Dissolving grade pulps from sesbania aculeata

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ABSTRACT

The pulping studies have bean done on S. aculeata for dissolving grade of pulps. The S. aculeata were analysed for their chemical and morphological characteristics. Liquor phase prehydrolysis have been carried out with and without mineral acid (0.20 gpl). The pulping studies were carried out by using different level of Sodium hydroxide (10,12,14,16 and 20%) and time at temperature (60, 90 and 120 mins). The pulps were bleached by using C-E H-D-SO_2 bleaching sequence. Viscose was formed by steeping, ageing and xanthation processes. The filteration constant was found to be KW 110.

It is well known fact that present forest resources can not meet the demand of raw material even for the existing units leaving aside the additional requirement for the future growth of pulp, paper and viscose industry. These industries in India have met the various challenges by adopting itself to the changed condition. The forest based raw materials are in tremendous short supply and cannot meet the projected pulp capacities. Further the depleting forest cover and the thrust to conserve ecological balance will force the industry to look alternative fibrous raw materials other than wood and bamboo. There is still wide scope for identifying some new potential non-wood fibrous plants for these industries. The present studies have been carried out on utilisation of Sesbania aculeata for dissolving grade pulp. Sesbania aculeata[1] (Dhaincha) is a agricultural byproduct, which is fast growing in nature. It attains a height of 3—6 M after 5—6 months. Like all legume, dhaincha also fixes atmospheric nitrogen with its root nodules. About 18,175 tonnes of dhaincha ploughed in one hectare yield about 77 kg. of nitrogen. The annual plant yield ranges from 11.3—13.8 tonnes of green matter/ha. The dhaincha is a light weight material with a packing density of 120—125 kgs/m^3.

EXPERIMENTAL

Raw Material

The S. aculeata were collected from local regions. The stalk of plant were chopped manually to get chips of acceptable size. The chips were screened and the size of accepted chips varied from 25 mm to 35 mm.

Chemical and Anatomical Studies

The air dried chips were disintegrated in a Laboratory WEVERK disintegrator. The —40+60 mesh portion was utilized for chemical analysis. All the analysis were carried out as per TAPPI methods. Section at different planes of the wood were cut on lietz base sledge microtome 1300. Fibre length, fibre diameter, lumen diameter and cell wall thickness were measured according to IS 5285. The results are reported in Table 1 and 2.

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TABLE—1

Proximate Chemical Analysis of S. aculeata

S. No.	Characteristics	Value %
1.	Cold water solubility	2.52
2.	Hot water solubility	3.44
3.	Alcohol-Benzene solubility (1:2 V/v)	3.60
4.	1% NaOH Solubility	21.35
5.	Lignin	20.20
6.	Pentosan	17.30
7.	Holocellulose	74.34
8.	Hemicellulose	25.40
9.	Alphacellulose	48 52
10.	Beta cellulose	11.75
11.	Gamma cellulose	13 20
12.	Ash	1.72
13.	Silica	0.50

TABLE—2

Anatomical Characteristics of S. aculeata

Sl. No.	Particulars		Value
1.	Colour		White
2.	Density	gm/c.c.	0.36
3.	Fibre length (Average L)	mm	0.843 (0.356—0.956)
4.	Fibre diameter (Average D)	micron	21.576 (10.836—31.240
5.	Lumen diameter (average d)	micron	14.82 (4.02—22.120
6.	Cell wall thickness (average W)	microm	3.38 (1.657—5.120
7.	Ratio of fibre length to diameter L/D		39.07
8.	Flexibility co-efficient (d/D) × 100		68 68

Prehydrolysis and Soda pulping

The prehydrolysis and pulping studies were carried out in WEVERK rotary autoclave digester of 20 liter capacity. Liquor phase prehydrolysis was done with and without addition of H_2SO_4(²). Cooking was done following soda process after draining and venting completely at the end of prehydrolysis. Prehydrolyzate and spent liquor were analysed for acidity, pH and free alkali. The extensive studies carried out in detail covering almost all the parameters that have a direct bearing on the resulting pulp properties. These include investigation of prehydrolysis and cooking at different time and temperature. Soda pulping was carried out at different level of active alkali (10 — 18%). The results are tabulated in table 3 to 6.

