The effect of hydrogen peroxide in the alkaline extraction stage during bleaching of kraft and soda pulp.

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INTRODUCTION—

In order to obtain a pulp of very high brightness and brightness stability, the lignin must be removed. This can not be done in digester because if the cooking is continued too long, the carbodydrates in the pulp break down and dissolve. The final delignification is, therefore, performed in the bleaching process with certain oxidising agents and the conditions of the reactions are adjusted so as to protect the carbohydrates as far as possible[1].

In India kraft and soda pulps are generally bleached by usage of following conventional sequences:

CHLORINATION (C)–EXTRACTION (E)–HYPOCHLORITE (H) i.e. CEH, or

CHLORINATION (C)–EXTRACTION (E)–HYPOCHLORITE (H)–HYPOCLORITE (H) i.e. CEHH,

or CHLORINATION (C)–HYPOCHLORITE (H)–HYPOCHLORITE (H) i.e. CHH.

The philosophy, behind this multistage bleaching process is, however, being affected by pollution abatement and energy conservation measures. This means bleaching must be carried out with new processing chemicals such as oxygen, ozone and hydrogen peroxide or by better ways of using chlorine, hypochlorite and chlorine dioxide must be implemented. If there are two bleaching processes producing the same end product and with the same operating cost, the one which yielding less chlorine containing effluent is likely to be preferred[1]. One has to see this from availability of suitable hypochlorite replacing chemicals and effective usages of it during bleaching process in the context of developing countries.

The usage of hydrogen peroxide at first extraction stage of kraft pulp has been reported in the literature[2-5]. Laboratory bleaching work on usage of hydrogen peroxide has been reported recently with respect to Indian fibrous raw material in Indian context[6-7].

During the chlorination of pulp, chlorolignins are formed. These chlorolignins are extracted in alkaline extraction stage. In alkaline extraction stage when compounds are dissolved and extracted, the colour of the pulp become darker. The objective of this stage is to make the pulp ready for subsequent oxidative bleaching, generally using hypochlorite. However, this particular period of extraction is not actually utilised for bleaching and is therefore, considered as a dead period.

By introducing hydrogen peroxide in an alkaline extraction stage, this dead period can be utilised to improve the brightness of the pulp. Due to this alkaline oxidative extraction of chlorinated pulp, kappa number or permanganate number of the pulp reduces and it becomes easier to bleach further with hypochlorite either in single stage or in two stages. There is also a possibility of eliminating second stage hypochlorite thus producing fully bleached brighter pulp. Sometimes, the pulp mill faces the problem of variation in quality of raw material or problem of temporary irregularities in cooking operations. However, under such flexible operations of the mill, by introducing hydrogen peroxide at extraction stages, the brightness level can be adjusted.

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In short, this chemical pulp bleaching consists of two basic steps. The first step is delignification which is realised in the first two stages. The seeond step is brightening of delignified pulp. Many different chemical reactions are utilised for the delignification and bleaching operations. The behaviour of hydrogen peroxide in kraft pulp bleaching is illustrated in FIG-1 The formation of molecular oxygen formed by catalysis of hydrogen peroxide may contribute to delignification of kraft or Soda pulp[1].

EXPERIMENTAL

In our Research Centre, during last five to six years extensive laboratory work has been carried out on bleaching of kraft and soda pulp. The conventional bleaching sequences like CEH, CHH, CEHH and CEHD were modified effectively to CE_pH, CE_pHH, and CE_pHD.

The pulp samples were chlorinated in the laboratory and washed thoroughly. These chlorinated samples were further divided into two parts one for normal EH, EHH, HH or EHD bleaching sequences and other for EpH, EpHH or EpHD bleaching squences. During initial laboratory work, at alkali extraction stage (FIG. 2-4) sodium silicate or proprietory organic stabilizer (ALCOBLEACH—HP, a product of S.M. Dye-Chem, Bombay) was used as stabilizer along with hydrogen peroxide. However, with our further experiences, we have discarded its use (FIG.5-8) as during extraction as the idea is to carry out delignification[3] and not bleaching of pulp. The usage of 0.3 to 0.5% H_2O_2 (100%) on O.D. pulp was restricted during delignification. The efforts were also made to reduce the usage of caustic soda during peroxide reinforced extraction as well as usage of hypochlorite in subsequent bleaching stages.

The bleached pulp samples were tested for brightness on Technibrite TB-1C. In some of the cases detail analysis on strength properties as well as determination of colour of extraction wash liquor was also carried out. The laboratory experiments were carried out on bamboo, and eucalyptus kraft as well as on bagasse soda pulp.

RESULTS AND DISCUSSIONS

During the experiments (FIG. 2, 3 and 4) efforts were made to reduce caustic soda. In case of mixed hardwood pulp (FIG. 4) efforts were also made to reduce chlorine dioxide. The results show that in all these cases brightness of final bleached pulp is on higher side. It shows that hydrogen peroxide is even effective during cold extraction, though with higher temperature, results would be still better.

FIG. 3 is a typical case which clearly shows the effect of hydrogen peroxide at extraction. With 0.3% H_2O_2 (100%) on O.D. pulp, the brightness level of alkali extracted pulp was elevated by 7.6° ISO even after reduction in caustic level, Thus pulp is made easy for subsequent bleaching.

By usage of 0.3% H_2O_2 (100%) on O.D. pulp, brightness of alkali extracted bagasse pulp was elevated by 14.7 °ISO (FIG. 5). This was achieved even after using 33% less caustic soda. It was further observed that 2°ISO higher brightness for final bleached pulp was achieved even after reducing hypochlorite dosages to 50%. The properties of the pulp were also comparable (TABLE NO. 1) with that of control CEH bleaching sequence. The colour of extraction wash liquor is also reduced by 40%.

FIG. 6 shows that it is possible to modify GHH bleaching sequence economically to CEpH sequence without disturbing the bleaching conditions. The pulp under study was mixed hardwood kraft pulp. In the laboratory entire hypo from 1st stage and 25% hypo of second stage, i.e. altogether 5% hypo was replaced by 0.5% H_2O_2 (100%) on O.D. pulp. The final brightness of CEpH bleached pulp is higher than that of CHH bleached pulp.

