

Studies on alkaline sulphite-anthraquinone pulping of bagasse

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ABSTRACT :

Cooking trials were made with partially depithed bagasse using AQ (0.05 to 0.2% on o.d. bagasse) with alkaline sulphite cooking liquor containing mostly Na_2SO_3 , and NaOH with little amount of Na_2CO_3 . The optimum activity ratio (ratio of Na_2SO_3 to total chemicals i.e. $\text{Na}_2\text{SO}_3 + \text{NaOH} + \text{Na}_2\text{CO}_3$) was found to be 80 to 85%. The total chemical charge varies from 12 to 16% (of o.d. bagasse). The cooking time varies from 80 to 120 minutes at a maximum temperature which varies from 140 to 160 °C, in order to know the optimum doses of cooking chemicals and to know the effect of cooking time and temperature during the course of pulping. On increasing alkalinity of the alkaline sulphite process, the rate of delignification also increased and approached to that of kraft. These pulps were evaluated for yield, selectivity of cooking, Kappa number and strength properties.

INTRODUCTION :

Cellulosic fibres are produced through permanent growth of not trees but also of large number of non-woody plants including agricultural residues. The non-woody plants represent one of the major sources of fibrous raw material for many developing nations of the world. In some countries, especially where wood is scarce, non-woody fibres are consequently more common as raw materials to be used for pulp, paper and board manufacture.

With the tremendous increase in population and literacy, the planning commission has indicated that the capacity creation of paper and board products will need to be increased to about 42.5 lakh tonnes by the end of this century. The biggest bottle neck in meeting the targetted amount of paper and board products will be the availability of cellulosic raw materials from existing forest resources which are already in short supply and are barely able to sustain the present level of production. To meet the increasing demand of paper and board

products, the requirement of fibrous raw materials is increasing day by day which is affecting the environment and natural ecosystem to conserve forest which is so essential for a stable eco-balance. Therefore, it is very much essential that in the developing nations, where the forest cover is already below the required level, the main emphasis should be given on the utilization of agricultural residues for the production of high and ultra high yield pulps with the latest pulping technology which will also help in reducing the environmental pollution load in the biosphere.

The structure of the group of the non-woody fibrous raw materials in India is significantly different from that of other countries and regions of the world, since in India bamboo along with other non-woody fibres including agricultural residues are the most important fibrous raw material for pulp and paper making.

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Among the many agricultural fibers utilized for pulp manufacture, sugar cane bagasse stands out, more than any other, as being one which promises to become a major fibrous raw material for the world's pulp and paper industry. This material is readily available and easily accessible in a great many countries of the world, and is especially abundant in some of the wood-poor countries. In fact, this raw material appears to satisfy the criteria, for being a successful fibrous raw material for manufacture of pulp, paper, paperboard and reconstituted panelboard, better than any other crop fiber. It has a distinct advantage over other agricultural residues in that it involves no problem of collection.

Over the past decades, high yield sulphite pulping of hardwoods has been investigated as a mean of making a low-cost pulp for the production of newsprint, printing papers and tissue papers. Different combinations of pulping variables have been tried (for example pre-steaming/liquor-impregnation procedure, cooking liquor pH, $\text{Na}_2\text{SO}_3/\text{NaHSO}_3$ content, cooking temperature and time, and liquor-to-wood ratio). Sulphite pulping under very alkaline conditions, uses caustic (NaOH) and Na_2SO_3 , to produce a pulp having strength properties similar to kraft pulp.¹⁻⁴ The alkaline sulphite (AS) approach does not produce odoriferous sulphides (such as CH_3SH and $[\text{CH}_3]_2\text{S}_2$) that are commonly produced in the kraft pulping/chemical recovery system. The anthraquinone (AQ), has also been found to be a most effective pulping aid as accelerator of delignification and protector of carbohydrates in alkaline sulphite environment. With the addition of anthraquinone, both the cooking kinetics and the composition and properties of the resulting pulps are profoundly altered^{5,6}. Because of the high paper strength, they produce, comparisons are generally made with the common kraft process with and without AQ but they are distinctly different also from this process in chemistry, reaction mechanism, lignin to carbohydrate ratio, and degradation of cellulose and hemicelluloses. The emergence of sulphite process at moderate alkalinity with AQ as its essential additive offers the industry a pulp of kraft like strength at higher yield and better bleachability, while substantially reducing the air pollution load.^{7,8} The capital cost required to convert an existing kraft pulp mill to an alkaline sulphite mill would be significantly less than that required to renovate an existing kraft mill

to comply with increasingly stringent air quality standards, including lower TRS levels at the recovery stack exit.

