Neutral Sulphite Chemimechanical Pulping of Eucalyptus Hybrid

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High yield Chemimechanical pulps were obtained in the Laboratory from Eucalyptus hybrid using the following stages (a) steaming of chips, (b) impregnation of steamed chips with sodium sulphite liquor, (c) vapour phase cooking, and (d) refining of semicooked chips. Unbleached pulp yields obtained were in the range of 81.5 to 85.2 percent with brightness of 41 to 51 percent. The maximum brightness was obtained when 9% $Na_2 SO_3$ was retained for vapour phase cooking and by adding 0.5% EDTA during impregnation. The pulp was stronger when 11.9% $Na_2 SO_3$ was retained for vapour phase cooking than when 6.9% $Na_2 SO_3$ was retained. These two pulps were respectively blended in different proportions with bleached bamboo kraft pulp. The standard sheets of the blend of bamboo and former pulp were stronger than those of the standard sheets of the latter pulp and blended bamboo pulp. The highest yield pulp obtained was the weakest in strength values.

The strength values of the pulps were moderate and the pulps had good bulk. When blended with bleached bamboo kraft pulp, standard sheets of satisfactory strength values, moderate brightness and good bulk could be obtained.

The impregnability tests revealed that the amount of $Na_2 SO_3$ retained increased by increasing the concentration of impregnation liquor. When steamed chips were impregnated with a Na_2SO_3 solution of 160 grams per litre, and draining off the liquor after retaining for 10 minutes at 100°C, 12.02% $Na_2 SO_3$ was retained: The corresponding amount when impregnated with a liquor of 100 grams per litre $Na_2 SO_3$ was 7.78 percent. The wood to liquor ratio after steaming and impregnation was almost constant at 1:1.22.

INTRODUCTION:

The neutral sulphite semichemical process is being used since many years for producing pulps in the yield range of 65-80 percent. Recent trend in this field is to obtain still higher yield(1). This has been made possible by impregnating the chips with liquor under pressure and heat and draining off the excess liquor and followed by vapour phase cooking and refining. Some of the commercial installations of this type are (a) Diamond National at Red Bluff, California for soft wood, (b) Stora Kopparberg at Falun, Sweden for birch, (c) Blandin Paper Co., using Balsam fir, and (d) Troirscosa Semichemical Plant at Saici, Italy(2) for hardwoods. These processes are gaining impetus because of decreasing wood resources and increasing wood costs. These high yield pulps are designated as Neutral Sulphite Chemimechanical pulps.

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In Diamond National(1) after 1st stage impregnation with sodium sulphite-sodium bisulphite (pH=7) at 150°F, and hydraulic pressure of 200 p.s.i., the chips go to pressafiners and finally to two stage double disc refining. A groundwood type of pulp which has long fibre , high tear, high bulk, good opacity and relatively low burst, is obtained.

At Stora Kopparberg(1) installation, the chips are steamed at atmospheric pressure, 2nd stage compression by screw presses and the pressed chips are fed, while still compressed through, a tower with cooking liquor. The expanding chips absorb the liquor and finally they are cooked in an atmosphere of steam, defibred and refined. The cooking liquor is a mixture of Na_2SO_3 and $NaHSO_3$ at a pH of about 7.3. The net pulp yield is 83 to 84 per cent with a brightness of 57 per cent (65 per cent with green wood). The pulp has high tear and burst, fairly low bulk and opacity.

Blandin Paper Company(1) uses Messing — Durkee digester for impregnation with a liquor of Na_2SO_3 and $NaHSO_3$ for 15 minutes at 280°F and 85 p.s.i. The impregnated chips are compressed by screw presses. The compressed chips are refined in three double disc refiners in parallel and then in a fourth refiner at 5 per cent consistency as second stage refining. The pulp has an average brightness of 56 per cent. The pulp has about twice internal tearing resistance than that of groundwood.

Saici(2) in Italy is producing 135,000 tonnes of pulp per year. The cooking system operates according to Defibrator process in the three following stages (i) steaming, (ii) impregnation of chips under pressure with cooking liquor after compression in screw press and (iii) the actual vapour phase cooking at temperature of upto 190°C and a pressure of 12 atmospheres. The cooking chemical is sodium sulphite, 4-10 pH and 100-200 gpl Na_2SO_3.

