Alkaline Sulphite Anthraquinone Methanol (ASAM) Pulping of Jute Fibre

M. Sarwar Jahan*, D.A. Nasima Chowdhury and M. Khalidul Islam

Pulp and Paper Research Division, BCSIR Laboratories, Dhaka, Dr. Qudrat-E-Khuda Road, Dhaka-1 205, Bangladesh

Keyword : Jute, Anthraquinane ASAM pulping, ECF and TCF bleaching, Kappa number, Oxygen delignification, Pulp yield, Strength properties.

ASAM pulping of whole jute plant was done with varying alkali ratio (Na_2SO_3/NaOH), alkali charge, methanol ratio in the pulping liquors, cooking time and temperature. The pulp yield and Kappa number were decreased with decreasing alkali ratio and with increasing alkali charge and temperature. Methanol in the pulping liquor increased selectivity. At optimum conditions of ASAM pulping, where alkali ratio was 80%, alkali charge 22%, MeOH 25% and cooking time 1 h at 170°C produced pulp yield of 56.6% with Kappa number 39.2. ASAM pulping of whole jute plant resulted in high yield, excellent strength properties and unbleached brightness than the kraft pulp. Oxygen bleaching reduced kappa number by 35% and increased brightness to 56%. Therefore, less chlorine dioxide was needed in subsequent ECF bleaching. ASAM pulp from whole jute plant retained superior strength properties after ECF bleaching. Jute fibre was also cooked in the same optimum conditions of whole jute plant. Pulp was produced with very low Kappa number (7.6), very yield (61.5%) and brightness (63%). The strength properties of ASAM pulp from jute fibre were very high. ECF bleaching showed higher strength and brightness as compared to kraft pulp from jute.

INTRODUCTION

Jute has a long historical role in the socioeconomic development of Bangladesh. Once jute was considered as golden fibre of Bangladesh. The export of jute and related products accounts for a significant portion of total export. In addition, it provides considerable employment opportunities to the country's work force. Traditionally jute was used in backing, sacking, gunny bag, hasein etc. In recent years, jute has faced stiff competition from synthetics. Therefore, traditional uses of jute have declined. As a result, its demand in local and overseas markets has been shrunk. This has caused jute prices drop and jute growers are the ultimate victims. Therefore, diversified usage is needed to regain the lost glory of jute. The chemical and morphological characteristics of jute favor it as pulping raw material [1]. Therefore, many studies have been done on the pulping of jute in home and abroad [2-6]. But a lot of problems like blowing, washing, screening, recovery etc. have risen to commercialize the pulping process of jute. Another drawback is the price of jute three times higher than the conventional raw materials of pulp mills. Therefore, it is utmost need to find out new pulping processes, which overcome all limitations in the conventional jute pulping.

The sulfite pulps have higher initial brightness, the process has become less competitive due to the restriction of concerning suitable raw materials and lower strength properties of the sulfite pulps. In 1985, a new pulping process was developed by Patt and Kordsachia [7] based on alkaline sulfite pulping with addition of anthraquinone (AQ) and methanol to achieve high delignification rate. The process is known as ASAM process. It is found that cooking liquor containing Na_2SO_3, NaOH or Na_2CO_3 and AQ in a water/methanol is an excellent lignin solvent. Sodium sulfite renders lignin soluble and anthraquinone enhance delignification by its catalytic effects. Therefore, lignin degradation reactions are not restricted to structures with benzyl alcohol or alkyl ether groups as in neutral or alkaline sulfite pulping and therefore, a strongly increased delignification is possible.

Softwoods and hardwood can be delignified with a sulphite liquor containing methanol and 0.5-3.0 w-% sulphur dioxide [8]. Compared to the kraft process, the ASAM process uses 10 % less inorganic chemicals. The pulp can be bleached without chlorine and it has strength properties superior to those of kraft pulp. The methanol content of the pulping liquor can be dropped from 35 % to 20 % without impairing the pulp quality. In the pressure relief of the digester 95 % of the methanol is recovered, which can be improved to over 97% by steam stripping. The recovery of the inorganic chemicals is similar to the recovery in sulphite pulping. The ASAM method could be used to produce beech pulps with very low residual contents [9].