One of the attractive features of the AS-AQ process, by comparison with the conventional kraft process, besides the inherent advantage of being able to produce strong pulp without kraft odour, is its low requirement of caustic soda. In kraft pulping, the charge of caustic soda is higher even than in the strongly alkaline sulphite pulping process and necessitates the operation and maintenance of large units for the re-causticization of the spent pulping liquor at considerable expense and fossil fuel consumption. With the moderately alkaline process, the necessary moderate alkalinity can be provided by soda ash, which is available directly from the recovery process and if needed, by a limited amount of caustic soda used as make-up chemical for losses in the process. Thereby the causticization unit can be completely eliminated, or if some free caustic is required elsewhere in the mill, for instance in the bleaching, a small causticization unit can be operated. The AS-AQ process has advantages over the common kraft process in economy, much greater product flexibility and complete elimination of kraft odour, but it requires AQ catalyst.

The present investigation relate to the evaluation of moderately alkaline sulphite-AQ pulps of bagasse with the help of statistical experimental design, to study the effect of various process variables during alkaline sulphite-AQ pulping of bagasse.

Experimental Methodology :

Raw Material Preparation and Analysis :

The air dried bagasse was collected from local unit of state sugar corporation. The total pith content was determined by TAPPI useful method UM 3-1981. The air dried bagasse was mechanically depithed, and the amount of pith so removed was determined. The mechanically depithed bagasse was further treated in a hydrapulper at 5% consistency for 10 minutes and the amount of pith removed by wet depithing method was also determined. The total amount of pith, pith removed by dry depithing method as well as by wet depithing method, alongwith the amount of pith still associated with bagasse, are reported in Table-I.

TABLE—I

Studies on Depithing of bagasse for pulping.

Sl.No.	Particulars	Percentage
1.	Total amount of pith.	33.0%
2.	Pith removed by dry depithing method.	7.29%
3.	Pith removed by wet depithing method.	4.45%
4.	Total amount of pith removed.	11.74%
5.	Amount of pith still associated with bagasse.	21.26%

The whole bagasse, depithed bagasse and pith were chemically analysed by TAPPI standard method. The chemical composition of different fractions of bagasse including whole bagasse, separated fiber (depithed bagasse) and pith is given in Table-II.

TABLE—II

Proximate Chemical Analysis of Bagasse.

Sl.No.	Particulars	Whole Bagasse	Depithed Bagasse (Fiber)	Pith
1.	Ash %	2.9	2.6	6.5
2.	Hot Water Solubles %	2.5	0.9	1.9
3.	Alcohol-Benzene Solubles%	4.1	1.8	2.8
4.	1% NaOH Solubles %	30.7	27.5	35.8
5.	Lignin %	20.2	20.8	20.2
6.	Pentosan %	26.7	27.9	28.4
7.	Holocellulose %	76.6	77.8	74.2
8.	Alpha Cellulose % (Corrected)	38.1	42.4	32.8

The morphological studies of different fractions of bagasse was done by Bureau of Indian Standard method (BIS) 5285-1969, and the dimensional characteristics of bagasse fractions are given in Table-III.

TABLE—III

Dimensional Characteristics of Bagasse.

Cellulosic	Length(mm)			Diameter(μ)			Ratio of average Length to diameter.
	Min.	Avg.	Max.	Min	Avg.	Mix.	
Sugar cane fibers	0.80	1.70	2.80	10.2	20.2	34.1	85:1
Parenchyma	—	—	0.84	—	—	140	—
Vessel Segments	—	—	1.35	—	—	150	—
Temperate Coniferous wood	1.2	—	7.3	22	—	50	75:1
Temperature deciduous woods	0.3	—	2.7	10	—	46	50:1

Pulping Studies :

The pulping studies were conducted in an electrically heated rotary digester of 0.02 M³ capacity having four bombs of one litre capacity, furnishing sufficient pulps for evaluation. During these studies, technical grade chemicals were used. During the course of pulping, the wood to liquor ratio of 1:4 was used and the following time schedule for digester heating was adopted.

