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Preliminary Investigation On Bamboo Pulping

by

Semi-Keebra Process)

(SENIOR THESIS)

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ABSTRACT

A preliminary investigation on bamboo pulping by Semi-Keebra process reveals that a higher pulp yield than that obtained by most of the known methods of pulping bamboo can be obtained by Semi-Keebra process.* The chemical requirement for pulping is lower than the conventional processes and the spent liquor also contains a higher per cent of solids. The pulp obtained by Semi-Keebra pulp would also require a considerable lesser time for obtaining the desired freeness value, resulting in lower power consumption for stock preparation, than that for the conventional sulphate bamboo pulp of bleachable grade. A comparison of the various hand-sheet properties of the two pulps show that all properties other than density and breaking length are, however, lower in the case of Semi-Keebra bamboo pulp. It is, however, hoped that further investigation on optimum conditions of Semi-Keebra process for bamboo pulping would show some further improvement in the strength properties.

DEFINITION OF PROBLEM

The various difficulties encountered in bamboo pulping by alkaline and other processes, particularly difficulties due to long cooking periods, use of liquor of high chemical content, highly colored pulp which require extra bleaching, low pulp yields, and trouble in recovery of chemicals from the spent liquor have necessitated the preliminary investigation on bamboo pulping by Semi-Keebra process, which has not yet been reported in literature for bamboo. The Semi-Keebra process for wood is reported in literature to be especially well suited for certain grades of both fine and other papers besides the benefits of an increase in the yield. In

*The Keebra Process has been developed and patented by E. P. McKeefe and L. Bradley of New York.

case of the wood the pulp is also reported to be more easily bleached.

LITERATURE SURVEY

Bamboo Pulping:

Most of the known methods of pulping has been applied to bamboo. Least promising of the conventional methods is mechanical pulping which is attributed to difficulty of holding the culm against the stone, the difference in structure of node and internode, and change in hardness with drying (1).

Alkaline cooking owing to their high content of H_2O -soluble material especially of pentosan in the stem are best suited for bamboo and sulphate process is reported to be the best (2-3). In the sulphate method best results are obtained by a mixture, in the total cooking liquor, of 60-70 per cent $NaOH$ and 30-33 per cent of Na_2S , which is about the limit attainable by the later in the recovery. But perfectly good results are obtained by 25 per cent of Na_2S . The check on hydrolysis imposed by the presence of Na_2S increases the yield slightly and is responsible for a marked improvement in the color of the pulp (4). About 20-22 per cent chemicals on the weight of the wood is required. The maximum pressure should be about 120 lb., temperature 162-170°C., and the time of cooking about 5 to 6 hours. The yield is about 41 to 43 per cent (2). A two stage cook is recommended for soda process, a mild cook at about 115°C. for 2 hours in 1.5 per cent caustic soda, followed by a more drastic cook in 5 per cent caustic for 3 hours at 140 to 160°C. After bleaching with 8 per cent bleach, a yield of 42 per cent pulp on the weight of bamboo is obtained (5).

Good pulps have been obtained by the modification of soda and kraft process. Two modifications, the Gruco process and the Raitt pro-

cess have yielded the best quality pulp. (1).

The Grucco method (1) consists of: (a) a cooking liquor composed of 75 to 80 grams of NaOH plus 12 to 15 grams of Na_2S per liter of liquor, (b) a total cooking period not in excess of 4 hours, (c) reduction of cooking temperature to range of 140 to 160°C.

In the Raitt process (4), the principle of "fractional digestion" is applied. The method aims at reducing the cost of digestion and improving the quality and yield of pulp. The process consists of removal of soluble substance in raw material by using a combination of spent liquor and caustic. The reason for this discussed in (4-6), is based on removal of the three undesirable components namely: (a) water soluble salts, starches, sugar etc., (b) soluble pectins, fats, waxes and gums in 1 per cent alkali at 100°C., and (c) alkali soluble lignin acid etc. in 4 per cent alkali at 130°C. Removal is accomplished in two stages (17).

