M. B. Jauhari, N. S. Jaspal and Roshan Lal Bhargava

High Yield Semibleached Pulps from Dendrocalamus Strictus using Sulphate Semichemical Process and Multistage Bleaching

High yield (69 to 75%) unbleached pulps were prepared from DENDROCALAMUS STRICTUS using first a prewashing stage followed by sulphate cooking and then refining in Sprout-Waldron refiner. The pulp cooked to 75% yield was bleached using non-conventional bleaching agents in combination with hypochlorite stages to brightness levels of 50 to 55% and the yields obtained were ranging from 66 to 69 percent. Bleaching improved the strength properties of the pulps. By using the sequence chlorine-sodium sulphite- H-E-H, a pulp having good strength in 63 percent yield was obtained.

Fibre fractionation studies carried out on a pulp cooked to 67%yield indicated 6.7% fraction (on basis of bamboo chips) passing through a 200 mesh sieve, which emphasises the importance of using closed water system to the maximum possible extent, especially if these high yield processes are to be adopted on a mill scale and implementation of the yield figures is required.

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INTRODUCTION

The semichemical process which was first developed at the Forest Products Laboratory, Madison, is now being gradually adopted in other countries, in order to conserve the fibrous resources, to bring down the cost of production and to make the paper pro-duct more competitive with with other materials. By this process and with its various modifications, it is now possible to produce a whole spectrum of pulps. Unfortunately the Indian Paper Industry is lagging far behind in adopting and giving impetus to such processes, inspite of the shortage of cellulosic resources, coupled with rising cost of chemicals, especially caustic soda, sodium sulphate and various other materials.

Successful efforts are being made at the Forest Research Institute, Dehra Dun to obtain pulps in high yields from indigenous fibrous materials by following semichemical processes. In a recent publication (1) Mukherjea and Guha described the hot caustic soda process, and obtained pulps in high yields from bamboo, Eucalyptus, jute sticks, etc. However, one of the disadvantages of high yield kraft pulps is that they suffer from strength losses and presents problem in bleaching. Work carried out (2) in this Laboratory revealed that bleaching of high yield kraft pulps by conventional bleaching agents to brightness levels of 77% to 78% was accompanied with considerable shrinkage and in many cases the consumption of chemicals was excessive and therefore uneconomical In the present study we have used nonconventional bleaching agents in combination with hypo-chlorite stage to reduce the shrinkage, but this has limited the brightness development.

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The production of high yield pulps from bamboo chips is attenuated with the problem of getting the liquor uniformly distributed inside the chips, so that the refining may be easier and the amount of uncooked cores or shives may be kept at minimum. Uniform impregnation of the chips with liquors is quite important for good strength development. Bhargava (3) suggested an improved pulping method especially for bamboo in which the chips were first treated with water or weak wash liquor, at a 20 to 25 lbs./sq. inch. After the treatment was over the liquor was drained out and then sulphate cooking was accomplished. The first treatment helped in raising the moisture content of the chips to about 30 per cent. A somewhat similar precedure has been followed in the study reported here.

Objective

The objective of this investigation was to prepare high yield unbleached pulps of light colour and then to bleach these pulps to moderate brightness with good strength and minimum shrinkage.

Experimental

The chips were prepared in the mill chipper from 10 months seasoned bamboo. After hand sorting of the chips to ensure size uniformity, a number of charges

Table I.	Prox	imate	Chemical
Analysis	of	Dend	Irocalamus
Strictus			

-	%
· · · · · · · · · · ·	
Ash	3.60
Silica	2.21
Cold water solubility	2.40
Hot water solubility	4.38
1% NaOH solubility	18.13
Alcohol-Benzene solubility	2.33
(1:2)	
Water solubility after Al-	
cohol Benzene extraction	3.20
Total Pentosans	17.40
Alpha cellulose ¹	49.16
Lignin ²	25.58
Calculated Holocellulose ²	65.30

Notes : All results are expressed on O.D. material basis.

> ¹ Alpha cellulose was estimated after preparing chlorite holocellulose from defatted material, and the result was corrected for ash.

² corrected for ash.

were weighed out equivalent to 1.0 kg. of OD chips.

A portion of the chips was used for proximate analysis, after powdering and sieving. The fraction retained on 80 mesh was used for analysis. The results of $t \ge a$ analysis are recorded in Table I.

Cooking experiments and the presoaking of the chips was carried out in an electrically heated rotary outoclave. The conditions used in presoaking and cooking stages together with the results obtained are recorded in Table II.