FIG. 7A and 7B show the usage of hydrogen peroxide at alkali extraction stage of bamboo kraft pulp. These particular bleaching experiments show that a optimum dosage of hypochlorite can be selected in order to get maximum efficiency in terms of final bright ness gain.

Now a days in Indian pulp industry, a blend of hardwood and bamboo is used for pulping due to scarcity of bamboo. A typical case is shown in FIG 8. and TABLE-2. In this case by usage of 0.3% H_2O_2 (100%) on O.D. pulp, a final brightness of bamboo mixed hardwood kraft pulp was elevated by 3.0 °ISO without sacrificing the strength properties.

(1) Large size integrated mills based on Bamboo and Hardwood : Suspended solids 100-150, BOD 40-60 & COD 150-200.

(2) Newsprint Mills : — Suspended solids 80-100, BOD 50-60, COD 130-140.

(3) Small Paper Mills, based on agricultural residues:- Suspended solids 100-250, BOD 100-270, COD 400-1000.

(4) Small Paper Mills, based on waste paper :—Suspended solids 50-90, BOD 20-40, COD 50-100.

Pollution due to small paper mills based on agricultural residue is more serious as recovery of chemicals for these Mills is still a big problem, and most of the Mills are not having a recovery system. Normally a 30 TPD small paper mill, based on agricultural residue, will have a BOD load equivalent to 100 TPD large integrated mill, as the integrated mills are having chemical recovery system.

Increased use of paper, paper board and newsprint has resulted in increased waste paper generation from various sources. Recovery of waste paper and waste paper products is hardly 20-25% in our country as compared to 40-50% in advanced countries. This necessiates urgent need to have better and efficient utilisation of waste paper, which will help in solving many inherent problems arising from waste paper contamination.

Various environmental protection measures include water pollution control, air pollution control, solid waste treatment and noise pollution control.

WATER POLLUTION

Paper Industry probably consumes a large volume of water per unit of consumer goods produced. The entire process of Paper making, right from the washing of fibrous materials to the drying of sheet of paper, depends upon water in some form or other and since the end product is practically free from water (5/7% max. moisture is there in finished paper) all the water consumed in pulp and paper making process, reappears as waste water. In our country, about 200-350m^3 of fresh water is consumed per ton of paper produced, resulting in about 200-250m^3 effluent per ton of paper.

The modern trend in paper and pulp Industries is towards "Zero Effluent Discharge" Compared to this, effluent discharge in our country in paper Industry is abnormally high. This not only pollutes the water resources to a high degree but also effects the economics of the paper mills and poses a problem for the treatment of the large quantities of effluent.

As most of our water resources are either in the form of rivers or streams, they get polluted by such discharge making it unfit for human consumption.

In the developed countries of the World, such as USA, the water pollution rate from Pulp and Paper Mills is of the order of 7 Million Gallons per day, where as the same in India is approximately 200 Million Gallons per day, when our total paper production in the year 1988 is only 1.8 Million Tons as against 60 Million Tons of paper production in USA.

A good water management is vital for economy of a mill. Cost of effluent treatment itself is about Rs. 80/- per ton or Rs. 1/- per kg of BOD removed in a 100 TPD Mill. Reduction in water consumption by about 10-20m^3 per ton of Paper will result in saving of about Rs. 2.5— 4.5 lakh per annum. By recycling maximum amount of back water, introduction of control measures and technological changes, it is possible to minimise water consumption to a very low level. This of course requires personal training and properly planned development programme, and arousing the awareness of pollution problems amongst all the working personal in the mill. Some of the in-plant control measures for controlling pollution are (a) Max. recycling of back water at various stages. (b) Recycling of condensate by strict monitoring (c) Plugging all the possible leakages point from the pump glands and valves, pipeline etc. (d) Monitoring and Metering the water consumption at different stages. (e) Use of back water in waste Paper and pulp system and (f) Good housekeeping.

Some of the technological changes which should be incorporated to cut down pollution are :

(a) Oxygen bleaching/chlorine dioxide bleaching.

(b) Use of Anthraquinone and lower sulphur high-yield pulping process.

(c) Modification of bleaching sequence to eliminate caustic extraction stage. and

(d) Total recycle concept.

The effluent should be treated in primary system for removal of suspended solids and in secondary system for removal of dissolved solids and adsorption for removal of colouring material. The most common practice is to have aerated lagoons and follow activated sludge treatment system for removal of dissolved solids. During recent years, anaerobic treatment has received considerable interest due to low power consumption, lower nutrient demand and low generation of sludge, in developing countries. Land application of Industrial effluent is very common and is very promissing for ecological and economic reasons. The land application system involves irrigation, infilteration, percolation and over land flow. Sandy soils give highest percolation, loamy soils are suitable for irrigation and clay loams for over land flow. The rate of application vary with soil, conditions, type of crops, climatic conditions and effluent quality.

In our country, many of the integrated Pulp and Paper Mills are having primary and secondary treatment systems. Some of the Mills are in the process of implementating these systems whereever they don't exist. However, the situation in small and medium size Mills, based on agriculture residues, is very alarming due to lack of recovery systems, and many mills are not having full fledged treatment system.

AIR POLLUTION

Air pollution control measures include inplant control measures, process modifications and external control measures as well. Some such control measures are :

(a) Minimisation of dust generation in Chipper house by spraying water on Bamboo/Wood and Chips.

(b) Controlled discharge of digestors.

(c) Proper care in chlorine handling.

(d) Maintaining low sulphidity in cooking liquor &

(e) Proper ventilation and providing exhaust fans at various dust generating points, such as lime and talcum handling plants.

Air pollution control measures are yet to be given due to consideration in our country. However, Aeration, ventury scrubbing, Electrostatic precipitation and condensation etc. are being practiced by many mills.

FUTURE ENVIRONMENTAL NEEDS :

The environmental pollution control measures are very expensive and many Mills find it very difficult to use them due to poor financial condition. However, with adoption of MINAS and rising public concern over environmental pollution, the mills have to go in for short term and long term pollution control programmes. Pollution control measures are a social responsibility. In plant control measures and good housekeeping, proper ventilation, effective safety measures and proper training of concerned persons, will go a long way in eliminating environmental hazards. Good water management, effective waste recycling and properly modified process improve the atmospheric conditions as a whole and will improve the economy of the operations.

