M. B. Jauhari, G. L. Bisani, N. R. Jangalgi and Roshan L. Bhargava

Within last 15 years the paper production in India has increased five fold. For the Fourth Five Year Plan, the target for pulp and paper production has been fixed at 1.70 million tonnes and by 1980-81 this is expected to be 4.20 million tonnes. The National Development Council has estimated that by 1980-81, when the per capita consumption of paper in India will be 7.0 Kg only, the fibrous raw material requirement will be about 10 million tonnes.

According to one estimate, the existing resources of bamboo in the country can hardly supply 0.80 million tonnes of paper. It is thus imperative that alternative fibrous raw materials must be made available to the paper industry. The other worth mentioning cellulosic raw materials are bagasse, straw, jute sticks and hardwoods. Besides, the suitability of these short fibre materials for paper manufacture which is yet to be established on a sound commercial basis, the problem is aggravated by the fact that paper is just only one use of cellulose. Manufacture of nonpaper cellulose would require substantial quantities of fibrous materials. To narrow the gap between the requirements of paper and non-paper cellulose and the availability of fibrous materials, the reasonable way is to adopt high yield pulping methods.

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High Yield Pulps from Bambusa arundinacea using kraft Semi-chemical Process and Multistage Bleaching.

Pulping experiments were carried out using kraft semi-chemical and kraft sulphite semi-chemical process to get high yield pulps from bamboo chips. Unbleached pulps yield was 61 to 69 percent and bleached pulps yield was 52 to 54 percent. With a four-stage bleaching 76 to 78 percent brightness was obtained.

Unbleached pulp of every cook having different lignin content was evaluated for strength properties. At lignin content of 8 to 9 percent the strength properties were better than at 13 to 15 percent. Bleaching was done using sequence $C|E|C + Ca(OH_2)|H|$ Acid and C|E|H|H|Acid. Bleached pulps were characterised by fairly good viscosity and brightness.

The semichemical process developed by Forest Products Laboratory at Madison, perimits greater retention of Carbohydrates and lignin in pulps, thereby making substantial improvements in both unbleached and bleached pulp yields. It is rather unfortunate that not a single paper mill in India, inspite of the shortage of fibrous raw materials, is following this process.

Considering the Indian conditions especially the dearth of sulphur and with little experience in sulphite pulping and chemical recovery methods the alkaline semichemical process are best suited. At the West Coast Paper Mills we have plans for kraft semichemical process and it is expected that this will not require considerable modifications and additions in existing plant equipment.

Bamboo which is the main fibrous raw material of Indian Paper Industry, is potentially rich in cellulose and lignin. The total carbohydrate content (Holocellulose) is about 65 per cent, which indicates that 65 per cent pulp yield could be obtained. In most of the Paper Mills in India the bleached pulp yield is about 40-45 per cent indicating that about 20-25 per cent useful constituents are lost during the pulping and bleaching processes. The unbleached pulp yield can be obtained over a wide range as the holo-cellulose and lignin together constitute about 90 per cent fraction of bamboo. Thus it is high time that semichemical processes should be given big fillip in Indian Paper Mills to meet the growing shortage of fibrous raw materials.

Literature survey revealed that very little work has been done on high yield from bamboo. Guha and Pant (1) used the neutral sulphite semichemical process to obtain high yield pulp from bamboo. Mukherjea and Guha (2) used hot Caustic Soda process to obtain high yield. Micolas (3) used the Cold Soda pulping for Philippine bamboo to obtain high yield.

EXPERIMENTAL

Chip samples—Mill chips of flowered Dowga (Bambusa arundinacea) were used in present study. The composite sample of chips used in cooks 1 to 4 was prepared after hand sorting to remove oversize chips. The chip length measurements are given below:

30 mm	 62%	
30 — 35 mm	 38%	

For cooks 5 to 7 and another pulp (Cook C) slightly bigger size chips were used. The chip

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measurements for the length composite sample are given below:

30 mm	•••	45%
30 - 40 mm		37%
40 — 45 mm		18%

From the composite sample of chips, a fraction was powdrered in a distintegrating mill and then analysed for some important con-stituents. The results are given in Table I.

