Alkaline Sulphite Pulping of Jute (JRC-321 Variety)

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ABSTRACT

Jute as a raw material for high quality paper is known to have many advantages. In contrast it has some constraints because of contaminants viz; husk, dirt, coarseness and difficulty in removing lignin. As various grades of jute are available, the choice of the right quality fibre in respect of fineness and flexibility is one such way. With this objective in view, JRC - 321 jute variety, which is known as the finest variety, has been tried for pulping by both Kraft and moderately alkaline sulphite process. The first set of pulping experiment of jute (JRC-321 variety) is carried out by kraft process at different percentages of total chemicals under same sulphidity (20%), liquor ratio (1:3), cooking temperature (160°C) and cooking time (2hrs.). It has been observed that at 9.35% total chemical (as Na_2O) gives higher yield and good strength properties. In the second set of experiment different ratios of sodium sulphite to sodium hydroxide are used with the same cooking time (3hrs.), cooking temperature (160°C) and liquor ratio (1:4). It is observed that acceptable pulping conditions are achieved at 6.4% sulphite (as Na_2O)and 2.33% NaOH (as Na_2O). The unbleached Kraft pulps are bleached by CEH sequence which gives 82% brightness (P.V.) whereas alkaline sulphite pulps are bleached by CEHH sequence which gives 81.5% brightness (P.V.). Both unbleached and bleached pulps are evaluated for various physical properties.

INTRODUCTION

Cellulose fibres are produced not only from trees but also from a number of non-wood plants including agricultural residues that are the source of cellulose. Non-wood plants are more common as raw material in some countries especially where wood is scarce. At present the Planning Commission has also indicated that paper and board production will have to be increased to about 42.5 lakh tonnes due to the tremendous increase in population and literacy. The existing forest resources which are already in shortage, are barely able to sustain the forest production rate. On the other hand the increasing demand of fibrous raw material for pulp production has affected the natural eco-system and environment due to deforestation. Therefore, it is necessary that attention should be given to utilization of agricultural residues and non-wood fibrous materials for the production of high yield pulp using latest technology which will help in reducing the environmental pollution.

Indian paper industries are set up to use forest based raw material and technology was established as such. Serious limitations for the availability of raw materials are observed, due to the poor forest cover which has been determined to be less than 16% of the total land. Attention should be given on the national priority for the conservation and regeneration of forest cover. With continued limitation of forest based raw material, Indian paper industries have to use non-conventional raw material like waste paper, bagasse, agricultural residues, jute etc.

Among the long fibre non-woody plants, jute is the major raw material which is available in plenty in our country. There are two major varieties of jute fibres viz; Capsularis (white) and Olitorious (tossa). The major Capsularis varieties are JRC -321, JRC-744, 7D -154 etc. It has been proved that white variety is finer than tossa variety. It has also been observed that in fibre quality characters other than fineness, the capsulary varieties do not differ significantly. Under identical agronomical conditions and uniform retting practices, JRC-321 would poduce better fibre than other standard Capsularis. The yarn quality characters, yarn tenacity(1) and strength, indicate the supremacy of JRC-321 variety.

Pulping of jute by conventional processes has been the subject of numerous studies for the last decades both domestically and abroad. There are certain technological barriers requiring R&D breakthrough. These are (1) effective removal of contaminants like bark, dirt, plastics, rubber and other trash materials, (2) increasing pulp brightness & (3) reducing stiffness of fibre without sacrificing strength. Studies along these lines have been carried out in this present investigation including the choice of right type of raw material. Some work on pulping[2-4] of jute has been done but no work has been done on pulping of this variety i.e., JRC-321.

Our present investigation has been carried out to make pulp from this variety by kraft process using Na_2S and NaOH as cooking chemicals and alkaline sulphite process under moderate alkaline condition using Na_2SO_3 and NaOH. This alkaline sulphite process has the advantages of both alkaline (kraft) and acidic (sulphite) pulping process. Laboratory trials so far showed encouraging results for alkaline sulphite process rather than Kraft process which is being reflected in this paper.

EXPERIMENTAL

Choice of raw material and sampling for pulping

There are different grades for each jute variety (viz; w w_2 w_3 w_4 w_5 w_6 w_7 and w_8. The gradation is given on the basis of six physical characters e.g. (1) root content (2) strength (3) defects (4) colour (5) fineness and (6) density.