Bleaching

The pulp was bleached using C—E—H—D—SO_2 sequence. Soft water was used in every stage except for SO_2 stage where demin water was used. Bleached pulp was centricleaned and sheets were made by hand moulds. These were processed and dried in steam heated drying chamber. Sheets were processed to form viscose by using xanthation procedure and clogging constant Kw determined. The bleached pulp was analysed for important characteristics. The results are reported in Table 8.

Results and Discussions

The results of chemical analysis listed in Table 1 reveal that S. aculeata contain 48.52% alpha cellulose, 17.30% pentosan, 20.20% lignin, which place this fibre closer to hardwood. The fibre length (0.843mm) is short compared to softwood and bamboo. Lumen diameter and fibre diameter are good. Table 3 and 4 showed the effect of varying time (33—120 min) and temperature (155–165°C) during liquor phase prehydrolysis, when the time to temperature is kept constant. The acceptable degree of purification was achieved in about 60 min (acid prehydrolysis), 90 min(water prehydrolysis) at 165°C. As the time at temperature increased, the acidity of condensate formed increased, pentosans, yield and viscosity of unbleached pulp decressed. The P. no. of unbleached pulp decreased with increasing time at temperature during prehydrolysis.

TABLE—3

Effect of time and Temperature on prehydrolysed S. aculeata and unbleached pulp

A. Prehydrolysis

1.	Acid (H_2SO_4)	Nil
2.	Material to liq. ratio	1:3.5
3.	Time to max. temp.	90 min.
4.	Relieving time	30 min.

B. Washing — No washing

C. Soda cooking

1.	A.A. as Na_2O	14%
2.	Material to liq. ratio	1:3.5
3.	Time to max. temp.	90 min
4.	Max. temp.	160°C
5.	Time at max. temp.	90 min.
6.	Relieving time	15 mim.

S No.	Characteristics		1	2	3	4	5	6	7	8	9
1.	Max. temperature	°C	155	155	155	160	160	160	165	165	165
2.	Time to temp.	min	60	90	120	60	90	120	60	90	120
3.	P.H. yield	%	96.20	94.20	91.26	94.30	91.37	87.52	88.40	84.28	82.56
4.	Pentosan in prehydrolysed S. aculeata	%	16.42	14.75	11.46	15.10	10.56	9.25	11.52	8.10	5.92
5.	Acidity of prehydrolysate as H_2SO_4	gpl	5.10	5.92	6.80	5.60	7.66	8.40	7.60	8.84	10.72
Spent liquor											
6.	Free alkali	gpl	2.62	2.18	2.16	2.70	2.12	2.17	2.50	2.02	1.98
7.	A.A. consumption	%	13.08	13.23	13.24	13.05	13.25	13.24	13.12	13.29	13.30
Unbleached pulp											
8.	Yield	%	43.72	40.60	37.92	40.85	37.60	36 45	37.10	35 14	31.90
9.	P. No.		13.7	9-2	8.7	9.4	8.2	7.8	8.1	7.2	6.9
10.	Pentosans	%	11.52	7.80	4.96	8.20	4.75	4.60	4.36	3.15	2.82
11.	Viscosity	cp	65.95	62.70	53.10	58.70	54.10	45.80	46.20	39.80	29 64

Table—4

Effects of Time and Temperature on prehydrolysed S. aculeata and Unbleached pulp

A. Prehydrolysis

1. Acid (H_2SO_4) 0.20 gpl
2. Material : liquor 1:3.5
3. Time to max. temp. 90 min
4. Relieving time 30 min.

B. Washing — No Washing

C. Soda cooking

1. A.A. as Na_2O 14%
2. Material to liquor 1:3.5
3. Trme to max. temp. 90 min
4. Max. temp. 160°C
5. Time at max. temp. 90 min
6. Relieving time 15 min