Table II. Presoaking and Cooking Conditions.

Presoaking	Cook No. 1	Cook No. 2
Chips : Water Lissapol PSN Conc.*	1:3	1:3
on chips.%	0.1	0.1
Temperature. °C	90	110
Time to temperature,	• •	
mts.	30	30
Time at temperature,		
mts.	45	60
Amount of water		
drained based on		
original water,%	73	72
Digester evacuated		
to 17" Hg for 5 mts.	Yes	—
Cooking chemicals		
(NaOH+Na₂S),%	10	11
Cooking Chemicals		
(NaOH+Na ₂ S+-		
Na ₂ CO ₃),%	11.5	13.4
Chips : Liquor	1:3	1:3
Initial Temperature.		
°C	70	100
Maximum Tempera-		
ture, °C	110	150
Time to get maxi-		200
mum temperature.		
mts.	10	45
Time at maximum		
temperature, mts.	60	nil
Sulphidity. %	$\tilde{20}$	18
Total yield, %	75	$\overline{67}$

* Lissapol PSN conc. is a product of I.C.I. (India) Pvt. Ldt., Bombay.

After the cooking stage, the chips were refined hot at a clearance of 35 thou in a Laboratory Sprout-Waldron refiner.

The spent liquor was removed after the first stage of refining in a hydra extractor and then the pulp was again refined at 5 thou after which it was washed, thickened and then used for calculating the yield.

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Bleaching

Bleaching of the pulps was carried out in plastic bottles using 50 g O.D. pulp. The pulp from Cook No. 1 was first bleached using conventional chlorination — Alkali extraction — Chlorination + Lime neutralization — Hypochlorite stages. The bleaching conditions and the results obtained are given in Table III.