ASAM process showed a good alternative to kraft process for both softwood and hardwood. But no report has been done on the ASAM pulping of jute. Therefore, in the present investigation, an effort has been made on the ASAM pulping of whole jute plant and jute fibre. Optimization of ASAM pulping for whole jute plant has been done with varying cooking variables such as Na_2SO_3/NaOH ratio; chemical charge; methanol ratio in the liquor; temperature and time. Elemental chlorine free (ECF) bleaching of ASAM pulps from jute also has been studied.

EXPERIMENTAL

Raw materials

Whole jute plant (WJP) and retted jute fibre (RJF) were collected from the BJRI, Dhaka. It was sun dried and dirt was removed. Then it was cut to 2-3 cm in length. The moisture content of raw materials was determined according to TAPPI Standard Methods (T 18m- 53). After determination of the moisture content of air dried raw materials equivalent to 250 gm oven dried (o.d.) was weighed separately in a polyethylene bag for subsequent cooking experiments. The lignin and a-cellulose contents of WJP was about 22 and 51 %, while 14 and 63 % for RJF respectively.

Cooking

All pulping experiments of WJP were perfomed in an autoclave of 5 liters capacity, made of stainless steel, rotating at 1 rpm, fitted with thermostat. The following parameter was maintained in ASAM process:

-Total alkali charge as sodium hydroxide was 18, 20, 22 and 24% on o.d. raw materials

-Na_2SO_3/NaOH ratios were 70/30, 80/20 and 90/1 0

-Liquor to fibre ratio was 5:1.

- AQ charge was 0.1 % on o.d. raw materials

-Temperature was 170 and 180°C.

-Cooking time was 1 and 2 h.

-Methanol was 0, 20, 25, 30% on the total volume of liquor. 20 / capacity digester was used to evaluate papermaking properties.

Bleaching

WJP and RJF pulps were bleached in elemental chlorine free (ECF) bleaching sequences. The details of bleaching conditions are given in Table 2. The kappa factor 0.22 was used for ECF bleaching.

Evaluation of pulps

WJP and RJF pulps of optimum conditions were beaten in a Valley beater. The samples were collected at different freeness and handsheets of about 60g/m^2 were made in a Rapid Kothen Sheet Making Machine according to German Standard Methods DIN 106. The sheets were tested for tensile (T 404os 61), burst (T 403m 53) and tear strength (T 414m-49) according to TAPPI Standard Methods.

All WJP pulps were beaten in a Wearing blender for about 15 min then handsheets were prepared for tensile, tear burst and folding strength as described above.

RESULTS AND DISCUSSION

ASAM pulping of whole jute plant was done with varying cooking variables namely chemical charge, alkali concentration in NaOH/Na_2SO_3 ratio, AQ charge, temperature methanol ratio and cooking time as shown in Table 1. It is seen that pulp yield, kappa number and tear index were decreased and breaking length and burst index were increased with increasing chemical charge at the conditions of 80% of Na_2SO_3/NaOH ratio, 25% methanol in the liquor, 1h of cooking at 170°C. Kappa number of 39.2 in 56.6% pulp yield was observed at 22% chemical charge. NS-AQ pulping of whole jute plant produced pulp yield of 53.0% in Kappa number of 35.0 when 22% of chemical charge [10] was used. Whole jute plant produced pulp of low Kappa number with the sacrifice of yield in the kraft process [11]. If chemical charge was increased from 22 to 24% kappa numbers did not reduced significantly but pulp yield lost by 2.2% on o.d. raw materials. The delignification rate was increased with increasing NaOH concentration in Na_2SO_3/NaOH

