

PER ACID BLEACHING OF KRAFT PULP

A DISSERTATION

*submitted in partial fulfilment of the
requirements for the award of the degree*

of

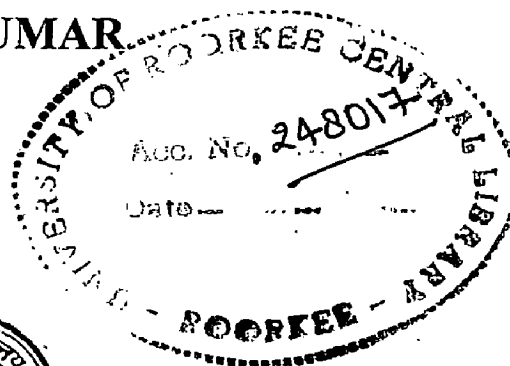
MASTER OF ENGINEERING

in

PULP AND PAPER TECHNOLOGY

By

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CANDIDATE'S DECLARATION

I hereby certify that the work which is being presented in the thesis entitled "PERACID BLEACHING OF KRAFT PULP" in the partial fulfilment for the award of degree of Master of Engineering in Pulp and Paper Technology submitted at Institute of Paper Technology (University of Roorkee), Saharanpur, is an authentic record of my own work carried out during the period from July 1997 to January 1998 under the supervision of Dr, Satish Kumar, Associate Professor, Institute of Paper Technology (University of Roorkee), Saharanpur. The matter embodied in this thesis has not been submitted by me for the award of any other degree.

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This is to certify that the above statement made by the candidate is correct to the best of my knowledge.

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ABSTRACT

Per acids may prove viable for use in ECF and TCF bleaching sequences. In the present study, the peracetic acid was used as a delignifying agent. Peracetic acid was used to delignify the oxygen treated and untreated pulp in ECF & TCF sequences. A comparison was done between Peracetic acid delignification and conventional Oxygen delignification stage.

The results indicate that the extent of delignification by Pa stage is lower than O stage. The O stage gives brightness, viscosity equivalent to Pa stage followed by Eop stage. However the COD load of O stage was found higher than Pa stage. P stage or D stage used after Pa Eop stages improved the brightness by 14-20 points and after O Pa stages by 13-14 points with reduction in pulp viscosity by 14-20% and 30 % respectively.

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CHAPTER-1

INTRODUCTION

Pulp Bleaching processes which are based on the chlorine and chlorine containing chemicals, produce chlorinated organic compounds. Environmental concerns and market pressure has forced the pulp and paper industry to explore alternatives to conventional bleaching practices. The pollution load from the bleaching processes can be reduced by the use of chlorine free compounds. The major drawbacks in chlorine free bleaching are the capital investment required, high chemical cost and difficulty in maintaining both high brightness and pulp strength properties. But due to environmental concerns, the demand is increasing for pulps bleached without chlorine containing chemicals, by ignoring the drawbacks associated with chlorine free bleaching sequences.

Oxygen and the Hydrogen peroxide are replacement chemicals that are firmly established in chemical pulp bleaching. However due to their low reactivity with certain lignin structures, they are unable to completely replace the chlorine containing chemicals. Ozone is a strong electrophile that attack many lignin structures and is now

being used commercially as a partial replacement for chlorine containing compounds. The drawbacks of three oxidants above particularly Oxygen and Ozone, is cellulose degradation if large amount of oxidants are consumed by the pulp. Thus there is an need for additional delignifying and brightening agent that are fairly inert towards the carbohydrate fraction of chemical pulps. Peracids like Peroxymonosulfuric acid (Caro's acid)(1-2) and Peracetic acid (3) have been identified as candidates. The pulp can be bleached to high brightness upto 80-85% ISO by the sequences containing peracids stage. Generally it is accepted that Per acids are less capital intensive ,easily retrofit and highly selective bleaching chemicals when used under the optimum conditions. Per acids have high oxidation potential which allows them to be used in bleaching reactions under more moderate conditions than those required for Hydrogen peroxide.