To make a complete environmental evaluation of a product, it is necessary to consider the entire product life cycle from raw material extraction to final disposal stage or recycle of the product. The ideal product can efficiently be recycled at a low cost but if it cannot, it should have safe disposal characteristics or be able to add fuel value in refuse burning. An attempt to identify the future environmental needs of the Pulp and Paper Industry should start with product life cycle. Technology is rapidly developed and implemented to bring down the emisson from the production and converting units to a very low and safe level. In Industry we have to be prepared to accept stringent environmental requirements as the public awareness is increasing day by day.

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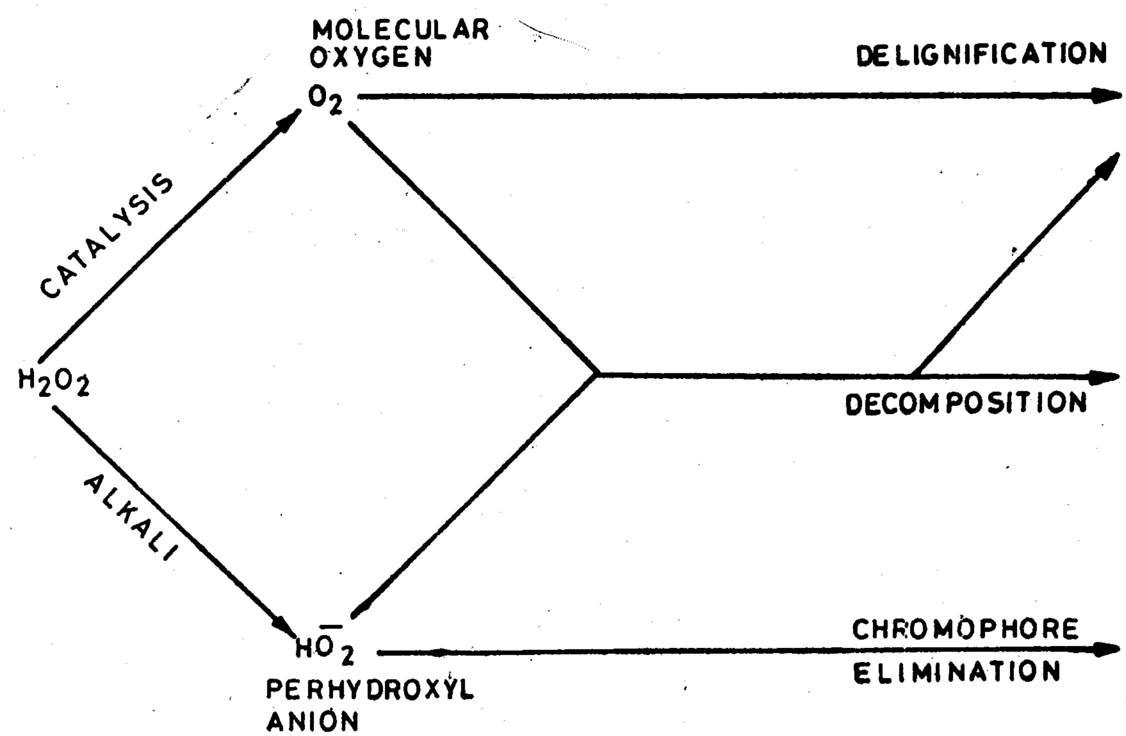
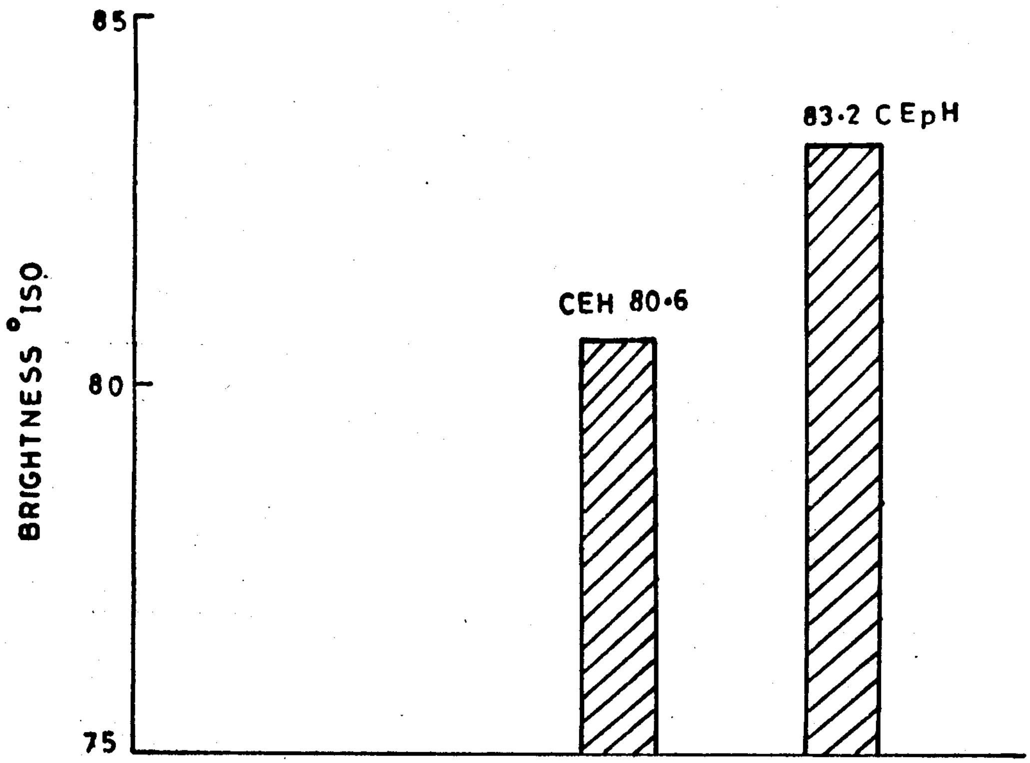


FIG.1 ROLE OF HYDROGEN PEROXIDE DURING BLEACHING OF CHEMICAL PULP

BAGASSE SODA PULP – UNBLEACHED BRIGHTNESS : 37·7 °ISO

STAGE	CONSISTENCY %	RETENTION TIME MINUTES	TEMP °C	pH INITIAL	pH FINAL
C	2	60	AMBIENT	2·2	2·2
E	10	60	AMBIENT	11·9	11·8
OR Ep				11·0	10·5
H	5	120	AMBIENT	10·1	8·7