Table 1: ASAM cooking conditions for WJP pulping and pulp properties

Chemical charge, as NaOH	Na_2SO_3/NaOH ratio	MeOH %	Temp. °C	Time, h	Pulp yield, %	Kappa number	°SR	Breaking length, m	Burst index, kPa.m²/g	Tear index, mN.m²/g
18	80	25	170	1	61.8	55.7	15	4410	3.0	13.1
20	80	25	170	1	58.9	48.8	15	4549	3.1	12.3
22	80	25	170	1	56.6	39.2	14	4657	3.2	11.2
24	80	25	170	1	54.4	39.0	14	4861	3.1	11.7
22	80	0	170	1	53.9	46.5	15	4049	2.9	11.3
22	80	20	170	1	56.1	40.3	15	4587	3.0	11.8
22	80	30	170	1	55.8	41.7	17	5113	3.9	12.4
22	70	25	170	1	51.2	37.1	15	4484	3.0	12.5
22	90	25	170	1	56.7	42.3	15	4841	3.7	12.7
22	80	25	170	2	53.8	41.1	16	4621	3.2	12.4
22	80	25	180	1	49.3	32.4	15	6273	3.8	12.7
22*	80	25	170	1	57.5	50.4	16	3814	2.7	12.3

* without AQ

ratio with the loss of pulp yield, at the ratio of 70% Na_2SO_3/NaOH, kappa number reduced to 37.1 with the loss of 5.5% pulp yield on o.d. raw material. The delignification was significantly improved with the huge loss of pulp yield if cooking temperature increased to 180°C. Kinetics study of Eucalyptus wood pulping showed that the higher temperature was required for ASAM process than kraft process [12]. Activation energy for ASAM process was 132.4kJ/mole and 83.5kJ/mole for kraft. At higher temperature physical properties were increased. ASAM pulping of spruce or pine chips at 175°C for 90min resulted in Kappa numbers of 35, but an increase in temperature and time accelerated the delignification to Kappa number of 14.5 [13]. If AQ was not used in the cooking liquor, no remarkable improvement in delignification was shown. Figure 1 represents the effect of methanol on the delignification and pulp yield. In this case, chemical charge and sulfite to alkali ratio were set at 22% and 80% for 1h at 170°C, respectively. The addition of methanol increased delignification, i. e. decreased Kappa number of the resulting pulp. Methanol supports the even distribution of AQ in the chips and its reactivity during the redox cycle. It also reduced the ionization of the cooking chemicals and the swelling of wood components [14]. The presence of methanol up to 25% in the cooking liquor accelerates the rate of delignification. Similar evidence was observed during ASAM pulping of spruce, beech and pine [15]. This was may be due to increasing polarity of the cooking liquor and by decreasing the viscosity of the liquor. These two factors enhance

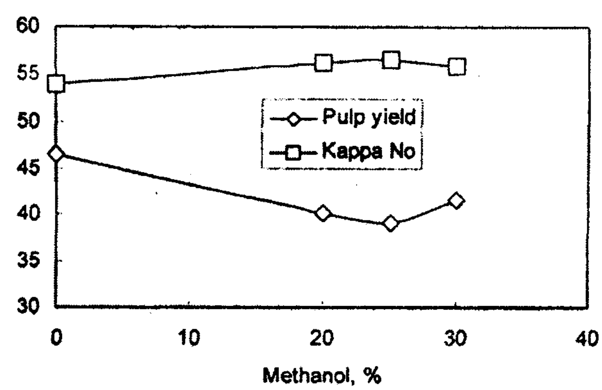

Figure 1 : Effect of methanol on pulp yield and kappa number of WJP

Table 2: Bleaching conditions and results of ASAM pulps from WJP and RJF

Sequence	Temperature °C	Time, min	Pressure Kg/cm²	Consistency %	Kappa number		Brightness, %		Viscosity, mPa.s		Yield, % on o.d. pulp	
					WJP	RJF	WJP	RJF	WJP	RJF	WJP	RJF
UB	-	-	-	-	39.2	7.6	45.0	63.0	21.8	35.6	100	100
Oxygen*	100	60	2.5	10	25.5	5.1	58.3	69.8	19.7	31.8	95.6	97.0
D1**	70	60	-	7	13.1	3.2	68.1	76.5	17.2	28.1	94.1	95.3
E	70	60	-	10	10.6	2.1	70.1	80.3	15.9	26.9	93.2	94.6
D2	70	60	-	7	2.8	1.8	83.5	85.3	15.3	25.7	92.8	93.6