Similarly, Kushiro Mills, Japan(2), Jajo Paper Co., Japan, etc. are also manufacturing high yield pulps (3).

At the West Coast Paper Mills, we have a Eucalyptus hydrid plantation of 5,500 acres. It will be ready for exploitation in 3 to 4 years' time. Hence, Eucalyptus hybrid wood of the proper age was collected to test its suitability for high yield pulping. The objective was to get high unbleached pulp yields with good brightness. The pulping was essentially on the following lines (i) steaming of chips, (ii) impregnation with the cooking liquor, (iii) vapour phase cooking, and (iv) refining. Unbleached pulp yields of 81.5-84.1 per cent were obtained. The pulps had moderate strength properties with good bulk. Hence, when these pulps were blended in different proportions with bleached bamboo sulphate pulp, paper of satisfactory strength with high bulk was obtained.

EXPERIMENTAL:

Eucalyptus hybrid billets were chipped in the mill chippers. The chips were sorted and chips of 15 to 30 mm length were used for pulping and impregnability tests. The chips (moisture content 20 per cent) were stored in air tight polythene bags. At this moisture content density of the wood was 0.6 g/cc.

To assess the response of Eucalyptus hybrid for high yield pulping, preliminary impregnability tests were carried out. The vessel used for impregnation, cooking, and steaming, etc., was a Laboratory autoclave of cylindrical shape of 16 litres capacity rotating at 2 r.p.m. and electrically heated. For direct steaming, low pressure saturated steam was used.

Chemical Analysis of Wood

The wood was analysed for proximate chemical analysis. TAPPI standard methods were used for analysis. For results, see Table I.

TABLE I Chemical Analysis

		Eucalyptus Hybrid %	Bambusa arundinacea %
Moisture		7.73	7.53
Cold water solubility	...	0.62	2.89
Hot water solubility	...	2.10	4.51
1 per cent NaOH solubility	...	13.4	22.6
Alcohol benzene solubility	...	1.48	2.99
Cross and Bevan cellulose	...	52.2	62.1
Holocellulose	...	65.8	68.3
Alpha cellulose	...	45.1	49.0
Lignin	...	30.9	25.8
Pentosans	...	14.1	18.3
Ash	...	0.44	4.87
Silica	...	0.03	2.70

Notes:

1. All values, except moisture, are expressed on O. D. sample basis.
2. For all analyses — 40 + 60 mesh sample was used.
3. Pentosans were determined volumetrically.
4. Holocellulose was determined by chlorite method. For others, TAPPI standard methods were used.
5. Cellulose and lignin have been corrected for ash.

Impregnability Tests

623 grams of chips (19.8 per cent moisture content) equivalent to 500 grams of O.D. chips were steamed in the Laboratory autoclave using low pressure saturated steam at 143°C for half an hour with continuous removal of condensate. The steamed chips were impregnated with hot sodium sulphite solution of varying concentrations for 10 minutes at 100°C. The liquor was drained off and was analysed for residual Na_2SO_3 content. The impregnated chips, in each case, were transferred as quickly as possible into weighed buckets with lids. The buckets, with its content were weighed again. The data and results are given in Table II.

Pulping

A series of pulping experiments were carried out using sodium sulphite as the cooking chemical. The liquor was prepared by dissolving commercial grade sodium sulphite in distilled water. A 200 g/l. solution had a pH of 10.0. The pulping schedule was as follows:

Direct Steaming

Steaming of chips with low pressure saturated steam for half an hour was done, condensate being continuously drained during steaming. Maximum temperature during steaming was 143°C.

Impregnation

The steamed chips were impregnated with liquor (sodium sulphite solution) by varying the concentration from 65 to 195.9 grams per litre. The temperature was maintained at 100-105 °C. After half an hour of retention time, the excess liquor was drained off. Volume of the drained liquor was measured and it was analysed. In all cooks except Cook No. 8, sodium sulphite solution was used as the impregnating liquor.

Cooking

The temperature of the autoclave containing the steamed and impregnated chips was raised to 150°C in 20 minutes and was maintained at 150°C for 0.5 to 1.5 hours.

