

Comparative Kraft Pulping of Pine and Eucalyptus Changes in Pulp Properties throughout Cooking Cycle

S. V. Singh
Rajesh Pant
Y. K. Sharma

Introduction

Pulping characteristics of various raw materials have been studied by various workers on a kinetic basis and data on changes in pulp properties throughout the cooking cycle reported; however, the conditions employed were not identical in all cases. The aim, of the present investigation was to provide comparative information on kinetics of kraft pulping of pine, eucalyptus and bamboo in regard of changes in yield, lignin to carbohydrate ratio and pulp properties throughout the cooking cycle under identical conditions of digestion. The results on bamboo were taken from earlier work¹.

Experimental

Raw Material Preparation

The species used in this investigation were *Pinus patula*, *Eucalyptus* hybrid (mainly *tereticornis*) and *Dendrocalamus strictus*. The chips were prepared in a pilot plant Waterous 4 knife chipper. After

S. V. Singh, Rajesh Pant and Y. K. Sharma, Cellulose & Paper Branch, Forest Research Institute, Dehra Dun.

Changes in pulp properties namely yield, lignin to carbohydrate ratio and strength were followed under identical conditions throughout cooking cycle consisting of a period of 90 min. to 170° and a period of a varying time (0-90 min.) at maximum temperature, and the data were compared with those on bamboo¹. Upto 100° the drop in yield followed the order bamboo > eucalyptus > pine. The same order persisted at the end of rise to temperature period. At the end of cook both pine and bamboo touched same level of yield and eucalyptus gave 5% higher yield. Conditions of selective delignification were identified in case of all the three, when changes in lignin to carbohydrate ratio were followed with yield. On an average best strength properties were obtained at about 50-52% yield in all the three cases. This limit was achieved more easily in bamboo than pine and eucalyptus, the later two required same time.

screening, the chips were air dried, packed in black paper bags and wrapped with polythene bags; each containing 500 g. oven dry weight. The chip size and moisture content affect the rate of delignification as they effect the rate of transport of chemicals to the site of reactions and of reaction products away from the site by diffusion. In the work described in this paper it was considered sufficient to use samples of chips for which the

average size and moisture content were constant.

Cooking Cycle

The cooking cycle was made up of the conditions as listed hereunder:—

Active alkali	15% as Na ₂ O on oven-dry chip
Sulphidity	25%
Material to liquor ratio	1:4
Initial temperature	50°C

Maximum temperature 170°C
 Time to maximum temperature 90 min.
 Time at maximum temperature 0 to 90 min.
 The temperature was raised at the rate of 4° per 3 min. to the maximum temperature or to a lower level temperature in those cooks where the cycle was interrupted before the maximum temperature was reached to achieve very high yield pulps.

Pulp Treatment

Pulps of yield below 50% were disintegrated in a disintegrator equipped with an impeller from 5 to 30 min. and then screened on a cut flat screen of 0.35 mm slots. Pulps of higher yields were prepared by fibrizing in Sprout Waldron refiner. The refined pulps were not screened.

Pulp Evaluation

Pulps yields were determined by the method described in earlier publication¹. Lignin content determination in pulp was done by Tappi Standard Method. The strength properties were determined after beating the pulps to a freeness of 250+25 CSF in Jokro Mill and conditioning the hand sheets made therefrom at 65% RH and 27°C.

Results

The variation in cooking conditions and results of analyses of various pulps are recorded in Tables I to III.

Discussion

Changes in yield with time and temperature

In order to obtain information

regarding changes in yield with time and temperature the standard cooking cycle was interrupted at regular intervals and increasing temperature levels. The results have provided some interesting information. These are listed in Tables I to III and represented graphically in Fig. 1. It will be noted from Fig. 1 that the original drop in yield followed the order Bamboo > Eucalyptus > Pine. Further, in case of Pine and Bamboo, apart from the original drop in yield in 37.5 min., the greatest rate of loss in yield occurred from approximately 40 min. to 90 min. or from 100°C up through the maximum 170°C; whereas in case of Eucalyptus the

TABLE I—Cooking conditions and pulp properties of *Pinus patula*.