From room temperature to 105°C = 30 minutes.

From 105°C to maximum temperature = 60 minutes.

At the end of cooking, the digester pressure was reduced by gas relief until the temperature reached 105°C. The charge was then blown from the digester. The pulp was washed on a laboratory flat stationary screen having 150 mesh wire bottom for the removal of residual spent liquor. The pulp was further disintegrated in the laboratory disintegrator, The disintegrated pulp was further screened through a vibratory flat screen and the screened pulp was further evaluated.

The pulping studies were made using various doses of cooking chemicals with different ratios of sodium sulphite to sodium hydroxide in order to know the

optimum ratio of these cooking chemicals. The cookings were also carried out at different temperature (140°C to 160°C) with different time at temperature (80 min. to 120 minutes), to know the effect of cooking temperature and time, during the course of pulping. The pulp yield and Kappa number were determined. The pH of the spent liquor was also determined. The results are reported in Table-IV to VII.

During the course of study, three process variables viz., cooking time, temperature and total chemicals doses have been varied, keeping the other factors like bath ratio, activity and the rate of increase of temperature etc. constants. The output variables which includes, yield, kappa number, tensile, tear and burst indexes were found to be a function of input variables (time, temperature and total chemical doses). The multiple regression equations so developed can be used for:

(i) Optimization of the system by mathematical analysis.

(ii) making prediction for conditions which have not been used for the experiments.

The theoretical values of strength properties were determined and compared with the experimental values for each conditions. The theoretical values are quite comparable with the experimental values for these properties.

Pulp Evaluation :

The unbalanced alkaline sulphite and alkaline sulphite-AQ pulps of bagasse were beaten in a PFI mills with a beating pressure of 1.8 kgs/cm. to different freeness levels and handsheets of 60 gms/m² were prepared on a standard British Sheet Forming machine. These hand sheet were evaluated as per BIS standard methods for their physical strength properties at a relative humidity of 65 ± 2% and a temperature of 27 ± 2°C. The pulp evaluation results are tabulated in Tables IV to VII.

TABLE—IV
Effect of Pulping Parameters on Kappa Number, Pulp Yield and Strength Properties.

Sl. No.	Temperature (°C)	Time at temperature (Min.)	Total Chemicals (% as Na ₂ O)	Kappa No.	Pulp Yield%	Tear Index	Burst Index	Breaking Length (Km.)
1.	140	80	12	78	80	5.9	7.4	3.9
			14	71	75	6.1	7.6	4.4
			16	65	72	6.2	7.8	4.7
		100	12	77	78	5.6	7.1	4.6
			14	66	71	5.7	7.2	4.9
			16	61	68	5.8	7.4	5.1
		120	12	68	69	5.2	6.6	4.9
			14	59	66	5.4	6.8	5.2
			16	55	63	5.5	6.9	5.4
2.	150	80	12	62	70	5.6	7.2	5.1
			14	58	66	5.8	7.3	5.2
			16	54	64	5.9	7.5	5.5
		100	12	52	68	5.2	6.8	5.3
			14	44	66	5.4	7.0	5.6
			16	41	62	5.5	7.1	5.8
		120	12	50	66	5.1	6.3	5.5
			14	41	62	5.2	6.5	5.7
			16	35	60	5.2	6.7	5.9
3.	160	80	12	39	63	5.3	7.0	5.9
			14	32	59	5.5	7.1	6.4
			16	28	57	5.6	7.3	6.9
		100	12	40	62	5.1	6.5	5.9
			14	36	58	5.2	6.7	6.1
			16	32	56	5.2	6.9	6.2
		120	12	38	60	4.4	6.0	5.9
			14	33	57	4.6	6.2	6.1
			16	30	55	4.9	6.5	6.3

TABLE—V
Effect of Different Doses of AQ During AS Pulping of bagasse with 16% Total Chemical dose
(With 80% Activity) at 160°C For 80 Minutes.