The choice of raw material for our present study of pulping was the medium grade, the fibres were mostly free from root content, fine, brownish to grey white in colour but contained major body defects and of heavy bodies. This gradation was confirmed from the following observations[5] which are shown in the Table -1 below.

The above grades (w_5 -33% up) JRC-321 jute fibre were collected from Purnea (Bihar) India. The whole fibre including barks, dust were chopped to the required size (Ca.2.5 inch in length). The chopped materials were mixed uniformly and 1.5 Kg air dry material was packed in several packets. Moisture content of the material was estimated. The same sample was also used for proximate chemical analysis and testing for morphological characteristics, which are shown below:

Proximate Chemical Analysis

The proximate chemical analysis of this variety of jute sample was carried out as per TAPPI Standard Method(6). The physical and chemical characteristics of this variety are recorded in Table -2.

Morphological Characteristics

The various morphological characteristics i.e., density, fibre length, fibre diameter, cell wall thickness, lumen diameter etc. of jute variety (JRC-321) and its comparision with Bagasse and Bamboo(7) are shown in Table -3.

Table-2

Proximate Chemical Analysis of Jute (JRC-321 Variety) and its comparision with Bagasse and Bamboo(7)

Particulars (%)	Jute (JRC-321)	Bagasse	Bamboo
Cold Water Solubility	2.45	5.91	7.6
Hot Water Solubility	1.42	7.85	8.5
Alcohol-Benzene Solubles (1:2 v/v)	0.9	6.3	3.4
1% NaOH Solubility	12.44	33.6	26.8
Lignin	14.9	20.3	24.3
Pentosan	16	23.85	18.3
Holocellulose	82.94	70.6	71.5
Hemicellulose	23.1	28.45	27.6
α-Cellulose	59.36	42.0	43.5
Ash	1.76	3.8	2.1
Acetyl Content	3.2	-	2.5

Table-1

Particulars	Root Content by Wt.%	Defect by Wt. %	Fibre Tenacity Gm/Tex	Fineness Tex	Colour (Brownish to Grey)	Bulk Density GM/cc	Total score Mark	Gradation	Remark
JRC-321	2	2	15	1.5	4.6	0.45	W_5-59	33% up	Medium Quality
Score Marks	33	4	14	2	4	2			

Pulping Studies

a. Kraft Pulping

Kraft pulping of above air dry packed (1.5Kg) materials were separately done in a 10 litre capacity electrically heated laboratory stainless steel rotary digester.

The pulpings were carried out at same cooking time, sulphidity and liquor ratio but at different percentages of chemicals. After cooking, the black liquors were collected by filtering through 100 mesh sieve and analysed for total solid and free alkali content. The cooked pulps were washed first with tap water and then simultaneously defibrilated and washed in a Valley Beater. The washed pulps were then analysed for yields, brightness and the permanganate numbers estimated. The pulping parameters and their results are shown in the Table -4.

Table-3

Morphological Characteristics of jute (JRC-321) fibre, bagasse and bamboo

Particulars (JRC-321)	Jute	Bagasse	Bamboo
Density gm/cc	1.42	-	0.52
Fibre Length L (mm)	2.6	1.2	1.7
Fibre Width (D) μ (micron)	18.0	-	23.6
Lumen Width (d) μ (micron)	6.5	20.00	9.3
Cellwall Thickness (w) μ (micron)	5	-	7.0
Flexibility Co-efficient d/D x 100	36.1	-	40.56
Ratio of length ot width L/D	144	85.0	72.03
Ratio of twice Cell wall Thickness to the fibre width 2 w/D	555	-	0.59
Wall Fraction 2 w/Dx100	55.5	-	59.3
Runkel 2 w / d	1.5	-	1.47
Ratio of Wall Thickness to lumen width w/d	0.77	-	0.74

Bleaching Study

A three stage (CEH) bleaching sequence (Chlorination, alkali extraction and hypochlorite treatment) by conventional method was applied to each kraft pulp and their results are recorded in Table - 6.