Refining

After cooking, the hot semi-cooked chips were refined in a 12" Sprout-Waldron Laboratory disc refiner. For the 1st pass the plate clearance was 0.050" (0.127 mm); and for the 2nd pass it was 0.010" (0.025 mm) at 2% consistency.

The refined pulp was thickened to about 25% consitency in a centrifuge, broken to pieces and was kept in an air tight polythene bag. The unbleached pulp yield, and brightness of the pulp were determined. Pulping conditions, pulp yield, brightness etc. are given in Table III. Cook No. 1 and Cook No. 3 were eva-

TABLE II: Impregnation Tests.

	Experiments				
	1	2	3	4	5
Chips (19.8% moisture) used, g ...	623	623	623	623	623
Equivalent OD chips, g ...	500	500	500	500	500
Weight of chips after steaming, g ...	1000	1000	1000	1000	1000
Volume of liquor (sodium sulphite solution) charged for impregnation, ml ...	1000	1000	1000	1000	1000
Concentration (as Na_2SO_3) of liquor, g/l ..	100	115	130	145	160
Concentration (as Na_2SO_3) of liquor drained off, g/l ..	63.2	100.8	113.4	128.5	134.8
Weight of chips after steaming and impregnation, g ...	1107	1107	1095	1120	1115
Wood to liquor ratio after steaming ...	1:1	1:1	1:1	1:1	1:1
Wood to liquor ratio for impregnation ...	1:3	1:3	1:3	1:3	1:3
Concentration of liquor (as Na_2SO_3) during impregnation, g/l ...	66.7	76.7	86.7	96.7	106.7
Wood to liquor ratio for vapour phase cooking ...	1:1.21	1:1.21	1:1.19	1:1.24	1:1.24
Concentration of liquor for vapour phase cooking, g/l ..	64.2	68.1	79.1	92.1	97.1
Na_2SO_3 retained by chips for vapour phase cooking, % ...	7.76	8.26	9.42	11.42	12.02

luated for strength properties. Beaten pulps of Cook No. 1 and Cook No. 3 were respectively blended in different proportions with beaten bamboo kraft pulp and the blends were evaluated for strength properties. The strength values for Cook No. 1 and 3 are given in Table IV and V respectively.

Analytical and Testing Methods

Sodium sulphite present in impregnating and the spent liquor was determined iodimetrically. The pH was determined using a Beckmann pH meter with general purpose glass electrode and saturated Calomel electrode. The brightness of the pulps were determined using a Dr. Lange Reflection meter. The beater used for beating the pulps was the TAPPI Valley beater. Standard sheets were made on British standard sheet forming machine. Strength values were determined using TAPPI standard instruments.

DISCUSSION

Pulp Yield

A high unbleached pulp yield (81.5 to 85.2%) has been obtained in all the Cooks (see Table III). In Cooks 2 and 3, all other conditions have been kept constant except concentration of impregnating liquor. For Cook 2 a concentration of 195.9 gpl as Na_2SO_3 was used and for Cook 3 a conecentration of 65 gpl as Na_2SO_3 was used; 19.5% was retained for cooking for Cook 2 and 6.9% for Cook 3. The decrease in yield from Cook 3 to Cook 1 is from 83.9 to 82.7 percent. Still higher yields can be obtained by decreasing the concentration of impregnation liquor at the cost of strength values.

pH

The pH of the condensate obtained after steaming was always acidic indicating that some of the wood acids are liberated during steaming. This may be the reason why the pH of the impregnating liquor did not drop to the acidic side. No buffering agents such as Na_2CO_3 or $NaHCO_3$ were added to the impregnating liquor because of their adverse effect on unbleached pulp brightness. This has been observed by C. B. Christiansen also (5). At constant sodium charge, he had varied sulphite to carbonate ratio. With a sulphite to carbonate ratio of 90%, he had obtained 29% Densicron brightness while with 100% Na_2SO_3 he has obtained 31.2% brightness for direct NSSC pulping.

