

Delignification of *Phragmites karka* – a Wetland Grass – by Soda Pulping Process

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Phragmites karka, a common wetland grass, was delignified by the soda pulping process, and all the operating parameters were optimized. The effects of both anthraquinone and surfactant on pulp yield and kappa number at optimum pulping conditions were also investigated. The pulp was beaten at different beating levels to optimize different mechanical strength properties. A detailed morphological study of soda-AQ pulp of *P. karka* was conducted by scanning electron microscopy.

Keywords: *Phragmites karka*; Soda pulping; Anthraquinone; Surfactant; Paper properties

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INTRODUCTION

Bob Flynn, Director, International Timber, RISI, stated in Brussels (Viewpoint) in 2007 that forest cover in India is about 67.8 million ha, or 20.6% of the total nation's surface area. It is rather disappointing that the per capita forest area is only 0.8 ha/person, one of the lowest in the world. Total fibre consumption for the production of paper and paperboard in India will nearly be doubled between 2006 and 2016, growing from 7.4 to 13.7 million tonnes. India's total wood fibre deficit is forecast to increase at an annual rate 11.3% by 2016 (Flynn 2007). The lack of wood fibers has forced the paper industry to use other non-conventional fibrous raw materials. To narrow the gap between demand and supply, many fast-growing annual and perennial plants with high biomass have been identified and their suitability for pulp production has been evaluated. These non-woody plants include: *Hibiscus cannabinus* and *Hibiscus sabdariffa* (Dutt *et al.* 2009), *Sesbania aculeata* and *Sesbania sesban* (Dutt *et al.* 2004), *Cannabis sativa* and *Ipomea carnea* (Dutt *et al.* 2008), *Arundo donax* (Shatalov and Pereira 2005), lemon and sofia grasses (Kaur *et al.* 2011; Kaur *et al.* 2013), *Cajanus cajan* and *Eulaliopsis binata* (Dutt *et al.* 2002; Dutt *et al.* 2005), as well as agricultural residues such as wheat straw (Singh *et al.* 2011), rice straw (Rodríguez *et al.* 2008), and sugarcane bagasse (Agnihotri *et al.* 2010), fast growing hardwoods such as *Leucaena leucocephala* (Malik *et al.* 2004) and *Anthocephalus cadamba* (Lal *et al.* 2010), and waste papers (Dutt *et al.* 2012). Due to short growth cycles, low lignin, and high hemicellulose contents, non-woody plants have several advantages over woody plants, resulting in decreased energy and chemical consumption during the cooking process (Hurter and Riccio 1998).

Phragmites karka, the common reed, is a large perennial grass of the family Poaceae found in wetlands throughout temperate and tropical regions of the world. *Phragmites karka* is a short-fibred plant (Kumar 2013). The admixture of these pulps with long-fibred pulps, such *Eulaliopsis binata* or bamboo, is essential for the successful use of this reed for the production of paper on a commercial paper machine. This grass is abundantly available in the northeastern part of India, but the transportation of reed grass

to places outside northeast India is not economically viable. *P. karka* contains holocellulose, α -cellulose, hemicelluloses, and lignin 75.67 ± 0.63 , 50.55 ± 0.4 , 25.12 ± 0.54 and $22.00\pm 0.32\%$, respectively (Kumar 2013). Whole plant production estimated by the maximum-minimum method was 9960 g/m^2 . *P. karka* is the most productive species of this wetland genus, showing a maximum production of 1091 g/m^2 as the net annual production (Hocking 1989). It would be better to establish a paper mill in that region for exploitation of reed grass. The availability of bamboo to that region could be used for admixing with short-fibred pulp of *Phragmites karka* for the manufacture of quality paper.

The present study is focused on delignification of *Phragmites karka*, a new, renewable, non-conventional, and hitherto unexploited source of cellulose fibers, by the soda-AQ pulping process. Various operating parameters were also optimized, such as alkali dose, cooking temperature, cooking time, AQ dose, surfactant doses, and AQ + surfactant doses.

EXPERIMENTAL

Pulping Studies

Phragmites karka was collected from the Mashkhara river, flowing in the vicinity of the village of Chilkana in Saharanpur district, located in the foothills of the Shivalik Hills, Western Uttar Pradesh (India) during the months of May and June. *P. karka* was manually disintegrated into small chips of length 4 to 6 cm and stored in polythene bags after drying in sunlight. The dried *P. karka* chips were delignified by the soda pulping process in a Weverk electrically-heated rotary digester of 0.02 m^3 capacity with four bombs, each of 1 L capacity. The *P. karka* was cooked at different cooking conditions. The active alkali doses were varied from 9 to 19% (as Na_2O), the maximum temperature was varied from 140 to 170 °C, the time at optimum temperature was varied from 30 to 220 min, and the liquor-to-raw material ratio was fixed at 4:1.