Table-4

Kraft Pulping Parameters

Particulars	Cooking No. 1	2	3
Total Chemical % (as Na_2O)	15.54	12.46	9.35
Sulphidity %	20	20	20
Bath Ratio	1:3	1:3	1:3
Cooking Schedule in Minutes			
I. Room Temperature to 120°C	45	42	50
II. 120°C to 160°C	80	80	70
III. At 160°C	2Hrs.	2Hrs.	2Hrs.
Pulp Yield (%)	54.43	59.62	62
Permanganate No. (using 25ml. 0.1N of $KMnO_4$)	9	11	16
Black Liquor Characteristics			
I) Total Solid (w/w) %	25	21.01	19.12
II) Free Alkali in GPL	16.64	6.66	4.2
Brightness of Cooked pulp (P.V. Cell)	36.5	36.4	26

Pulp Evaluation

Unbleached and bleached paper sheets from each type of pulp were prepared by sheet making machine. The sheets were pressed in between blotting papers under auto compression for 5 & 3 minutes consecutively at 3.5 Kg/cm^2 pressure. Then the sheets were dried in air. The physical strength properties of both bleached and unbleached airdry sheets for each of the cooked pulps at different beating times under the same condition were evaluated. The results for unbleached sheets are shown in the Table -5 while for

Table-6

Bleaching Results of Kraft Pulp

Particulars	Cooking No. 1	2	3
Total Cl_2 added on pulp %	3.3	4.71	7.55
Total Cl_2 consumed on pulp %	2.81	4.1	6.45
Bleaching Loss %	5	6.2	5.1
Brightness % P.V.	80.2	83.9	82
Cu No. of Bleached Pulp	2.1	2.22	1.22

bleached sheets are given in Table -7.

b. Alkaline Sulphite Pulping

Moderate alkaline sulphite pulping for the same raw material was separately done in the same rotary digester. The work involved the different ratios of sodium sulphite to sodium hydroxide under same cooking time (3Hrs.), temperature (160°C) and liquor ratio (1:4). The washing of the pulps and collection of the black liquor were done using same procedure as for the kraft process. The black liquors were used for analysing total solid content, residual sulphite and pH respectively. The yields and permanganate nos. of each type of pulp were also estimated. The pulping parameters and their results are shown in the Table -8.

Bleaching Study

A four stage (CEHH) (Chlorination, Alkali Extraction, Hypochlorite - Hypochlorite Treatment) bleaching sequence for the cook number 5&6 and a three stage (CEH) bleaching sequence for cook number 7&8 were followed by the conventional methods and their results are shown in the Table -10.

Pulp Evaluation

Unbleached and bleached pulps for each cooked material were beaten at different beating times under same conditions. The same procedures were followed for preparing paper sheets from both unbleached and bleached pulps as it was done for kraft process. The physical strength properties of both unbleached and bleached sheets were evaluated and their results are shown in the Table -9 & Table -11 respectively.

RESULTS & DISCUSSION

The results of proximate chemical analysis indicate that the ash and lignin content of this variety of jute is lower in comparison to that of bagasse and bamboo but the holocellulose and α cellulose contents are on the higher side. As holocellulose content is high and 1% NaOH solubility is low, this material will yield higher percentage of bleached pulp. As lignin, pentosan and ash content are lower in comparison to those of bamboo and bagasse, it indicates that this jute variety will require milder cooking condition.

Table-5

Unbleached Kraft Pulp Evaluation

Cook No	Stretching (%)	Beating time in min.	Freeness in °SR	Bursting Index kPam²/g	Tensile Index mNm²/g	Tearing index MN m²/g	Double fold K mollion
1	x	0	15	x	x	x	x
	x	15	18.9	2.1	27.16	12.45	9
	x	30	25.2	2.6	38.48	10.4	20
	x	45	33	2.8	43.3	9.05	20
	x	60	43.3	2.823	43.72	8	21
	x	75	56.6	3.46	48	7.3	28
	x	80	65	3.53	50.2	6.43	21
2	0	0	15	x	x	x	x
	4	15	18.6	2.36	37.25	17.35	56
	3.125	30	23.7	2.88	40.2	17.54	261
	4	45	32	4.41	58.82	20.0	1100
	4.25	60	40	4.42	63.72	18.3	1944
	4	90	59	5.2	74.3	16.5	>2500
3	x	0	16.5	x	x	x	x
	2.81	15	21	3.64	61.2	23.853	218
	3.0	30	27	5.2	69	21.8	870
	3.37	45	36	6.6	80.8	20.3	914
	3.81	60	43	7.3	88	19.54	1210
	4.12	75	55	8.0	95	17.55	1245
	4.44	90	65	8.1	98	17.08	>2000