(a) Crushed bamboo chips are extracted for about 2 hours with a partially exhausted alkaline solution containing about 2 per cent of active alkali from a previous cook at a temperature of 100 to 115°C..

(b) The predigested material is then cooked in a fresh caustic solution containing about 5 per cent for 3 hours, one hour at 150°C. and the remaining two at 140°C. Best results are obtained by the sulphate process, reusing the spent liquor.

Several other methods besides the alkaline process and its modifications have been tried for cooking bamboo. The acid methods fail to deal satisfactorily with starch and pectin, although they handle lignin well enough; but the residue of these remain present in the pulp as a yellow coloration. Raitt (7) reported calcium-bi-sulphite to be a total failure for bamboo cooking, but obtained fair results with magnesia-bi-sulphite. The acid treatment of bamboo gives a fair unbleached

pulp, good enough for newsprint and similar low colored papers but bleaching difficulty remains if pure white papers are wanted. A later work on cooking of crushed bamboo chips by calcium-bi-sulphite liquor (8) shows that well digested nearly uniform and light colored pulp having 42.25 per cent yield on A.D. chips can be obtained; which can be bleached to a good white shade, retaining a yield of 40.43 per cent. The time required for digestion was 12 hours (4 hours for reaching the maximum temperature of 162°C., and 8 hours cooking at the maximum temperature), which makes the use of this process doubtful for large scale production. The above process when carried out with uncrushed chips gave undigested pulp containing a large number of uncooked pieces. This partially digested pulp could not be bleached within economic limits.

Neutral sodium sulphite method has also been used (9) for cooking bamboo (sino-take). The chips (3-4 cm. length) when pulped at 163°C. and a pressure of 6-5 Kg. per sq cm. gave a better yield than by soda or sulphate method. Lignin removal was rapid during the first 2 hours of cooking but slowed down and became almost constant during the next 3 hours. Alpha-cellulose per cent based on total charge was found to increase progressively with cooking time; however, pentosans remained in the pulp in large quantities despite prolonged cooking. The strongest paper, however, was obtained from pulp cooked for 4 hours to a lignin content of 10.9 per cent.

A preliminary investigation on bamboo pulping by cold soda process (10) reveals that out of the various chemicals applied for pretreatment at room temperature and at atmospheric pressure water steeping of bamboo give better results. Both bleached and unbleached papers having fairly good strength can be prepared by this method and the strength of the

bleached paper can very well be compared with those obtained by pressure digestion sulphite process for bamboo investigated by Bhargave and Batra (11).

The soda cooked semi-chemical pulp is reported to be of poor quality (1).

Bamboo being much less receptive to the penetration of liquid than wood give several pulping difficulties. The major difficulties encountered are: long cooking periods, the use of liquor of high chemical content, non homogeneous pulp, degenerated surface cellulose, highly colored pulp requiring extra bleaching, and further trouble in soda recovery because of insufficient alkali being left in the pulp. Both the conventional overhead (single stage) and fractional (two stage) cooking system suffer from some or all of these faults. Bhargave (12) has presented a new method which solves all these problems and produces a superior pulp in higher yield. In the production of easy bleaching and bleachable pulp the bamboo is first chipped into pieces of 1/8 to 1 inch in size. These chips are then steamed in water or very weak liquor to open up their fibers and then subjected to an action of super-heated pressurized cooking liquor. Pressure and temperature are brought down after the requisite period and there after a conventional digestion process is followed. In the production of high yield chemical or semi-chemical pulp a mechanical defibration step follows the super cooking step.

Chaudhuri (13) has also suggested the steaming of chips before alkaline pulping for reducing the cooking cycle in both one stage and two stage process. The bleach ability of the pulps by the two processes is reported to be the same because of steaming.

The other proposed process (14) involve steeping of the bamboo in spent liquor from an earlier cook for 2 to 5 hours at 115 to 120°C.,

followed by cooking in a 5 per cent solution of NaOH for 2 more hours at 140 to 160°C.

