Alkali/Oxygen delignification and bleaching of soda bamboo pulp, bamboo + mixed hardwood pulp (70 : 30) and mixed hardwood pulp--Part-II

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SUMMARY

Bamboo chips. Bamboo + mixed hard woods chips (70 : 30) and Mixed hard woods chips were Soda cooked and refined in a sprout waldron disc refiner. Bamboo pulp (Kappa No. 45.4), Bamboo + Mixed hard woods pulp (Kappa No. 40.2) and mixed hard woods pulp (Kappa No. 38.4) were bleached under C/E/H and O/C/E/H sequences to get a pulp of 78-80% P. V. brightness. On alkali/oxygen treatment the brightness of Bamboo pulp improved moderately whereas the brightness of mixed hard woods pulp improved significantly. Shrinkage (%) at alkali/oxygen bleaching stage was low

The alkali extracted effluent of C/E/H sequence bleached pulp has BOD, COD > O/C/E/H sequence alkali extracted effluent. The total chlorine consumption for bleaching these three pulps under C/E/H Sequance > O/C/E/H bleaching Sequence. C/E/H Sequence bleached pulps take longer time in beating than O/C/E/H bleached pulps. The pulp viscosity and P. C. No. of C/E/H bleached pulps < O/C/E/H bleached pulps and reverse trend was observed for copper No This indicates that with Oxygen bleaching the degradation of the pulp is reduced.

Tensile Index of O/C/E/H Sequence bleached bamboo + mixed hard wood pulp and mixed hard wood pulp > C/E/H Sequence bleached pulps whereas for bamboo pulp it remained same in both the bleaching Sequences. Burst index of these three pulps under these two bleaching Sequences were nearly same but tear index for all these pulps bleached under O/C/E/H Sequence > C/E/H bleached pulps. Tensile Index and Tear index of Bamboo pulp, Bamboo + mixed hard woods pulp bleached under O/C/E/H Sequence > mixed hard woods pulp under O/C/E/H bleaching Sequence.

It has been knowm for many years, that lignin can be degraded and solubilized with Oxygen in alkaline media[1]. Recent development of Oxygen bleaching process has stimulated other researchers[2,3,4] to explore the possibility of extending this system to a Sulfur free pulping process. Such a system would consist of a high yield Soda stage followed by defibrization and subsequent Oxygen alkali treatment.

A world wide drive to reduce water pollution from pulp plants has resulted in mill scale application of Oxygen/alkali delignification in Sweden, France, U.S.A., Japan and South Africa by partially replacing chlorination stage by Oxygen/alkali stage, BOD, COD and colour in effluent water from these mill is reduced considerably[5]. The waste water from washing of Oxygen pulp is recirculated and used in brown stock washing. Thus most of the organic matter dissolved during Oxygen/alkali stage goes into the mill recovery. In commercial operation Magnesium salts as degradation inhibitor are added in alkali/Oxygen bleaching.

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Studies have shown that there can be reduction in total cost of bleach chemicals if a Sequence is employed which contains an Oxygen stage[6].

In Orient Paper Mills Amlai we are using 25 to 30% mixed hard woods and 70-75% bamboo for producing 200 tonnes of different grades of papers. *Shorea Robusta* (Sal), *Boswellia Serrata* (Salai) and *Terminalia Tomentosa* (Saza) are the main hard wood species used in our mill alongwith mixture of hard woods. Oxygen bleaching studies were conducted on Bamboo (100%), Bamboo mixed hard woods (70:30) and Mixed hard woods (100%) Soda pulps to see how these raw materials respond to Oxygen bleaching and also to explore its possibilities for future application.

EXPERIMENTAL AND RESULTS :

Soda cooking of Bamboo chips, Bamboo+ mixed hard wood chips (70:30) and hard wood chips (−22+10 m m. size) were carried out using 21.0%, 23 0% and 25 0% alkali (as NaOH) respectively keeping bath ratio at 1:4. Cooking conditions and results are recorded in Table-1. The resulting pulps were passed through Sprout waldron disc refiner at a clearance of 254 microns using refiner plate D A-501. The refined pulps were analysed for Kappa No. and yield (%). Black liquor samples were analysed for their characteristics and results are tabulated in Table-1.

Unbleached Bamboo pulp, Bamboo + mixed hard wood pulp and mixed hard wood pulp were bleached under C/E/H Sequence to achieve 78-80% P.V. pulp brightness. BOD, COD of alkali extracted effluent was analysed as per standard methods. Bleaching conditions and results are recorded in table-2. Bleached pulps were analysed for P C. No. pulp brightness, Copper No. viscosity and pulp shrinkage (%). These bleached pulps were beaten at 25, 35, 45 and 55 °SR freeness and physical strength properties were determined. Bamboo pulp. Bamboo + mixed hard wood pulp and mixed hard wood pulp were delignified with alkali Oxygen using 4% NaOH and 0.57, $MgCO_3$ maintaining 7% consistency. Oxygen was injected at 120°C (9.0 kg/Cm^2 pressure) for 90 minutes in an autoclave through a side bottom flange. The resulting pulp was analysed for shrinkage (%), brightness (%), Kappa No. and black liquor was also analysed (table-3).