Sl. No.	AQ Dose %	Pulp Yield %	Kappa Number	Tear Index	Burst Index	Breaking Length (cms)	Brightness (% Elrepho)
1.	0	57	28	5.6	7.3	6.9	24
2.	0.05	58	25	5.9	7.2	7.0	26
3.	0.1	59	22	6.2	7.2	7.2	30
4.	0.15	59	20	6.3	7.2	7.3	30
5.	0.2	60	19	6.4	7.2	7.3	32

TABLE—VI
Effect of Cooking Time During AS Pulping of Bagasse With 16% Total Chemical Dose
(With 80% Activity), 0.1 % AQ at 160°C For 80 Minutes.

Sl. No.	Time at temperature	Pulp Yield %	Kappa Number	Tear Index	Burst Index	Breaking Length (Kms.)	Brightness (% Elrepho)
1.	60	60	25	6.0	7.0	7.1	28
2.	80	59	22	6.2	7.2	7.2	30
3.	100	58	21	6.3	7.2	7.3	31
4.	120	57	20	6.4	7.3	7.3	32

TABLE—VII
Comparison Of AS, AS-AQ and Kraft Pulping of bagasse.

Sl. No.	Particulars	AS	AS-AQ	$\Delta\%$, AS/AS—AQ AS=100	Kraft	$\Delta\%$, Kraft/ AS—AQ Kraft=100
1.	Total chemicals	16	16	—	16(AA)	—
2.	Alkali ratio	0.8	0.8	—	—	—
3.	AQ, % on o.d. wood	—	0.1	—	—	—
4.	Maximum temperature, °C	160	160	—	160	—
5.	Time at temperature	80	80	—	80	—
6.	pH of the cook (start-end)	13.1-9.4	13.1-9.4	—	13.5-12.1	—
7.	Total yield, %	57	59	+3.5	50	+18
8.	Kappa No.	28	22	-21.5	21	+4.7
9.	Tear Index	5.6	6.2	+10.7	6.0	+3.3
10.	Burst Index	7.3	7.2	-1.4	7.1	+1.4
11.	Breaking length (Km.)	6.9	7.2	+4.3	7.0	+2.8
12.	Brightness	24	30	+25	22	+36.3

Results And Discussions :

The results of proximate chemical analysis of bagasse clearly revealed that the alpha cellulose content is higher in depithed fiber and ash content is higher in the pith fraction. Pentosan content is almost 50% higher than in typical temperate hardwoods and exceeds by three to four times that present in coniferous softwoods. Lignin content is about 75% that of softwoods and about equal to that of hardwoods, indicating the possibility of producing high yield pulp from depithed bagasse under optimum pulping conditions.

In dimensional characteristics, the bagasse fibres are similar to those of hardwoods but are shorter than those of the coniferous woods.

The most important factors influencing the result of AS-AQ cooking were found to be total alkali charge, activity ratio ($\text{Na}_2\text{SO}_3/\text{total chemicals}$, both as Na_2O) and amount of AQ on wood. Especially when the aim is to produce fully defibered pulps, thorough impregnation of cooking liquor into prepared bagasse is necessary. The pH (cold) of moderated alkaline sulphite is slightly lower than for highly alkaline process at the start and drops to about 9.5 at the end of the cook. The starting pH depends on the ratio of carbonate to hydroxide used.

The influence of total alkali charge and activity ratio on bagasse is shown in Fig. 1. As the figure shows, the activity should be in the range of 0.80 to 0.85 to achieve as low a Kappa number as possible. An increase in the total alkali charge accelerates delignification. It was also found out that the Na_2SO_3 charge has a greater influence on delignification than the total chemical charge. Delignification becomes slower at a kappa number level around 40. with an increase in the Na_2SO_3 charge and cooking time, a lower kappa number may possibly be achieved. The amount of rejects increases rapidly with increasing kappa number and vice-versa.

The results of AS pulping studies on bagasse (Table-IV) indicated that on increasing the total chemical charge at a particular level of temperature and time, the kappa number and pulp yield showed a continuously decreasing trend. The effect of increasing the temperature at constant time (at temperature) also