Table 1. Proximate chemical analysis of Bambusa arundinacea

		%
Ash		3.12
Silica		2.50
Cold water solubility		3.12
Hot water solubility		4.21
1% NaOH solubility		21.85
Alcohol Benzene		
solubility $(1:2)$		3.42
Water solubility after		
alcohol Benzene		
extraction		3.73
Total Pentosans		18.12
Chlorite Holocellulose	*+	66.5
Lignin		24.15
Alpha cellulose°		44.82

- Note: Dust of -60+80 fraction was used for all tests. Moisture, % 8.21.
- determined according to the method of Sen Gupta, AB Majumdar, SK., and MacMil-lan, WG, Indian J. Appl. Chem. Vol. 21, No. 3, 1958.
- + The values are corrected for ash.
- Corrected for resistant pentosans.

Four chlorite treatments (1 hour each) were given with wash-ing after each stage of treatment.

Analysis for ash, lignin, pentosans content (volumetric deter-mination) (T223m-58) and the solubility in alcohol benzene, cold and hot water and 1% NaOH solubility were made according to standard TAPPI procedures.

PULPING

Pulping tests were carried out in an electrically heated rotary autoclave of 16 litre capacity tumbling at 2 r.p.m. For each experiment 1.0Kg of 0.D. Bamboo chips were used.

It may be stated that in these pulping tests fairly uniform chips were used. This was done to improve the uniformity of cooking and defibering in Sprout Waldron Refiner.

For making up the chips to liquor ratio, water was used as The temperature schediluent.

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dules for every cook were follow-ed as given in Table II. In order to expel air, the digester was re-lieved momentarily at 110°C to O p.s.i. The procedure of long rise of temperature and slow

was followed to improve the uniformity of dissolving and loosen-ing of binding material from fibres. The cooking data for kraft semichemicals is given in Table III.

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Table II. Temperature Schedules for Cook No. 1 to 7.

Cook No.	1	and	2:	22-110°C +	$110-140^{\circ}C + 140^{\circ}C$
				75 mts.	105 mts. 225 mts.
Cook No.	2			10% chemi	g liquor was added in two stages cals was first added and the tem- aised to 140°C as indicated be-
				25-100°C	+ <u>100-140°C</u>
				60 mts.	105 mts.
				Immediatete atmospherei cals was ac	ely the digester was relieved to ic pressure and 4% more chemi- dded.
				105-140°C	14 0°C
				15 mts.	+
Cook No.	4			25-115°C	+ <u>115°C</u> + <u>115-150°C</u> <u>150°C</u>
				90 mts.	+ <u>120 mts.</u> <u>135 mts.</u> <u>195 mts.</u>
Cook No.	5			25-120°C	120°C 120-150°C 150°C
Cook No.	6			90 mts.	+ $ + +$
1st Stage				25-120°C	120°C
				90 mts.	+ $$ 120 mts.
2nd Stage	9			100-150°C	150°C
· Cook No.	7			20 mts.	$+$ $\frac{1}{80 \text{ mts}}$
1st Stage				25-135"C	135°C
				90 mts.	+
2nd Stage	÷			110-150°C	150°C
				20 mts.	+ $$

Table III Pulping conditions of kraft semichemical pulps

Cook No.	1	2	3	4	5
Chips : liquor ratio Active Alkali	1:3	1:3	1:2.5	1:3	1:2.5
(NaOH+Na2S), %	12.0	14.0	10 + 4	14.5	15.0
Max. Temp. °C Time to rise to	140	140	140	150	150
Max. Temp. mts. Time at Max.	105	105	105	135	150
Temp., mts. Total pulp yield	120	105	90	60	60
% on chips	65.4	62.5	62.5	61.4	61.7
Kappa No.	101	80	78	72	72.4
Lignin in pulp * Yield of lignin	13.1	10.4	10.1	9.4	9.4
free pulp on					
Chips, $\hat{\%}$	56 .8	56.0	56.2	55.2	55.9

Notes

Moisture in chips was 11.0%,liquor sulphidity 24.0% and Causticity 85.0%.

* Calculated from Kappa Number Lignin = Kappa No. X 0.13.