Brightness

Unbleached pulp brightness has varied from 41 to 51 percent. Highest brightness has been obtained for Cook No. 6, where 9.0% Na_2SO_3 was retained for cooking. Lowest is for Cook No. 4 where 7.3% Na_2SO_3 was retained for cooking. But the cooking temperature of the latter was 162°C while that of the former was 150°C. High temperature of cooking has an adverse effect on brightness . Other conditions maintained constant, the brightness of the unbleached pulp has decreased as the concentration of impregnation liquor was increased (compare Cooks 3,1 and 2). At the same conditions of impregnation, time and temperature of cooking, addition of 0.5% EDTA on chips during impregnation has increased the brightness only by 1 point (Cooks 5 and 6). Anyhow better improvement can be expected in commercial installations where raw water, instead of distilled water will be used. But better improvements in brightness can be effected by adding chelating agent during refining. Addition of 0.5% EDTA during refining has given 2 points brightness increase for pulp of Cook No. 4.

All the pulps had a slight pink tint. To increase the brightness further, without affecting the yield, adversely, bleaching with H_2O_2 or hydrosulphite or both is necessary.

Strength Properties

A study of Table IV and V indicates that the pulp of Cook No. 1, where 11.9% Na_2SO_3 was retained for cooking, is stronger than that of Cook No. 3 where 6.9% Na_2SO_3 was retained. This leads to the inference that if the chemicals retained for cooking is less, i.e. if the unbleached pulp yield is more, a pulp more like a ground-wood pulp results. Hence, the pulp of higher yield tends to be weaker. G. H. Chidester and co-workers have come to

TABLE III. Pulping Conditions, Yield, and Brightness

Cook No.	L.P. Saturated steam: Steaming temp. °C	Steaming time, min.	pH of condensate	Impregnation: Volume of Na_2SO_3 solution added, ml.	Concentration of liq. added, g/l.	Time, min.	Temp. °C	pH of drained liquor	Vapour Phase Cooking: Concentration of drained liquor as Na_2SO_3 g/l.	Na_2SO_3 retained for cooking on OD chips %	Time of (a) cooking mts.	Temp. of cooking	Unbleached Pulp: Unbleached Pulp yield on OD chips basis, %	Brightness, %
1	143	30	4.8	2000	120	30	100	7.5	83.2	11.9	20+90	150	82.7	48
2	143	30	5.3	3000	195.9	30	100	7.9	141.8	19.5	20+90	150	82.2	46
3	143	30	5.3	3000	65	30	100	7.5	47.5	6.9	20+90	150	83.9	50
4	143	30	5.2	3000	65	30	100	7.5	49.8	7.3	30+90	162	—	41/43(b)
5	143	30	5.2	2000	96	30	100	7.5	68.0	9.2	20+90	150	84.1	50
6	143	30	5.6	2000	96	30	100	7.4	66.2	9.0	20+90	150	83.2	51(c)
7	143	30	5.6	2000	96	30	100	7.45	65.5	8.9	20+90	150	85.2	50
8	143	30	5.2	2000	157.5	30	100	6.9	109.0	9.7	20+90	150	81.5	50(d)

Notes

(a) The first figure indicates the time required to raise to maximum temperature; the second figure indicates the time at maximum temperature.

(b) 41% without adding EDTA and 43% after adding 0.5% EDTA during refining.

(c) 0.5% EDTA was added during impregnation stage.

(d) Mixture of Na_2SO_3 and $NaHSO_3$ was used for impregnation (initial pH=7.0).

TABLE IV. Strength Properties of Eucalyptus Hybrid Pulp from Cook No. 1 and Blends with *Bambusa arundinacea* Pulp.

Particulars	100% Pulp A	80% Pulp A +20% Pulp B.	60% Pup A +40% Pup B.	40% Pup A +60% Pulp B.	20% Pup A +80% Pulp B.	100% Pulp B.
Initial freeness, °SR ...	15.0	—	—	—	—	17.0
Final freeness, °SR ...	42.0	—	—	—	—	42.0
Beating period, min. ...	46.0	—	—	—	—	18.0
Brightness of standard sheets % ...	45.0	46.5	48.5	50.0	55.5	68.0
Basis weight, g/m^2 ...	59.8	60.0	60.7	60.9	61.4	61.3
Thickness, microns ...	143.0	134.0	124.0	118.0	106.0	105.0
Bulk, cc/g ...	2.39	2.23	2.04	1.94	1.73	1.71
Breaking length, km. ...	4.06	4.82	4.96	5.62	5.57	6.12
Stretch, % ...	2.3	2.6	2.7	2.8	2.9	3.2
Burst factor ...	19.9	26.5	28.3	33.0	34.2	37.5
Tear factor ...	37.6	51.7	58.5	56.7	58.6	71.8
Folding endurance, Double Folds ...	3	5	6.	6	6	7
Burst factor + ½ tear factor ...	38.7	52.3	57.6	61.4	63.5	73.4

Notes

1. Pulp A is semichemical Eucalyptus Hybrid Pulp from Cook No. 1 (Table III). Brightness 48 Percent.
2. Pulp B is kraft bleached *Bambusa arundinacea* pulp. Brightness 68 Percent.

