

Studies on Pulp and Paper Making Characteristics of *Saccharum Spontaneum*

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ABSTRACT

The study aims at assessing the suitability of *S. spontaneum* for pulp and paper making. The proximate chemical analysis indicates that it is a bulky material having comparatively lower extractives and lignin content with higher holocellulose content. It requires lower cooking chemicals and shorter cooking cycle in order to produce chemical grade pulp. The unbleached pulp shows good response towards O₂ delignification and reduces kappa number by 62 per cent. The pulp shows good response towards multi-stage bleaching sequence and produces a pulp of high brightness ceiling.

Key Words: *Saccharum spontaneum*, soda pulping, proximate chemical analysis, O₂ delignification, multi-stage bleaching

INTRODUCTION

Indian paper makers are facing a serious lack of better quality of fibrous raw materials. This inadequate supply of better fibrous material has forced the paper makers to spend heavily on imported wood fiber. With modernization of process equipment, revamping of product quality along with cost reduction aspects; the paper technocrats have to think seriously on sustainable availability of cost-effective raw materials. However, paper manufacturers have thought intensely. Besides agro-based residues, plenty of other non-woody plants are available abundantly in India. It has been proven that by selecting the approximate mixture of non-woody plant fibers and the appropriate pulping process, any quality of paper and paper-board can be manufactured with or without addition of any wood pulp. The augment use of non-woody fibrous raw materials for pulp and papermaking may solve this problem up to some extent. In today's environment, it is evident that non-woody plant fiber will play an important role in the global scenario for pulp and papermaking.

Saccharum spontaneum linn is commonly known as 'Kans grass' and is a perennial grass with slender culms, growing in stools or forming continuous canebreaks with most often aggressive rhizomatous tillering, distributed widely in the sub-tropical and tropical parts of Asia, Africa and

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ascending up to an altitude of 1800 m. The culms are green, grey, white, hard and often hollow in the center, varying in the diameter from 5 to 15 mm; often rooting at the nodes; internodes usually long, and nodes always thicker than the internodes; leaves long, linear, narrow or very narrow or sometimes reduced to the midrib, the 'leaf module' or ratio of breadth to length varying from 1:24 to 1:300 or more in the different forms of the species; inflorescence a panicle varying in length and in color from pale and grayish white to purplish gray, spikelets in pairs, one pedicelled and the other sessile the pedicelled spikelet of the pair always blooming first; glumes always four or lodicules ciliate¹.

Pilot plant trials with the grass have shown that by the soda process yields of 42 per cent of unbleached and 37.8 per cent of bleached pulps suitable for writing paper of satisfactory strength. The grass can be used in admixture with other grasses. *S. spontaneum* can also be used for the production of hardboards, rayon grade pulps and activated carbon^{2,3}. The pulp of kans grass is suitable for wrapping, writing, printing and greaseproof papers.

The present study aims at pulping and bleaching of *S. spontaneum* in order to develop writing and printing papers.

Experimental Methodology

Raw material procurement-*S. spontaneum* was collected from nearby regions of Saharanpur. The procured

raw material was chopped by grass cutter. The chips were air dried under atmospheric conditions and packed in polythene bags.

Fiber morphology- The plant sample was subjected to physicochemical maceration with a solution of KClO₃ and concentrated hydrochloric acid to separate the individual fibers to gather without damage for removing the most of the lignin and the other binding materials. The results are reported in Table 1.

Proximate chemical analysis- The air dry chips were disintegrated in the laboratory wood mill. The portion passing through the 40 mesh but retained on 80 mesh sieve, was utilized for proximate chemical analysis and analyzed for as per standard Tappi procedure. The results are reported in Table 2.

Pulping studies- Soda pulping of *S. Spontaneum* was carried out in WEVERK rotary, electrically heated digester of capacity 0.02 m³. The pulping studies were carried out at different alkali doses i.e. active alkali 6-14% (as Na₂O). In order to see its effect on pulp yield and kappa no 0.1% anthraquinone (on o.d. raw material basis) was added at optimum alkali dose i.e. 12%. At the end of cooking the charge was blown from the digester. The cooked chips were defibered through a refiner. The digested chips were defibered through a Bauer refiner with a plate clearance of 0.15 mm