Table-7

Unbleached Kraft Pulp Evaluation

Cook No	Beating time min.	Freeness (°SR)	Stretching (%)	Bursting Index kP_am^2/g	Tensile Index Nm/g	Tearing index $mN\ m^2/g$	Double fold K mollion
1	0	16.5	x	x	x	x	x
1	15	24.5	x	2.41	40.5	5.72	x
1	30	46	x	2.46	43.2	3.92	x
1	40	59	X	2.26	44.46	3.22	x
2	0	15.5	x	x	x	x	x
2	15	20.5	2.44	3.0	30.04	10.5	15
2	30	31.0	2.37	3.7	52.55	8.2	25
2	45	43.0	2.75	3.63	59.7	7.26	23
2	60	57	2.94	4.3	62.9	5.93	19
2	70	65	3.31	4.1	64.56	5.63	28
3	0	17	x	x	x	x	x
3	15	22.5	3.31	3.34	43.63	18.14	76
3	30	32	3.87	4.22	55.4	16.3	1800
3	45	43	4.12	5.05	63.14	15.44	>2000
3	60	59	4.0	6.25	68.4	14.12	>2000
3	70	65	4.0	6.06	70.83	13.1	>2000

Table-8

Effect of Amount of Na_2SO_3 at Constant Alkali Dose

Cook No	Chemical change (as Na_2O)		Time at Temp. 160°C in Hr.	pH of Cooking Liquor	pH of black Liquor	Total solid in Black Liquor (w/w)	Residual Na_2SO_3 in Black Liquor	Yield of Pulp in %	Permanganate no: (Using 40 ml. N/10 $KMnO_4$ Used)
	Na_2SO_3	NaOH							
4	7.874	2.33	3	12	8	14.5%	1%	67.8	15
5	6.4	2.33	3	11.5	7.5	13.9%	0.22%	69.7	17
6	4.93	2.33	3	10.5	6.5	13.15%	-	70	18
7	7.874	1.94	3	11.5	7.2	12.8%	1.26%	69.4	17
Effect of Amount of NaOH at Constant Na_2SO_3									
8	6.4	1.55	3	10.5	7.5	13%	0.13%	71.8	19
9	6.4	2.33	3	11.5	8	13.9%	0.22%	69.7	17
10	6.4	3.87	3	12.5	8.5	15.18%	0.131%	65.64	23.5
11	4.93	3.87	3	12.5	8	14.8%	0.03%	69	25

Table-9

Unbleached AS-Pulp Evaluation at different Chemical %

Cook No	Cooking Chemical % (as Na_2O) Na_2SO_3	NaOH	Beating Time in min.	Consistency %	Substance in gm	Freeness °SR	Bursting Index KP_am^2/g	Tensile Index Nm/g	Tearing Index mNm^2/g	Double Fold K Mollion	Stret-ching %
			0	1.67	x	16	x	x	x	60 4	x
			15	1.67	60.5	20.5	4.47	54	25.12	23.00	1.7
5	6.4	2.33	30	1.53	62	31	7.40	66	22.45	22100	2.32
			45	1.51	64	36.5	8.14	72	20.21	22500	3.25
			60	1.51	63.4	45.5	8.7	79	19.48	22500	3.75
			0	1.5	77.3	18	2.76	27.45	22.84	47	2.87
6	4.935	2.33	15	1.67	62.3	34	4.12	52.74	22.06	818	3.12
			30	1.6	62.7	34	4.97	69.06	20.6	>2500	3.56
			45	1.56	62.7	45	6.9	76.86	18.72	2850	3.94
			0	1.56	51	17.6	3.31	38.1	21.53	58	4.0
			15	1.46	59.2	25.3	5.82	54.1	25.2	1600	3.7
7	7.874	1.93	30	1.45	58.6	36	7.44	63.82	18.4	1900	3.5
			45	1.45	54	51.5	8.23	88.92	17.25	2500	4.125
			60	1.4	60	64	7.9	87.25	16.34	>2500	4.0
			0	1.52	64.7	17.5	1.77	19.46	16.67	8	3.12
			15	1.5	59.0	28.5	5.55	46.53	25.92	1380	3.87
8	6.4	1.55	30	1.46	59.3	37.0	6.66	66.67	22.16	>2500	5.00
			45	1.42	60.6	50	7.14	67.16	20.68	>2500	4.12
			60	1.43	61.3	64	7.225	74.63	19.5	>2500	4.56