Investigation on influence of liquor ratio (15) shows that lower liquor ratio favor the removal of lignin and the increase of the added alkali. The yield of the pulp is reduced little by the decrease of the liquor ratio; but variation of liquor ration has no influence on the removal of pentosans.

Nodes also constitute a problem in pulping because they are denser than the rest of the stock and are penetrated with great difficulty. Cooking of bamboo is facilitated (8) by crushing the bamboo stems between heavy iron rollers, which crush the internodes and separate the fiber bundles. The thoroughly crushed chips are found to give highest per cent of both unbleached and bleached pulps and are comparatively more uniform in quality and require the least per cent of standard bleaching powder for the production of good white shade. Although crushing of bamboo chips is the ideal method its use on a large scale is doubtful because of high power requirement and more irregular size of the crushed chips.

Semi-Keebra Process:

The Semi-Keebra pulp is reported (16) to have characteristics between soda and Full Keebra pulps. The Full Keebra pulp is reported to rival kraft pulp in strength and with abnormally high tear and folding properties. The Semi-Keebra pulp is reported to be especially well suited for certain grades of both fine and other papers, besides the benefits of an increase in the yield due to the protective action of the sulphite. The resulting pulp also keeps its strength and color without deterioration. In case of wood the pulp is reported to be more easily bleached.

The reason why Semi-Keebra process in particular has been selected over Full-Keebra process is that in the later the recovery and reuse of the spent liquor has not been demonstrated to be practical if it is used by itself. However, if a proportion of Semi-Keebra cooks are made at the same time there are no difficulties in recovering the two spent liquors together. In working the Semi-Keebra process no difficulties whatsoever are reported (16) in obtaining sufficient liquor suitable for sulphiting, however high the recovery. The black liquor is evaporated in the usual way, and losses in the system are made up by fresh soda ash used for sulphiting. The Semi-Keebra process is also reported to require lower cooking time and pressure than Full-Keebra process. The cooking liquor in the Semi-Keebra process consists of caustic soda and sodium sulphite in a quantity of 20 to 30 per cent of total soda present.

EXPERIMENTAL PROCEDURE

Bamboo (*Dendrocalamus Strictus*) chips of equal size (2 to 2.5 Cm. long and 1 to 2 Cm. wide) were cooked in a stainless steel digester provided with an arrangement for constant liquor circulation through the heat-exchanger. The cooking liquor consisted of 7.2 per cent Sodium hydroxide, 1.8 per cent Sodium Sulphite, and 0.256 per cent Sodium Sulphide, based on the total liquor volume. The ratio of liquor to chips was kept at 8 to 1. The results of cooking liquor analysis appear on Table I of the report. The chips were cooked for a total period of $3\frac{1}{2}$ hours ($\frac{1}{2}$ hour to raise the temperature to $144^{\circ}\text{C}.$, and a further 3 hours at $144^{\circ}\text{C}.$) and at pressure of 56 psig.

The cooked chips in order to defiberate were passed through a laboratory Bauer refiner, fitted with coarse plates, thrice, keeping the load at 2, 3, and 4 amperes respectively. The pulp was then washed in a

drainer box and screened through a 0.01 cut flat screen to remove sieves etc. The screened pulp was washed in the drainer box again and filtered through the pulp mat formed by the fibers, till clear washing was obtained. After dewatering the per cent yield of the pulp was determined. The Permanganate number and initial brightness of the pulp and per cent solids in black liquor were also determined according to Tappi standards.

The refining of the pulp was carried out in a Mead laboratory refiner at 2 per cent consistency, using 25 pounds thrust on the plates. The pulp was refined at four different refining times and hand sheets were made on British sheet mold according to Tappi Standard Method at the four different freeness values obtained. The hand sheets were tested for various sheet properties according to Tappi Standards and the results appear on Table III of the report.