*$MgSO_4$ 0.5% on o.d. pulp, ** Kappa factor 0.22, WJP-whole jute plant, RJF-retted jute fiber

penetration of the cooking liquor and diffusion of the chemical ions to the reaction sites [16-18]. An addition of 6.3% methanol in the kraft liquor reduced Kappa number by 2 points in softwood pulping [19]. Pulp yield remained almost similar from 20 to 30% methanol in the cooking liquor. Kraft pulp yield slightly increased with methanol and without AQ. Norman et al. [20] showed that addition of methanol in the high sulphidity kraft liquor accelerated the delignification and the degradation, but the effect of delignification was greater. Breaking length, burst index and tear index were increased with increasing methanol ratio in the cooking liquor (Table 1)

Table 3: ASAM cooking conditions and results for RJF pulping

Chemical charge as NaOH on o.d. RJF, %	22
Cooking time at max. temperature, min	60
Maximum temperature, D°C	170
AQ charge on o.d. RJF, %	0.1
Na_2SO_3/NaOH,	80/20
Pulp yield, % on o.d. RJF	61.5
Kappa Number	7.6
Brightness, %	63.0

Retted jute fibre (RJF) pulping

The retted jute fibre pulping was done in 22% chemical charge (Na_2SO_3/NaOH, 80/20) at 170°C for 1 h. The AQ charge and liquor ratio were 0.1 % on o.d jute 5, respectively. Very high pulp yield with exceptionally low Kappa number was obtained in ASAM pulping of retted jute fibre. Similarly, ASAM pulping of poplar chips resulted to Kappa number of 6.5, whereas the corresponding Kappa number for birch chips was 13.0 [13]. As shown in Table 3, RJF produced Kappa number 7.3 in pulp yield of 61.5% in ASAM process. The brightness of unbleached pulp was 63%, which indicated easier bleachability. The higher Kappa number during whole jute plant pulping (Table 1) may be attributed that the whole jute plant contains bark and core in the ratio of 1 :2.5. Core contains more lignin and less holocellulose than bark. During retting, extractive and resinous matters were dissolved from the bark. Therefore, during unretted whole jute plant pulping chemicals was consumed by extractives

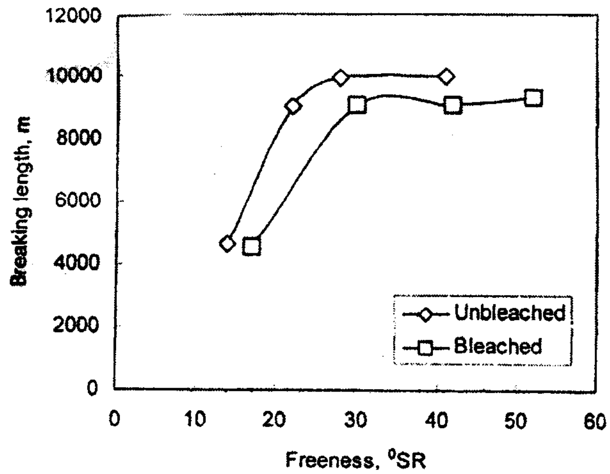

Figure 2 : The relationship between freeness and breaking length of ASAM pulp from WJP

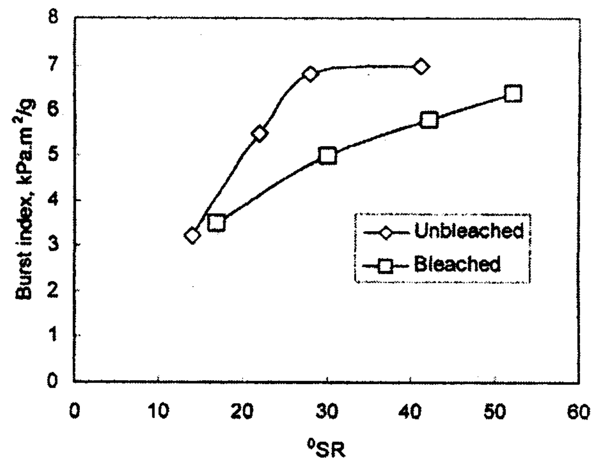

Figure 3 : The relationship between freeness and burst index of ASAM pulp from WJP

and densely lignified core portion. The viscosity of ASAM jute pulp was 30.1, which predicted good strength properties.