Alkali/Oxygen bleached pulps of Bamboo, Bamboo+mixed hard woods and mixed hard woods were further bleached under C/E/H Sequence. Caustic extracted effluent was analysed for BOD and COD. Bleaching conditions and results recorded in table-4. Bleached pulps were analysed for Copper No. viscosity brightness, P. C. No. and pulp shrinkage. Results are recorded in table-4. These O/C/E/H bleached pulps were beaten at 25, 35. 45 and 55°SR freeness. Standard sheets were made and tested as per Tappi standards.

Physical strength properties, tensile Index, Burst Index and Tear Index of O/C/E/H Sequence bleached pulp against different °SR freeness for Bamboo pulp are represented in Fig 1-3, Bamboo+ Mixed hard wood pulp (Fig. 3-6) and for mixed hard wood pulp (Fig. 6-9). Beating time for Bamboo pulp, Bamboo + Mixed hard wood pulp and Mixed hard wood pulp (bleached under C/E/H and O/C/E H Sequence) against different °SR freeness is represented in Fig. 10,11 and 12 respectively.

DISCUSSIONS :

The pulp yields of Bamboo. Bamboo + mixed hard woods and mixed hard woods were 47.78%, 45 60% and 45.44% respectively and the Kappa No of these pulps were 45.4, 40.2 and 38.4 respectively when cooked with 21.0%, 23% and 25% alkali. Cooking conditions and black liquor characteristics are given in table-1.

Soda Bamboo pulp (Kappa No. 45.4), Bamboo + mixed hard wood pulp (Kappa No. 40.17) and Mixed hard wood pulp (Kappa No. 38.44) were bleached under C/E H Sequence using different chlorine and hypo chlorite percentage in the first and third stage bleaching respectively. The total chlorine consumption increases with increase in Kappa No. as expected. The BOD 5 and COD of caustic extracted effluent of Bamboo pulp, Bamboo+mixed hard wood pulp and mixed hard wood pulp was high. These bleached pulps under C/E/H Sequence have high P.C. No. Copper No. and low pulp viscosity (Table-2).

Bamboo, Bamboo+mixed hard woods and mixed hard woods pulps were alkali/Oxygen delignified using 4% alkali. The resulting pulps have Kappa No 23.61, 15.54 and 12 53 respectively. The brightness of alkali/Oxygen delignified pulps improved with increase in mixed hard wood percentage Bamboo pulp (28 4% PV), Bamboo+ mixed hard wood (34.0% P.V) and Mixed hard woods (40 0% P.V). Shrinkage in alkali/Oxygen delignified pulps was low. Bleaching conditions and results are recorded in table-3.

Alkali/Oxygen delignified Bamboo pulp, Bamboo+ Mixed hard woods pulp and mixed hard woods pulps were bleached under C/E/H Sequence

TABLE—1. CAUSTIC COOKING OF BAMBOO, BAMBOO+MIX HARD WOODS (70 : 30) AND MIXED HARD WOODS.

S. No.	Particulars	Expt. No. 1 Bomboo (100%)	Expt. No. 2* Bamboo+Mixed hardwoods (70 : 30)	Expt. No. 3* Mixed hardwoods (100%)
1.	Weight of chips taken in Kgs (OD basis)	2.5	2.5	2.5
2.	Caustic applied (%) as NaOH	21.0	23.0	25.0
3.	Bath ratio	1:4	1:4	1:4
4.	**Cooking Cycle**			
	(i) Upto 135°C (mts)	60	60	60
	(ii) At 135°C (mts)	30	30	30
	(iii) From 135-162°C (mts)	60	60	60
	(iv) At 162°C (mts)	120	120	120
5.	Yield (%)	47.78	45.60	45.4
6.	Kappa No of pulp	45.4	40.2	38.4
7.	**Black liquor characteristics**			
	(i) °TW at 60°C	16.5	15.5	17.0
	(ii) R.A.A. (g/L)	17.05	16.5	17.05
	(iii) Inorganics (%)	28.73	29.42	22.20
	(iv) Organic (%)	71.27	70 58	77.80
	(v) Total solids (%)	21.37	20.32	25.13

*Unbleached pulp of Expt. No. 1, No. 2, and No. 3 were passed through Sprout waldron disc refiner at a clearance of 25 microns using refiner plate D—2A—501.