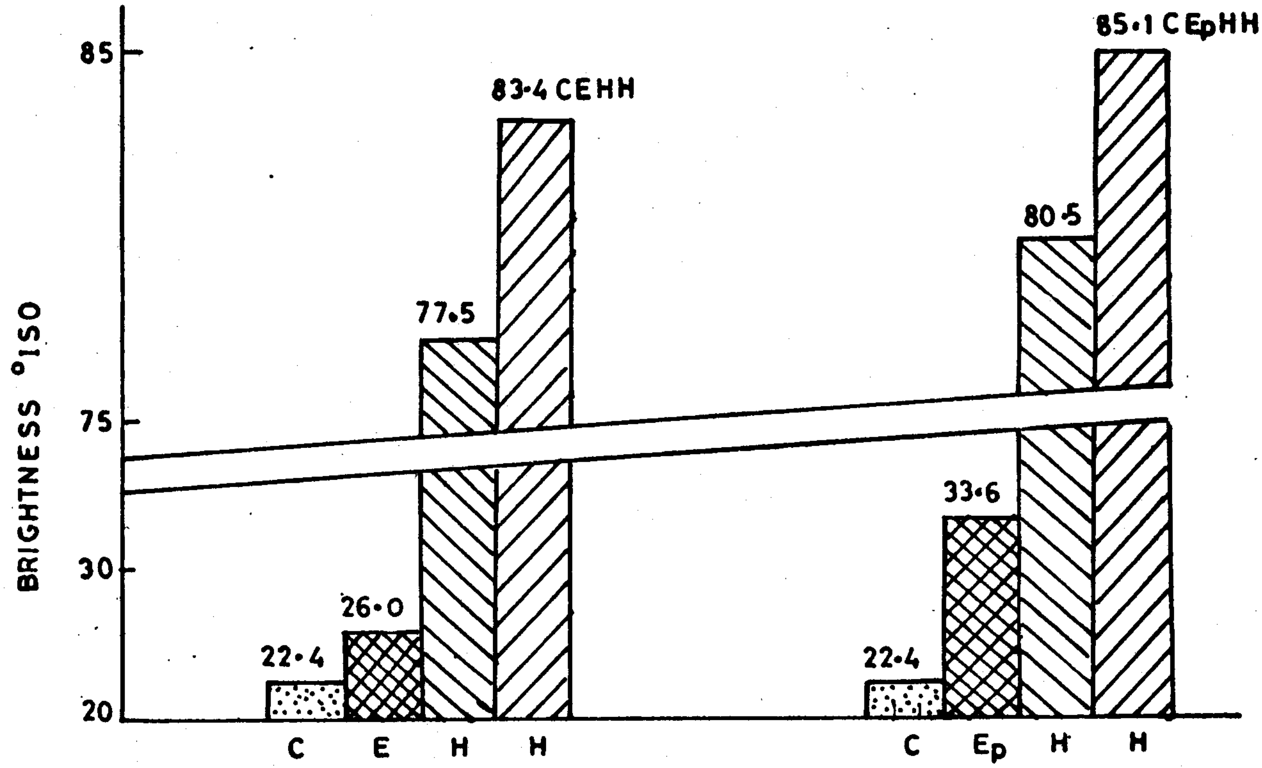


STAGE	CHEMICALS	GM/100GM ON O.D. PULP	
C	AVAILABLE CHLORINE	4·5	4·5
E	NaOH	1·2	0·65
OR Ep	H_2O_2 (100%)	—	0·25
	SOD. SILICATE (38°Be)	—	0·5
H	AVAILABLE CHLORINE	2·0	2·0

FIG·2 LABORATORY STUDIES ON USAGE OF HYDROGEN PEROXIDE AT ALKALI EXTRACTION STAGE WITH SODIUM SILICATE AS STABILIZER FOR BAGASSE SODA PULP

BAMBOO KRAFT PULP — UNBLEACHED BRIGHTNESS : 21·4 °ISO
UNBLEACHED KAPPA NO : 24·1

STAGE	CONSISTENCY %	RETENTION TIME MINUTES	TEMP. °C	pH	
				INITIAL	FINAL
C	2	45	AMBIENT	2·2	2·2
OR E	8	90	60	11·4	11·30
OR Ep				11·1	10·6
H	6	180	40	10·4	7·0
H	6	120	40	10·1	8·0



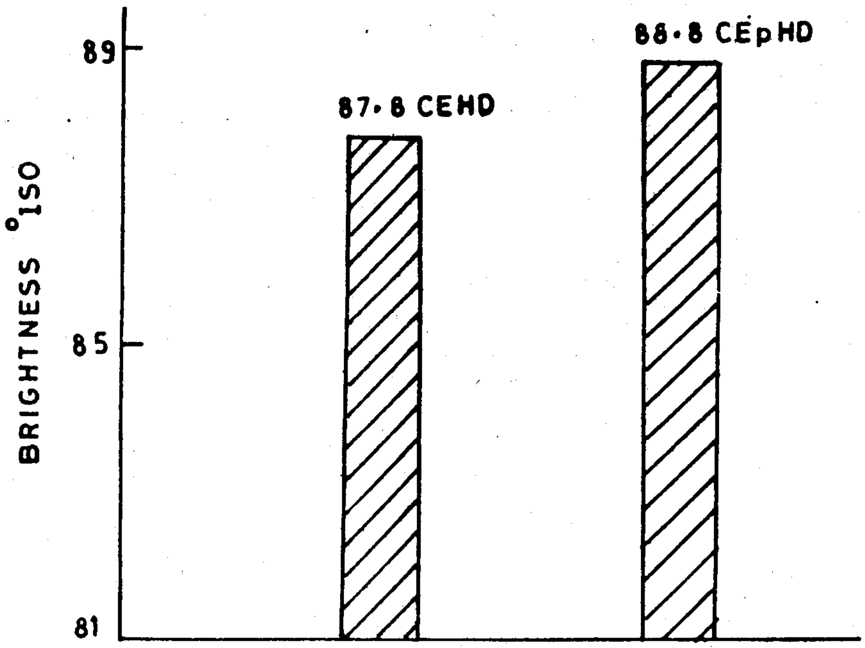
STAGE	CHEMICALS	GM/100GM ON OD PULP	
C	AVAILABLE CHLORINE	5·7	5·7
OR E / Ep	NaOH	2	1·4
	H_2O_2 (100%)	—	0·3
	SOD. SILICATE (38°Be)	—	2·0
H	AVAILABLE CHLORINE	2·85	2·85
H	AVAILABLE CHLORINE	0·95	0·95