Table1— Fiber morphology and proximate chemical analysis of *S. spontaneum*

Sl no	Parameters	Values
1	Cold water solubility, per cent	7.8
2	Hot water solubility, per cent	10.2
3	Lignin, per cent	16.0
4	Pentosans, per cent	19.4
5	Holocellulose, per cent	76.7
6	A-cellulose, per cent	38.0
7	Alcohol Benzene soluble, per cent	5.8
8	Hot water soluble, per cent	4.8
9	Ash, per cent	3.6
10	Silica, per cent	1.8
11	Fiber diameter, μm	
	Average	16
	Variation	8 – 24
12	Fiber Length, mm	
	Average	1.52
	Variation	0.98- 2.10

Table2— Cooking conditions and results soda pulping of *S. spontaneum* at different alkali doses

Sl no	Alkali dose, per cent	Kappa number	Total yield, Per cent	Screening rejects, per cent	Screened yield, per cent	Residual alkali, per cent
1	6.0	40.0	48.7	10.4	38.3	0.08
2	8.0	38.3	46.3	5.8	40.5	0.51
3	10.0	32.5	46.0	3.6	42.4	0.93
4	11.0	30.6	45.3	2.5	42.8	1.18
5	11.5	28.4	45.7	2.1	43.6	1.28
6	12.0	26.5	45.7	1.8	43.9	1.39
7	12.5	26.3	44.7	1.3	43.4	1.47
8	13.5	24.6	42.3	1.1	41.2	1.59
9	14.0	17.0	41.1	0.9	40.2	1.81

Cooking conditions: Alkali dose = 6-14 per cent (as Na_2O), Time from ambient temp to max temp = 45 min, Time from 105 °C to 155 °C = 45 min, Time at 155 °C = 60 min, Liquor to wood ratio = 5:1 and digester pressure = 6.0 kg/cm^2

Table3— Effect of AQ on screened pulp yield, screening rejects and kappa number during soda pulping of *S. spontaneum*

Sl no	Pulping processes	Alkali dose, per cent	Kappa number, per cent	Total yield, per cent	Rejects, per cent	Screened yield, per cent
1	Soda	12	26.5	45.7	1.8	43.9
2	Soda - AQ	12	21.0	46.9	0.9	46.0

followed by second pass at 0.07 mm plate clearance. The pulp was screened through WEVERK vibratory flat screen with 0.15 mm slits and the screened pulp was washed, pressed and crumbled. The pulp was analyzed for kappa number (T 236 om-99), pulp yield, screening rejects and residual alkali. The results are reported in Tables 2 and 3.

Oxygen prebleaching- The unbleached soda- AQ pulp having kappa number

21, was prebleached with oxygen in laboratory digester of capacity 0.02 m^3 . First the pulp sample was mixed with 1 % MgSO_4 (on O.D. pulp basis) to mitigate the carbohydrates degradation and then with 2% NaOH (on O.D. pulp basis) and pulp consistency of 10% was maintained. The prebleaching was carried out at O_2 pressure of 5 kg/cm^2 at 90 °C for 90 min. The results are reported in Table 4.

Pulp bleaching The soda- AQ pulp was bleached by using single stage hypochlorite bleaching sequences as well as by multi- stage bleaching sequences viz., HH, CEH and CEHH, OC(EO)H, OC(EOP)H, O(CD)(EO)H, O(CD)(EO)D, O(CD)(EO)HD, O(CD)(EOP)H, O(CD)(EOP)D and O(CD)(EOP)HD. The results are reported in Table 4.

Pulp evaluation- The bleached pulps were beaten to a beating level of 40 ± 1 °SR in PFI mill as per ISO DP 5264 method. During pulp beating a pressure of 17.7 N/cm^2 , pulp consistency of 10% and a relative speed of 6.0 ms^{-1} was maintained. Laboratory hand sheet of 60 g/m^2 were prepared on British Sheet Former. Laboratory hand sheets were prepared according to Tappi Test Methods (T 205 sp 02) and tested for various physical strength properties like burst (T 403 om-97), tear (T 414 om-98) and tensile indexes (T 404 cm-92). The results are reported in Table 5.