TABLE—2. BLEACHING OF BAMBOO, BAMBOO MIXED HARDWOOD AND MIXED HARDWOOD PULP UNDER C/E/H SEQUENCE.

S. No.	Particulars	Expt. No. 1 Bamboo Pulp	Expt. No. 2 Bamboo+Mixed hardwood pulp	Expt. No. 3 Mixed hardwood pulp
1.	**Chlorination Stage**			
	(i) Chlorine applied (%)	9.0	9.4	9.0
	(ii) Chlorine consumed (%)	9.0	8.88	8.80
	(iii) End pH	1.8	1.8	1.8
2.	**Alkali Extraction Stage**			
	(i) Alkali added (%)	2.5	2.5	2.5
	(ii) BOD_5 (mg/L)	440	460	440
	(iii) COD (mg/L)	1900	1933	1896
	(iv) End pH	10.8	10.95	10.95
3.	**Hypochlorite Stage**			
	(i) Hypochlorite added (%) as available chlorine	6.5	5.0	5.0
	(ii) Hypochlorite consumed (%)	6.0	3.15	2.67
	(iii) Pulp brightness (%) P.V.	77.0	81.0	77.0
	(iv) End pH	7.7	8.1	7.8
	(v) Total pulp loss (%)	11.25	11.4	10.23
	(vi) P.C. No.	8.29	6.57	6.67
	(vii) Viscosity of pulp (0.5% CED) cps	5.63	5.58	5.67
	(viii) Copper No.	1.72	1.44	1.43
4.	**Final Results**			
	(i) Total chlorine added (%)	15.5	14.4	14.0
	(ii) Total chlorine consumed (%)	15.0	12.03	11.47
	(iii) Total pulp loss (%)	11.25	11.4	10.23

TABLE—3. OXYGEN BLEACHING OF BAMBOO PULP (KAPPA NO. 45.4), BAMBOO MIXED HARDWOOD PULP (KAPPA NO. 40.17) AND MIXED HARDWOOD PULP (KAPPA NO. 38.44).

S. No.	Particulars	Expt. No. 1 Bamboo Pulp	Expt. No. 2 Bamboo+Mixed hardwood puld (70:30)	Expt. No. 3 Mixed Hardwood pulp
1.	Pulp taken in Kgs	800.0	1.00	1.00
2.	Consistency of pulp (%)	7.0	7.0	7.0
3.	Magnesium carbonate added (%)	0.5	0.5	0.5
4.	Alkali added (%)	4.0	4.0	4.0
5.	Oxygen injected at 120°C (Kg/cm^2)	9 0	9.0	9.0
6.	Oxygen injected (mts)	90	90	90
7.	Kappa No.	23.61	15.54	12.53
8.	Brightness of pulp (%PV)	28.5	34	40
9.	R.A.A. of wash liquor (g/L)	2.32	2.32	3.1
10.	Original brightness of pulp (%PV)	23.0	25	26
11.	Shrinkage of pulp (%)	2.32	3.36	3.35

TABLE—4. BLEACHING OF BAMBOO-MIXED HARDWOOD OXYGEN PULP AND MIXED HARDWOOD OXYGEN PULP UNDER C/E/H SEQUENCE.

S.No.	Particulars	Expt. No. 1 Bampoo pulp	Expt. No. 2 Bamboo+Mixed hardwood oxygen pulp	Expt. No. 3 Mixed hardwood pulp
1.	**Chlorination Stage**			
(i)	Chlorine applied (%)	5.5	3.5	3 0
(ii)	Chlorine consumed (%)	4.89	3.35	2.72
(iii)	End pH	2.0	1 8	I.8
2,	**Alkali Extraction Stage**			
(i)	Alkali added (%)	2.5	2.5	2.5
(ii)	BOD 5 (mg/L)	160	180	140
(iii)	COD (mg/L)	940	857	857
(iv)	End pH	11.3	11.5	11.5
3.	**Hypochlorite Stage**			
(i)	Hypochlorite added (%) as available chlorine	3.0	2.0	1.5
(ii)	Hypochlorite consumed (%)	2.08	1.43	1.36
(iii)	Pulp brightness (% PV)	80.0	78	81
(iv)	End pH	7.8	7.7	7.9
(v)	Total pulp loss (%)	9.5	8.88	7.63
(vi)	P.C. No.	1.87	1.675	1.207
(vii)	Viscosity of pulp (0.5% CED) cps	6.35	6.26	6.21
(viii)	Copper no.	1.372	1.008	1.145
4.	**Final Results**			
(i)	Total chlorine added (%)	8.50	5.50	4 5
(ii)	Total chlorine consumed (%)	6.97	4.78	4.08
(iii)	Total pulp loss (%) (Oxygen+C/E/H sequence)	12 25	12.10	10.98