S.No.	Characteristics		1	2	3	4	5	6	7	8	9
1.	Maximum temp.	°C	155	155	155	160	160	160	165	165	165
2.	Time at temp.	min	30	60	90	30	60	90	30	60	90
3.	P.H. Yield	%	95.90	93.98	91.02	93.95	91.10	87.80	88 50	84.60	82.32
4.	Pentosan in prehydrolysed S. aculeata	%	16.12	13.80	11.26	15.34	10.10	9.14	11.50	8.16	5.80
5.	Acidity of prehydrolyzate as (H_2SO_4)	gpl	3.96	4.10	4.65	4.94	5.25	6.86	7.40	8.72	10 12
Spent liquor											
6.	Free alkali as Na_2O	gpl	2 40	2.10	2.15	2.32	2.06	2.10	2.14	1.92	1.84
7.	A.A. consumed	%	13.16	13.26	13.24	13.18	13.28	13.26	13.24	13.33	13.36
Unbleached pulp											
8.	Yield	%	43.20	40.00	37.80	40.40	37.10	36.00	36.75	34.98	30.60
9.	P. No.		13.2	8.9	8.2	9.1	7.8	7.3	7.9	6.8	6.2
10.	Pentosan	%	11.06	7.15	4.26	7.90	4.10	3.96	4.04	2.92	2.56
11.	Viscosity	CP	63.70	60.20	49.92	52.10	50.61	41.74	42.35	38.46	29.40

TABLE—5

Effect of 'H' Factor on prehydrolysed S. aculeata and Unbleached pulp

A. Prehydrolysis

1	Acid (H_2SO_4)	0.20 gpl
2.	Material to liquor	1:3.5
3.	Time to max. temp.	90 mins.
4.	Max. temp.	165°C
5.	Time at max temp.	60 min.
6.	Relieving time	30 min.
7.	P.H. yield	84.60%
8.	Acidity of prehydrolyzate as H_2SO_4	8.70 gpl

B. Washing — No washing

C. Soda cooking

1.	A.A. as Na_2O	14%
2.	Material to liquor	1:3.5
3.	Time to max. Temp.	90mins.
4.	Relieving time	15 min

S. No.	Characteristics		1	2	3	4	5	6	7	8	9
1.	H factor		358	489	619	553	753	954	840	1145	1450
2.	Max. temp.	°C	155	155	155	160	160	160	165	165	165
3.	Time at temp.	min	60	90	120	60	90	120	60	90	120
Spent liquor											
4.	Free alkali	gpl	3 20	2.94	2.40	2.38	1.92	1.98	2.19	1.80	1.76
5.	A.A. consumption	%	12.88	12.97	13.16	13.16	13.33	13.31	13.23	13.37	13.38
Unbleached pulp											
6.	Yield	%	39.20	36 90	34.62	36.40	35.00	32.20	36.00	33.10	30.85
7.	P. No.		9.7	8.6	7.2	8.4	6.8	6.0	8.1	6.1	5 4
8.	Pentosan	%	4.90	4 40	3.87	3.30	2.94	2 70	3.22	2.82	2.56
9.	Viscosity	cp	54 34	46.10	40.21	47.80	38.46	34.24	42.65	36.92	29.20

TABLE—6

Effect of varying Active alkali charge on prehydrolysed S. aculeata and Unbleached pulp

A. Prehydrolysis

1.	Acid (H_2SO_4)	0 20 gpl
2	Material to liquor	1:3.5
3.	Time to Max. temp.	90 mins.
4.	Max. temp.	165°C
5.	Time at Max. temp.	60 min.
6.	Relieving time	30 min.
7.	P.H. Yield	84.25%
8.	Acidity of prehydrolyzate as H_2SO_4	8.74 gpl.

B. Washing — no washing

C. Soda cooking

1.	A.A. as Na_2O	14%
2.	Material to liquor	1:3.5
3.	Time to max. temp.	90 mins
4.	Max. temp.	160°C
5.	Time at max. temp.	90 mins
6.	Relieving time	15 min.