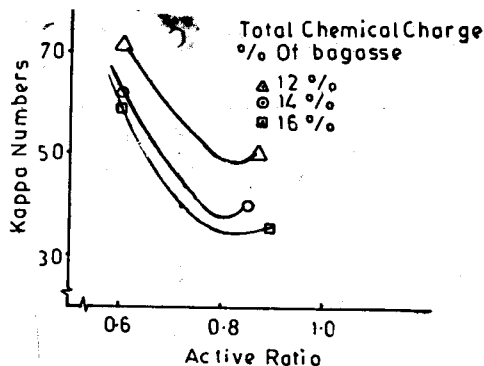


FIG. 1: THE INFLUENCE OF TOTAL CHEMICAL CHARGE AND ACTIVITY RATIO DURING AS-AQ PULPING OF BAGASSE AT 150°C 90 MINUTES TIME AND 0.1% AQ DOSE

showed continuously decreasing trend of kappa number and pulp yield. The effect of increasing the cooking time, keeping the temperature constant also showed the continuously decreasing trend of Kappa number and pulp yield (Fig 2). The continuously decreasing trend of Kappa number and pulp yield on increasing the total chemical charge, temperature and cooking time, is due to the fact that the enhancement in these process variables increases the delignification thereby decreasing both the pulp yield and kappa number. These results indicated that a temperature of 160°C, for 80 minutes with 16% total chemical charge may be considered as optimum set of process variable for the AS pulping of bagasse,

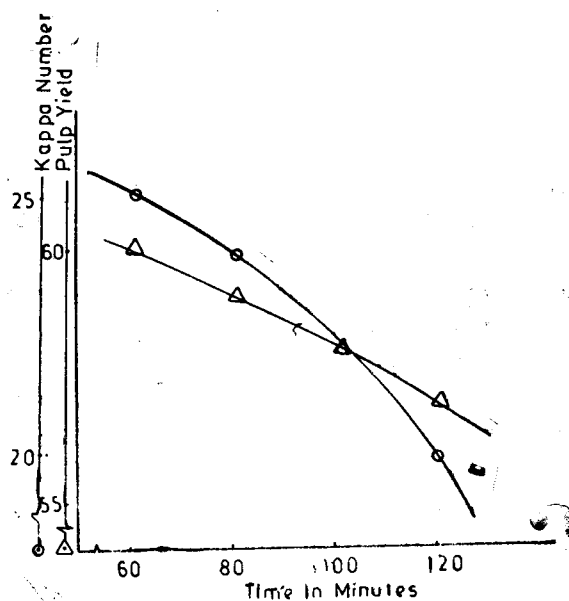


FIG. 2: EFFECT OF TIME ON KAPPA NUMBER AND PULP YIELD

To study the effect of AQ during AS pulping at optimum set of process variables, various doses of AQ ranging from 0.5 to 2% (on o.d. bagasse) was charged and the results are indicated in Table-V. These results clearly indicated that on increasing the AQ dose, although the pulp yield not increased substantially but the Kappa number reduced substantially (Fig. 3). These results also indicated that there is no gain of using more AQ dose beyond 0.1%. On further studying the effect of cooking time at optimum level of temperature and total chemical charge with 0.1% AQ dose, again indicated that on increasing the temperature, the rate of delignification increases thereby decreasing the pulp yield and Kappa number continuously (Table-VI). Based on strength properties and brightness level of pulp, it seems that a time of 80 minutes at cooking temperature, could again be considered as optimum level of time. The curves between varying dose of AQ versus strength properties (Fig. 4) clearly indicated that all the strength properties including brightness showed an increasing trend, except for burst index which decreased minutely (compared to AS pulping without AQ) and remains almost unaffected with any doses of AQ; on increasing the AQ doses. The magnitude of increase in strength properties is high upto a AQ dose of 0.1% and beyond that the magnitude of increase is not so pronounced, therefore 0.1% AQ dose may again be considered as an optimum dose of AQ.

The results of comparative studies on pulping of bagasse by alkaline sulphite, alkaline sulphite with AQ and kraft process at equal chemical dose, temperature