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In cooks No. 6 and 7 (kraft/ sulphite) the first stage liquor was blown out at maximum pressure and temperature, after which, the hot sodium sulphite solution was introduced and further cooking accomplished as given in Table IV.

Table IV. Kraft/Sulphite Semichemical Pulping Conditions

Cook No.	6	7
1St. Stage		
Chips : Liquor	1:2.5	1:2.5
Active Alkali (NaOH+Na2S)%	8.0	10.0
Max. Temperature, °C	120	135
Time to raise to Max. Temperature, mts.	90	90
Time at Max. Temp, mts.	30	60
Active Alkali in spent liquor as Na20,g/l	5.9	7.2
2nd. Stage		
Na ₂ SO ₃ , added on bamboo chips,	6	6
Chips : Liquor	1:2.0	1:2.0
Max. temperature, °C	150	150
Time to raise to Max.		
temperature, mts.	20	20
Time at Max. temp, mts.	60	1 2 0
Final pH of liquor	10.2	10.5
Total yield on Chips, %	69.0	64.1
Kappa number	120	78
Lignin in pulp, %	15.6	10.1
Yield of lignin free		
pulp on chips, %	58.3	57.6

TABLE V. Holcocellulose, Alphacellulose and Pentosans of Pulpfor Cook No. 2, No. C and No. 6

	Kraft Semi- chemical Cook No. 2	Cook No C	Kraft/ Sulphite semi- chemical Cook No. 6
Total unbleached pulp			
yield on O.D. chips, % Chlorite Holocellulose	62.5	51.5	69.0
in pulp, % Holocellulose on	88.90	93.30	84.10
chips, % Pentosans in	55.5	48.05	58.0
Holocellulose, %	21.7	19.8	24.1
Pentosans in pulp, %	19.3	18.50	20.3
Pentosans on chips, % Alpha cellulose on	12.1	9.53	14.0
chips, % +	42.8	39.0	43.1

Notes * Determined according to the method of Wise, L. E. Murphy, M., and D' Addie Co., A. A. Paper Trade Journal 122, No. 2, 35 (1946). The values are not corrected for ash.

+ Corrected for ash and resistant pentosans.

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In each experiment after the cooking was over, the digester was relieved to atmospheric pressure, and the lid was removed and the partly delignified pulp were immediately quenched in large volume of hot water. It was observed for cooks No. 1 and No. 6 that the chips after the chemical treatment, when removed from digester, were quite stiff and that is why some modifications in cooking procedure were introduced in subsequent cooks to get soft chips.

The hot chips were then passed through a Laboratory Sprout Waldron Refiner, having a 12" disc and 1465 r.p.m. at a clearance of 40 thou. The consistency during refining was maintained at about 6.0 percent. After the first stage of refining was over, the spent liquor was removed by thickening the pulp in a hydraextractor.

The second refining was also done at about 6 percent consistency but in the absence of any spent liquor, water was used for making up the consistency. The clearance this time was 20 thou only. The unbleached pulp yield in Table III and IV refers to total unscreened pulp yield. The pulps were analysed for Kappa number according to TAPPI standards (T236m-60).

For making a comparative study, an unbleached pulp, having about 6 percent lignin content, was also prepared under the following conditions:

Cook 'C'	
Chips : Liquor	1:3
Active Alkali	
$(NaOH+Na_S), \%$	18.5
Liquor sulphidity, %	24
Maximum temperature°C	150
Time to raise to Max.	
temperature, mts.	120
Time at max.	
temperature, mts.	75
Total yield, %	55
Screened unbleached	
yield, %	51.5
Rejects, %	3.5
Kappa No.	45
Lignin in pulp, %	5.8
Yield of lignin free pulp	510
on bamboo chips, %	48.5

DISCUSSION

It can be seen from Table III and IV that with slight variations in chemical charges (14-15% and temperature 140-150°C) pulps of varying lignin contents could be obtained. The yield of lignin free pulp is varying from 48.5% for cook 'C' to 56.3% for kraft semichemical and 58.3% for kraft/ sulphite semichemicals, indicating greater retention of carboTABLE VI. STRENGTH PROPERTIES OF UNBLEACHED PULP