Comparative microscopic examinations of fibre indicates that JRC-321 jute variety is longer in fibre length than those of bagasse and bamboo. This resembles softwood and produces pulp of higher strength particularly tear strength[8]. As the length is high the fibre is finer. But the width and lumen diameter of the fibre are lower than those of bagasse and bamboo which resembles hardwood. Increased density generally means increased pulp strength and high yield per digester. So high density is desirable both for improved pulp quality and reduced manufacturing cost. As JRC-321 variety shows high density in comparison to that of bagasse and bamboo, the pulp quality and yield should be higher and manufacturing cost should be lower. As the cell wall thickness is very low which indicates low wall fraction and Runkel ratio, this type of fibre gives stronger pulp. Due to the thin-wall fibres, they are more plastic and give rise to more bonding area on beating and produce high strength paper.

From the cooking parameters of kraft pulp it indicates that with increasing the cooking chemicals, the pulp yield and permanganate number decreases. The pulping results at different percentages of chemicals are shown in Table -4. The strength properties of the unbleached paper sheets made from kraft pulp are shown in Table-5. The results of cook no:1 show that it is overcooked under same condition. With decreasing the cooking chemical, the strength

Table-10

Bleaching Results of AS-Pulp

Particulars	Cooking Numbers 5	6	7	8
Total Chlorine added in Pulps on %	5.95	9.81	10.35	8.32
Total Chlorine consumption on Pulp (%)	5.14	7.76	8.78	7.2
Bleaching Loss %	5	6	8	8
Brightness % P.V.	80.4	77.5	86.5	86.3
Copper No. of Bleached Pulp	0.85	0.9	1.12	1.1

Table-11

Bleached AS-Pulp Evaluation at Different Chemical Percentages

Cook No	Cooking Chemical % (as Na_2O)		Beating Time in min.	Consistency %	Substance in gm	Freeness °SR	Bursting Index $KP_a m^2/g$	Tensile Index Nm/g	Tearing Index mNm^2/g	Double Fold K Mollion	Stret-ching %
	Na_2SO_3	NaOH									
			0	1.69	x	16.5	x	x	x	60.4	x
			15	1.57	61.5	22.5	3.98	42.51	290	22.94	1.75
5	6.4	2.33	30	1.54	63.0	31.0	5.9	60.2	>25000	19.3	3.14
			45	1.53	61.6	40.5	7.3	75.4	>25000	16.86	3.75
			60	1.53	67.7	53	7.25	76.45	>25000	16.1	3.87
			0	1.69	66.8	18	1.65	20.8	18	17.55	2.81
6	4.935	2.33	15	1.43	63.4	25	2.82	44.31	256	19.2	3.19
			30	1.53	63.0	35	4.7	47.06	925	16.76	3.56
			45	1.5	63.3	48	5.4	68.63	1220	16.1	3.87
			0	1.75	51	17.5	2.48	25.47	10	14.03	3.19
			15	1.59	50.3	27.6	3.46	51	195	11.1	3.69
7	7.874	1.93	30	1.43	50.0	48.3	5.41	62.1	335	8.82	3.8
			47	1.45	51.5	68	5.52	68.7	226	7.04	3.81
			0	1.63	62	19.5	2.3	24.8	17	19.4	3.94
			15	1.53	56.5	29.0	5	47.86	456	21.2	3.75
8	6.4	1.55	30	1.48	58	41.5	6.6	63.95	1986	18.1	4.25
			48	1.48	64	63	7.14	68.92	>2500	17.45	4.25

properties increase and at 9.35% total chemicals (as Na_2O) it gives better strength properties and high yield although the permanganate no. is slightly high (Cook no:3). As pulp yield is high which is one of the indications of low cost factor and strength properties are also high, so 9.35% cooking chemicals for kraft pulping is most acceptable under these conditions.