Table III...Bleaching Conditionsand Results

Chlorination	L
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Consistency, %2.5Temperature, °C23Cls added, %18Cls consumed, %18Time, mts.60Alkali Extraction60Consistency, %5Temperature, °C $55 + 1$ NaOH added, %4Time, mts.60Final pH8.1Chlorination + LimeNeutralization6Consistency, %4Temperature, °C $30 + 1$ Cls added, %6Ca(OH)s added after1.5 mts., %1.5 mts., %7Total time, mts.30Final pH9.7Hypochlorite5Consistency, %5Temperature, °C $35 + 1$ Cls added, %8.8Cls consumed, %8.2NaOH added, %2.0Final pH9.2Total Cls added, %2.8NaOH added, %2.0Final pH9.2Total Cls consumed, %6.2Total Cls consumed, %6.2Total Cls consumed, %6.2Cls consumed as celemental, %2.2Cls consumed as ocl., %8.2Ca(OH)s consumed, %60Yield of bleached pulp77Shrinkage, %23Yield of bleached pulp77Shrinkage, %58.1		
Temperature,C $33 + 1$ NaOH added,%4Time, mts.60Final pH8.1Chlorination + Lime8.1NaOH added, + Lime8.1Chlorination + Lime8.1NeutralizationConsistency, %Cla added, *6Ca(OH)a added after1.51.5 mts., %7Total time, mts.30Final pH9.7Hypochlorite9.7Cla added, %8.8Cla consumed, %8.2NaOH added, %8.2NaOH added, %2.0Final pH9.2Total Cla added, %2.2Final pH9.2Total Cla consumed, %6.2Total Cla consumed, %32.2Cla consumed as elermental, %24.0Cla consumed as ocl., %8.2Ca(OH)a consumed, %7Brightness, %60Yield of bleached pulp77Shrinkage,%23Yield of bleached pulp58.1	Consistency, % Temperature, °C Cl ² added, % Cl ² consumed, % Time, mts. Alkali Extraction Consistency, %	2.5 23 18 18 60 5 55 + 1
Neutralization Consistency, % 4 Temperature, °C $30 + 1$ Cl ¹² added, % 6 Ca(OH) ² added after 1.5 mts., % 7 Total time, mts. 30 Final pH 9.7 Hypochlorite Consistency, % 5 Temperature, °C $35 + 1$ Cl ¹² added, % 8.8 Cl ² consumed, % 8.2 NaOH added, % 2.0 Final pH 9.2 Total Cl ² added, % 32.8 NaOH added, % 2.0 Final pH 9.2 Total Cl ² added, % 32.8 NaOH added, % 6.2 Total Cl ² added, % 32.8 NaOH consumed, % 6.2 Total Cl ² consumed, % 6.2 Cl ² consumed as ele- mental, % 24.0 Cl ² consumed as ele- mental, % 7 Brightness, % 60 Yield of bleached pulp on unbleached pulp, % % 77 Shrinkage,% 23 Yield of bleached pulp on chips.% 58.1	Temperature, °C NaOH added, % Time, mts. Final pH Chlorination + Lime	55 + 1 4 60 8.1
Ca $(OH)_2$ added after 1.5 mts., % 7 Total time, mts. 30 Final pH 9.7 Hypochlorite Consistency, % 5 Temperature, °C 35 + 1 Cl ² added, % 8.8 Cl ² consumed, % 8.2 NaOH added, % 2.0 Final pH 9.2 Total Cl ² added, % 32.8 NaOH added, % 6.2 Total Cl ² added, % 32.8 NaOH consumed, % 6.2 Total Cl ² added, % 32.8 NaOH consumed, % 6.2 Cl ² consumed as ele- mental, % 24.0 Cl ² consumed as ele- mental, % 7 Brightness, % 60 Yield of bleached pulp on unbleached pulp, % 77 Shrinkage,% 23 Yield of bleached pulp on chips.% 58.1	Chadded %	$\frac{4}{30} + 1$
Consistency, %5Temperature, °C $35 + 1$ Cl ² added, % 8.8 Cl ² consumed, % 8.2 NaOH added, % 2.0 Final pH 9.2 Total Cl ² added, % 32.8 NaOH consumed, % 6.2 Total Cl ² consumed, % 32.2 Cl ² consumed as elemental, % 24.0 Cl ² consumed as ocl., % 8.2 Ca(OH) ² consumed, %7Brightness, % 60 Yield of bleached pulp on unbleached pulp, % 77 Shrinkage, % 23 Yield of bleached pulp on chips, % 58.1	Ca(OH) ² added after 1.5 mts., % Total time, mts. Final pH Hypochlorite	7 30 9.7
Cl_2 added, %8.8 Cl_2 consumed, %8.2NaOH added, %2.0Final pH9.2Total Cl_2 added, %32.8NaOH consumed, %6.2Total Cl_2 consumed, %32.2Cl_2 consumed as ele- mental, %24.0Cl_2 consumed as ocl., %8.2Ca(OH)_2 consumed, %7Brightness, %60Yield of bleached pulp on unbleached pulp, %77Shrinkage, %23Yield of bleached pulp on chips, %58.1	Consistency, % Temperature, °C	35 + 1
NaOH consumed, % 52.8 NaOH consumed, % 6.2 Total Cl₂ consumed, % 32.2 Cl₂ consumed as ele- mental, % 24.0 Cl₂ consumed as ocl., % 8.2 Ca(OH)₂ consumed, % 7 Brightness, % 60 Yield of bleached pulp on unbleached pulp, % 77 Shrinkage,% 23 Yield of bleached pulp on chips.% 58.1	Cl ² added, % Cl ² consumed, % NaOH added, % Final pH	8.8 8.2 2.0 9.2
mental, %24.0Cl* consumed as ocl., %8.2Ca(OH)* consumed, %7Brightness, %60Yield of bleached pulp on unbleached pulp, %77Shrinkage, %23Yield of bleached pulp on chips.%58.1	NaOH consumed, % Total Cl ² consumed, % Cl ² consumed as ele-	6.2 32.2
Brightness, %60Yield of bleached pulp on unbleached pulp, %77Shrinkage,%23Yield of bleached pulp on chips.%58.1	mental, % Cl ² consumed as ocl., % Ca(OH) ² consumed, %	24.0 8.2 7
Shrinkage,% 23 Yield of bleached pulp on chips.% 58.1	Brightness, % Yield of bleached pulp on unbleached pulp,	6U 77
	Shrinkage,% Yield of bleached pulp on chips.%	23 58.1

The results indicate that the consumption of bleach chemicals was excessive, and the shrinkage was also high.

Other bleaching sequences which were studied are:

1. SO₂ + Hydrosulphite - Hypo - SO₂ + Hydrosulphite -Hypo.

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- 2. Hypo SO_2 + Hydrosulphite - Hypo - H_2O_2 .
- 3. Cl Na₂SO₈ H E H.
- 4. SO_2 + Hydrosulphite Hyp - H₂O₁ - SO₂ + Hydrosulphite.
- 5. SC² + Hydrosulphite Hypo - H₂O² - Hypo

The conditions of bleaching in hydrosulphite and hydrogen peroxide stages were maintained constant. — Table IV. The conditions used in hypochlorite stages are given in Table V, and the other relevant data such as, the brightness, shrinkage, during bleaching and the consumption of chemicals are recorded in Table VI. Table IV. Bleaching Conditions with Hydrosulphite and Hydrogenperoxide.