Sl. No.	Cook			Yield		Chemical properties		Physical Properties		
	Time, Min.	Blow Temp. °C	'H' factor	Total %	Rejects %	Klason lignin %		Breaking length Km.	Burst factor	Tear Factor
1.	37.5	100	4	87.5	—	23.9		1.12	3.8	14.0
2.	45.0	110	19	—	—	—		—	—	—
3.	52.5	120	64	74.5	—	24.3		2.12	6.6	39.1
4.	60.0	130	192	77.5	—	22.0		3.06	10.1	23.2
5.	67.5	140	533	73.4	—	21.2		2.87	8.5	55.0
6.	75.0	150	1399	69.0	—	18.8		3.64	12.4	69.4
7.	82.5	160	3522	65.9	—	18.3		5.82	36.8	67.2
8.	90.0	170	8502	60.3	—	16.2		7.46	41.6	103.5
9.	105.0	170	22407	61.0	—	16.1		8.09	49.8	117.0
10.	120	170	36312	60.8	—	16.4		7.39	44.2	120.4
11.	135	170	50217	53.0	0.04	6.5		9.37	40.0	171.2
12.	150	170	64122	49.9	6.0	5.70		9.01	53.5	142.6
13.	165	170	78037	46.8	5.0	4.71		8.41	47.8	157.8
14.	180	170	91932	44.1	4.0	3.56		7.56	52.5	91.4

TABLE-II
Cooking conditions and Pulp Properties of Eucalyptus hybrid

Sl. No.	Cook		'H' factor	Yield		Chemical Properties		Physical Properties		
	Time Min	Blow Temp. °C		Total %	Rejects %	Klason lignin %		Breaking length Km.	Burst Factor	Tear Factor
1.	37.5	100	4	84.4	—	24.4		1.33	3.3	12.5
2.	45.0	110	19	82.3	—	22.0		1.97	8.6	22.5
3.	52.5	120	64	83.0	—	22.4		2.83	10.2	21.6
4.	60.0	130	192	81.4	—	20.4		3.06	10.0	34.8
5.	61.5	140	533	77.6	—	18.6		3.07	18.3	32.2
6.	75.0	150	1399	71.8	—	16.8		4.30	26.4	60.0
7.	82.5	160	3522	65.0	—	12.6		5.26	27.8	80.2
8.	90.0	170	8502	55.0	14.0	6.96		7.04	48.3	118.3
9.	105	170	22407	53.5	0.58	2.67		5.95	39.0	116.2
10.	120	170	36312	53.0	0.40	2.08		5.87	45.6	114.7
11.	135	170	50217	52.5	0.39	1.96		7.56	53.1	116.6
12.	150	170	64122	51.0	0.40	1.82		7.26	48.6	115.4
13.	105	170	78037	50.2	0.04	1.82		6.93	40.2	111.1
14.	150	170	91932	50.2	0.08	1.80		7.06	43.3	100.0

Table—III
Cooking conditions and pulp properties of bamboo (D. Strictus)