FIG·3 LABORATORY STUDIES ON USAGE OF HYDROGEN PEROXIDE AT ALKALI EXTRACTION STAGE WITH SODIUM SILICATE AS STABILIZER FOR BAGASSE KRAFT PULP

MIXED HARDWOOD : KRAFT PULP

UNBLEACHED BRIGHTNESS : 32·9°ISO

STAGE		CONSISTENCY %	RETENTION TIME MINUTES	TEMP. °C	pH	
					INITIAL	FINAL
	C	3	40	AMBIENT	2·3	2·1
OR	E	9	90	60	12·1	11·9
	Ep				11·2	10·6
	H	9	105	60	10·6	8·1
	D	9	120	80	6·1	5·9

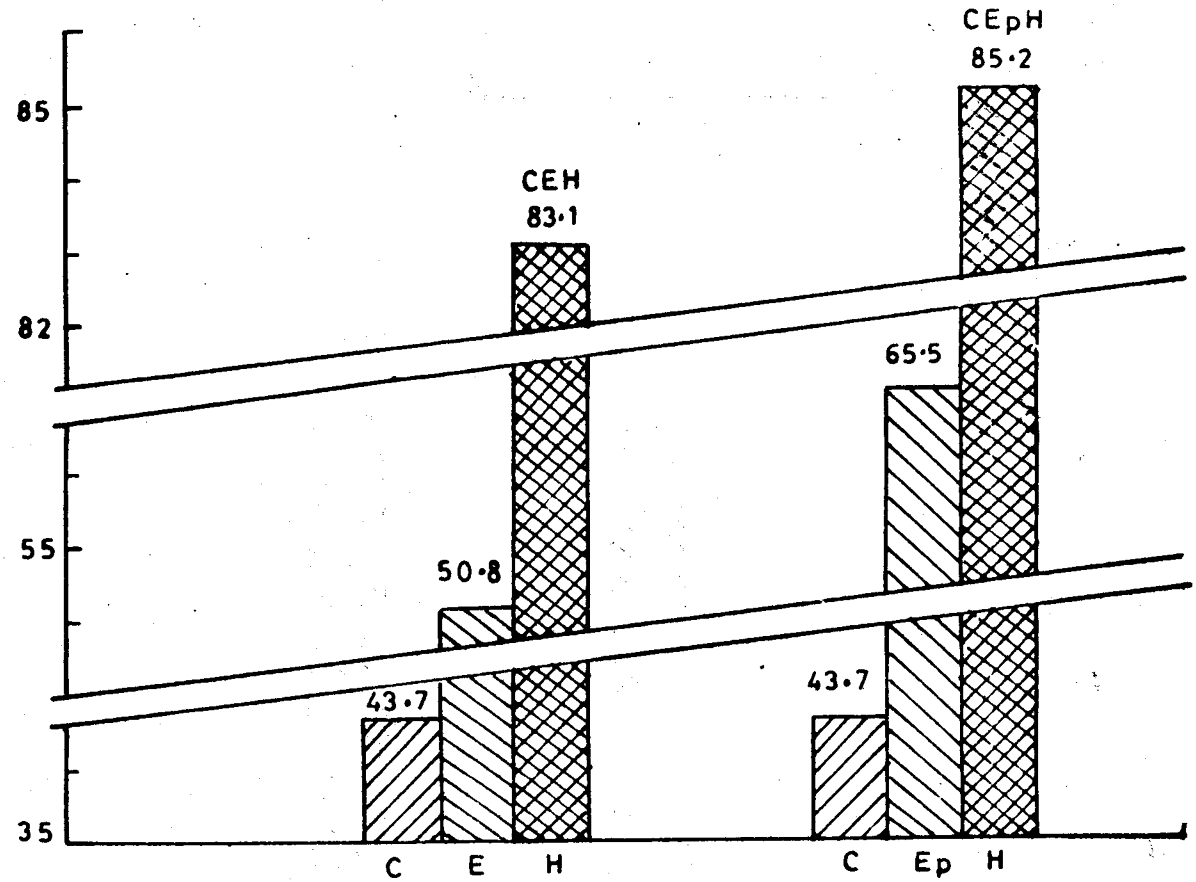


STAGE		CHEMICALS	GM/100GM ON OD PULP	
	C	AVAILABLE CHLORINE	3·5	3·5
OR	E	NaOH	2	1·0
	Ep	H_2O_2 (100%)	–	0·2
		ORGANIC STABILIZER	–	0·06
	H	AVAILABLE CHLORINE	1·5	1·5
	D	AVAILABLE CHLORINE	0·65	0·5

FIG·4 LABORATORY STUDIES ON USAGE OF HYDROGEN PEROXIDE AT ALKALI EXTRACTION STAGE WITH ORGANIC STABILIZER FOR MIXED HARDWOOD KRAFT PULP (RAYON GRADE PULP)

BAGASSE SODA PULP – UNBLEACHED BRIGHTNESS = 40·7° ISO
UNBLEACHED K NO = 8·76

STAGE	CONSISTENCY	RETENTION TIME MINUTES	TEMP, °C	pH INITIAL	pH FINAL	K. NO.
C	3	40	AMBIENT	2·85	2·71	3·40
or { E	8	90	50	11·9	11·5	2·0
Ep				11·35	10·6	1·23
H	8	120	AMBIENT	10·9	8·9	-