S. No.	Characteristics		1	2	3	4	5
1.	Active alkali as Na_2O	%	10	12	14	16	18
Spent liquor							
2.	Free alkali as Na_2O	gpl	1.22	1.68	1 92	2.64	2.96
3.	A. A. consumption	%	9.57	11.41	13.33	15.07	16.96
Unbleached pulp							
4.	Yield	%	42 10	36.52	34 98	33.40	30.00
5.	P. No.		11.20	7.9	6.8	6.2	5.8
6.	Pentosans	%	8 12	4.30	2.92	2.86	2.42
7.	Viscosity	CP	56 45	48.67	38.46	34.20	26.92

TABLE 7

Optimized Soda pulping

A.	Prehydrolysis	
1.	Acid (H_2SO_4)	0.20 gpl
2.	Material to liquor	1 : 3.5
3.	Time to max. temp.	90, mins.
4.	Max. temp.	165°C
5.	Time ai max. temp.	60 mins.
6.	Relieving time	30 mins
7.	P. H. yield	84.72%
8.	Acidity of prehydrolyzate as H_2SO_4	8.74 gpl
B.	Washing	No washing
C.	Cooking	
	A.A. as Na_2O	14%
	Material to liquor	1 : 3.5
	Time to max. temp.	90 mins.
	Max. temp.	160°C
	Time at Max. temp.	90 mins.
	Relieving time	15 mins.

S. No.	Characteristics		Particulars*
Spent liquor			
1.	Free alkali	gpl	1.90
2.	A.A. consumed	%	13.34
Unbleached pulp			
3.	Yield	%	35.06
4.	P.No.		6.8
5.	Pentosans	%	2 95
	Viscosity	CP	38.51

*Avg. of 10 digestion

TABLE 8

Bleaching conditions and Bleaehed Pulp characteristics

S. No.	Stages	Chemical as av. Cl_2, NaOH and SO_2 on o.d. pulp %	Consistency %	Temperature °C	Retention time min
1.	Chlorination	2.0	3	30	30
2.	Alkali extraction	2.0	10	65	90
3.	Hypochlorite	1.5	10	40	120
4.	ClO_2	0.25	10	75	180
5.	Acid (SO_2)	1.0	3	45	30

Pulp Characteristics		Value
6. P.No.		0 3
7. Viscosity	CP	9.2
8. 1% NaOH solubility	%	2.82
9. 7.14% NaOH solubility	%	9.60
10. 10% NaOH solubility	%	3.80
11. Copper Number		0.58
12. Pentosans	%	3.12
13. Alpha Cellulose	%	93.12
14. Beta cellulose	%	6.16
15. Gamma cellulose	%	1.05
16. Brightness (EL)	%	92.0
17. Clogging constant $Kw_{20,\ 40}$		110
18. Yield	%	33.4

The soda cooking were performed at different level of active alkali charge (10—18% as Na_2O) indicated that the total pulp yield is in indirect proportion with active alkali charge i.e. as the active alkali charge increases the total pulp yield decreases. Soda cooking were also performed at different H factor (358—1450). The pulp yield, P. no., pentosans and viscosity decreased as temperature and time at temperature increased. By considering the optimum yield, pentosan and viscosity, I4% active alkali (as Na_2O), temperature 160°C and time at temperature 90 min. was found suitable for the soda cooking of S aculeata Ten batches of pulps were prepared by using optimized conditions. It was observed that the pulps were readily bleachable under the condition chosen. The Table 8 showed that pulp having brightness about 92% (El), alpha cellulose 93.12% and pentosans 3.12% can be produced easily.

CONCLUSIONS

The results of chemical analysis, particularly high alpha cellulose content and low ash are encouragingly in favour of processing the S. aculeata to rayon grade pulp. Anatomical results are quite suitable except fibre length . Fibres present in S. aculeata wood are short in length compared to softwoods and bamboo. Short fibres of the pulp do not seem to affect viscose filterbility, and the fibres are highly reactive toward xanthation. Acid prehydrolysis was found to be most suitable for such raw material for the production of dissolving pulp. Bleached pulps when prepared using C—E—H—D —SO_2 sequence gave pulp of acceptable quality for the production of normal staple fibre. From the foregoing study it may be remarked that S. aculeata can be processed to yield rayon grade pulp of satisfactory characteristics. A clogging constant Kw (110) was found quite satisfactory and acceptable for rayon grade pulp. On the basis of the above experimental work, it was concluded that S. aculeata is quite suitable non-wood fibrous raw material for dissolving pulps. It can be used as a partial replacement of conventional raw material.

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