RESULT AND DISCUSSION

The results of proximate chemical analysis as given in table 1 indicate that total water solubles in *S. Spontaneum* are 10.2 per cent, which is towards on higher side. The water and alcohol benzene solubles come under the category of extractives and totally undesirable for pulp and paper making. The lignin content in *S. Spontaneum* is about 16 per cent. It indicates that *S. Spontaneum* requires comparatively low cooking chemicals and shorter cooking cycle. The pentosan content is 19.40 per cent, which is towards on higher side. However, holocellulose in *S. Spontaneum* is 76.7 per cent, which is towards on higher side. The ash and silica contents in *S. Spontaneum* are 3.6 per cent and 1.8 per cent respectively, which are slightly higher in comparison to wood. The above results clearly indicate its suitability for pulp and paper making. Table 1 also shows the morphological characteristics of *S. Spontaneum*. The fiber length of *S. Spontaneum* varies from 0.98 to 2.1 mm with an average of 1.6 mm as compared to 0.89 mm (average) in eucalyptus³. The fiber width varies from 8 to 24 μm with an average of 16 μm . The above results indicate that the fiber collapse readily into ribbons during sheet formation and provides maximum

Table 4— Bleaching conditions and results of soda pulp of *S. spontaneum*

S.L	Particulars	H	HH	CEH	CEH	OC(E)H	OC(E)H	OC(E)H	OC(E)H	OC(E)H	OC(E)H	OC(E)H	OC(E)H	OC(E)H	OC(E)H	OC(E)H	OC(E)H	OC(E)H	
1	Chlorination stage (C)																		
	Cl ₂ applied (avail Cl ₂),%	—	—	2.65	2.65	1.5	1.5	—	—	—	—	—	—	—	—	—	—	—	—
	COD load, mg/L	—	—	229	—	228	228	—	—	—	—	—	—	—	—	—	—	—	—
2	E stage																		
	NaOH applied, %	—	—	1.35	1.35	—	—	—	—	—	—	—	—	—	—	—	—	—	—
	COD load, mg/L	—	—	1218	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
3	(C-D) stage																		
	Cl ₂ applied, %	—	—	—	—	—	—	0.60	0.60	0.60	0.60	0.60	0.60	0.60	0.60	0.60	0.60	0.60	0.60
	ClO ₂ applied, %	—	—	—	—	—	—	0.30	0.30	0.30	0.30	0.30	0.30	0.30	0.30	0.30	0.30	0.30	0.30
	COD load, mg/L	—	—	—	—	—	—	376	376	376	376	376	376	376	376	376	376	376	376
4	H ₁ Stage																		
	Ca(OCl) ₂ applied, %	5.0	3.0	2.65	1.50	1.0	1.0	1.0	—	—	0.75	0.75	—	—	—	—	—	—	0.75
	COD load, mg/L	571	—	448	—	457	331	280	—	—	323	323	—	—	—	—	—	—	323
5	H ₂ stage																		
	Ca(OCl) ₂ applied, %	—	2.0	—	1.15	—	—	—	—	—	—	—	—	—	—	—	—	—	—
	COD load, mg/L	—	382	—	476	—	—	—	—	—	—	—	—	—	—	—	—	—	—
6	D ₁ stage																		
	ClO ₂ applied	—	—	—	—	—	—	—	—	—	0.70	0.70	0.70	0.70	0.70	0.70	0.70	0.70	0.70
	COD load, Mg/L	—	—	—	—	—	—	—	—	—	341	341	341	341	341	341	341	341	341
7	D ₂ stage																		
	ClO ₂ applied	—	—	—	—	—	—	—	—	—	0.40	0.40	0.40	0.40	0.40	0.40	0.40	0.40	0.40
	COD load, Mg/L	—	—	—	—	—	—	—	—	—	—	256	256	256	256	256	256	256	256
8	E ₁ O stage																		
	O ₂ pressure, kg/cm ²	—	—	—	—	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
	COD load, Mg/L	—	—	—	—	876	—	—	—	—	501	501	501	501	501	501	501	501	501
9	1.7EOP stage																		
	O ₂ pressure, kg/cm ²	—	—	—	—	—	—	—	—	—	2.0	2.0	—	—	—	—	—	—	2.0
	H ₂ O ₂ applied, per cent	—	—	—	—	—	—	—	—	—	1.5	1.5	—	—	—	—	—	—	1.5
	COD load, Mg/L	—	—	—	—	—	—	—	—	—	885	885	—	—	—	—	—	—	885
10	O ₂ stage ^{??}																		
	O ₂ pressure, kg/cm ²	—	—	—	—	6.0	6.0	6.0	6.0	6.0	6.0	6.0	6.0	6.0	6.0	6.0	6.0	6.0	6.0
	COD load, Mg/L	—	—	—	—	4685	4685	4685	4685	4685	4685	4685	4685	4685	4685	4685	4685	4685	4685
	Brightness, % (ISO)	68.9	69.6	78.3	82.1	83.2	84.5	85.6	86.2	86.5	86.5	86.2	86.9	87.5	88.6	89.6	89.6	89.6	89.6
	Opacity, per cent	82.08	81.32	74.19	73.82	75.84	72.47	74.01	74.67	74.03	71.28	72.77	71.28	72.77	73.71	73.71	73.71	73.71	73.71
	Bleaching conditions	C	E	H ₁	H ₂	C: D	H ₂	O ₂	E ₁ O	EOP	D ₁	D ₂							
	Consistency, per cent	3.0	10	10	10	10	10	10	10	10	7	7							
	Temp, °C	Ambient	70±2	45±2	45±2	70±2	45±2	70±2	105±2	70±2	70±2	70±2	70±2	70±2	70±2	70±2	70±2	70±2	70±2
	Time at temp, min	—	—	—	—	—	—	—	75	—	—	—	—	—	—	—	—	—	—
	Reaction time, min	60	60	120	120	90	120	90	30	60	60	180	180	180	180	180	180	180	180
	pH	2.5	11.4	11.2	11.2	4.2	11.2	4.2	11.8	10	10	4.4	4.4	4.4	4.4	4.4	4.4	4.4	4.7
	?? = NaOH 0.9 % (on o.d. pulp basis), ?? = NaOH 2%, MgSO ₄ 1.0%, Kappa number reduction 62%, Pulp yield after O ₂ stage 38.5%																		