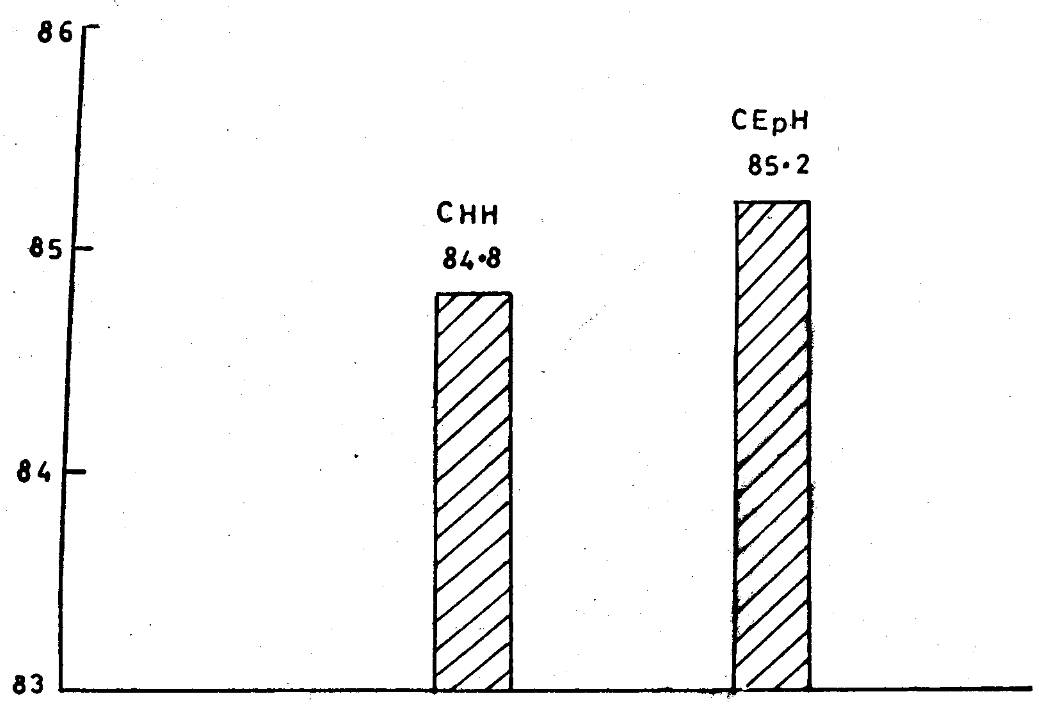


STAGE	CHEMICALS	GM / 100 GM ON OD PULP	
C	AVAILABLE CHLORINE	4·5	4·5
OR { E	NaOH	1·5	1·0
Ep	H_2O_2 (100%)	—	0·3
H	AVAILABLE CHLORINE	4·0	2·0

FIG·5 LABORATORY STUDIES ON USAGE OF HYDROGEN PEROXIDE AT ALKALI EXTRACTION STAGE WITHOUT STABILIZER FOR BAGASSE SODA PULP (LOW KAPPA NO)

MIXED HARDWOOD KRAFT PULP - UNBLEACHED BRIGHTNESS 23·5 °ISO

STAGE	CONSISTENCY %	RETENTION TIME MINUTES	TEMP. °C	pH INITIAL	pH FINAL
C	2·5	45	AMBIENT	2·25	2·10
OR { H	10	120	50	11·60	8·31
OR { Ep	10	120	50	11·15	10·10
H	10	120	40	10·53	8·12



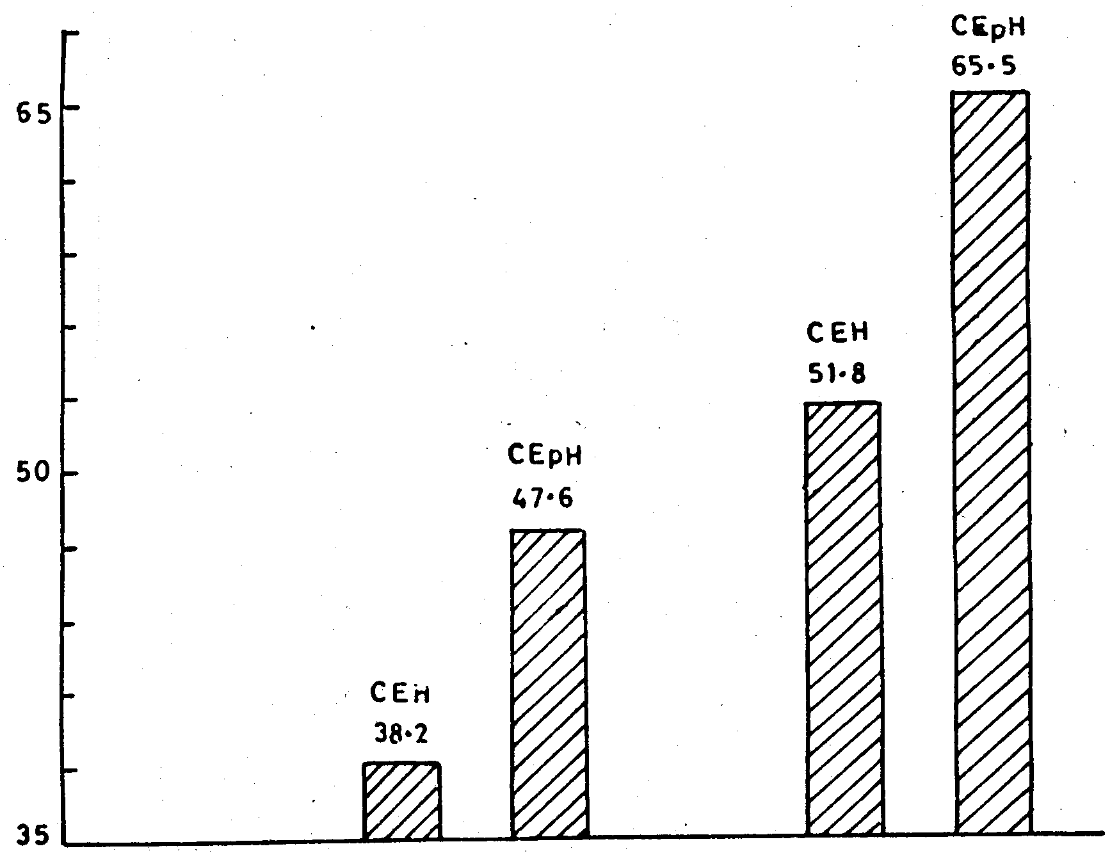
STAGE	CHEMICALS	GM/100GM OD PULP	
C	AVAILABLE CHLORINE	8·0	8·0
H	AVAILABLE CHLORINE	4·0	—
Ep	NaOH	1·5	1·5
	H_2O_2 (100%)	—	0·5
H	AVAILABLE CHLORINE	4·0	3·0