In order to make a comparison in the strength properties of the Semi-Keebra pulp and the conventional sulphate bamboo pulp of bleachable grade, hand sheets were made at the same freeness ranges as in the case of Semi-Keebra pulp and the hand sheets were tested for the various sheet properties and the permanganate number and the initial brightness of the pulp were also determined according to Tappi Standards. The Sulphate bamboo pulp of bleachable grade was obtained for this purpose from Orient Paper Mills Ltd, India. The strength properties of the hand sheets as tested and the pulping conditions as reported appear respectively on Table IV and Table II of this report. A comparative study of the various properties of hand sheets, made from Semi-Keebra and sulfate bamboo pulp, at various freeness levels can be made by referring to Figures I, II, and III of this report.

EXPERIMENTAL DATA

Table I Cooking Conditions and Pulp Properties of Semi-Keebra Pulp.

Total Cooking Time	3½ hours
Time to reach 144°C.	½ hour
Cooking at 144°C.	3 hours
Pressure	56 psig.
Liquor Volume	12 liters
Liquor to Wood ratio	8 : 1
Chemical Concentration:	
NaOH	69.35 gpl., as chemical
Na ₂ SO ₃	16.9 gpl., as chemical
Na ₂ S	2.41 gpl., as chemical
Na ₂ CO ₃	7.11 gpl., as chemical
Per cent solids in Black Liquor	17.8 per cent
Pulp Yield	51.6 per cent
Kno ₄ Number	23.2
Brightness of unbleached pulp (IPC)	17.06

Table II. Cooking Condition and Pulp Properties of Sulfate Bamboo
Pulp (Bleachable cook)*

Total Cooking Time	2½ hours
Time to reach 130°C.	1 hour
Cooking at 130°C.	½ hour
Time to reach 165°C.	1½ hour
At 165°C.	½ hour
Chemical Concentration:	
NaOH	70 gpl., as chemical
Na ₂ S	29 gpl., as chemical
Na ₂ CO ₃	12 gpl., as chemical
Per Cent Solids in Black Liquor	10 - 15 per cent
Pulp Yield	45 per cent
KMnO ₄ Number	20.2
Brightness of unbleached Pulp	19.37

*Cooking Conditions and Chemical Concentration figures obtained
through courtesy of Orient Paper Mills Ltd., India.

Table III. Properties of hand sheets made from Semi-Keebra Bamboo Pulp.

Refining Time, Minutes	0	$\frac{1}{2}$	$1\frac{1}{2}$	$2\frac{1}{2}$
Freeness, C.S.F.	535	451	318	197
Density, gm/cc.	0.44	0.48	0.51	0.57
Burst Factor *	16.24	21.20	28.82	34.80
Tear Factor **	101.4	97.45	101.5	86.85
Breaking Length, meters	3.500	4.032	5.300	5.762
Folding Endurance				
(MIT, Double Folds)				
Average	8	13	50	82
Max.	10	18	63	116
Min.	7	10	35	52

* Burst Factor = $50 B/R$

** Tear Factor = $71.1e/R$

Where, B = Bursting strength in lbs / sq. inch;

R = Basis Weight in lbs per 500 sheets 25 x 40 inches.

e = Force in grams to tear a single sheet.

Table IV. Properties of Hand Sheets made from Sulfate Bamboo Pulp
(Bleachable grade)*

Refining Time, Minutes	1½	2½	3½	4½
Freeness, C.S.F.	590	491	306	226
Density GM/cc	0.43	0.41	0.50	0.51
Burst Factor **	17.07	23.38	36.9	35.5
Tear Factor ***	140.4	135.2	132.8	128.5
Breaking Length, meters	3,350	3,666	5,275	5,933
Folding Endurance (MIT, Double Folds)				
Average	8	17	106	177
Max.	9	20	145	232
Min.	7	13	74	122