Sl. No.	Bleaching sequence	Initial °SR	Pulp brightness, % (ISO)	Opacity, %	Breaking length, m	Tear factor	Burst factor	Double fold no
1	H	13.5	66.9	82.06	7450	81.67	60.00	132
2	HH	12.5	69.6	81.32	7485	81.82	60.87	140
3	CEH	13.5	78.3	80.76	7375	80.88	60.33	136
4	CEHH	15.0	82.1	80.52	7340	80.44	59.87	130
5	OC(EO)H	14.0	83.2	82.85	7465	81.48	60.95	137
6	OC(EOP)H	12.5	84.5	82.90	7493	81.88	61.02	140
7	O(CD)(EO)H	13.0	85.6	81.87	7375	80.84	60.75	134
8	O(CD)(EO)D	12.0	86.5	81.63	7350	80.56	60.50	133
9	O(CD)(EO)HD	14.5	88.2	81.50	7345	80.45	60.55	130
10	OC(EOP)H	12.5	86.9	83.40	7477	81.67	60.70	134
11	OC(EOP)D	13.0	87.5	83.55	7485	81.30	60.80	132
12	OC(EOP)HD	13.0	89.6	82.78	7336	80.28	60.32	126

produce dense and uniform sheet with high burst and tensile strength.

Table 2 shows the results of soda pulping of *S. Spontaneum*. Since the raw material is having low lignin content, therefore it requires lower dose of alkali to get pulp of optimum yield with low kappa number. When, *S. Spontaneum* is cooked with soda pulping process at different alkali doses i.e. 11 to 14 per cent (as Na₂O), maximum cooking temp of 155 °C for 60 min, liquor to wood ratio 5:1 and digester pressure 6.0 kg/cm², the screened pulp yield and kappa number both increases with increasing alkali dose from 11 to 12 per cent. Further, on increasing alkali dose both screened pulp yield and kappa number both decreases. The reason is that carbohydrates fractions degraded due to peeling reaction⁴. At an alkali dose of 12 per cent (as Na₂O) *S. Spontaneum* produces screened pulp yield of 43.9 per cent at kappa number 26.5. As the raw material is bulky in nature therefore, less amount of material will be loaded per digester, and per digester yield will also be low.

0.1 per cent AQ was added into the digester to reduce kappa number further at optimum cooking conditions. Table 3 indicates that the screened pulp yield increases by 2.1%, while kappa number reduces by 20.8 per cent. The soda AQ pulp was further prebleached with oxygen under conditions mentioned in Table 4. The oxygen pretreatment reduces the kappa number by 62 per cent whereas, pulp yield losses by 5.5 per cent.

Table 4 reveals the bleaching results of sodaAQ pulp bleached by single stage hypochlorite bleaching as well as by multi stage HH, CEH and CEHH bleaching sequences. The oxygen prebleached soda-AQ was also

bleached by OC(EO)H, OC(EOP)H, O(CD)(EO)H, O(CD)(E_o)D, O(CD)(E_o)HD, O(CD)(E_{op})H, O(CD)(E_{op})D and O(CD)(E_{op})HD bleaching sequences.