FIG·6 LABORATORY STUDIES ON USAGE OF HYDROGEN PEROXIDE AT ALKALI EXTRACTION STAGE WITHOUT STABILIZER FOR MIXED HARD-WOOD KRAFT PULP

BAMBOO KRAFT PULP – UNBLEACHED BRIGHTNESS : 20·1° ISO

UNBLEACHED K.NO : 25·1

STAGE	CONSISTENCY	RETENTION TIME MINUTES	TEMP., °C	pH INITIAL	pH FINAL
C	2	30	AMBIENT	1·92	1·80
OR {E	8	60	55	11·47	11·30
OR {Ep				11·1	10·6
H	6	120	40	9·5	7·8



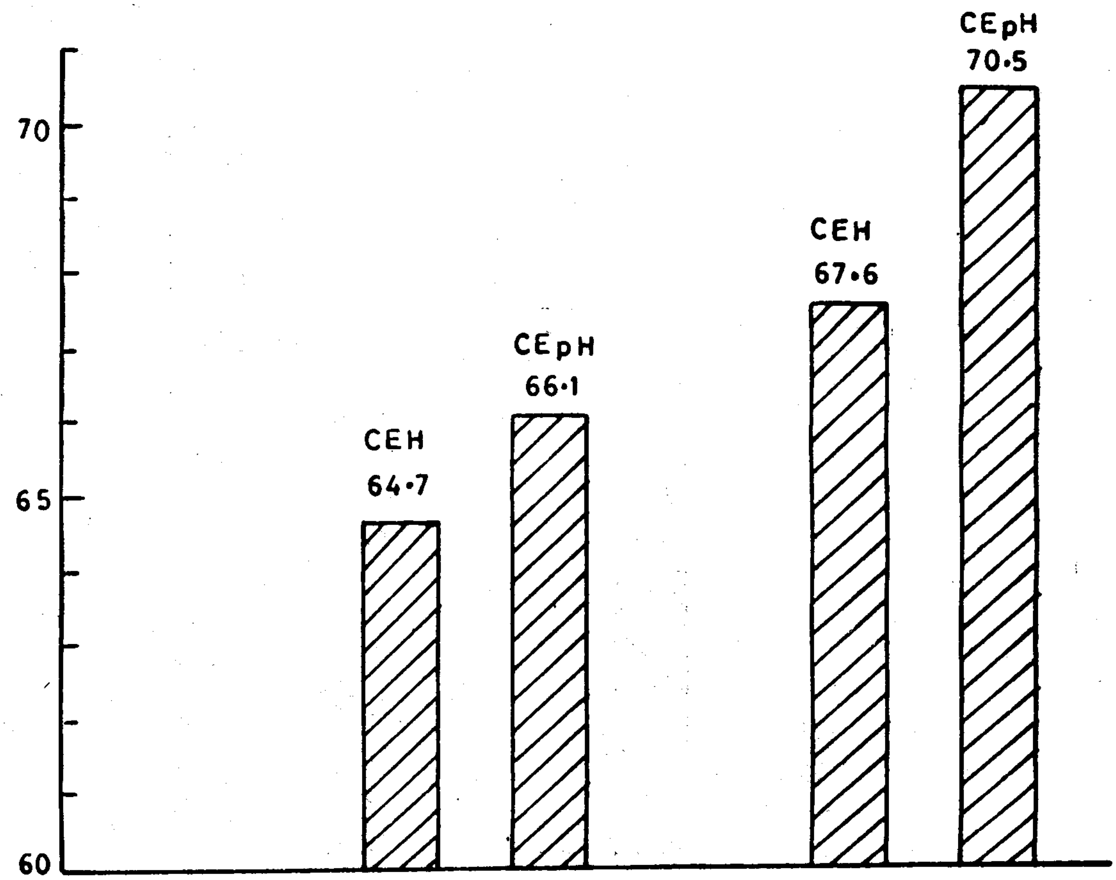
STAGE	CHEMICALS	GM / 100GM ON CD PULP	
C	AVAILABLE CHLORINE	6	6
OR {E	NaOH	2·5	2·5
OR {Ep	H_2O_2 (100%)	0·3	0·3
H	AVAILABLE CHLORINE	1·0	2·0

FIG. 7.A – USAGE OF HYDROGEN PEROXIDE AT ALKALI EXTRACTION STAGE WITH DIFFERENT LEVELS OF CALCIUM HYPOCHLORITE AT FINAL STAGE

BAMBOO KRAFT PULP – UNBLEACHED BRIGHTNESS : 20·1° ISO

UNBLEACHED K. NO. : 25·1

STAGE	CONSISTENCY %	RETENTION TIME MINUTES	TEMP. °C	pH	
				INITIAL	FINAL
C	2	30	AMBIENT	1·92	1·80
OR { E	8	60	55	11·47	11·30
OR { Ep				11·1	10·6
H	6	120	40	9·5	7·8