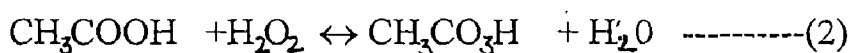
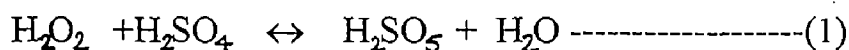
CHAPTER-2

LITERATURE REVIEW

A per acid or Peroxy acid is an acid that contains a perhydroxyl group in place of the hydroxyl group of its parent acid. These are weaker acid than their parent acids viz. Peracetic acid (Pa), Caro's acid (Px) or Peroxymonosulfuric acid and Dimethyl dioxirane(DMD). Caro's acid is a dibasic acid. The ionisation constant of the perhydroxyl proton pK_2 , is much higher than the pK_2 for sulfuric acid; therefore, the Caro's acid anion is a much weaker acid than the parent acid(4). Peracetic acid has a sharp pungent odor, having a boiling point of 103 °c and a vapour pressure of 20 m.m. Hg at 25 °c. Long term exposure to Peracetic acid can lead to liver damage, decreased white blood count, and lung tumors(4). Personal protective equipment should include a respirator in addition to splash proof goggles.

2(a)MANUFACTURE OF PER ACIDS

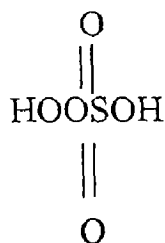
Per acids are generally produced by reacting a concentrated solution of Hydrogen peroxide with a concentrated solution of Sulfuric acid or Acetic acid or Formic acid according to the equation 1&2(5).



Excess acid is normally used to drive the equilibrium to the right. If the Caro's acid is to be used under acidic condition, the excess sulfuric acid is not a problem. The separation of Peracetic acid and Acetic acid by distillation is feasible. The higher the molar ratio of the Sulfuric acid with Hydrogen peroxide, higher the relative amount of Peroxymonosulfuric acid and higher the molar ratio of acetic acid to the Hydrogen peroxide, higher the relative amount of Peracetic acid. If Acetic acid is added to Caro's acid, a mixture of Peracetic acid and Caro's acid can be formed which can be called as Mixed peroxymonosulfuric acid (Pxa). One of the benefits of Pxa is the flexibility that can be achieved in the production of the per acid to suit the individual requirement of the mill. If the bleach plant effluents are to be recycled and the sulfur balance is an issue, low molar ratio of Sulfuric acid can be used. If the effluent are to be discharged and BOD is a problem, lower molar ratios of Acetic acid can be used. High conversion ratio i.e. beyond 70% are necessary for maximum pulp brightness gain and kappa number reduction.

2(b) BASIS FOR PER ACID CHARGE

All the Per acids are expressed in terms of equivalent amount of "active oxygen" (AO) or as an equivalent amount of a representative Per acids, such as H_2SO_5 (Caro's acid) (6). Caro's acid with a molecular weight of 114.08 gm., is represented as follows



In calculating the percent active oxygen of a Per acid, only one of the oxygen atom in the O-O linkage of a Per acid is considered to be active. For example, H_2SO_5 is 14% by weight active oxygen ($16.0 / 114.08 * 100 = 14\%$). Similarly Peracetic acid has a molecular weight of 76.05 and is 21% AO.

Similarly a weight of any Per acid or solution containing Per acid, can be converted to an “equivalent H_2SO_5 ” weight basis by multiplying by the percent per acid active oxygen in the solution and dividing by 14.0 (the percent active oxygen in H_2SO_5). For example 5.72% active oxygen is equivalent to $5.72/14 = 0.406$ Lb of H_2SO_5 .

2© SELECTION OF PER ACID FOR BLEACHING IN A MILL

Selection of any Per acid for bleaching of pulps depends upon the following points(6)

- Delignifying and brightening efficiency of Per acid.
- Chemistry of the on-site manufacturing process, including economics and chemical effluent load.
- Operational issues for the on-site processes, including chemical safety, handling and process utility.