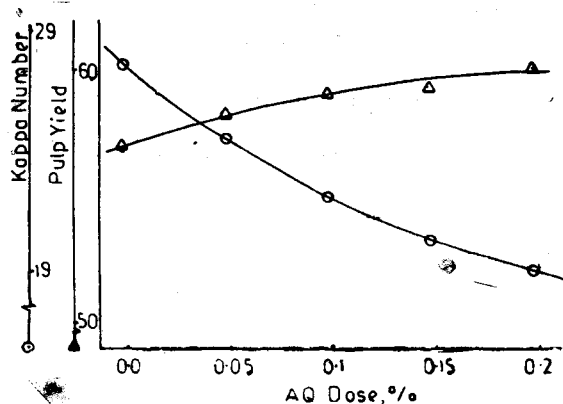


FIG. 3. EFFECT OF AQ DOSE ON KAPPA NUMBER AND PULP YIELD

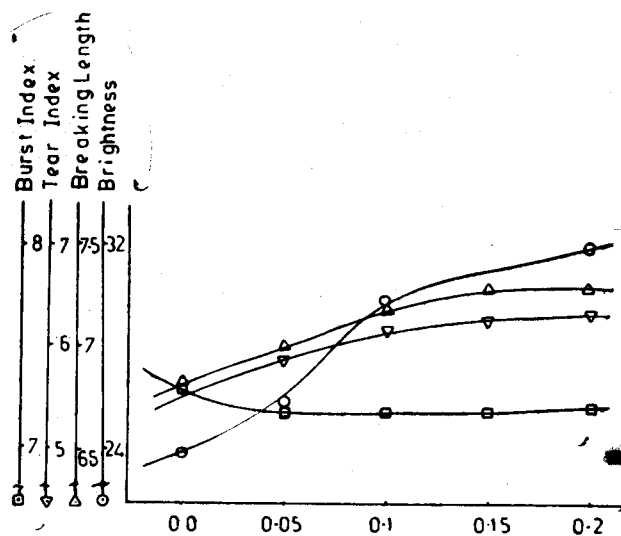


FIG. 4. EFFECT OF AQ DOSE ON TENSILE, TEAR BURST AND BRIGHTNESS

and time are shown in Table-VII. It is seen that the addition of anthraquinone to the alkaline sulphite cook abruptly increases the rate of delignification while protecting cellulosic components against degradation. The resulting pulp beats much more easily and reaches comparatively higher levels of mechanical strength. The efficient kraft cook still retain some advantage of time and temperature in delignification rate and in viscosity, but is deficient in tensile strength properties. There is always a gain in brightness level of pulp prepared by AS or AS-AQ process over pulp prepared by kraft process. Regarding the effect of AQ during moderately alkaline sulphite process, it seems to be twice as great effective than with the strongly alkaline system. In the presence of AQ, the known action of alkalinity to accelerate cooking is replaced by a similar action of sulphite.

With the same charge of cooking chemicals, kraft pulping retain its superiority as a most efficient delignification medium, requiring lower temperature and less time to reach a given level of pulp yield. The superiority of the moderately alkaline sulphite anthraquinone process on the other hand, lies for bleached pulp grades in carbohydrate protection affording high pulp yield and viscosity, very easy bleaching to a higher brightness ceiling with less reversion, and higher strength

of bleached pulp. For packaging grades such as liner pulp for containers and bag papers, the new process has the additional advantages of lower lignin content of the pulp at a given yield, one half the energy requirement for refining, higher burst and tensile strength which decrease little on raising the yield substantially, and higher brightness.

A high initial rate of delignification was observed which decreased steadily with an appreciable amount of lignin being quite resistant. This initial high rate of delignification which is common in a varying degree, to nearly all delignification process, is undoubtedly partly due to high initial alkali concentration in the cooking liquor. It may also correspond to easily assessable and less condensed lignin where its high rate of dissolution coincides with that of bagasse polyloses. The interaction of lignin and hemicelluloses in the bagasse was probably one of the major factor affecting the rate and selectivity of the delignification during the later stages, whether this involves a primary or secondary valence bond or both.

Conclusion :

The present investigation was undertaken to study the AS-AQ pulping of bagasse -a potential non-wood fibrous plant for pulp and paper making and to know the effect of fibre dimensions on the formation and structure of paper. The results of the present studies indicated that on increasing the alkalinity of the alkaline sulphite AQ process, the rate of delignifications also increased and approached to that of kraft. The selectivity at moderate alkalinity was found to be superior and at high alkalinity equal to kraft. The total yield was found to be a function of Kappa number. These results also showed that the AS-AQ process produced pulp superior to kraft process. It resulted in increased pulp yield combined with higher pulp strength, high

pulp viscosity with easier bleachability and a higher brightness ceiling combined with less colour reversion, besides lower power and fuel consumption. It provides a simplified and more economical pulping process with less environmental pollution load.

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