Hydrosulphite

· · · · · · · · · · · · · · · · · · ·	
Consistency, %	3.5
Temperature, °C	60
SO_2 added, %	0.5
Initial pH	5.5
Sodium tripoly-phos-	
phate, %	0.5
Sodium hydro-sulphite,	1.5
Time, mts.	60
H_2O_2	
Temperature, °C	60
H_2O_2 added, %	1.5
Na:SiO: added, %	4.0
Time, mts.	90
MgSO4 added, %	0.05

Table V...Bleaching Conditions in Hypochlorite Stage Pulp consistency: 5%; Time: 60 minutes

ხეჟა	,	Hypo Stage	1		Hypo Stage	II
Bleachin Sequence	Temp. °C	Av. Cl² added %	NaOH added %	Temp. °C	Av. Cl₂ addeď %	NaOH added
1	30	15	1.5	40	5	0.8
<u>2</u>	30	10		40	10	1.5
3*	30	10	1.0	40	5	1.2
4	30	15	1.5	·		
- 5	30	10	1.0	40	10	1.5

*Chlorination	
Consistency, % Cl ² added, %	3.0 12.0
Na2SO3 treatment	
Consistency, % Temperature, °C Na:SO: added, % Time, mts. Final pH	$5.0 \\ 60.0 \\ 1.5 \\ 30 \\ 4.5$
Alkali Extraction	

Consistency, %	5.0
Temperature, °C	60.0
`NaOH added. %	1.5

The semi-bleached pulps were evaluated for strength properties using Lampen Mill under constant conditions of consistency, temperature and number of revolutions. The results are recorded in Table VII.

It could be seen from the results given in Table VI that the maximum brightness which could be obtained was 55 and the shrinkage was varying from 8 to 16 per cent. Further it could be observed from sequence 1, and 5 that no advantage was obtain-

ed by replacing partly the hydrosulphite stage by the hydrogen peroxide. The strength characteristics of the two pulps are also very near. However, when compared to unbleached pulps, the strength properties of all the semi-bleached pulps are better and quite good strength was obtained for the pulp bleached by sequence 3.

Fibre Fractionation and Conventional Bleaching of Pulp from Cook No. 2

The fibre vascular bundles in bamboo are dispersed in thin walled parenchyma cells and these cells persist even after converting the bamboo chips to pulp. As these cells make up a large proportion (29 to 32%) (4), it is imperative that they will contribute significantly to yield of pulps. In high yield pulping processes, the amount of these cells remaining with the pulp may be quite high, as in full chemical pulping processes, some of these cells may be dissolved out because of high alkali concentration and high cooking temperatures.

Table VI. Bleaching Conditions

Bleaching sequence	1	2	3	4	5	
Brightness, % Yield of bleached pulp on unbleached pulp.	53	55	54	42	52	
%	91	88.3	83.3	91.8	91.0	
Shrinkage, %	9	11.7	16.5	8.2	9.0	
Total Cl ² consumed, %	20	20	27	15	20	
NaOH consumed, %	2.3	1.5	3.7	1.5	0.5	
SO ² consumed, %	1.0	0.5	-	1.0	2.5	
Sodium hydro-sulphite						
consumed, %	3.0	1.5	-	3.0	1.5	
H_2O_2 consumed, %		1.5	-	1.5	1.5	

For this reason we have estimated for this particular pulp, the fraction which would easily pass through a 200 mesh sieve. The separation was achieved by putting a known weight of the pulp on a standard 200 mesh sieve and partly submerging it in a large volume of water. No attempt was made to separate out completely the parenchyma cells from the fibres, only the fraction which was passing easily, was estimated after drying. The results are recorded below :

Table VII. Lampen Mill evaluation of Unbleached and Semibleached pulps.