Equal amount of Per acid Active oxygen,give essentially equal bleaching and delignification whether it is from Pa,Pxa orPx. The ingredient costs to make and use per acids on -site can vary significantly depending on the per acid made ,the recipe and the process used,because the yield on each gradient changes with the recipe and process. These differences are also the function of the relative prices of the ingredients used. Different safety issues are associated with equilibrium per acids from 50% and 70% Hydrogen peroxide and from distilled Peracetic acid . The use of 70% H₂O₂ and distillation of Peracetic acid require more precautions. Sulfer,Sodium and BOD in the Per acid stage effluent could be deciding factor depending on a mill's ability to manage each. These and other differences should be reviewed before deciding which acid to used.

2(d) USES OF PER ACID IN BLEACHING SEQUENCES

The applications of Per acids and laboratory data comparing their performance have been reviewed by N. Liebergott (7). Per acids or Peroxy acids can be used in a bleaching sequence as

1. Delignification agent

- Wood (pulping process such as Milox)
- Pulp (replacement or reinforcement in an initial chlorination stage), reinforcement of an Eop stage (7)

2. Activation agent

- Activation of lignin before or between two O₂ stages(7)

- Activation of Hydrogen peroxide together with Oxygen under acidic or mildly alkaline conditions (7)

3. *Brightening agent in later oxidizing stage*

- In ECF bleaching as a replacement for a chlorine dioxide or hypochlorite stage (6).
- In TCF bleaching to replace a Hydrogen per oxide stage (8)

Peracetic acid is used first time as a delignifying agent by poljak in 1948 as a method for preparing hollocellulose from wood (4). Leopold B(9). found that Peracetic acid is highly selective in the removal of lignin from the wood. Bailey and Dence investigated the effect of pH (in the range 3-9) , temperature (in the range 50-80 °c) and consistency ,on delignification and on the properties of kraft pulp (3). Kappa number reduction of pulp oxidized with Peracetic acid at pH 7-9 was found essentially equivalent to the kappa number of similar pulp samples oxidized with an eqimolar quantity of chlorine. At pH range 5-9, reaction pressure and pulp consistency had a minimal impact on pulp strength. However at pH 3, the viscosity and strength did decrease with increasing temperature with Peracetic acid treatment.

It has been shown that Peracetic acid is capable of hydroxylating the aromatic ring of a phenolic compound to form a hydroquinon. Hydroquinones are readily oxidized to quinones which then undergo ring opening to yield muconic, maleic and fumeric acid derivatives of the starting phenolic compounds (10).Hydroxylation of phenolic

compounds, epoxidation of olefinic bonds, and Baeyer-Villiger oxidation of ketones, all involves oxidation of functional groups that are normally present in lignin. Lignin containing pulps therefore should be susceptible to oxidation with Per acids (11).

Kraft pulps can be further delignified to low kappa number by an oxygen stage followed by a caroate stage (7). A halide, preferable chlorine can be added to the caroate stage which accelerates lignin degradation and simultaneously retard cellulose depolymerisation. In the process, the chloride is oxidized to chlorine by Per acid. Chlorine involvements results in an effluent containing 0.5 to 0.7 kg. of AOX per ton of pulp.

N. libergott found that the peracid pretreatment can result in an increase in deliginification before an oxygen stage or between oxygen type stages (7). peracetic pretreatment of an unbleached softwood pulp can increase Eop deliginification from 35% (Q Eop) to 48.9% (PaEop). On an Oxygen pretreated pulp the use of peracetic acid treatment also increased the effectiveness of the following Eop Stage O [Pa Eop] from 20% deliginification to 40% deliginifications. When the washing stage between Pa & Eop stage is eliminated, it was also found that all the peracid treatments done under oxygen pressure, after Eop Extractions, were more effective in kappa number reduction than per acids alone. [Px - O Eop] & [Pxa-O Eop] treatment resulted in

delignification of 63% and 67% respectively (7) further bleaching of [pxa-O Eop] pulp in a peroxide stage resulted in ISO brightness of 77 to 80%.

Rapson reported that the optimum pH range for both chlorine dioxide and peracetic acid are close to each other (12). The optimum temperature for both chlorine dioxide & peracetic acid are in the same range and they do not react with each other. So it becomes possible to use the chlorine dioxide and peracetic acid together in the same solution for bleaching pulp. Higher brightness up to 90-92% GE can be achieved when both are used combinedly than when either bleaching agent used alone.