Cook No.				2		ŝ	. 4		сı			U	9			
Initial Freeness °SR	11.5			1.0	6	9.0	9.5	10	9.5		8.5	10	10.5	5	10.0	0
Final Freeness "SR	29	39	27	38	24	38	29	40	32	43	30	40	26	35	30	38
Beating time mts	27	36	26	36	27	34	30	40	22	29	35	40	28	41	41	53
Basis wt. g/m2	60.2	62.4	61.2	59.7	61.4	61.1	60.2	61.1	58	59.9	59.3		60.2	60.5	61.4	61.6
Thickness, microns	141.2	137.8	142.2	129.4	141.0	134.0	130.8	126.2	134.2		115.8		178.0		139.8	134.0
Bulk, cc/g.	2.34	2.21	2.32	2.17	2.30	2.19	2.17	2.07	2.31	2.19	1.95	1.86	2.96		2.28	2.18
Breaking length, km	4.54	5.08	4.90	5.28	4.97	5.25	5.12	5.53	5.38		5.85		3.87	4.20	4.98	5.37
Burst factor	29.7	33.0	29.4	34.2	32.1	35.2	34.2	36.3	31.2	36.6	37.1		23.4		29.6	31.3
Tear factor	67	94.2	112.4	104.5	101.6	96.9	108.0	97.7	92.6	86.3	104.7	93.9	106.8	100.8	88.4	81.2
Stretch, %	2.9	3.2	3.1	3.1	3.4	3.9	3.4	3.8	3.5	4.0	3.5	3.9	3.1	3.2	3.4	3.8
Folding endurance D.F.	18	19	43	55	61	56	77	82	27	20	45	46	33	42	24	19
					-											

hydrates. However, this includes slight increments in ash and extractives also. In order that the nature of pulps may be more revealing, some of the pulps were analysed for Holocellulose, Alpha cellulose and Pentosan. The results are recorded in Table V.

The results show that greater retention of holocellulose and pentosans with less degradation of Alphacellulose were obtained in Cooks No. 2 and 6 compared to Cook 'C'

The unbleached pulps were evaluated for strength properties by a Valley Beater run according to TAPPI procedure. No attempt is made in present study to correlate the strength properties of unbleached pulps to lignin content. As in pulping tests the variables introduced were many and the side effects of these variables cannot be ignored. Each pulping test is in itself a separate study and the strength properties are compared with only Cook 'C'.

The pulps are evaluated at two freeness levels and the results are recorded in Table VI. It can be seen from results that pulps of satisfactory strength properties could be obtained in yield range of 62-64 percent. Except for pulps of Cook No. 1 and 6, other pulps show quite promising results.

BLEACHING

Bleaching of pulp was carried out using two sequences. The first sequence consists of chlorination, caustic extraction, chlorination and lime neutralization, hypochlorite and acid souring.

The second sequence consists of chlorination, caustic extraction, Hypochlorite, Hypochlorite and acid scouring.

For each experiment 50 gm O.D. pulp was used. After each stage of treatment the pulp was thoroughly washed with filtered tap water. The conditions of bleaching used and the results obtained are recorded in Table VII and VIII.

Bleaching of pulps by sequence chlorination, caustic extraction chlorination and lime neutralization and Hypochlorite was tried for the obvious reason that these pulps are quite rich in lignin and, therefore, require substantial amounts of caustic soda for buffering in hypochlorite stages. Considering the cost of caustic soda, which is quite high, the use of milk of lime was tried which is much cheaper, for the unbleached pulp of Cook No. 4