Sl. No.	Time, Min.	Cook		'H' factor	Yield		Chemical Properties			Physical Properties			
		Blow Temp. °C.			Total %	Rej-ects %	Klason lignin %	Pento-sans %	Cellu-lose %	Kappa Num-ber	Break-ing length Km.	Burst factor	Tear factor
1.	37.5	100		4	81.2	—	23.0	16.0	80.2	—	2.50	15.0	70.0
2.	45.0	110		19	75.0	—	22.0	15.5	80.0	—	3.00	18.0	75.0
3.	62.5	120		64	70.0	—	21.0	16.0	80.8	60.0	3.40	20.0	76.0
4.	60.0	130		192	68.0	—	19.5	15.3	80.6	59.0	—	—	—
5.	62.5	140		533	62.0	—	16.0	15.3	80.4	51.0	3.40	20.0	80.0
6.	75.0	150		1399	57.6	—	13.0	15.0	80.0	45.2	—	—	—
7.	82.5	160		3522	56.0	—	12.0	15.0	80.4	41.0	3.66	25.0	120.0
8.	90.0	170		8502	50.0	4.0	8.6	14.5	80.6	36.3	4.46	30.2	151.0
9.	100.0	170		17772	49.8	3.4	7.4	14.0	181.0	33.8	—	—	—
10.	110.0	170		27042	49.4	2.4	6.4	14.2	82.4	34.0	4.73	30.3	151
11.	120.0	170		36312	48.9	1.6	5.8	14.0	83.0	32.0	5.01	34.0	152
12.	130.0	170		45822	48.4	1.1	5.1	13.8	84.1	33.0	4.56	34.0	155
13.	140.0	170		54852	42.6	0.6	4.8	13.6	84.0	32.0	4.59	35.0	180
14.	150.0	170		64172	46.8	0.2	4.0	13.5	84.4	30.2	4.50	30.6	155
15.	160.0	170		73392	45.4	0.1	3.5	13.6	84.8	28.2	4.75	31.3	152
16.	170.0	170		82662	44.4	0.0	3.0	13.2	85.1	27.1	4.17	32.1	165
17.	180.0	170		91932	43.6	0.0	2.6	13.0	85.0	20.0	4.54	31.3	170

rate of greater loss in yield started from approximately 60 min. of time; and the rate of loss being more than that of Pine and Bamboo. Beyond 90 min., the Eucalyptus and Bamboo followed almost similar trend; whereas in case of Pine the yield remained almost constant in the region 90-120 min. and thereafter it dropped with a faster rate to the level of Bamboo when compared with the other two. Eucalyptus gave pulp of higher yield (about 5%) than Pine and Bamboo under similar conditions of cooking.

Change in lignin to carbohydrate ratio

The rate of removal of lignin to carbohydrate ratio and the ratio in which they are retained in pulp is of prime importance in assessing the progress of pulping reactions and the behaviour of pulp in subsequent treatments. This ratio is a characteristic of interest in the field of high yield pulps for use as such and of potential interest in the field of bleached pulps.

The representation of the data on changes in lignin to carbohydrate ratio loses its meaning all to, easily when merely assembled in column of figures. Therefore in order to follow change in pulp composition with respect to lignin and total carbohydrate as the yield changes, the results were plotted according to the Ross diagram in fig. 2.

The Fig. 2 has revealed various important facts about the stages

of dissolution of lignin and carbohydrates. In case of Pine, down upto 78% yield the loss of carbohydrates was more than lignin; thereafter upto 68% of yield carbohydrates were removed

in same proportion. Again from this point down to 60% yield, carbohydrates were dissolved more than lignin. From 60% to the yield of 54% both lignin and carbohydrate were removed