At the optimum cooking conditions, anthraquinone (AQ) was added from 0.0 to 0.2% (on an oven-dry raw material basis) to study its effect on pulp yield, screening rejects, and kappa number. *P. karka* was also delignified at different surfactant (polyethylene glycol, PEG 1000) doses, varying from 0.0 to 0.2% (based on o.d. raw material) with and without AQ at optimum cooking conditions. After the completion of cooking, the pulps were washed on a laboratory flat stationary screen with a 300 mesh wire bottom for the removal of black liquor with tap water. After washing, the pulp was disintegrated and screened through a Weverk vibratory flat screen with a slot size of 0.15 mm. The screened pulp was washed, pressed, crumbled, and air dried. The pulps were evaluated for kappa number (TAPPI T236 cm-85), pulp yield, lignin (TAPPI T 222 om-02), and screening rejects. The yield of pulp and screening rejects were calculated as a percentage by weight on the moisture-free basis.

Preparation of Laboratory Handsheets and Testing

The unbleached pulp of *P. karka* was beaten in a PFI mill (TAPPI T 248 sp-00) at different beating levels. Laboratory handsheets of 70 g/m^2 were prepared on a British sheet former (TAPPI T205 sp-02), pressed, air-dried under atmospheric conditions, and tested for various physical strength properties, such as tear index (TAPPI T 414 om-98), tensile index (TAPPI 494 om-01), burst index (TAPPI T 403 om-97), and double folds

(TAPPI T 423 cm-98). The laboratory handsheets were preconditioned at a relative humidity of $65 \pm 2\%$ and temperature of $27 \pm 1^\circ\text{C}$ (TAPPI 402 sp-03).

Scanning Electron Microscopy

The detailed morphological study of soda-AQ pulp of *P. karka* was conducted using a scanning electron microscope (SEM, Leo 435 VP, England). A pulp sample was taken and subjected to fixation using 3% (v/v) glutaraldehyde and 2% (v/v) formaldehyde (4:1) for 24 h. After primary fixation, samples were washed thrice with double-distilled water and then treated with alcohol gradients of 30, 50, 70, 80, 90, and 100% for dehydration. The samples were kept for 15 min at each alcohol gradient up to 70%, and for 30 min at each subsequent alcohol gradient. After treatment with 100% alcohol, samples were air-dried and examined under SEM using the gold shadowing technique (Gabriel 1982). Electron photomicrographs were taken at 15.00 kV using detector SE1 at the desired magnifications.

RESULTS AND DISCUSSION

Influence of Alkali Charge

Active alkali charge (AA) is one of the main parameters in the soda pulping process, as it is directly related to delignification and the breaking down of carbohydrates. It thus has a major effect on pulp yield, screening rejects, and kappa number. Figure 1 reveals that the screened pulp yield increased with increasing active alkali doses from 9 to 15% (as Na_2O) and then declined. Both kappa number and screening rejects decreased sharply and, after the sharp decline, both parameters remained almost constant.