Per acids can be used in place of hypochlorite without making significant modification to its bleach plant (6). Bleach plants residence time is unchanged. Although there is a very minor difference in brightness with peracid bleaching as compared to hypochlorite stage, but peracid treated pulp suffer less colour reversion and the oven dried pulps brightness are virtually equal. Pulp viscosity from the peracid sequence is higher than that from the hypochlorite sequence.

At neutral pH, the use of peracetic acid has also been evaluated as a bleaching agent for mechanical pulps (13). Ultra violet and infra red spectroscopy of mechanical pulps, bleached at pH 7-8, have shown that bleaching results from modifications of the side chain groups of lignin (α,β unsaturated aldehydes,

conjugated double bonds, and α Carbonyl groups). Oxidation of the phenolic rings is normally minimal in this pH range (14). A combination of peracetic acid and Hydrogen peroxide in the PaP sequence with no wash stage between Pa & P to brighten a groundwood or chemical pulp is more effective in reaching higher brightness than either of the two chemicals used alone (14,8).

CHAPTER - 3

EXPERIMENTAL

3 (a) SAMPLE :- Unbleached mixed kraft pulp (80% Eucalyptus, 10-15% Bamboo 5-10% Pine) was taken from Star Paper Mills Ltd., (Brightness 19% ISO, Kappa number 21.5 & viscosity 47.9 cp.)

3(b) PREPARATION OF PERACETIC ACID :- The peracetic acid was prepared by adding Hydrogen peroxide to acetic acid in the presence of sulfuric acid (5), in the molar ratio Hydrogen per oxide : sulfuric acid : acetic acid 1 : 1: 0.5 to get high conversion ratio.

Hydrogen peroxide was taken in a flask kept in a ice bath. Drop wise addition of sulfuric acid (98%) with continuous agitation, was done through a funnel. Then the glacial acetic acid was added dropwise to the solution of hydrogen peroxide and sulfuric acid. This mixture was stored overnight in refrigerator to reach equilibrium. This equilibrium solution was then assayed for peracetic acid by the method proposed by Green span & mackeller.

3© ANALYSIS OF PER ACETIC ACID :- The per acetic acid was assayed by the method proposed by the Green span & Mackellar (15).

0.3 to 0.4 gram of per acetic acid was accurately weighed and placed in a 500 ml Erlenmeyer flask containing 150 ml of 5% sulfuric acid and sufficient cracked ice to maintain a Temperature of 0 to 10° c. Two or Three drops of ferrion indicator were then added to the flask (Solution turns reddish oragne in colour) It was titrated immediately with 0.1 N ceric ammonium sulfate to a pale blue end point. Approximately 10 ml of 25% KI was then added to the flask and the mixture was titrated with 0.1 N sodium thiosulfate until the dark radish colour of iodine began to fade. At this stage 2-3 ml of the starch indicator was added and titration was continued until the dark blue colour changed to pale reeddish orange colour end point. The concentration of H₂ O₂ and peracid active oxygen were calculated by using the following formula.

$$\% \text{ H}_2\text{O}_2 = \frac{V_{\text{ceric}} \times 0.1 \times 1.7}{\text{Sample weight}}$$

$$\% \text{ AO} = \frac{V_{\text{thio}} \times 0.1 \times 0.8}{\text{Sample weight}}$$

3(d) ANALYSIS OF HYDROGEN PEROXIDE : One ml of H₂ O₂ is pipetted to 250 ml. Erlenmeyer flask containing 100 ml. of distilled water and 10 ml. of 20%

sulfuric acid. One to three drops of ferrion indicator were added and it was titrated with 0.1 N ceric ammonium sulfate to a pale blue point. Concentration of Hydrogen peroxide was calculated by using following formula.

$$\text{gpl H}_2\text{O}_2 = V_{\text{ceric}} \times 0.1 \times 17$$

3 (e) ANALYSIS OF CHLORINE DIOXIDE CONCENTRATION :- Some of distilled water was taken in a 250 ml. Erlen meyer flask and 15 ml.. normal KI solutions was than added to 5 ml. of 20% sulfuric acid. 5 ml. ClO₂ solution was then transferred from pipette below the liquid level in the flask .