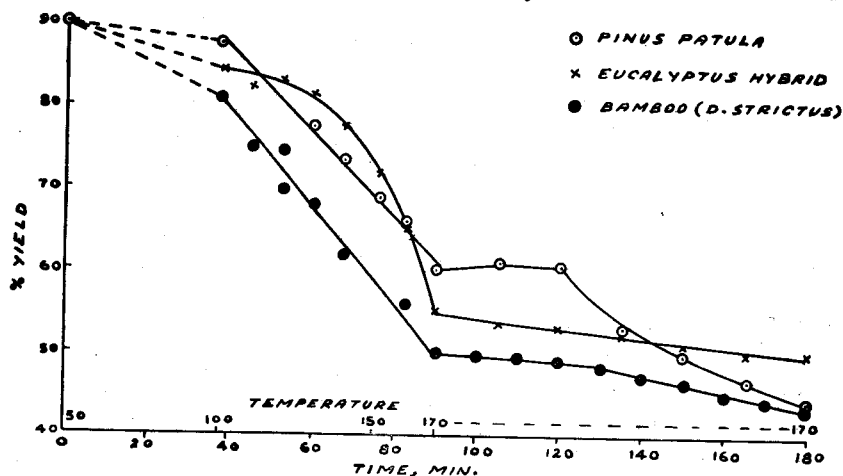


Fig. 1 % Yield Versus-Cooking Time and Temperature

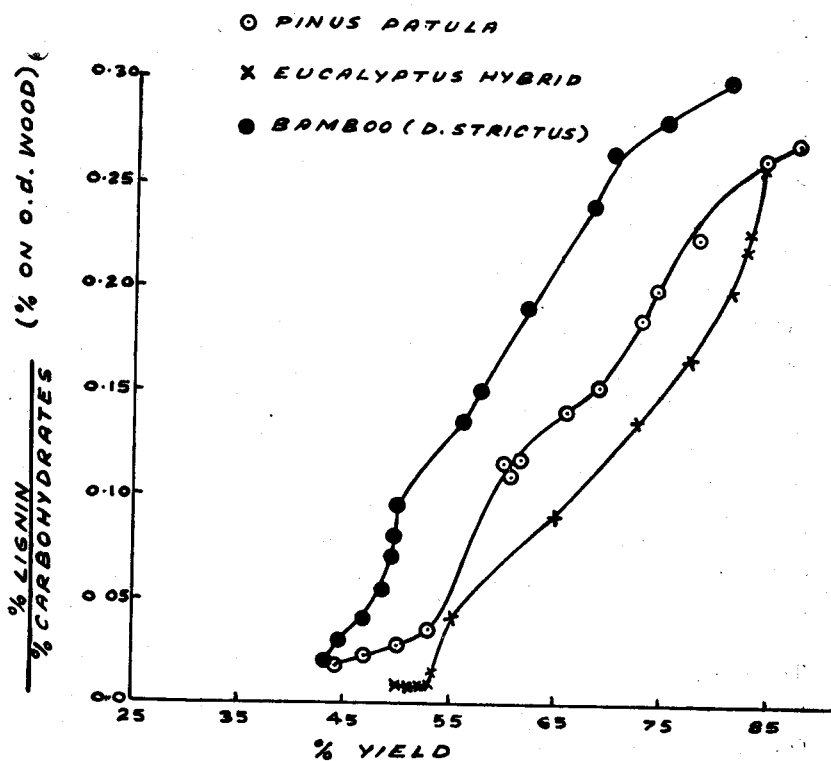


Fig. 2 Pulp Yield Versus Composition (Lignin to Carbohydrate Ratio)

rapidly but in same proportion. Beyond this point to the lowest yield for the most part the loss was carbohydrates and only little lignin was dissolved.

In case of Eucalyptus at early stages that is upto a yield of 82% both lignin and carbohydrates appeared to have been removed in same proportion. Thereafter also the removal characteristic was similar (upto 55% of yield) but the amounts dissolved were less than that gone into solution at the first stage. The decrease of yield from 54% to 50% indicated the loss of all carbohydrates and practically no delignification occurred.

In case of bamboo down to 70% of yield the loss was carbohydrate for the most part. From 70% down to 50% carbohydrate and lignin were removed in approximately same proportion. From 50% down to 43% again carbohydrate and lignin were removed in approximately equal proportions but in lesser quantities as compared to second stage.

Thus it was observed that during the regular kraft cycle employed in this investigation there were quite a few conditions that gave rise to selective removal of lignin in case of all the three raw materials. On the basis of this data pulps of desired composition can be chosen and cooking cycle terminated accordingly.