Consistency 3%; Temperature 25°C; No. of revolutions 11000

Bleaching sequence	Unbl. Pulp	1	2	3	4	5
Brightness, % Yield of bleached pulp	18	53	55	54	42	52
on chips, %		68.7	66.7	63.0	69.3	68.7
Final freeness, °SR	60	58	60	64	55	60
Basis weight, g/m ³	55.2	57.0	58.0	56.7	58.5	58.0
Thickness, microns	130	120	115	110	120	110
Bulk, cc/g.	2.35	2.1	2.0	1.94	2.05	1.90
Breaking length, km	3.64	4.74	4.48	6.40	4.20	4.80
Stretch %	2.7	3.5	2.7	3.8	2.5	3.0
Burst factor	20.8	27.4	31.2	49.5	25.3	31.5
Tear factor	71.7	84.2	79.3	79	82	79.3
Double Folds	7	19	15	40	21	17

Table	VIII.	Bleaching	Conditions	of	Whole	Pulp	and	+200	mesh
		-	Fraction	of	Pulp	-			

Chlorination		Cook No. 2 whole pulp +200		
Consistency, %	3	Brightness	57	60
Temperature, °C	25	Shrinkage %	16.6	14.5
Cl ² added. %	16			
Cl ₂ consumed. %	16			
Time, mts.	60			
Extraction				
Consistency, %	5			
Temperature, °C	50+1			
NaOH added, %	2.5			
Time, mts.	45			
Hypo I				
Consistency, %	5			
Temperature, °C	30			
Av. Cl ₂ added. %	10			
NaOH added. %	1			
Hypo II				
Consistency. %	5			
Temperature. °C	40 + 1			
Time. mts.	90			
Av. Cl ₂ added. %	4			
NaOH added, %	0.6			

Total unbleached
yield, %67--200 on pulp basis, %10--200 basis bamboo
chips, %6.7Yield of pulp on chips
after separating --
200 mesh fraction, %60.3

This emphasizes the importance of using perfectly closed water system, in order to minimize the losses of fines.

Bleaching studies were carried out on the whole pulp and also from which the zero fibre fraction (-200) was removed. The bleaching data is recorded in Table VIII.

It could be seen from the results that except for slightly better brightness and less shrinkage with +200 fraction, there is no appreciable difference in the bleaching characteristics.

Bleaching of the whole pulp was also carried out using sequences Hypo-Al-Hypo; (H+C)-Al-H and single stage hypochlorite stage. The bleaching conditions are recorded in Table IX.

The semi-bleached pulps were evaluated for strength properties and the strength data is recorded in Table X.

The results indicate that the strenth properties of semi-bleached pulps were not improved by single stage hypochlorite bleaching except for slight increments in burst factor and breaking length. Removal of non-cellulose impurities (lignin) to a certain degree is essential for good strength development.

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Bleaching sequence	H-E-E	(H+C)-E-]	нн
Hypochlorike Constant conditions Consistency, % 5 Temperature, °C 30 Av. Cls, % NaOH added, % Time, mts.	8 0.6 15	10 0.6 15	15 1.0 60
Elemental Cl₂ added, % Total time, mts.		5 45	
Extraction Consistency, % 5 Temperature, °C 50 Time, mts. 90 NaOH added, %	1.0	1.5	
Hypochlorite Consistency, $\%$ 5 Temperature, °C 40 Time, mts.120 NaOH added, $\%$ 1 Av. Cl. added, $\%$	10	8	
Acid wash Brightness, % Total Cl₂ consumed, % NaOH consumed, % Shrinkage, % Yield on chips, %	42 18 2.6 7.50 62	$50 \\ 23 \\ 3.1 \\ 10.4 \\ 60$	30 15 1.0 1.6 65 9

 Table IX. Bleaching Conditions of Whole Pulp under Different Sequences

Table X. Strength Properties of Semi-bleached Pulp

Consistency 3.0%; Temperature 30°C; No. of revolutions 8250.

	IInhl	Bleaching Sequence			
· .	Pulp	H-E-H (H+C)-!		Е-Н Н	
Final freeness °SR Basis Wt. g/m ² Thickness microns Bulk, cc/g Breaking length, km Stretch, % Burst factor Tear factor Double folds	46 59 130 2.2 3.12 3.0 17 84.7 8	48 57 130 2.28 3.56 3.3 24.5 124.5 19	46 58 125 2.15 4.00 3.7 27.6 100 50	47 58.5 130 2.22 3.45 3.0 22.4 91 12	

Conclusions

- 1. Pulps in high yield could be prepared from Dendrocalamus strictus by following the above process.
- 2. The pulps were lighter in colour compared to conventional kraft cooks.
- 3. Pulps contain a high percentage of fines, which requires that means are to be adopted for retaining them, as otherwise the possibility of these being lost in back water system is high.
- 4. Pulps could be bleached to moderate brightness by conventional and non-conventional bleaching agents. However, for good strength development, dissolution of a certain amount of lignin is essential in bleaching processes.

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