From Table-6, it is seen that a slightly higher dose of Cl_2 is required for CEH bleaching sequence for the cooked number -3. Although the Cl_2 consumption is high in comparison to that of other cooks, the copper no. is low for achieving high brightness. This indicates that carbonyl group formation during bleaching is probably low. In other words, less degradation of pulp during bleaching is taking place. This is further confirmed from the high strength values of the bleached sheets which are shown in Table -7. Thus the above observations help to consider that the conditions for cook number -3 are quite acceptable in comparison to other cooks.

The strength properties of both unbleached and bleached sheets for the cook no.3 are plotted against the freeness (°SR) which are shown in the Fig.1 and II respectively. These curves indicate that 55°SR and for bleached sheets 60°SR give the maximum strength properties for unbleached and bleached sheets respectively.

The results of sulphite pulping experiments at different doses of sodium sulphite keeping NaOH dose constant indicate that the pulping rate is dependent on the sodium sulphite charge. Table - 8 indicates that both pulp yield and permanganate number follow a continuous decreasing trend with increasing sulphite dose which is responsible for enhancing delignification. On increasing the NaOH charge but keeping the sodium sulphite dose constant, the pulp yield and permanganate number decrease. As the residual sulphite and NaOH in the black liquor are in very little amounts, further increasing the cooking time under same condition is not necessary. So 3 hrs. cooking is the acceptable cooking time at this condition. It is also found that with increasing the alkaline dose from 2.33 to 3.87% (as Na_2O) the yield decreases and permanganate number increases. The yield decreases due to more loss of hemicellulose at this higher alkali dose (3.87%). But permanganate number, instead of decreasing, is increasing because a reddish colour is developed after washing the cooked