* Pulp obtained through courtesy of Orient Paper Mills Ltd., India

** Burst Factor = $50 B/R$

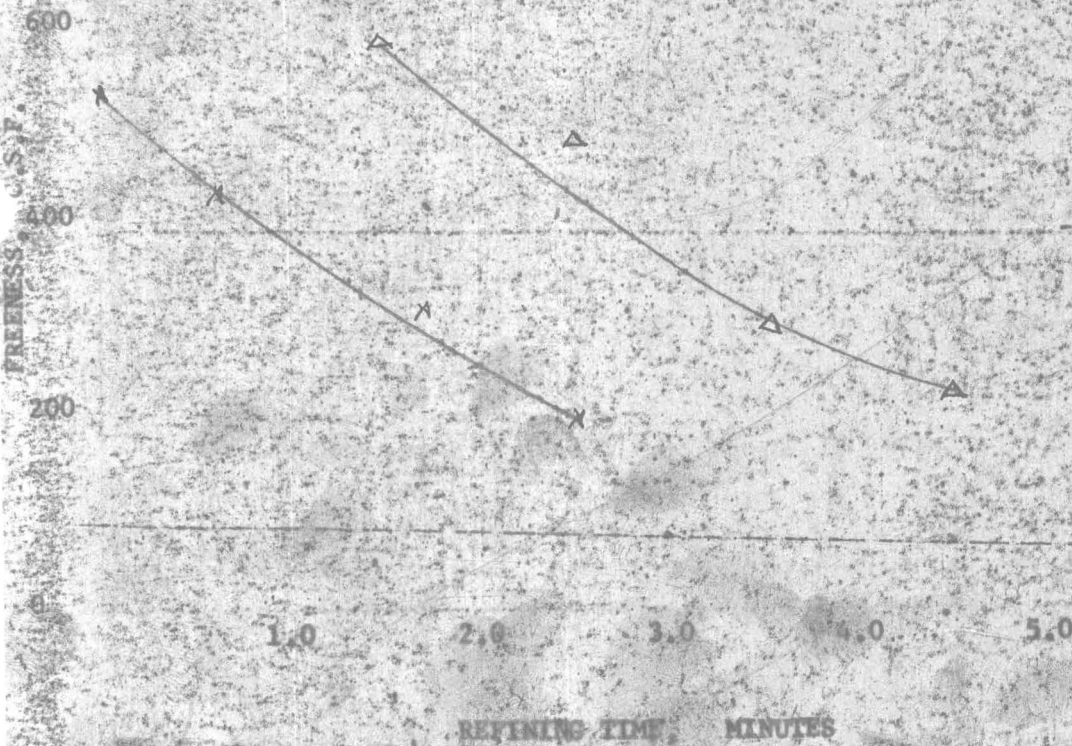
*** Tear Factor = $71.1e/R$

Where, B = Bursting strength in lbs/sq. inch;

R = Basis wt. in lbs per 500 sheets 25 x 40 inches.

e = Force in grams to tear a single sheet.

Fig. 1 Variation of Freeness of Semi-Keebra and Sulfate pulp with Refining Time.



LEGEND

x — x Semi-Keebra Pulp
△ — △ Sulfate Pulp

Fig. 2 Variation of Tear Factor, Folding Endurance, and Density of Semi-Keebra and Sulphate Bamboo Pulp with Freeness.

