AQ, this transition point occurs at a lignin yield of 1.29 i.e., a chlorine number of 2.98 or a kappa number of about 18. Similarly soda + AQ cook causes the transition point to shift at a lignin yield 1.34 i.e. a chlorine number of 3.11 or a kappa number about 19.

Normally, kraft pulping of muli bamboo to produce a bleachable grade pulp is continued to a kappa number of 20. Hence use of any of the above pulping processes seems to restrict the cook within the bulk delignification phase.

CONCLUSIONS

The study shows that three phases of delignification can be distinguished in alkaline pulping of muli bamboo. The rate of delignification is enhanced by an increase in sulphidity or an addition of AQ. There is practically very little variation in the rate of delignification between 15-25% sulphidity. In the bulk and residual phase, the delignification rate constant is in the order $kraft_{15}$ + AQ > soda + AQ > $kraft_{25}$ > $kraft_{15}$ cook.

The first and second transition points turn out at the lowest lignin yield with AQ in $kraft_{15}$ pulping. These points shift to the highest lignin content in normal $kraft_{15}$ pulping. Soda-AQ behaves almost similar to that of in the $krafts_{25}$ in these aspects.

$Kraft_{15}$ + AQ leads to the highest carbohydrate yield with the lowest alkali consumption. On the contary, normal $kraft_{15}$ shows the lowest carbohydrate yield and the highest alkali consumption. Soda + AQ and $kraft_{25}$ pulping are almost indentical in carbohydrate retention and alkali consumption. The loss of carbohydrates is maximum in the initial and residual phases and minimum in the bulk phase.

ACKNOWLEDGEMENTS

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