Bleaching of WJP and RJF

The WJP and RJF pulps of Kappa number 39.2 and 7.6, respectively, were bleached in elemental chlorine free bleaching (ECF) sequences. After oxygen treatment, Kappa number of WJP pulp was reduced to 25.5 with the sacrifice of viscosity 2.1mPa.s. At this time pulp brightness was increased to 56.0% from 45.0%. Kappa number and viscosity of RJF were reduced to 5.1 from 7.6 and to 31.8mPa.s from 35.6mPa.s, respectively. Oxygen treatment reduced about 40% of chlorine demand, therefore, reduced chlorinated compound in the effluent. Oxygen prebleached pulps were subsequently bleached by DED bleaching sequences. The final brightness and viscosity of the ASAM pulps were 83.5% & 15.3 mPa.s for WJP and 85.3% & 25.7mPa.s for RJF, respectively. ASAM pulp from douglas-fir, spruce and beech showed more easily bleachable than that of other conventional pulps in ECF and TCF bleaching [21, 22].

Physical properties of ASAM pulp from WJP

The strength properties of bleached and unbleached ASAM pulp, from whole jute plant are shown in Figures 2-6. It is seen from Figure 2 that breaking length of unbleached ASAM pulp from WJP reached to about 10,000m after beating to about 30°SR. At this beating degree the breaking length of bleached pulp reached to about 9,000m.

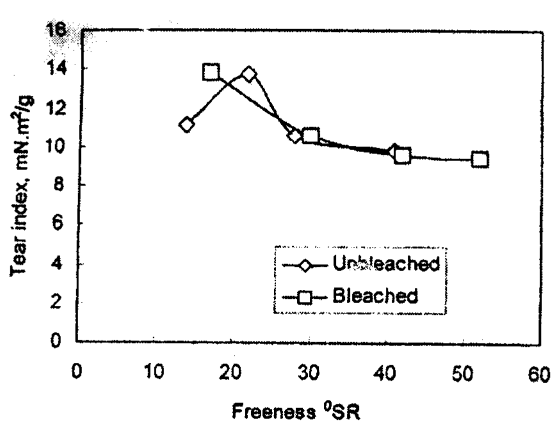

Figure 4 : The relationship between freeness and tear index of ASAM pulp from WJP

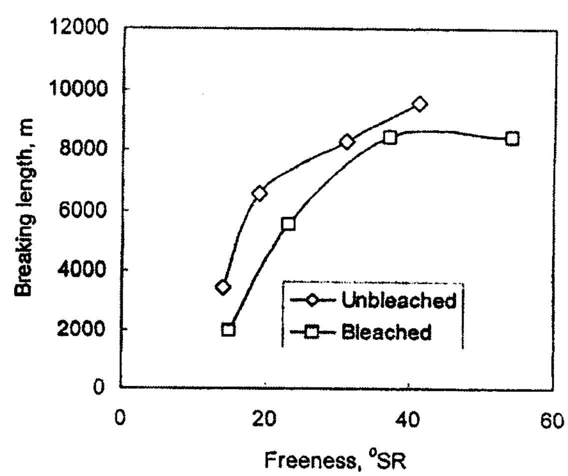

Figure 5 : The relationship between freeness and breaking length of ASAM pulp from RJF

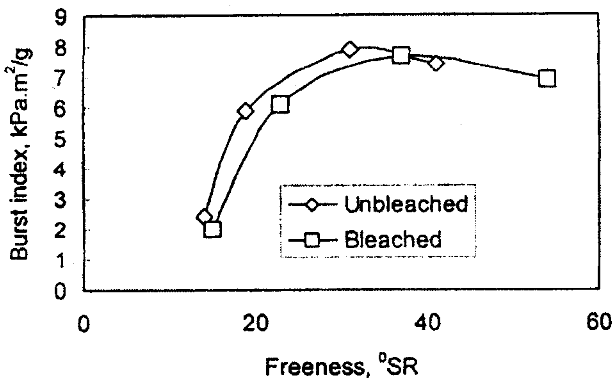

Figure 6 : The relationship between freeness and burst index of ASAM pulp from RJF