Physical Properties

The results of changes in strength properties with yield throughout cooking cycle are represented in

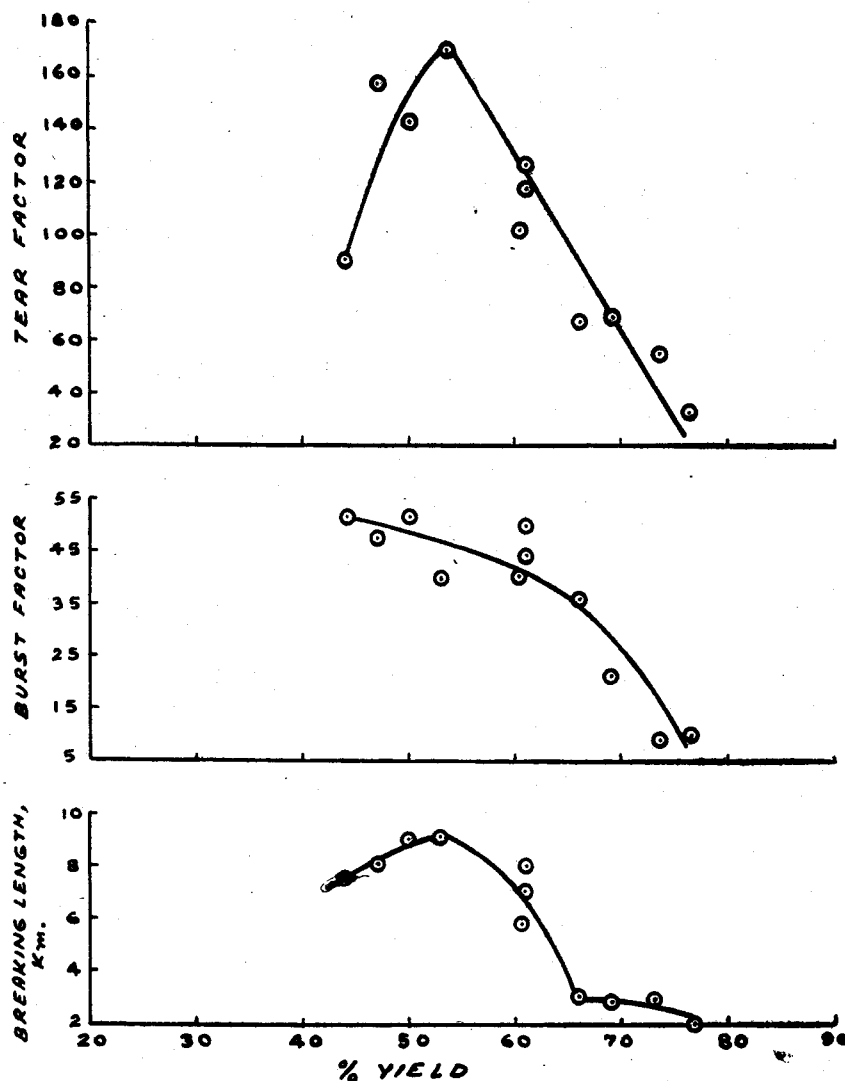


Fig. 3. Developed Strength Properties VS Yield
(Pinus Patula)

Fig. 3, 4 and 5 respectively for Pine, Eucalyptus and Bamboo.

Pinus Patula

Breaking Length : The lowest yield of 44% gave a breaking of 7.56 Km. This increased gradually to the value 9.91 Km. upto a yield 50% and then decreased with increasing yield in two different pattern, a rapid one followed by slow.

Burst Factor : The lowest yield gave a burst factor of 52.5 which decreased gradually with increasing yield.

Tear Factor : The tear factor increased sharply from the value 91.4 at lowest yield of 44% to a value of 171.2 at 53.0% yield. Beyond this it decreased with increasing yield.

Eucalyptus hybrid (mainly tereticornis)

Breaking Length : The lowest yield of 50% gave a developed breaking length of 7.06 Km. This rose slightly to 7.56 Km. at 52.5% yield and then decreased with increasing yield.

Burst Factor : Similarly the lowest yield gave a burst factor of 43.3 which also rose slightly to a value of 53.1 at a yield of 52.5%; beyond which it decreased with increasing yield.