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TABLE VII BLEACHING SE	EQUENCE (C.E.C.	+	Ca(OH)2H.Ac.
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TABLE VII DELAOIIING	BEQUENCE (С.Б.С. +		^{211.}
Cook No.	1	2	3	4
Unbleached Pulp Kappa Number	101	80	78	72
Total unbleached yield on OD chips. %	65.4	62.5	62.5	61.4
1st Stage				
C1, added, %	20	18	18	16
C1, consumed, %	20	18	17.2	15.9
Final pH	0.95	1.0	1.0	1.1
2nd Stage				
Final pH	7.4	8.3	9.3	9.6
3rd Stage	8	e	4	4
Cl ² added, % Cl ² consumed, %	° 7.7	6 4.8	3.5	3.5
Retention time, mts.	3	3	3	3
Ca(OH) added, %	10.4	7.4	4.9	5.0
Final pH	9	8.8	8.5	8.8
4th Stage Cl [_] added, %	5.0	3.0	2.0	2.0
Cl_2 consumed, %	3.33	1.60	1.05	1.01
NaOH added, %	1.0	0.80	0.60	0.50
(for maintaining pH) Final pH	9.5	9.2	9.5	9.6
H:SO4, Stage Consistency, % 2.5 Temp. °C 28 Retention time, mts. 30				
H,SO, added, %	0.8	0.8	0.6	0.6
Final pH	4.5	4.6	4.6	4.5
Total Cl2, added, % Total Cl2, consumed	33.0	27.0	24.0	22.0
on unbleached pulp, % Total Cl ² consumed bleach-	31.0	24.4	21.7	20.4
ed pulp %	38.0	29.3	25.9	24.1
NaOH consumedon unbleached pulp, %	5.0	4.8	4.6	4.6
Ca(OH) ² , consumed, %	10.4	7.4	4.9	5.0
Yield of bleached pulp on unbleached pulp, %	81.6	83.2	83.7	8 4.6
Shrinkage during bleaching, %	18.4	16.8	16.3	15.4
Bleached yield based on	2012	1010	1010	
total unbleached yield on OD bamboo chips, %	53.4	52.0	52.3	51.9
TAPPI viscosity, cP	44	58	55	58
Brightness, %	76	78.5	78	77
Chlorination	Caustic	extraction	n	
Constant Conditions		t Conditio		
Consistency, % 2.4	Temp. °	ncy, % 5 C 50		
Temp. °C 30 Time, mts. 45	NaOH a Time, n	dded, %	4.0	
Chlorination and lime				
Neutralisation	Coloium	Hypochlo	vrita	
Constant Condition Consistency, % 4.0		t Conditio		
Temp. °C 30	Consiste	ncy, % 5.	0	
Total retention	Temp. °	C 40		
time, mts. 60	Retentio	n time, m	ts. 120	

* On unbleached pulp basis.

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TABLE VIII. BLEACHING SEQUENCE (C.E.H.H.Ac.)

Cook No.	4	5	6	7	С
Unbleached Pulp Kappa Number	72	72.4	120	78	45
Total Unbleached yield on OD bamboo chips, %	61.4	61.7	69.0	64.1	51.5
1st Stage					
Chlorination Consistency, % 24 Temp. °C 30 Time, mts. 45					
Clª added, % Cl, consumer, % Final pH	$16.0 \\ 15.8 \\ 1.0$	$16.0 \\ 15.9 \\ 1.1$	20.0 19.9 1.0	18.0 17.9 1.0	12.5 12.1 1.1
2nd Stage					
Caustic Extraction Consistency, % 5 Temp. °C 50 Time, mts. 60					
NaOH added, % Final pH	4 9.0	4 10.0	5 10.1	4 10.0	3 10.8
3rd Stage					
Consistency, % 5 Temp. °C 40 Time, mts. 60					
Cl ² consumed, % Cl ² added, % NaOH added, % Final pH	3 3.0 0.6 8.2	4 3.72 1.0 8.8	6 5.25 1.65 8.6	4 3.92 1.0 8.7	2.5 2.4 0.5 8.6
4th Stage					
Consistency, % 5 Temp. °C 40+1 Retention Time, mts. 120 Cl ² consumed, % Cl ² added, % NaOH added, %	3.0 1.90 0.4	2.0 0.92 0.5	2.0 1.21 0.5	2.0 1.22 0.5	1.5 0.8 8.6
Consistency, % 2.5 Temp, °C 28 Retention time, mts. 30					
H₂SO₄ added, % Final pH Total cl₂ added %	$0.5 \\ 5.0 \\ 22$	$0.6 \\ 5.1 \\ 22$	0.6 5.4 28	0.6 5.1 24	0.6 4.8 16.5
Total Cl₂ consumed on unbleached pulp, % NaOH consumed %	$\begin{array}{c} 20.7 \\ 5.0 \end{array}$	$\begin{array}{c} 20.5\\ 5.5\end{array}$	26.4 7.15	$\begin{array}{c} 23.1 \\ 5.5 \end{array}$	15.3 3.8
Yield of bleached pulp on unbleached pulp, %	85.1	8 3.2	78.9	85.0	91.0
Shrinkage during bleaching, %	14.9	16.8	21.1	15.0	9.0
Total Cl., consumed on bleached pulp, %	24.3	24.6	33.5	27.2	16.8
Bleached yield on OD bamboo chips, %	52.2	51.3	54.4	54.5	46. 8
Cuprammonium disperse Viscosity, cP Brightness, %	39 77	46 77	37 76	43 77	44 77