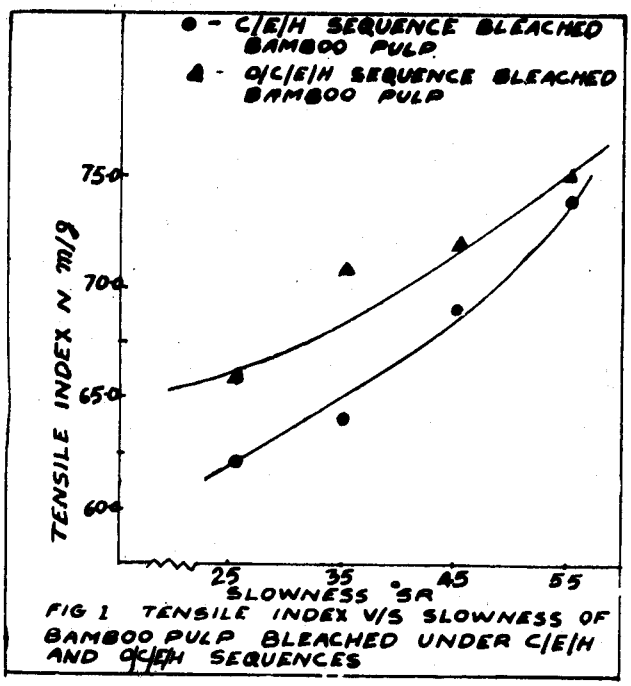

FIG 1 TENSILE INDEX V/S SLOWNESS OF BAMBOO PULP BLEACHED UNDER C/E/H AND O/C/E/H SEQUENCES

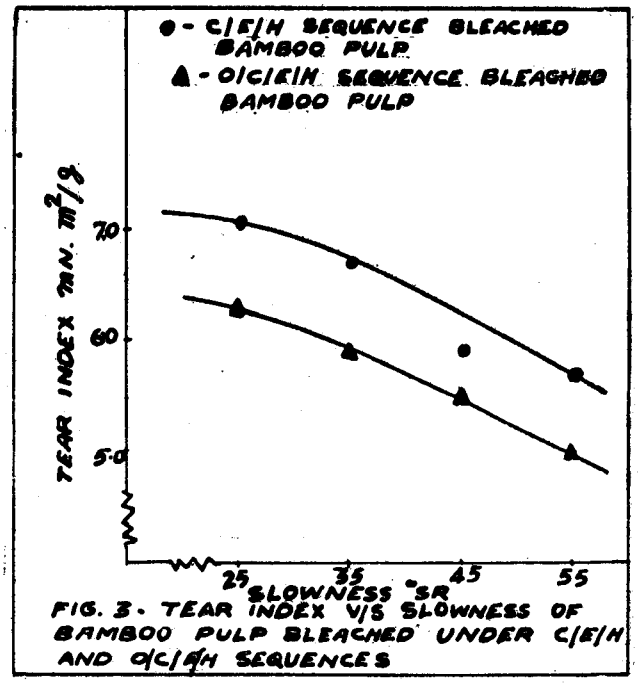

FIG. 3 - TEAR INDEX V/S SLOWNESS OF BAMBOO PULP BLEACHED UNDER C/E/H AND O/C/E/H SEQUENCES

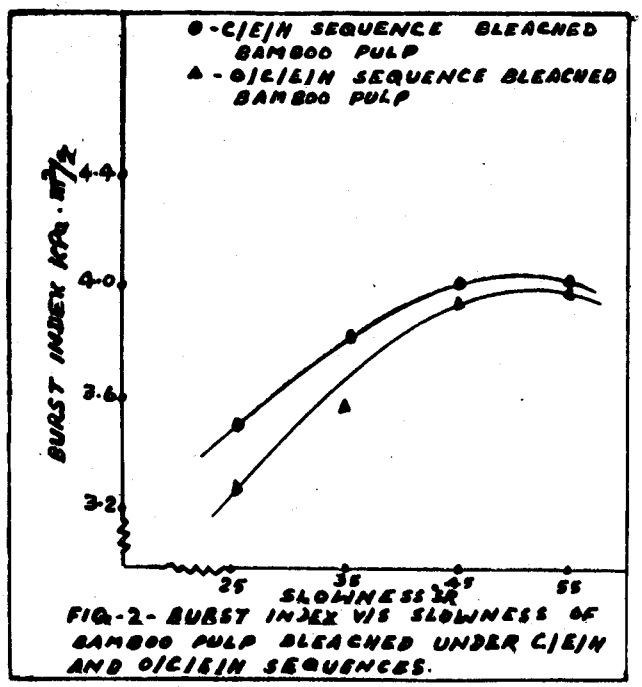

FIG-2- BURST INDEX V/S SLOWNESS OF BAMBOO PULP BLEACHED UNDER C/E/H AND O/C/E/H SEQUENCES.