Tear factor : The tear factor also increased slowly from a value of 100 at lowest yield to 118.2 at 55% yield and then decreased with increasing yield.

Bamboo (Dendrocalamus strictus)

Breaking Length : The lowest yield,

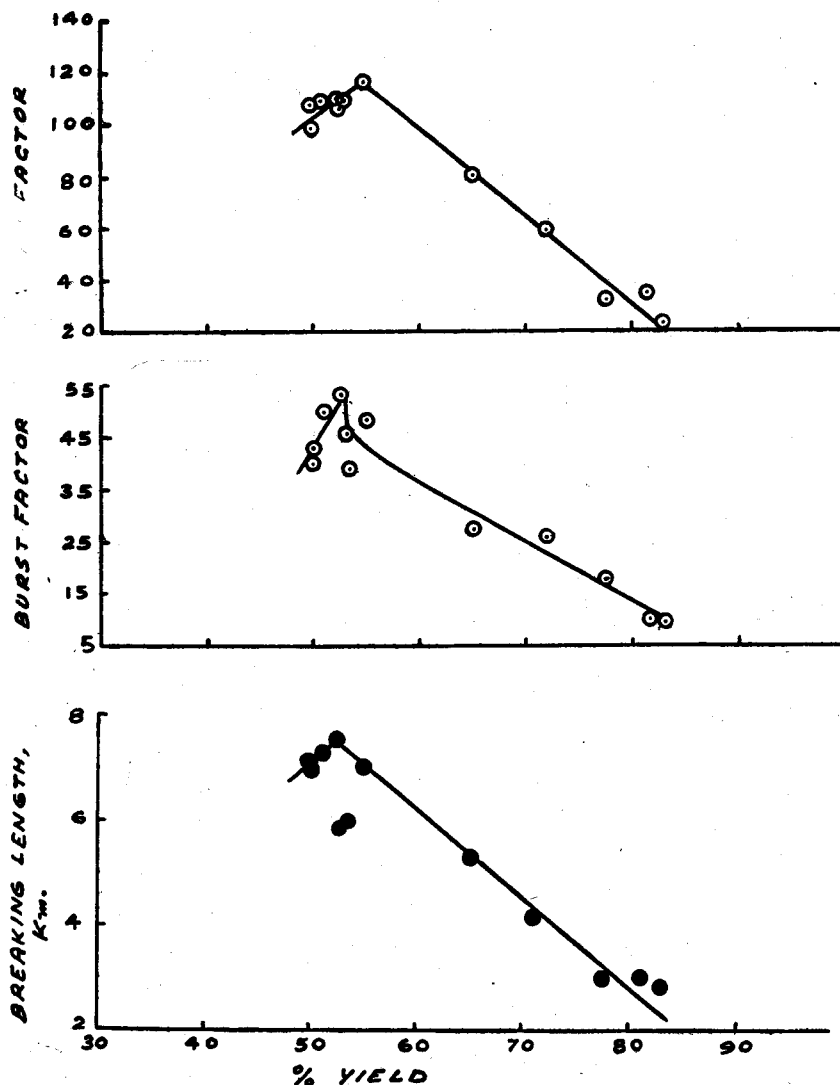


Fig. 4 Developed Strength VS Yield
(Eucalyptus Hybrid)

of approximately 43%, gave a developed breaking length of 4.54 Km. This rose slightly to 5.0 Km. at 49% yield and then decreased with increasing yield.

Burst Factor : Similarly the lowest yield gave a developed burst factor of 31.3 which also rose slightly to a value of 35.0 at a yield of 47.6%; beyond which it decreased slowly with increasing yield.

Tear Factor : The tear factor decreased gradually with increasing yield but there were two distinct phases of decreasing order. From the lowest yield of 43% to a value of approximately 62%, the decrease in tear factor followed one rapid pattern, beyond that yield the decrease was with a different slow pattern. An exceptional value of 180 was obtained at 47.6% of yield.