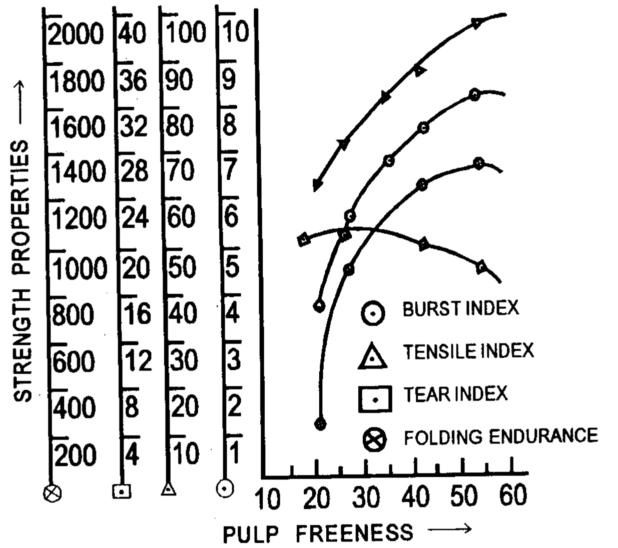

FIG. : 1 Plots of Strength Properties vs. Freeness of Unbleached Kraft Pulp

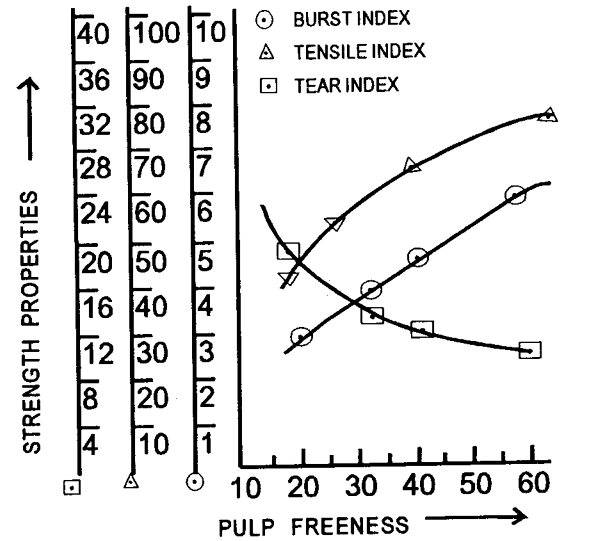

FIG. : 2 Plots of Strength Properties vs. Freeness of Unbleached Kraft Pulp

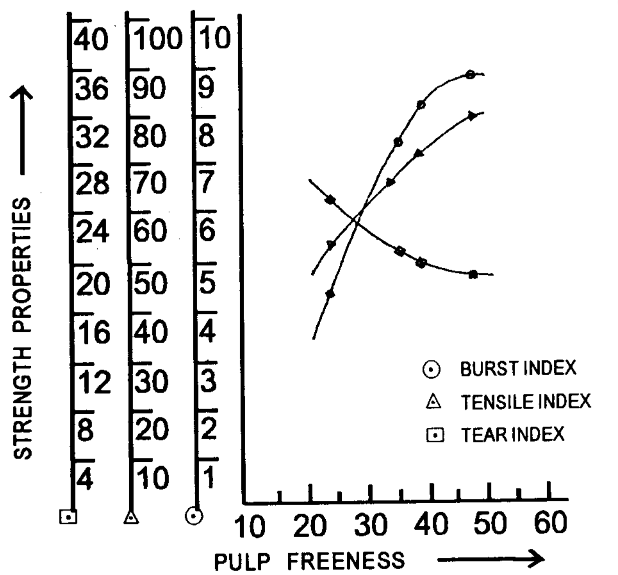

FIG. : 3 Plots of Strength Properties vs. Freeness of Unbleached Pulp

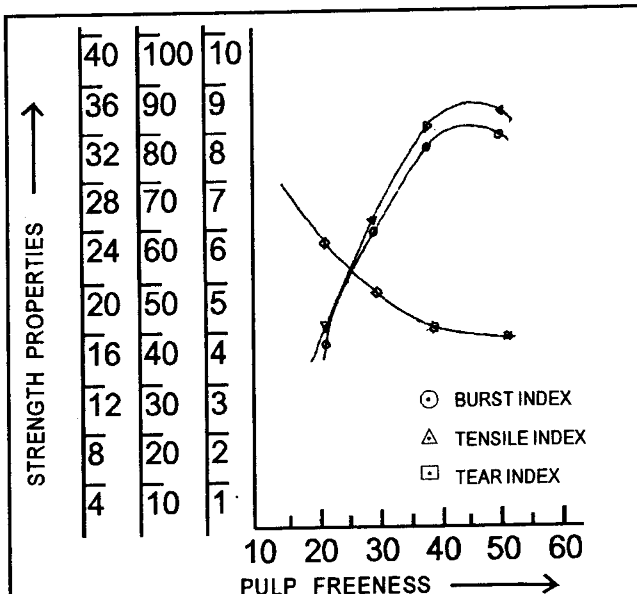

FIG. : 4 Plots of Strength Properties vs. Freeness of Unbleached Pulp

pulp which is not found at low alkali dose (2.33%). Naturally, more permanganate will be needed to oxidize this colour and ultimately it shows high permanganate value. So from the consideration of yield and permanganate number, the optimum dose of sulphite is 6.4% (as Na_2O) and NaOH is 2.33% (as Na_2O) respectively which is also supported from strength properties of unbleached and bleached sheets. The strength properties of both unbleached and bleached pulps for cook no:5 are plotted against the freeness (°SR) which are shown in the Fig. III & IV. From the graph it is indicated that for unbleached pulp gives the maximum strength properties at 46°SR whereas for bleached pulp gives the maximum strength value at 48°SR.

It has been observed that if the middle portion of the fibre (w_3-w_4 grade) are used for moderate alkaline sulphite pulping[9], higher yield (74.5%) and high brightness (86.5%) bleached pulp (using CECEH bleaching sequence) with good strength properties could be obtained.

CONCLUSION

The present investigation illustrates the advantage of alkaline sulphite process over kraft process. It also does not produce any odoriferous sulphides such as CH_3SH and (CH_3) S which are usually produced in the kraft process that creates atmospheric pollution. It provides a simplified and more economical pulping process although it requires more digestion time. As lignin, pentosan and ash contents for this fibre are low, mild cooking can give pulp of required strength. Although alkaline sulphite process requires higher cooking time than kraft process, high yield and high strength properties give the low cost factor.

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