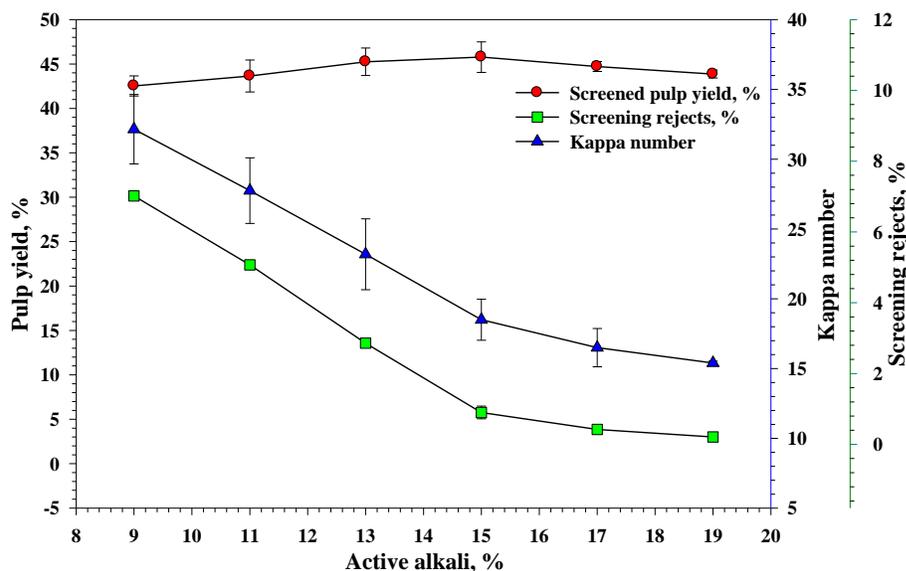


Fig. 1. Effect of active alkali doses on screened pulp yield, screening rejects, and kappa number during soda pulping of *P. karka* (Cooking conditions: Liquor to raw material ratio: 4:1, temperature: 150°C , digester pressure: 5.0 kg/cm^2 , time from room temperature to $105 \pm 2^\circ\text{C}$: 45 min, time from 105 to maximum temperature $150 \pm 2^\circ\text{C}$: 55 min; and time at maximum temperature, $150 \pm 2^\circ\text{C}$: 120 min)

In an alkaline solution, breaking of glycosidic bonds occurs, which leads to the separation of lateral chains and the main chains of carbohydrates. These cleavages consequently result in the formation of soluble compounds with low molecular weight and a reduction in pulp yield and kappa number. The active alkali charge of 15% (as Na_2O) is considered to be optimal for *P. karka* with 45.78% screened pulp yield and a kappa number of 18.50.

Influence of Temperature and Time

Figure 2 shows the curves plotted from residual lignin vs. reaction time at reaction temperatures varying from 140 to 170 °C, cooking time from 0.5 to 4.0 h, with 15% active alkali (as Na_2O), and a liquor-to-wood ratio of 4:1. The curves with steeper slopes are related to rapid solubilization of the bulk of lignin (bulk delignification). The curves with gentler slopes are related to the slow solubilization of the residual lignin (residual delignification). The bulk delignification phase represents the removal of easily accessible lignin mainly located in the middle lamella region. The residual delignification phase relates to the removal of lignin in the primary wall, secondary wall layers (S_1 , S_2 , S_{12} , S_3 , and S_{23}), and the central inter-connecting cavities.

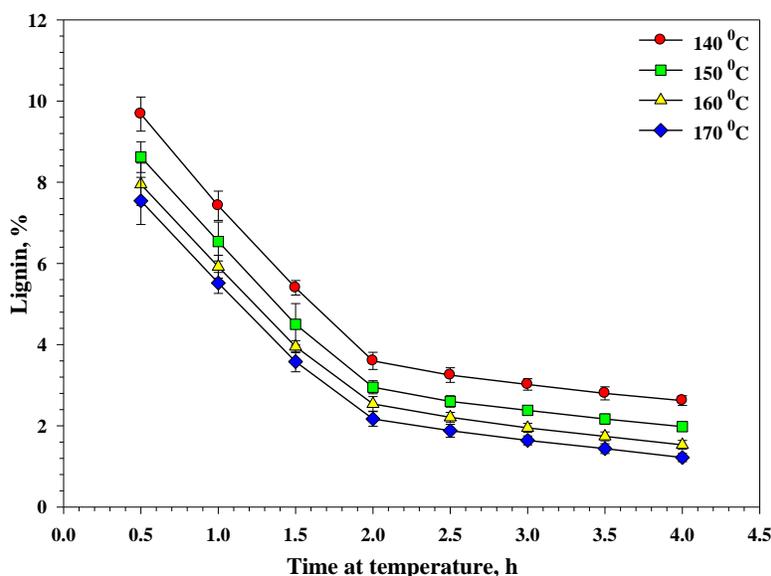


Fig. 2. Curves of lignin vs. different reaction times at different cooking temperature during soda pulping of *P. karka* (Cooking conditions: Liquor to wood ratio: 4:1, temperature: 160, °C, active alkali: 15% (as Na_2O), digester pressure: 5.0 kg/cm^2 , time from room temperature to 105 ± 2 °C: 45 min, and time from 105 to maximum temperature: 55 min)