This solution was titrated with 0.1 N sodium thio Sulfate solution to starch and point (16).

$$\text{gpl ClO} = V_{\text{thio}} \times 0.27$$

3 (f) PROCESS CONDITIONS

The Various process conditions for different bleaching stages are shown in Table 1.

TABLE-1 Process Variable for different stages

S.No	Stages	Pa	Eop	O	P1/P2	D
1.	Chemical Charge (% on O.D Pulp)					
a.	Peracetic acid					
	as H ₂ O ₂	1.0	-	-	-	-
	as AO	0.44	-	-	-	-
b.	Na OH	-	3.3	2.5	3.5	-
c.	H ₂ O ₂	-	carry over from Pa	-	2.5	-
d.	MgSO ₄ .7 H ₂ O	0.025	-	0.025	0.025	-
e.	ClO ₂ as Active Cl ₂	-	-	-	-	1.0
2.	Oxygen Pressure Kg/Cm ²	-	6.00	8.00	-	-
3.	Temperature (°C)	50	50 85	110	90	70
4.	Retention Time (min.)	30	40 20	60	240	180
5.	End pH	2.5	11.0	-	11.0	4.5
6.	Consistency %	11	10	10	10	10

3(g) PROCEDURE:- The unbleached Kraft Pulp containing 100 gm. O.D. Fiber and 80% moisture was taken for each bleaching sequence. An equilibrium peracetic acid solution with 2.8% Active oxygen was used through out for bleaching. The peracetic acid was added to the Mg SO₄ solution and then this mixture was added to the pulp slurry in polythene bags. The pH 2.5 was maintained with the help of NaOH. After Kneading for two three minutes, the bag was sealed and kept in a constant temperature bath maintained at 50° C. After 30 minutes of retention time, the Eop stage was conducted in stainless steel pressure reactor (Bomb). The required amount of NaOH (3.3 % on O.d Pulp) was added as an aqueous solution to the pulp slurry and per oxide was carried over from the pa stage. The oxygen was applied at a temperature of 85° C. After 20 Minute retention the pressure was released and 50° C. temperature was maintained for 40 minutes. The Pulp was then washed with water.

Oxygen delignifications was performed in stainless steel pressure reactor. Magnesium sulfate (0.025% On OD Pulp) was dissolved in distilled water and sodium hydr oxide (2.5% on OD Pulp) was added to this solution of Mg SO₄. This colloidal solutions was added to the reactor vassel for mixing with the Pulp. After the required retention time (60 Min.) at desired temperatures (110° C) the pulp was washed with water.

The peroxide stages were performed in the polythene bags. The bleach liquor was prepared by adding in order, the required amounts of magnesium sulfate (0.025% on OD Pulp) Sodium hydro oxide (3.5%) and hydrogen peroxide (2.5%) to a beaker. Subsequently the bleach liquor was added to the pulp in Polythene bag and then

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kneaded for two minute. After required retention time, the samples were taken for analysis of final pH and the residual peroxide. The Pulp slurry was treated with sulphuric acid solution and washed with water.

In the Chlorine dioxide stage the sodium chlorite solution was prepared and assayed and charged to the pulp slurry in required amount (1% as Active Chlorine) in polythene bag . pH (4.5) was maintained with the help of NaOH. The Pulp slurry was well kneaded and put into the thermostat water bath at temperature 70° C for the retention time 180 Min. The Pulp Slurry was then washed with water.

After each stage the liquor is separated from the pulp slurry and the COD values were determined with the help of thermoreactor and spectro meter. The kappa number of treated and untreated pulps were determined according to Tappi Test method T 236 cm 85. The Pulp viscosities were measured by using Tappi Test method T 206 OS 63. Hand made sheets were made and air dried determine the sheet brightness.

CHAPTER-4

RESULTS AND DISCUSSION

The various bleaching sequences performed are Pa Eop D, Pa Eop P₁ P₂, O Pa P D and O Pa P₁ P₂ and the results for the same are given in the table 2 to 6. Peracetic acid has been used as a delignification agent.

The results in table 2 indicate that kappa number of the pulp gets reduced from 21.5 to 13, giving an approximate 40% reduction in kappa number. The brightness get improved from 19 to 34% ISO, again of 15 points. However the viscosity remain unaffected i.e. around 47.3 cp..