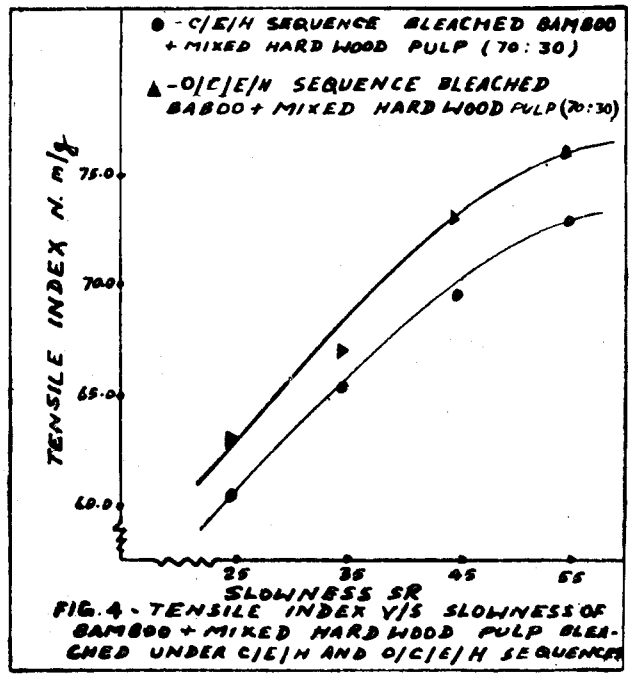

FIG. 4 - TENSILE INDEX V/S SLOWNESS OF BAMBOO + MIXED HARD WOOD PULP BLEACHED UNDER C/E/H AND O/C/E/H SEQUENCES

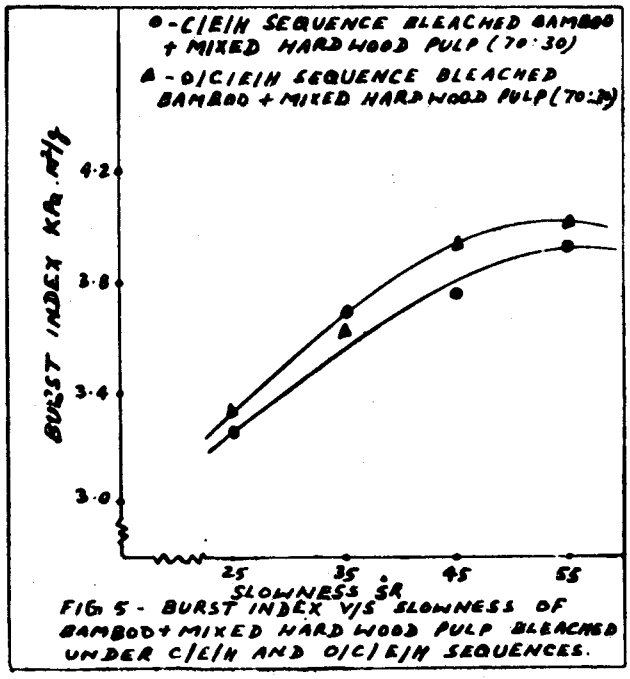

FIG. 5 - BURST INDEX V/S SLOWNESS OF BAMBOO + MIXED HARD WOOD PULP BLEACHED UNDER C/E/H AND O/C/E/H SEQUENCES.

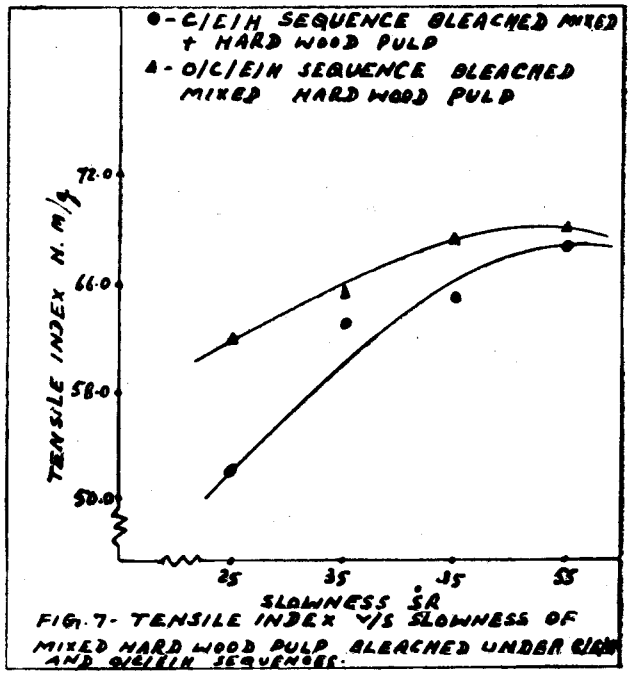

FIG. 7 - TENSILE INDEX V/S SLOWNESS OF MIXED HARD WOOD PULP BLEACHED UNDER C/E/H AND O/C/E/H SEQUENCES.