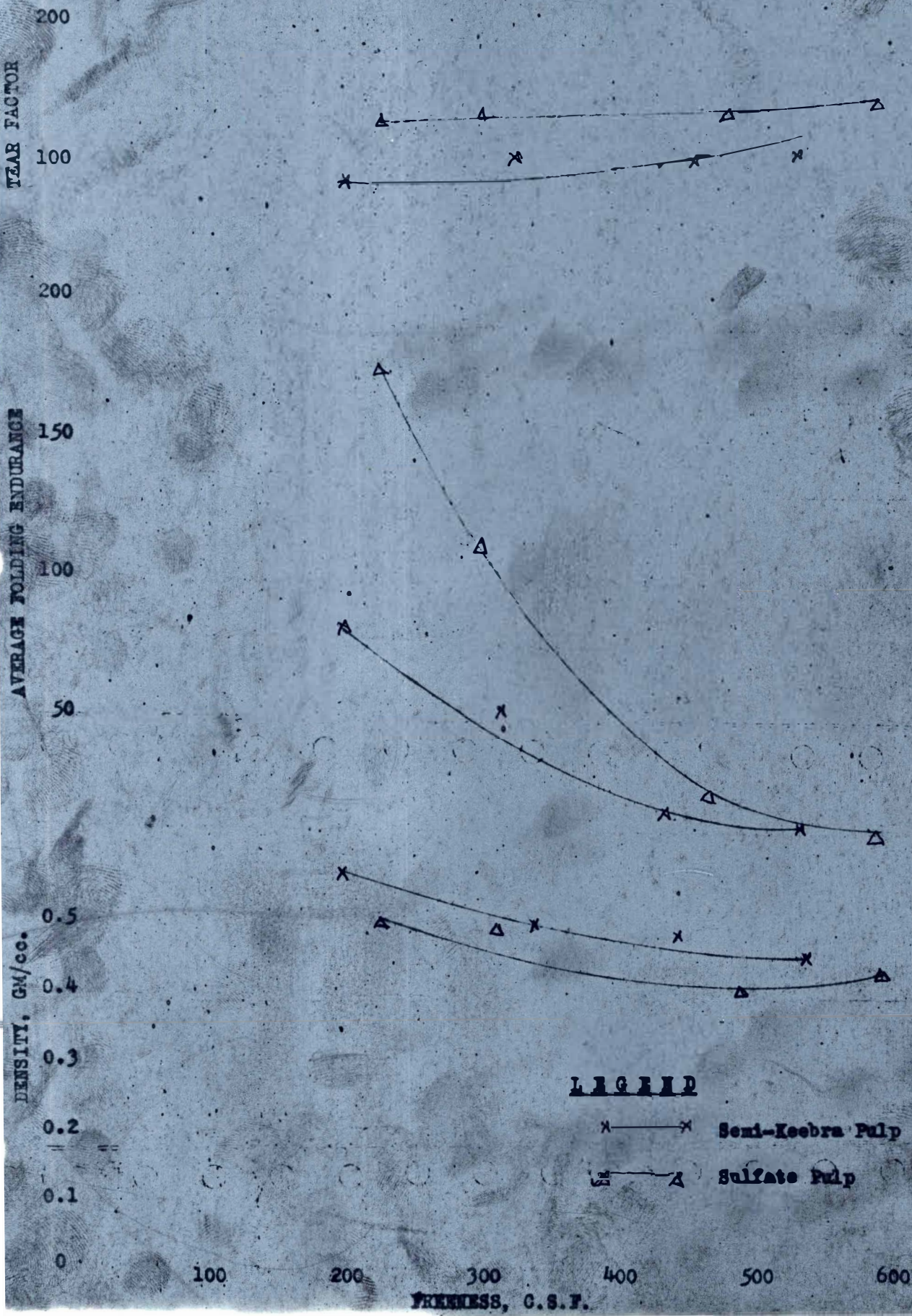
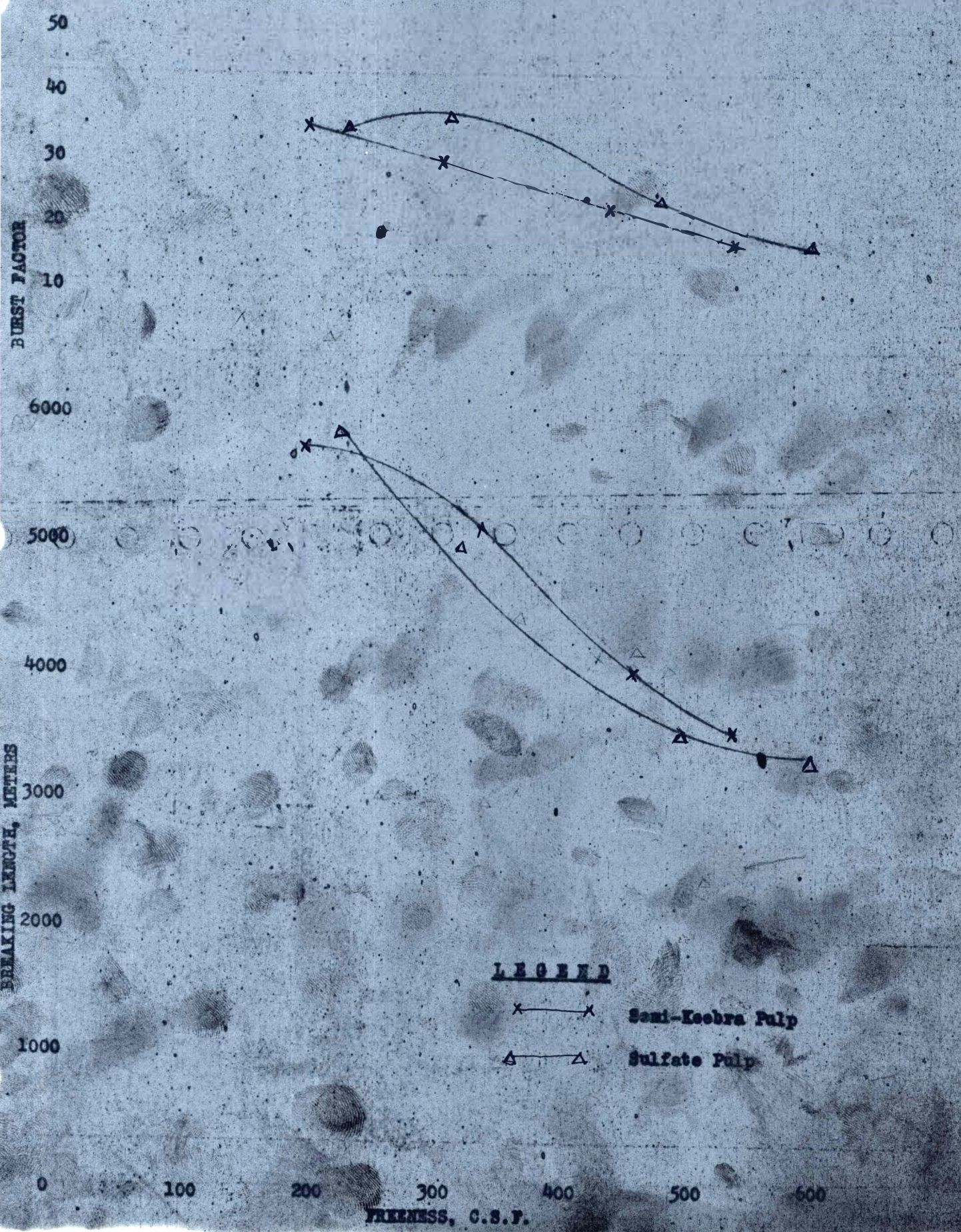