A lower pulp lignin content was obtained at 150 ± 2 °C; therefore, this temperature may be taken as optimum. Beyond 150 ± 2 °C, in addition to the peeling reactions, alkaline hydrolysis (depolymerization) of carbohydrates takes place more rapidly and the pulp is subjected to further degradation reactions, *i.e.* secondary peelings (McGinnis and Shafizadeh 1980). After transition points, the curves are almost straight lines, indicating that bulk delignification is over. There was clearly less lignin at any given time when the temperature was higher than 150 °C. Therefore, it did not make sense to continue pulping longer than 2 h. The curves plotted from residual lignin and reaction time (Fig. 2) and pulp yield and reaction time (Fig. 3) reveal that the drop in pulp yield beyond the

maximum cooking time of 2.0 h was minimal and reduction in lignin content was insignificant. Therefore, based on these findings, a maximum cooking time of 2.0 h and cooking temperature 150 °C may be considered as the optimum cooking conditions for the soda cooking of *P. karka*. Other researchers have also reported similar findings from reaction kinetics studies on various agro-based and non-woody cellulosic fibrous raw materials. For example, Dutt *et al.* (2008) reported an optimum cooking time of 2.0 h at 165 °C for *Ipomea carnea* and 2.0 h at 165 °C for *Cannabis sativa*; Kaur *et al.* (2011) reported a cooking time of 1.5 h at 150 °C for *Cymbopogon flexuosus* (lemon grass), and Singh *et al.* (2011) reported a cooking time of 1.0 h at 150 °C for *Triticum aestivum* PBW-323 L. (wheat straw).

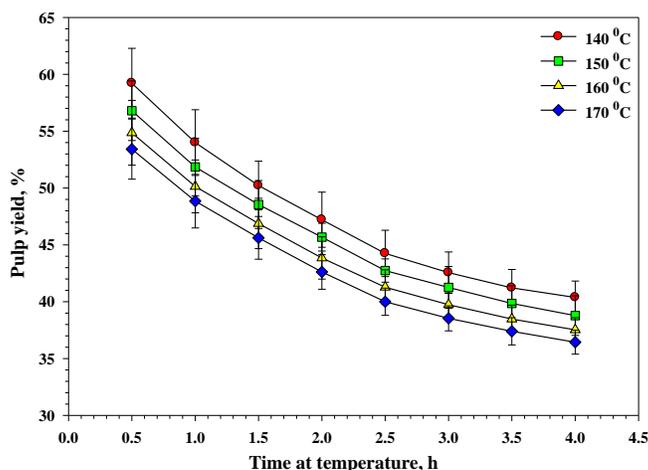


Fig. 3. Curves of pulp yield (%) vs. different reaction times at different cooking temperatures during soda pulping of *P. karka* (Cooking conditions: Liquor to wood ratio: 4:1, temperature: 160, °C, active alkali: 15% (as Na₂O), digester pressure: 5.0 kg/cm², time from room temperature to 105±2 °C: 45 min and time from 105 °C to maximum temperature: 55 min)

Influence of Time

Figure 3 shows the effect of maximum cooking time on screened pulp yield, screening rejects, and kappa number during soda pulping of *P. karka*, while other variables were kept constant, such as an alkali dose 15% (as Na₂O), a liquor-to-raw material ratio of 4:1, and a maximum cooking temperature 150 °C. The screened pulp yield increased from 42.53 to 45.72% and kappa number decreased from 32.84 to 18.52 units when the cooking time was varied from 30 to 120 min. A decrease in screened pulp yield was observed, while kappa number remained almost constant. This is due to increased contact time between alkali and carbohydrates, which means that the extent of alkaline hydrolysis taking place during soda cooking also increased. This results in carbohydrate degradation, and consequently a reduction in the pulp yield. Hence, the optimum cooking time for soda pulping of *P. karka* is 120 min (Fig. 4).

Effect of AQ Dose on Soda Pulping Process

Different doses of anthraquinone, varying from 0.00 to 0.2%, were applied at the optimal pulping conditions for *P. karka*. The use of 0.1% anthraquinone resulted in a major reduction in kappa number and screening rejects with a slight improvement in the pulp yield (Fig. 5). The kappa number and screening rejects for *P. karka* decreased by 5.7

units and 0.80% respectively, with an increase of 0.66% in screened pulp yield. Other researchers also have reported a reduction in kappa number and improvement in pulp yield such as 6 units and 0.30% for *Ipomea carnea* and 7 units and 0.40% *Cannabis sativa* at an AQ dose of 0.1% (Dutt *et al.* 2008), 9.6 units and 0.20% *Hibiscus sabdariffa* and 8.7 units and 0.10% for *Hibiscus cannabinus* at an AQ dose of 0.05% (Dutt *et al.* 2009), and in 3.6 units and 2.6% for *Saccharum officinarum* - CO 89003 (sugarcane bagasse) with a 0.1% AQ dose (Agnihotri *et al.* 2010).