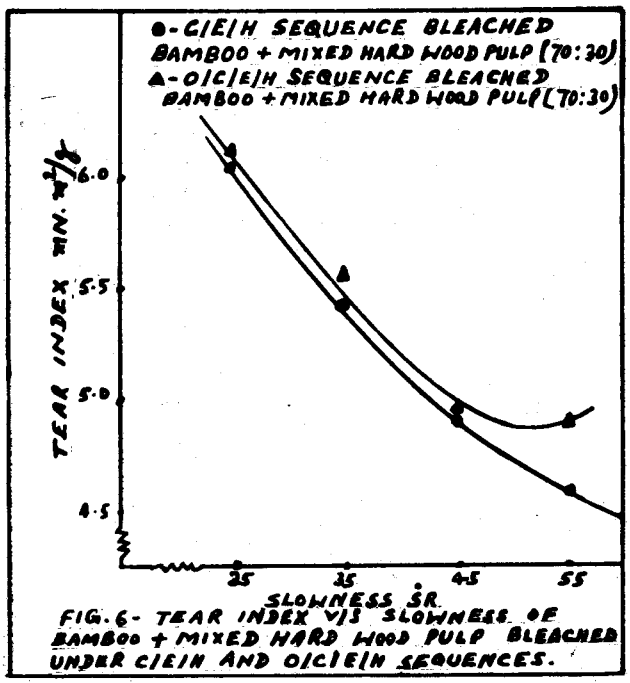

FIG. 6 - TEAR INDEX V/S SLOWNESS OF BAMBOO + MIXED HARD WOOD PULP BLEACHED UNDER C/E/H AND O/C/E/H SEQUENCES.

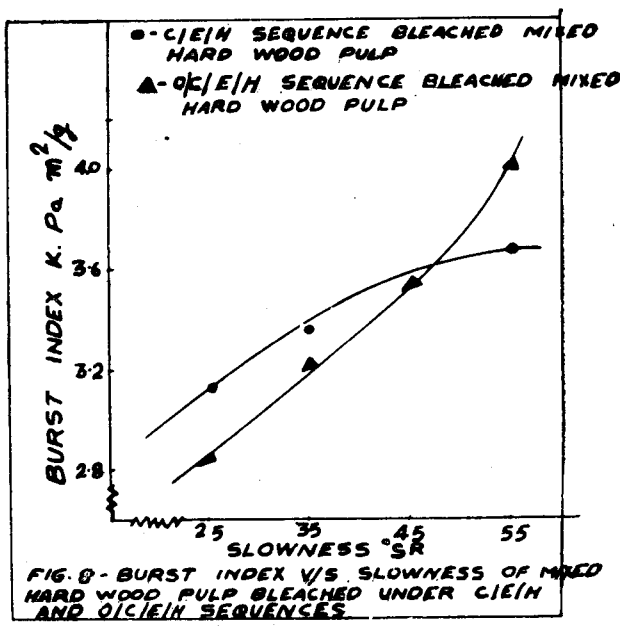

FIG. 8 - BURST INDEX V/S SLOWNESS OF MIXED HARD WOOD PULP BLEACHED UNDER C/E/H AND O/C/E/H SEQUENCES.