TABLE V. Strength Properties of Eucalyptus Hybrid Pulp from Cook No. 3 and Blends with *Bambusa arundinacea* Pulp.

Particulars	100% Pulp A1	80% Pulp A1 +20% Pulp B1	60% Pulp A1 +40% Pulp B1	40% Pulp A1 +60% Pulp B1	20% Pulp A1 +80% Pulp B1	100% Pulp B1
Initial freeness, °SR ...	9	—	—	—	—	—
Final freeness, °SR ...	45	—	—	—	—	—
Beating period, min ...	105	—	—	—	—	—
Brightness of standard sheets, % ...	49	—	—	52	59	69.5
Basis weight, g/m² ...	61.7	60.7	60.0	61.2	61.0	60.1
Thickness, microns ..	174.7	147.7	127.6	120.4	105.8	98.8
Bulk, cc/g	2.91	2.42	2.08	2.01	1.74	1.60
Breaking length, km ...	1.68	3.54	4.28	5.28	5.77	6.24
Stretch, % ...	1.7	2.3	2.7	3.7	3.7	3.8
Burst factor ...	9.0	17.1	23.4	31.3	37.1	40.8
Tear factor ...	23.3	45.8	57.6	64.7	73.8	78.4
Folding endurance, Double folds ...	1	3	7	12	19	41
Burst factor + ½ tear factor ...	20.7	40.0	52.2	63.7	74.0	80.9

Notes

1. Pulp A1 is semichemical *Eucalyptus Hybrid* Pulp from Cook No. 3 (Table III).
2. Pulp B1 is kraft bleached *Bambusa arundinacea* Pulp. Brightness 69.5 Percent.

the same conclusion (4). The pulps had very good bulk. The higher yield pulp had a better bulk than the lower yield pulp (2.43 for Cook No. 1 and 2.91 for Cook No. 3).

To obtain pulps of satisfactory strength properties, it is better to use a liquor of higher concentration during impregnation so that sufficient Na_2SO_3 is retained for cooking.

As the amount of bleached bamboo kraft pulp in the blend has increased, the strength values are improved greatly. Also the blend of pulp of Cook No. 1 and bleached bamboo kraft pulp has better strength than the blend pulp of Cook No. 3 and bleached bamboo kraft pulp. This indicates that lower yield semichemical pulp should be useful for blending with other pulps to obtain satisfactory strength properties. The exact quantity of the pulp to be blended depends upon the strength values and brightness required of the paper to be made.

All the pulps have very good bulk (2.43 to 2.91).

Chemical Composition

From Table I it can be observed that the wood has very low solubilities compared to bamboo. The ash and silica are also lower than those of bamboo. The lignin content is higher and cellulose content is lower.

CONCLUSIONS

High yield (81 to 85%) pulp can be obtained from Eucalyptus hybrid wood with brightness up to 50% using sodium sulphite as the sole impregnating liquor.

Because of pre-impregnation method, the impregnating liquor can be used over and over again after replenishing the used up chemicals. When it will be found to contain sufficient used chemicals, it can be pumped to Soda Recovery Department. This liquor can be efficiently absorbed in the Kraft Recovery System, if the ratio of kraft to semichemical production is 3 : 1 or greater (6). This is possible only when semichemical pulping is carried out jointly in a kraft mill. The cooking sulphite liquor can be obtained by sulphitation of the green liquor (6).

For papers, where very high brightness is not required (e.g. Newsprint, magazine papers, paper back books, etc.) papers of satisfactory strength can be obtained by blending 80% high yield Eucalyptus hybrid pulp with 20% bleached bamboo pulp. The brightness of the unbleached pulp can be improved further by mild bleaching with peroxide or hydrosulphite or both without sacrificing yield.

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