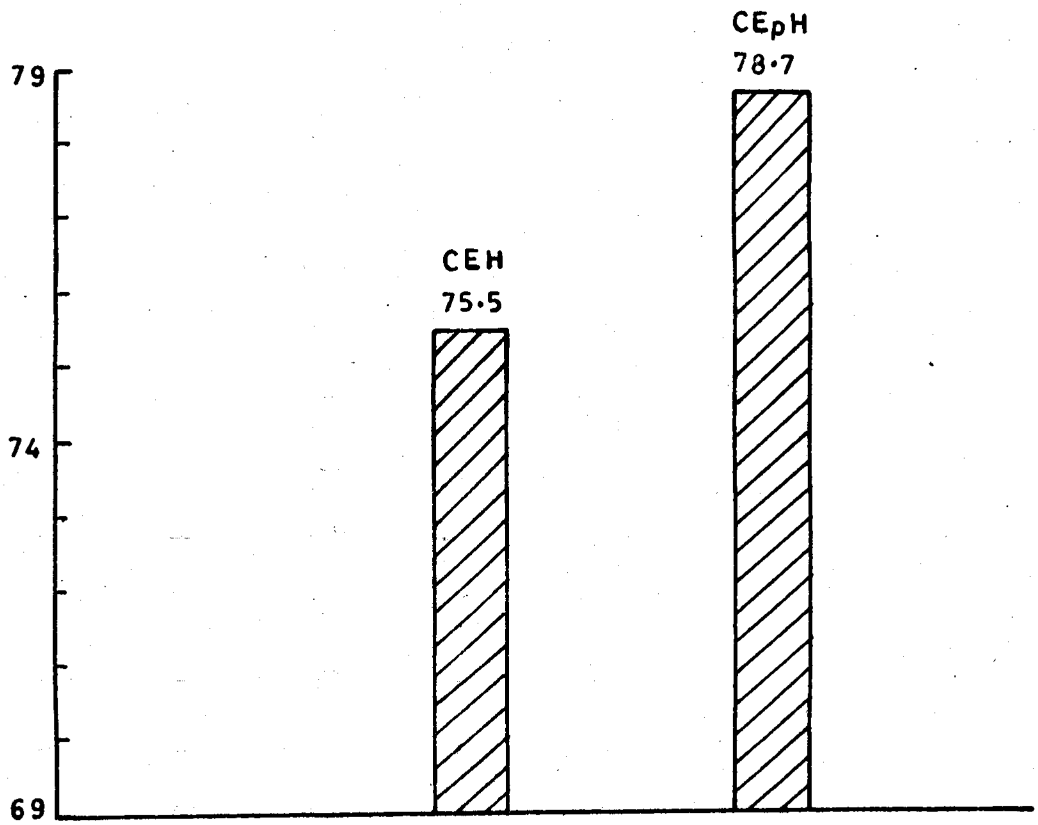


STAGE	CHEMICALS	GM/ 100GM OD PULP	
C	AVAILABLE CHLORINE	6	6·0
OR { E	NaOH	2·5	2·5
OR { Ep	H_2O_2 (100%)	0·2	0·3
H	AVAILABLE CHLORINE	2·5	4·0

FIG·7·B – USAGE OF HYDROGEN PEROXIDE AT ALKALI EXTRACTION STAGE WITH DIFFERENT LEVELS OF CALCIUM HYPOCHLORITE AT FINAL STAGE

BAMBOO HARD WOOD – UNBLEACHED KAPPA NO. : 24.8
KRAFT PULP

STAGE	CONSISTENCY %	RETENTION TIME MINUTES	TEMP °C	pH	
				INITIAL	FINAL
C	2	45	AMBIENT	2.30	2.31
OR E	10	60	60	11.8	11.51
OR Ep				11.2	10.9
H	8	120	40	9.51	7.59



STAGE	CHEMICALS	GM / 100 GM OD PULP	
C	AVAILABLE CHLORINE	5.0	5.0
OR E	NaOH	2.0	2.0
OR Ep	H_2O_2 (100%)	—	0.3
H	AVAILABLE CHLORINE	1.5	1.5

FIG.8. LABORATORY STUDIES ON USAGE OF HYDROGEN PEROXIDE AT ALKALI EXTRACTION STAGE FOR BAMBOO HARDWOOD PULP

TABLE—1

BAGASSE CHEMICAL PULP : 1) Unbleached brightness : 40.7° 150
2) Unbleached K. No. : 8.76

	Sequence	CEH			CE_pH		
	Parameters	C	C	H	C	E_p	H
1.	Chlorine % O. D. pulp	4.5	—	—	4.5	—	—
2.	Caustic % „ „	—	1.5	—	—	1.0	—
3.	H_2O_2 (100%) % „ „	—	—	—	—	0.3	—
4.	Ca-Hypochlorite (A. C.) O. D. pulp	—	—	4.0	—	—	2.0
5.	Consistency %	3	8	8	3	8	8
6.	Temperature °C	Amb.	50	Amb.	Amb.	50	Amb.
7.	Retention time (min.)	40	90	120	40	90	120
8.	pH : Initial	2.85	11.9	10.9	2.85	11.35	10.8
	Final	2.71	11.5	8.9	2.71	10.6	8.4
9.	Colour of extraction liquor (Hazen units)	—	3125	—	—	1750	—
10.	K. No.	3.45	2.0	—	3.45	1.23	—
11.	Freeness °SR	—	—	24	—	—	23
12.	Brightness °ISO	43.7	50.8	83.1	43.7	65.5	85.2
13.	Post Colour Number	—	—	0.32	—	—	0.28
14.	Strength Properties : TF	—	—	46.6	—	—	49.3
	BF	—	—	23.16	—	—	23.75
	BL	—	—	2756	—	—	2800

TABLE NO. 2

BAMBOO+MIXED HARDWOOD CHEMICAL PULP (KAPPA NO. 24.8)

	Sequence Parameters	Blank CEH	Peroxide CEpH
	O.D. Pulp	400 gm	400 gm
I.	**Chlorination**		
	% Chlorine	5.0	5.0
II	**Extraction**		
	% NaOH	2.0	2.0
	% H_2O_2 (100%)	—	0.3
III.	**Hypo (I)**		
	% Ca-Hypochlorite A.C.	1.5	1.5
	% Sulphamic Acid	0.07	0.07
	Total Chlorine added	6.5	6.5
	Total chlorine consumed	6.33	6.27
1.	Brightness % Elrepho	75.5	78.7
2.	Beating time (min)	16	20
3.	Initial slowness	18	18
4.	Final slowness	29	29
5.	G.S.M.	59.82	59.32
6	Burst factor	38.7	38.8
7.	Tear factor	77.7	77.5
8.	Breaking length	5550	5600

CONCLUSIONS

1. It is possible to elevate the brightness of fully bleached kraft and soda pulp of Indian tropical special like bamboo, hardwood (eucalyptus) and agro residue like bagasse by usage of 0.3 to 0.5% H_2O_2 (100%) at alkali extraction stage.
2. It is also possible to reduce the consumption of caustic at alkali extraction stage and hypochlorite during subsequent bleaching stages by usage of hydrogen peroxide at alkali extraction stage.
3. Due to oxidative properties of hydrogen peroxide, the colour of extraction wash liquor gets reduced during bleaching.
4. It is also expected that the strength properties of fully bleached pulp should show the improvement as the reduction in usage of hypochlorite is possible during bleaching. This will no doubt also improve the reversion characteristics of the pulp.

ACKNOWLEDGEMENTS

The authors wish to thank Dr. S.K. Datta, Vice-President- Technical Development, National Peroxide Limited, for his permission to publish this paper. Cooperation from various paper mills for pulp samples for the laboratory experiments is gratefully acknowledged.

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