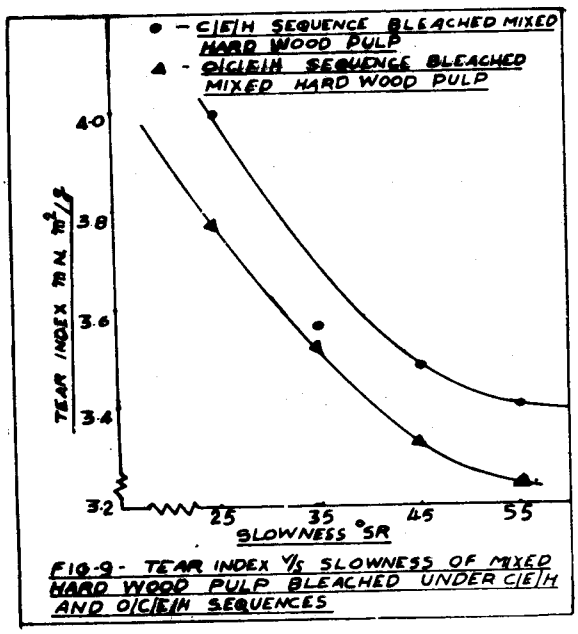

FIG-9 - TEAR INDEX V/S SLOWNESS OF MIXED HARD WOOD PULP BLEACHED UNDER C/E/H AND O/C/E/H SEQUENCES

using chlorine in the first stage and hypo chlorite in the third stage bleaching. The alkali extraction stage effluent of these pulps have low BOD_5 and COD as compared to alkali extracted effluent of C/E/H Sequence. The alkali extraction stage effluent of O/C/E/H Sequence bleached Bamboo pulp, Bamboo mixed hard wood pulp and Mixed hard woods pulps have high BOD reduction (63.63%, 60.80% and 68.10% respectively) and COD reduction (50.50%, 55.60% and 54.80% respectively) as compared to C/E/H Sequence of these pulps.

O/C/E/H Sequence bleached Bamboo pulp, Bamboo+mixed hard wood pulp and mixed hard wood pulp have lower P. C. No. lower Copper No. and higher viscosity than their counter part in C/E/H Sequence bleached pulps. Total chlorine consumption for Bamboo pulp, Bamboo+Mixed hard wood pulp and Mixed hard wood pulps under O/C/E/H Sequence was low as compared to C/E/H Sequence (Table 2 & 4) The total pulp shrinkage was higher in O/C/E/H Sequence bleached pulps than C/E/H Sequence bleached pulp. The pulp shrinkage of mixed hard wood pulp in O/C/E/H Sequence was lower than Bamboo pulp in O/C/E/H Sequence.

The effect of beating of Bamboo bleached pulp, Bamboo+mixed hard woods bleached pulp (under C/E/H and O/C/E/H Sequences at different °SR freeness against tensile Index, Burst Index and Tear Index are depicted in Fig. 1 to 9. Tensile Index and Burst Index of Bamboo bleached pulps of C/E/H and O/C/E/H Sequences are nearly same but tear index of O/C/E/H Sequence < bamboo bleached pulp O/C/E/H Sequence (Fig 1,2 & 3). Tensile Index of O/C/E/H Sequence bleached bamboo+mixed hard woods pulp> C/E/H Sequence bleached bamboo+mixed hard woods pulp but burst Index in both the bleaching Sequences were nearly same (Fig. 4, 5 & 6). Tensile Index and Burst Index of O/C/E/H Sequence bleached mixed hard woods pulp > C/E/H Sequence bleached mixed hard woods pulp but tear index of O/C/E/H Sequence < C/E/H Sequence bleached pulp Fig. 7, 8 & 9). Beating time of Bamboo pulp, Bamboo+mixed hard woods pulp and mixed hard woods pulp (bleached under C/E/H and O/C/E/H Sequences) against freeness °SR are dipected in Fig. 10, 11 & 12 The C/E/H Sequence bleached pulp takes longer time in beating than O/C/E/H Sequence bleached pulps.

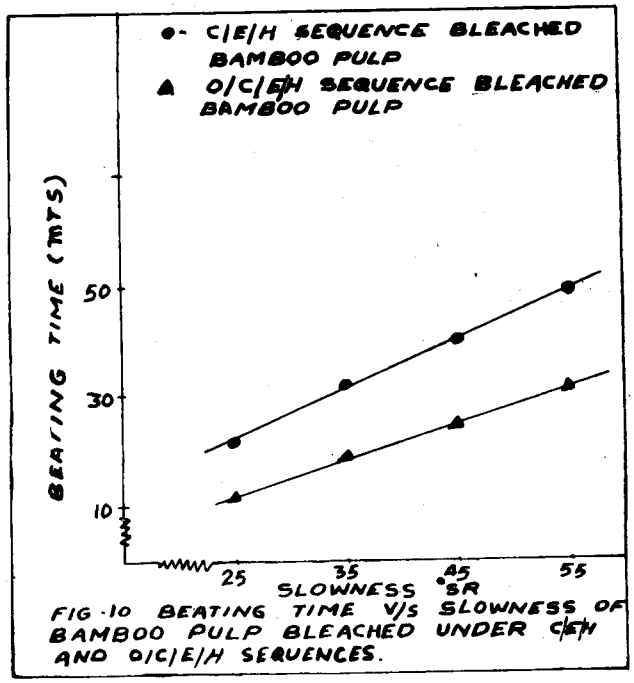

FIG-10 BEATING TIME V/S SLOWNESS OF BAMBOO PULP BLEACHED UNDER C/E/H AND O/C/E/H SEQUENCES.