On comparing peracetic acid stage with the conventional O stage, upto 50% reduction in kappa number is feasible with an additional 7.5 points gain in the brightness. However the viscosity drops by another 25%.

The use of Eop stage after Pa stage improves the brightness by another 7 points but the viscosity drops by another 25%. Thus peracetic acid stage followed by oxidative extraction stage Eop gives brightness and pulp viscosity approximately equivalent to an conventional oxygen stage. Another gain of 14 or 18 points can be achieved by using peroxide or chlorine dioxide, however the viscosity drops further by another 20% or 14%.

The delignification can further be improved by another 20% (kappa number reduces from 21.5 to 6.4) and brightness improves by another 13 points (from 42% to 54%)

with an additional reduction in pulp viscosity by merely 5%. The sequence Pa Eop P gives nearly the same brightness as O Pa . The brightness can be improved by another 13 points i.e. 67% ISO by using P stage after O Pa with another reduction in pulp viscosity by another 25%. The use of second peroxide stage after O Pa P , does not give any further improvements in brightness but viscosity drop further by another 5%. Using a D stage after P stage in O Pa P sequence gives an additional brightness of 6.5 points with a reduction in pulp viscosity by another 2%.

The pollution load from the various bleaching stages have been determined by estimating the COD values of the waste water generated in each sequence. The COD value indicate that COD load from Pa + Eop stage is lower than O stage , the values being 9.54 kg/t and 10.53 kg/t respectively . The COD load from bleaching sequences Pa Eop P or Pa Eop D are approximately same around 16-17 kg/t. However the COD load from O Pa P is higher i.e. 22.59 kg/t. Using an additional D stage increases the COD value to 29.43 kg/t.

Table 2 : Peracetic acid compared to Oxygen as delignification agent

Mixed kraft pulp	Kappa No.	Brightness (% ISO)	Viscosity (cp)	COD kg/t
Brown stock pulp	21.5	19	47.9	-
Pa stage	13.02	34	47.3	-
Eop stage	10.53	41	34.78	9.54
O stage	10.58	41.5	34.9	10.53

Table-3 ECF Sequence-Pa Eop D

Stage	Kappa No.	Brightness (% ISO)	Viscosity(cp)	COD kg/t
Pa	13.02	34	47.3	-
Eop	10.53	41	34.78	9.54
D	-	59	27.8	6.48
				16.02

TABLE-4 TCF Sequence Pa Eop P₁ P₂

STAGE	KAPPA NO.	BRIGHTNESS (%ISO)	VISCOSITY(cp.)	COD kg/t
Pa	13.02	34	47.3	-
Eop	10.53	41	34.78	9.54
P ₁	-	55	28.82	-
P ₂	-	55	25.62	7.92
				17.46

TABLE-5 ECF Sequence O Pa P D

STAGE	KAPPA NO.	BRIGHTNESS(% ISO)	VISCOSITY (cp.)	COD kg/t
O	10.58	41.5	34.9	10.53
Pa	6.4	54	33.2	7.02
P	-	67	20.46	5.04
D	-	73.5	19.68	6.84
				29.43

TABLE -6 TCF Sequence O Pa P₁ P₂

STAGE	KAPPA NO	BRIGHTNESS (%ISO)	VISCOSITY(cp)	COD (kg/t)
O	10.58	41.5	34.9	10.53
Pa	6.4	54	33.2	7.02
P ₁	-	67	20.46	5.04
P ₂	-	67	18.08	3.24
				25.83

CHAPTER-5

CONCLUSIONS

From the present studies the following conclusions may be drawn:

1. Peracetic acid can be used as delignifying agent.
2. The extent of delignification by Pa stage is lower than O stage.
3. The O stage gives brightness, pulp viscosity equivalent to Pa stage followed by Eop stage. The COD load of O stage is higher.
4. The use of P stage or D stage after Pa Eop improves the brightness by 14-18 points and after O Pa stage by 13-14 points with reduction in pulp viscosity 17-20% and 30%.
5. The second P stage does not give any improvement in brightness.
6. The O stage give somewhat higher COD load as compared to Pa stage.

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