Fig. 3 Variation of Burst Factor, Breaking Length of Semi-Keebra and Sulphate Bamboo Pulp with Freeness.



SUMMARY AND CONCLUSIONS

This being a preliminary investigation, no attempt was made to determine the optimum values for cooking conditions and chemical concentrations. The following conclusions, however, can be drawn from the present work:

1. A higher pulp yield than most of the conventional process can be obtained by using Semi-Keebra process for Bamboo pulping. The high yield may be due to the protective action of Sulphite which has a very little action on the more easily resolvable celluloses and consequently their presence results in a high yield.

2. The Semi-Keebra bamboo pulp would require a considerable lower refining time to achieve the same Freeness value as sulfate bamboo pulp.

3. The breaking length and density of the paper made from Semi-Keebra pulp is found to be higher than that made from the sulphate bamboo pulp of bleachable grade. The other strength properties, however, are found to be lower than paper made from the sulfate pulp of bleachable grade.

4. The spent liquor of Semi-Keebra pulping process is found to have higher per cent solids than that reported for the sulfate pulping process.

5. The permanganate number of the Semi-Keebra pulp is found to be higher indicating higher bleach requirement than that for sulfate pulp. The initial brightness of Semi-Keebra pulp is also found to be lower than Sulfate pulp.

It is felt, however, that further investigation on optimum cooking conditions and liquor concentrations for Semi-Keebra process would show more promise for making papers having fairly good strength along with high pulp yield.

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