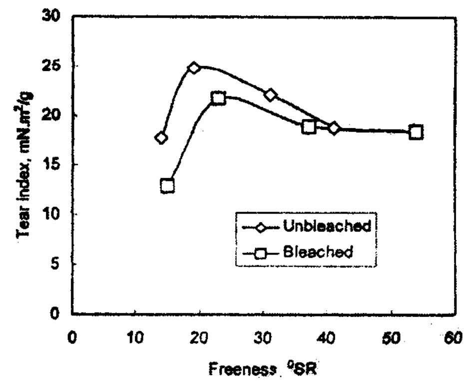

Figure 7 : The relationship between freeness and tear index of ASAM pulp from RJF

The burst index of ASAM pulp from WJP was increased during beating (Figure 3). The burst index of the unbleached pulp was improved from 3.2kPa.m²/g in the unbeaten state to a value of 6.8 kPa.m²/g at about 30°SR then level off. But burst index of bleached pulp was increased linearly from 3.5kPa.m²/g to 6.5 at 52°SR.

The tearing strength of bleached pulp was decreased with beating, whereas unbleached pulp increased in the initial degree of beating then decreased (Fig. 4). After 30°SR, bleached and unbleached pulps showed almost similar tear index. Similar results were observed in ASAM pulping of Douglas fir [21].

Physical properties of ASAM pulp from RJF

Strength properties of ASAM pulp from retted jute fibre are shown in Figures 5-7. The breaking length of unbleached pulp was increased to 9547m from 3437m with increasing SR number from 14 to 41 (Fig. 5). On ODED bleaching breaking length was reduced by 11 %. The bursting strength of both bleached and unbleached pulps were increased rapidly up to certain SR number then decreased. The bursting strength ASAM bleached and unbleached pulps from RJF reached to maximum value of 7.7kPa.m²/g at °SR 37 and 7.9kPa.m²/g at °SR 31, respectively. The tear index of ASAM pulp was increased with increasing SR number initially then decreased. The maximum tear index of unbleached pulp was obtained 24.9mNm²/g at 19°5R and for bleached was 21.7 mNm²/g at 23°SR. The correlation of tear-tensile of ASAM pulps from WJP and RJF is given in Fig 8. Figure shows that a very significant breaking length was gained at the expense of only a small drop in tear. RJF pulp exhibited higher tear-tensile than that of WJP pulp. At 8000m breaking length, RJF showed tear index of about 20 mN.m²/g and 23 mN.m²/g whereas WJP showed 11 mN.m²/g and 13.5 mN.m²/g for bleached and unbleached pulps, respectively. A high amount of hemicellulose improves tensile, whereas extended delignification contributes to tear strength. The correlation tear-tensile depends on the hemicellulose content of the pulps. ASAM jute pulp showed the highest tear-tensile relation than the any other process studied so far [2-6].

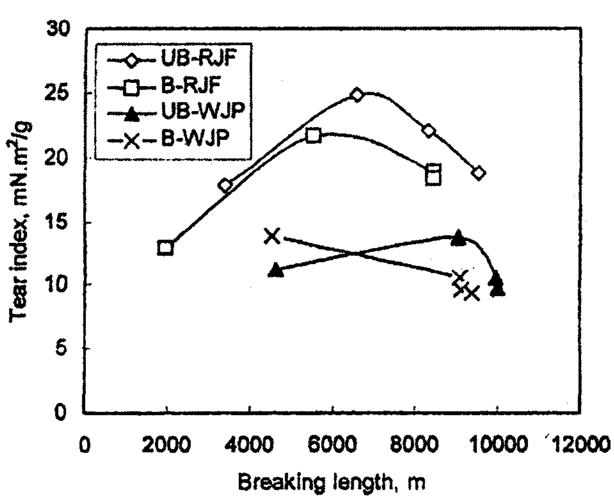

Figure 8 : The relationship between tear and tensile of ASAM pulps from WJP and RJF

CONCLUSIONS

Whole jute plant and jute fibre produced in high pulp yield than the any other processes. The Kappa number of WJP pulp was higher and RJF pulp was significantly lower. This was because WJP consists of highly lignified jute stick as compared to RJF. Initial brightness of RJF was 63%, which indicated easy bleachability. The breaking length and burst index of WJP pulp reached to about 10,000m and 6.8, respectively with increasing beating to 30°SR. At this SR number tear index was I3.8mN.m^2/g. But tear index of RJF was about 22 mN.m^2/g. On oxygen bleaching both WJP and RJF reduced Kappa number by about 40%. The final brightness after ODED bleaching of WJP and RJF were about 83 and 85%, respectively.