These results demonstrate that under the conditions of cooking employed in this investigation all the three raw materials, on an average, gave best strength properties at about 50-52% yield. The decrease in strength properties in pulp of yield lower than this is an indicative of further degradation of carbohydrate fraction of pulp at later stage of cooking period. This optimum value of yield in relation to strength properties of pulp was

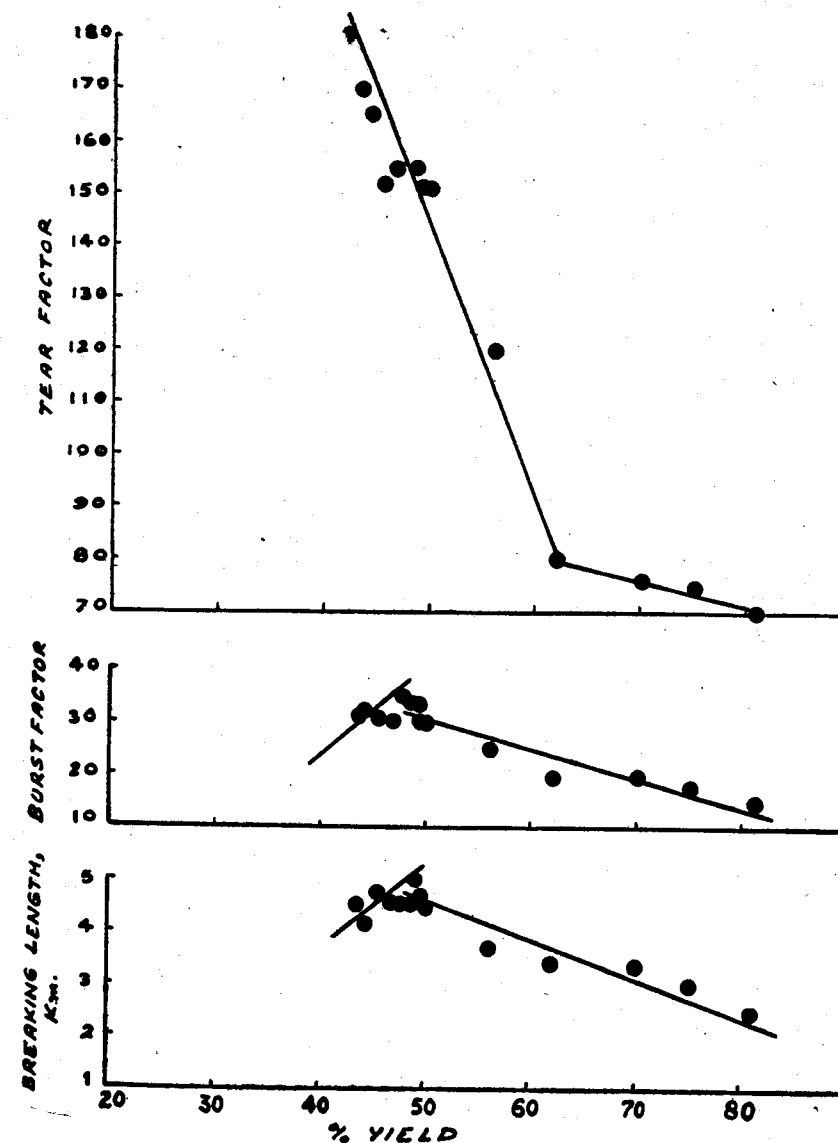


Fig. 5 Developed Strength Properties Vs Yield
(Bamboo-D. Strictus)

obtained by terminating the cooking cycle after 35 min. at 170 in case of Pine and Eucalyptus both whereas in case of Bamboo, a period of 10 min. at 170°C was sufficient.

References

1. Singh, S.V. and Guha, S.R.D., *Indian Pulp and Paper* 30, No. 3, 15 (1975).