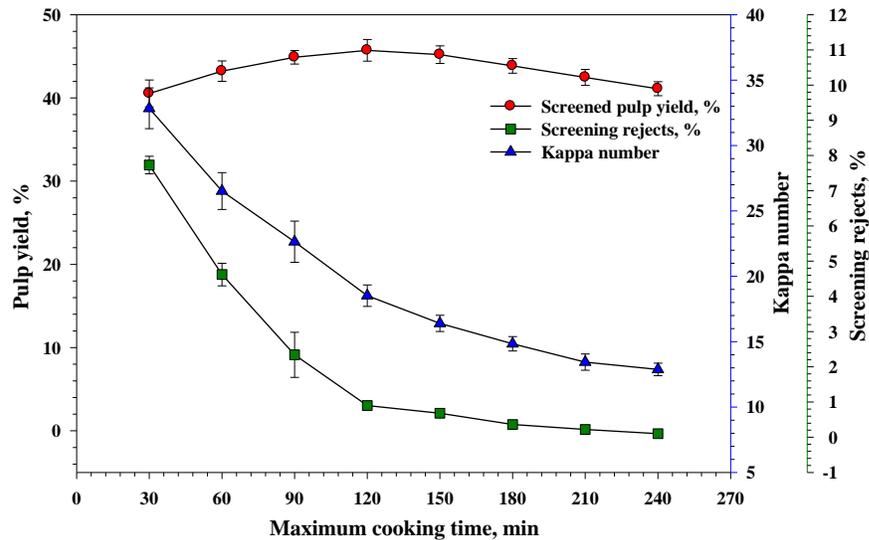


Fig. 4. Effect of cooking time on screened pulp yield, screening rejects, and kappa number during soda pulping of *P. karka* (Cooking conditions: Liquor to wood ratio: 4:1, active alkali: 15% (as Na_2O), digester pressure: 5.0 kg/cm^2 , time from room temperature to 105 ± 2 °C: 45 min and time from 105 °C to maximum temperature: 55 min)

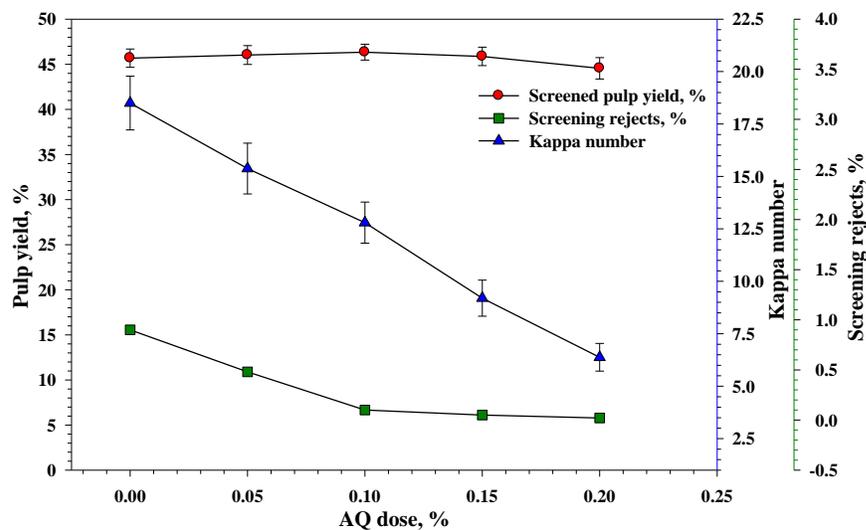


Fig. 5. Effect of anthraquinone (AQ) dose on screened pulp yield, screening rejects, and kappa number at optimum pulping conditions during soda pulping of *P. karka* (Liquor to raw material ratio: 4:1, active alkali: 15% (as Na_2O), digester pressure: 5.0 kg/cm^2 , time from room temperature to 105 ± 2 °C: 45 min, time from 105 °C to maximum temperature 150 ± 2 °C: 55 min and time at maximum temperature, 150 ± 2 °C: 120 min)

The reduction in kappa number and increase in pulp yield may be explained by the redox catalytic activity of AQ. AQ reacts with the reducing end of a carbohydrate polymer chain, stabilizes the end group (stopping reaction) against alkaline peeling, and produces the reduced form of AQ, *i.e.*, anthrahydroquinone (AHQ), which is soluble in an alkaline pH. In the second part of a reaction, the AHQ reacts with the quinone-methide segment of lignin polymer and thus increases the rate of delignification. Concurrently, AHQ is converted back to AQ, which can participate further in the redox cycle. AQ is typically used at about 0.1% on an oven-dry weight basis of the raw material as it goes through a cyclic process, and results in an increase in pulp yield and reduction in kappa number (Dutta and Biermann 1989).