REFERENCES

1. Nahar, N. Studies on carbohydrates in jute and pigeon pea. Ph.D. Dissertation. Swedish University of Agricultural Sciences. Uppsala, Sweden 1987.
2. Jahan, M. S., Farouqui. F. I. Pulping of whole jute plant (C. Capsularis) by soda amine process. Holzforschung 54(6): 625-630,2000.
3. Jahan, M. S., Farouqui. F.I. Soda-quinone pulping of whole jute plant (Corchorus capsularis). J. Tropical Forest Products. 7(1): 37-48,2001.
4. Jahan, M. S., Farouqui. F. I. Pulping of jute with amine. Cellulol. Chem. Technol 35(1-2): 177-187,2001.
5. Jahan, M. S. Evaluation of additive in soda pulping of jute. Tappi 84(8), 2001. Also available in www.tappi.org
6. Roy, T. K., Mohindru, V. K., Behera, N. C., Kulkarni, A. G., Ajoy, P. 1998. Jute for speciality pulp Ippta 10(3): 81-86.
7. Patt, R., Kordsachia, O. Herstellung von zellstoffen unter verwendung von alkalischen sulfitlosungen mit zusatz von anthrachinon und methanol. Papier 40(10A):V1-V8, 1986.
8. Chiang V. L., Moffitt G. E., Puumala R. J., Sako T. Linear regression modeling and response surface analysis of sulfur dioxide organosolv pulping. Solvent Pulping-Promises & Problems, Appleton, WI, p 68-92, 1987.
9. Kordsachia O., Part R. Untersuchungen zum aufschluss von buche nach dem ASAM verfahren und zur bleiche der zellstoffe in untershiedlichen sequenzen. Papier41(7): 340-351,1987.
10. Shafi, M. A. NS-AQ Pulping of jute. Ph.D. Dissertation, Dhaka University, 1994.
11. Jahan, M. S. Studies on the pulping of jute based raw materials using various additives and their characteristics. Rajshahi University, 1999.
12. Miranda, I., Pereira, H. Kinetics of ASAM and Kraft Pulping of Eucalypt Wood (Eucalyptus globulus) Horzforschung 56(1):83-90, 2002.
13. Part, R, Kordsachia, O. Knoblauch, J. The ASAM process - alkaline sulfite, anthraquinone, methanol pulping. Fourth International Symposium on Wood and Pulping Chemistry, Paris, France, 1: 355-360, 1987.
14. Part, R., Knoblauch, J., Faix, O., Kordsachia, O., Puls, J. Lignin and Carbohydrate Reactions in ASAM Pulping. Das Papier 45(7): 389-395, 1991.
15. Schubert, H. L., Kordsachia, O., Shackford, L. D. High yield and easy bleachability- The ASAM process. 1994 Tappi Pulping Conference Procds. San Diag, 1994.
16. Rabar, D., Rabar, N. Organic Chemistry, West Publishing, St. Paul, p.618, 1988.
17. Nakano, J., Daima, H., Hosoya, S., Ishizu, A. Studies on alkali-methanol cooking. Proc. 1st ISWPC Conf., Stockholm, p.72, 1981.
18. Sarkanen, K. V. Ludwig, C. H.- Lignin Occurrence, Formation, Structure and Reactions, John Wiley & Sons, New York, p. 687,1971.
19. Leduc, C., Destine, J., Daneult C. Sulfidity reduction in a kraft process by adding methanol and/or anthraquinone. Appita J. 56(1): 35,2003.
20. Norman, E., Olm, L., Teder, A. Methanol reinforced kraft pulping. Tappi J. 76(3):125, 1993.
21. Zimmermann, M., Part, R., Kordsachia, O. ASAM pulping of Douglas-fir followed by a chlorine-free bleaching sequence. Tappi J. 74(11): 129-134, 1991.
22. Knoblauch J., Roderweg, L. W., Kordsachia O. Comparison of pulp properties from the kraft, sulfite and ASAM process. Das Papier, 1: T1-T5, 2000.