The soda-AQ pulp was bleached by a single stage hypochlorite at a dose of 5.0 % on OD pulp basis. The brightness of pulp improves to 66.9 % (ISO) with a COD load of 571 mg/L. In another set, the pulp was bleached with the same dose of hypochlorite in two distinct stages i.e., 60% in H₁ and 40% in H₂ stages (Table 4). The pulp brightness was improved by 2.7% compared to single stage hypochlorite bleaching. The hypochlorite anion acts as reductant in alkaline medium. OCl⁻ reacts with lignin present on surface but it does not react with hidden lignin due to steric hindrance. Therefore, application of hypochlorite in stages shows an improvement in brightness. The *S. spontaneum* pulp was further bleached by CEH and CEHH bleaching sequence. The pulp brightness in CEH and CEHH bleaching sequence was observed to about 78.3 and 82.1 % (ISO) respectively. The chlorination following alkaline extraction stages together can selectively remove 75-90 % of the lignin in the fiber after pulping³. It can be safely stated that no other chemical can achieve this performance as economically. It was also observed that the C stage was generally the first point where dioxins appeared. Of the bleaching filtrates, the E-stage filtrate was found to have the highest concentrations^{4,6}. Dioxins are known to cause several different tumor types at a number of sites within the body including the liver and thyroid⁷. The generation of dioxins may be reduced by extending delignification prior to bleaching. The kappa number was further reduced by oxygen delignification. An oxygen pressure of 5 kg/cm² reduces the kappa number by

62%. OC (EO)H and OC(EOP)H bleaching sequences produce pulps of brightness of 83.2 and 84.5 % (ISO) with a total COD load of 6226 and 6119 mg/L respectively. The dose of molecular Cl₂ was reduced by 56.7 % compared to CEHH bleaching sequence. The O₂ and H₂O₂ reinforced alkaline extraction not only improves the pulp brightness but decreases the kappa number of the pulp compared to non-oxidative extraction without affecting pulp viscosity⁸. The O(CD)(EO)H, O(CD)(EO)D and O(CD)(EO)HD bleaching sequences produce pulp of brightness of 85.6, 86.5 and 88.2% (ISO) with a total COD load of 5808, 5883 and 6086 mg/L respectively. ClO₂ is an oxidant which accepts five electrons per molecule in being reduced to Cl⁻ ion. The four electrons participate in oxidation reaction (oxygen) and only one electron in substitution reaction (chlorine) with lignin. The substitution of chlorine with ClO₂ increased the delignification efficiency and maximum delignification efficiency was achieved when the ClO₂ and Cl₂ were added to the pulp sequentially, the ClO₂ first, the benefit was even greater^{9,10}. The substitution of ClO₂ for Cl₂ reduced the formation of chlorinated dioxins¹¹⁻¹³. The bleaching sequences O(CD)(EOP)H, O(CD)(EOP)D and O(CD)(EOP)HD produce a pulp of brightness 86.9, 87.5 and 89.6 % (ISO) with a COD load of 6600, 6277 and 6856 mg/L respectively. The introduction of H₂O₂ in the oxygen reinforced extraction stage enhances the brightness of pulp.

Table 5 shows the mechanical strength properties of *S. spontaneum* pulps bleached by different bleaching sequences at 40±1 °SR. These results indicate that there is not much variation in physical properties of pulps bleached by oxygen based multi- stage bleaching sequences.

CONCLUSIONS

S. spontaneum is fast growing non-wood fibrous plant available abundantly throughout the year; therefore, it seems to be a potential nonwood fibrous plant as the cellulosic raw material for tropical countries like India with high population density and limited land resources for small scale

nonwood fibrous plant as the cellulosic raw material for tropical countries like India with high population density and limited land resources for small scale industries and supplement raw materials for large paper industries. *S. spontaneum* has thin cell wall with wide lumen and possess good flexibility, such fiber collapse well to form ribbon like structure providing more surface area for bonding. *S. spontaneum* has low lignin content and comparatively an open fiber structure. Hence, it requires lesser cooking chemicals and shorter cooking cycle. *S. spontaneum* shows good response toward oxygen pre-bleaching followed by multi-stage bleaching sequences. The bleach chemicals requirement was low in comparison of bleaching of wood pulps. The pulp can be bleached up to a brightness level of 90±1 % (ISO). Any one of the bleaching sequence may be chosen depending upon end use of the product and requirement of brightness. There is not much variation in the mechanical strength properties of paper produced by various bleaching

sequences.

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