Effect of Surfactant Dose on Soda Pulping Process

Different doses of surfactant (PEG 1000) varied from 0.00 to 0.2% was applied under the optimized cooking conditions for *P. karka*. A surfactant dose of 0.1% reduces the kappa number and screening rejects with a slight improvement in pulp yield of *P. karka* (Fig. 6). The kappa number and screening rejects for *P. karka* were decreased by 1.72 units and 0.82% respectively, with an increase of 0.79% in screened pulp yield.

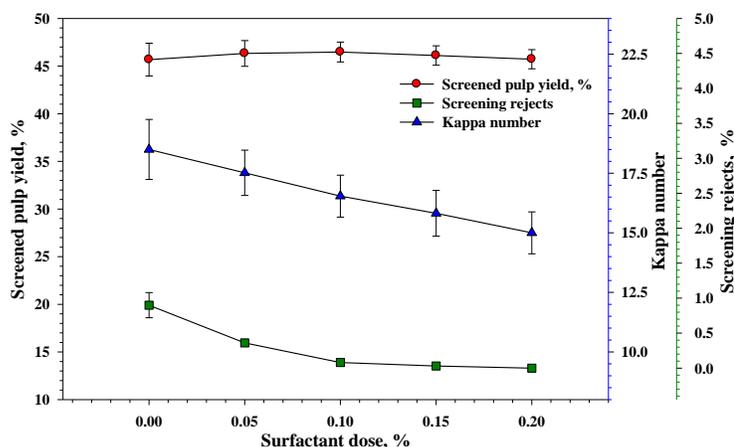


Fig. 6. Effect of surfactant (PEG 1000) doses on screened pulp yield, screening rejects, and kappa number at optimum pulping conditions during soda pulping of *P. karka* (Liquor to raw material ratio: 4:1, active alkali: 15% (as Na₂O), digester pressure: 5.0 kg/cm², time from room temperature to 105±2 °C: 45 min, time from 105 to maximum temperature 150±2 °C: 55 min and time at maximum temperature, 150±2 °C: 120 min)

Surfactants do not behave as chemical reactants, such as sodium hydrosulphide and sodium hydroxide of white liquor, nor do they behave as pulping redox catalysts, such as AQ. Surfactants affect the physico-chemical aspects of pulping (Chen 1996), which involve phase-boundary interactions of the exterior surfaces and interior lumen openings of wood chips with the cooking liquor. The addition of surfactant as a cooking additive reduces the surface tension between the cooking liquor and chips, allowing more thorough wetting of the chip surfaces and facilitating rapid penetration of liquor into the inner matrix of the chip. This gives rise to more uniform cooking with pulps of lower kappa number and screening rejects and improves residual active alkali in black liquor (Duggiralla 2000). The products of thermal degradation of extractives are deeply colored and add a dark color to the unbleached pulp (Forsskahl *et al.* 1998). Surfactant stops the

deposition of these products on the surface of fibers. When surfactant (PEG 1000) was used from 0.05 to 0.3% under optimum cooking conditions, it caused a further reduction in kappa number (0.48 units) and screening rejects (0.10%) with a slight improvement (0.06%) in pulp yield of *P. karka* (Fig. 7).