which had been bleached by both sequences, the differences in the amount of caustic consumption are not appreciable, but for other pulps having the same Kappa Number the differences do exist. However, the choice for a particular bleaching sequence in any paper mill will be governed by a number of other factors also, such as the availability of equipment, the cost of various chemcals and the ease of operation of bleaching sequence.

It can be also seen from the results given in Table VII and VIII that by using first sequence the bleached pulps of slightly higher viscosity are obtained and ultimately may show slightly better strength 'properties' (only COOK No. 4 is bleached by both sequences). However, this is not true for bleached pulp of Cook No. 1. The possible explanation for this is that beyond a certain point the chlorine dosages whether as elemental chlorine or as hypochlorite proves detrimental to cellulose, and especially so when they are transferred to pulp in limited bleaching stages.

CONCLUSIONS

- 1 Unbleached pulp of 62 to 65 percent yield and satisfactory strength properties can be obtained using kraft semichemical process. The strength properties were better at 62 percent yield than at 65 percent.
- 2 By using Kraft/Sulfite process unbleached yield of 65 percent and 69 percent were obtained. The strength properties were better at 65 percent yield compared to that of 69 percent.
- 3 By using conventional bleaching agents, bleached yield of 52 to 54 percent at brightness levels of 76 to 78 can be obtained. From the point of view chlorine consumed and the bleached yield obtained the conditions of pulping and bleaching used in cooks 3 to 5 are optimum. The question is largely of balancing the cost of bamboo against the cost of chemicals involved in bleaching. Other indirect advantages such as the increase in productivity and the reduction in fibrous raw material consumption should also be taken into consideration.

4 While bleaching these pulp by conventional bleaching agents, shrinkage (15 to 18%) was appreciable. In order to reduce this high shrinkage

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other bleaching agents such as peroxides, hydrosulphites, etc. may have to be used. The usefulness of these bleaching agents will depend to a large extent on the cost of these chemicals and the increase in brightness and bleached yield obtainable. Using some of these chemicals for the bleaching of Kraft semichemical pulps, a separate study will be undertaken and that will constitute the subject matter for a separate publication.

- 5 With conventional bleaching of pulp, it is desirable to segregate pulp for unbleached and bleached varieties of paper.
- 6 It may be stated here that bleaching of high yield Kraft Semichemical pulps presents some difficulties in bleaching to high brightness and high bleached yields because of their dark colour. To get high bleached yields it is necessary that losses of carbo-

hydrates should not take place. Not only that it is preferable to retain even a part of the lignin in modified colourless form by destroying chromophoric groups and thus contributing to improvement in bleached yields. Giertz (4) has emphasised the importance of "Surface Bleaching" (Decolourising the coloured matter) to obtain high bleached yields.

7 The fact that high unbleached and bleached yields can be obtained using kraft semichemical process and conventional bleaching agents will strengthen the claims that a judicious use of national forest wealth is one way out to meet the shortage of fibrous raw material, especially at a time when the paper industry is faced with the problem of finding additional raw materials.

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Presented at the Seminar on 'Improvement of yield from Indian Raw-materials' of the Indian Pulp and Paper Technical Association, Madras, March 14 to 15, 1969.

DISCUSSION

K. C. Viramani: General Comment:

It may be of great interest to note that the bamboo pulps prepared under unconventional conditions described in this paper may be advantageously employed for developing better shades of coloured papers by employing the same dyes as used with the conventional bamboo pulp.

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