Tensile Index and Tear Index of Bamboo pulp and Bamboo + mixed hard woods pulp (bleached under O/C/E/H Sequence> mixed hard woods pulp (bleached under O/C/E/H Sequence. Burst index of Bamboo pulp, Bamboo+mixed hard woods pulp and mixed hard woods pulp (bleached) under O/C/E/H Scquence were nearly same.

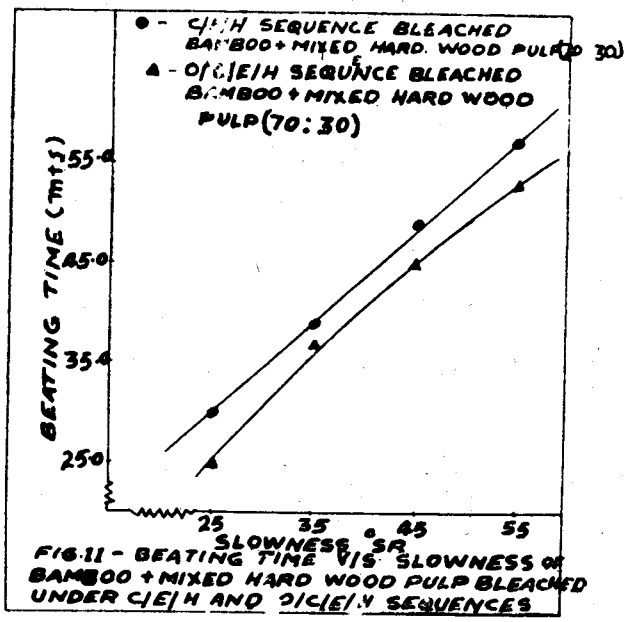

FIG 11 - BEATING TIME V/S SLOWNESS OF BAMBOO + MIXED HARD WOOD PULP BLEACHED UNDER C/E/H AND O/C/E/H SEQUENCES

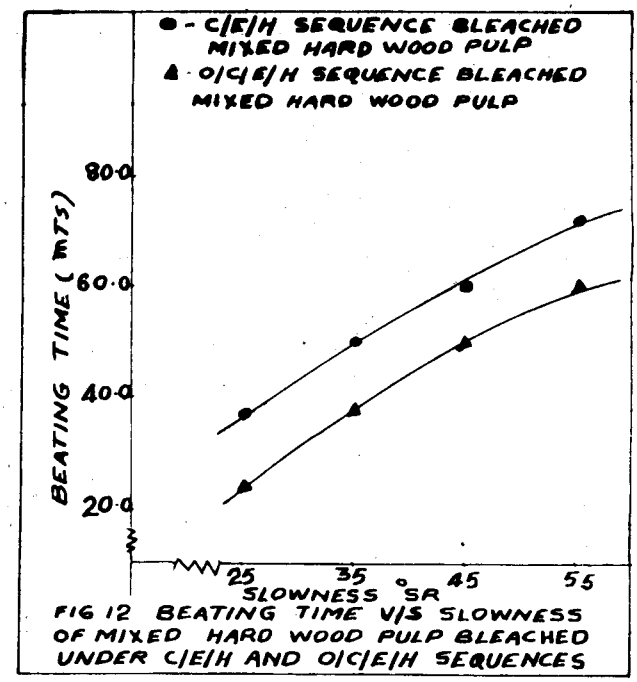

FIG 12 BEATING TIME V/S SLOWNESS OF MIXED HARD WOOD PULP BLEACHED UNDER C/E/H AND O/C/E/H SEQUENCES

CONCLUSION :

The use of Oxygen as a stage of bleaching not only reduces BOD & COD of the effluent but also improve the quality of pulp as reflected by the higher viscosity and lower P.C. No. The beating time is also reduced.

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REFERENCES :

1. Braun, F.E., "Chemistry of lignin", Academic Press, New York pp 52, pp 546-549.
2. Kleppe, P J., Chang, H-m and Eckert, R.C., paper presented at the third Canadian wood chemistry Symposium, Vancouver, B.C., June 1970.
3. Palenius, T. and Hissvirta, L, *Pulp Paper Mag. Can* 71 (21) : 63 (1970).
4. Worster, H.E., Pudek, M F. and Harison, R.E. *Pulp Paper Mag. Can* 72 (12) : 69 (1971).
5. Kleppe, P.J., Backlund, A, and Schildt Y. *Tappi* 59 (11), (1976).
6. Jamieson, A. and Smedman, L. *Tappi* 56 (6) : 107 (1973).
7. Rowlandson, G., *Tappi* 54 (6) : 962 (1971).
8. Evans, J.C.W., *Paper. Trade. J* 157 (42) : (Oct. 15, 1973).
9. "Standard Methods of Examination of water and waste water" p 415 421, 12th Edn 1965, American Public Health Association, Inc, Broadway, N.Y. 10019.