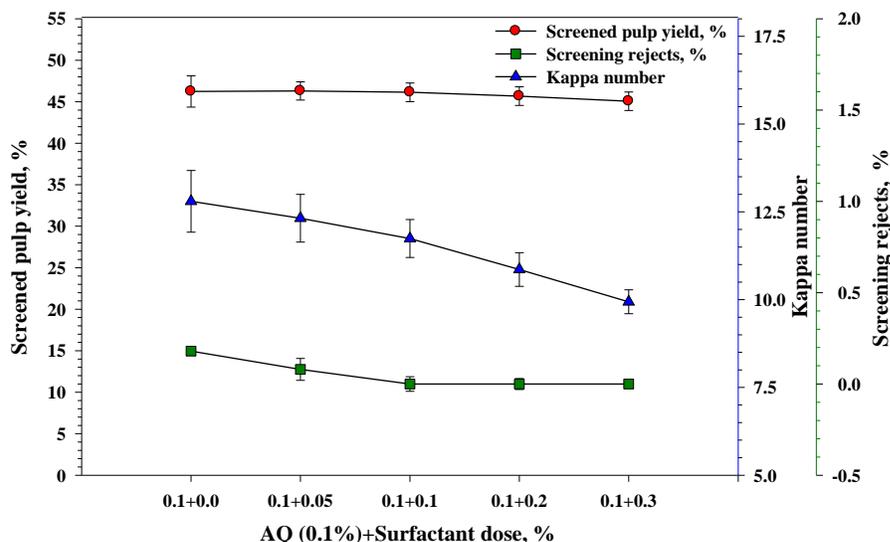


Fig. 7. Effect of different surfactant (PEG 1000) doses on screened pulp yield, screening rejects, and kappa number at optimum pulping conditions during soda pulping of *P. karka*, keeping AQ dose constant (0.1%)

The pulp evaluation results obtained at the optimum pulping conditions for soda, soda-AQ, and soda-AQ + surfactant pulping of *P. karka* are shown in Table 1.

Table 1. Optimum Pulping Results for Soda, Soda-AQ, and Soda-AQ + Surfactant Pulping of *P. karka*

Total pulp yield, %	Screened pulp yield, %	Screening rejects, %	Kappa number
Soda pulping			
46.58±0.60	45.68±1.72	0.90±0.18	18.50±1.28
Soda-AQ pulping			
46.42±1.11	46.34±1.88	0.10±0.02	12.80±0.98
Soda-AQ + surfactant pulping			
46.38±1.07	46.30±1.10	0.08±0.01	12.32±0.68

± refers standard deviation (Liquor to raw material ratio: 4:1, active alkali: 15% (as Na₂O), digester pressure: 5.0 kg/cm², time from room temperature to 105±2 °C: 45 min, time from 105 °C to maximum temperature 150±2 °C: 55 min and time at maximum temperature, 150±2 °C: 120 min, AQ dose 0.1% and surfactant dose 0.1%)

Mechanical Strength Properties

Table 2 presents the mechanical strength properties of *P. karka* pulp produced at an active alkali dose of 15% (as Na₂O) and maximum cooking time of 2.0 h at 150 °C. All mechanical strength properties, such as tensile index, double folds, and burst index,

increased with increasing beating level, up to 35 °SR, and then declined. During pulp beating, removal of the primary wall exposed the secondary wall layers. The primary wall is permeable to water but does not participate in bond formation. On the other hand, tear index (4.15 mNm²/g) increased with pulp beating, up to a beating level of 30 °SR, and then it declined. The reason for this is that the tearing energy required to pull the fibers from the mesh was slightly greater due to the development of hydrogen bonding among fibers after the removal of the primary wall. The work that has to be done to pull the fibers loose depends on the length of the fibers and the bond strength. At a beating level of 30 °SR, the inter-fiber bond strength will improve slightly. Therefore, the work done to pull the fibers from the mesh will be greater, and at a higher beating level, the fibers will start to break instead of being pulled out intact. Conversely, tensile index, double fold, and burst index depend on hydrogen bonding and improve with pulp beating. Therefore, a beating level of 35 ± 1 °SR is optimal for *P. karka*. Further, the addition of 0.1% AQ improved all of the mechanical strength properties (Table 2). This is due to the fact that AQ accelerates the delignification rate without degrading carbohydrates.

Table 2. Mechanical Strength Properties of Unbleached *P. karka* Soda Pulp at Optimum Pulping Conditions

Sl. No.	Drainage time (s)	Apparent density	Beating level, °SR	Tensile index, Nm/g	Tear index, mNm ² /g	Burst index, kPa·m ² /g	Double fold, number
Soda pulping							
1	6	0.62	15	18.52 ± 0.48	2.11 ± 0.09	0.7 ± 0.08	8.25 ± 0.82
2	16	0.67	30	40.20 ± 1.32	4.15 ± 0.16	2.62 ± 0.12	36.50 ± 1.2
3	22	0.70	35	43.80 ± 2.13	4.11 ± 0.22	3.05 ± 0.13	41.85 ± 2.2
4	24	0.72	40	42.68 ± 2.38	3.98 ± 0.25	2.96 ± 0.08	40.24 ± 2.2
5	28	0.74	45	39.22 ± 2.32	3.82 ± 0.15	2.72 ± 0.21	35.28 ± 2.1
Soda-AQ pulping							
6	6	0.63	15	19.28 ± 0.11	2.36 ± 0.11	0.82 ± 0.07	8.64 ± 0.92
7	15	0.68	30	45.62 ± 1.22	4.54 ± 0.17	3.28 ± 0.11	38.24 ± 1.4
8	21	0.72	35	49.22 ± 1.88	4.52 ± 0.24	3.61 ± 0.14	42.82 ± 1.9
9	23	0.75	40	47.18 ± 2.11	4.42 ± 0.19	3.48 ± 0.12	40.66 ± 2.1
10	27	0.76	45	43.86 ± 2.08	4.22 ± 0.16	3.18 ± 0.10	36.42 ± 2.2

± refers to standard deviation, pulp viscosity 28.46 cps.

SEM Studies of *P. karka* Fibers

The average fiber length, fiber width, cell wall thickness, and lumen diameter of *P. karka* were 0.65 mm, 16.8 µm, 4.86 µm, and 7.08 µm, respectively. SEM studies revealed that fibers in the soda-AQ pulp of *P. karka* were non-uniform; the cell wall was distinguished by longitudinal striations and transverse fractures with somewhat swollen fissures. Plates A and B show the lumen of the *P. karka* soda-AQ pulp fibers. As pulping continued, the association between the fibers was loosened firstly along radial planes, and ultimately the fibers remained stuck together only along those edges where several cells met and delignification was still incomplete. In addition, fibers showed signs of external fibrillation or formation of fibrils (plate B), which resulted in an increased area of contact for bonding. During alkaline cooking, some hemicelluloses (mainly xylan) were

solubilized and re-precipitated onto the surface of the fibers, as shown in plate A. Fibers lost their rigidity and collapsed readily after the removal of most of the lignin (plate C).

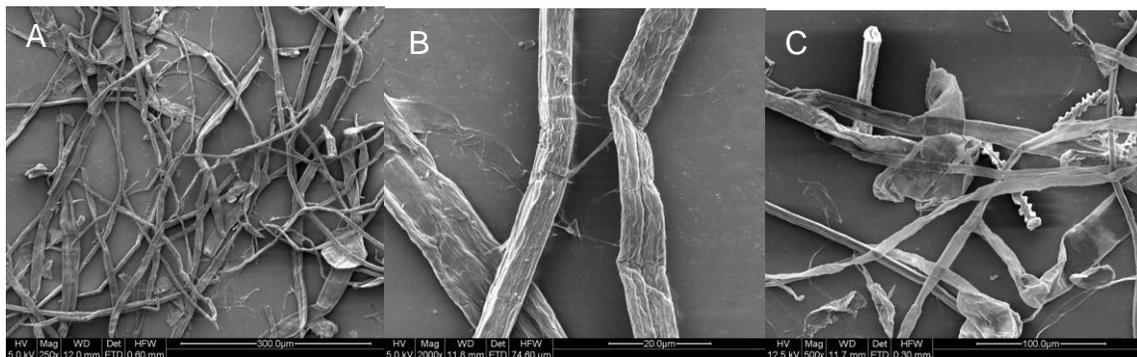


Fig. 8. Microphotographs of (A) completely smooth fibers of unbeaten soda-AQ pulp of *P. karka* with flattened fibers after pulping (250x), (B) fibers showing incomplete fibrillation and cracks (2000x), and (C) unbeaten soda-AQ fibers of *P. karka* showing flattened lumen (500x)

CONCLUSIONS

1. *P. karka* produced a screened pulp yield of 45.78% with kappa number 18.50 at an active alkali charge of 15% (as Na₂O), a maximum cooking time of 2.0 h, and a cooking temperature 150 °C.
2. An introduction of 0.1% AQ mitigated kappa number by 5.7 units and improved pulp yield by 0.66%. Further, the addition of 0.1% AQ improved all of the mechanical strength properties. Use of 0.1% surfactant (PEG 1000) mitigated the pulp kappa number by 1.72 units and improved pulp yield by 0.79%.
3. A beating level of 35 ± 1 °SR was taken as the optimum for *P. karka*.
4. SEM studies showed that as pulping continued, the association between the fibers was loosened, firstly along radial planes, and ultimately the fibers remained stuck together only along those edges where several cells met and delignification was still incomplete.

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