

BLEACHING OF MIXED KRAFT PULP FOR SOUTHERN PAPER MILL (SPM).

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ABSTRACT

Wood chips obtained from Southern Paper Mill (SPM) were mixed in the ratio of 80% softwood (pine) and 20% hardwood (eucalyptus) by weight and then cooked using a mixture of sodium hydroxide and sodium sulphide as a cooking liquor at 170 °C. Pulp obtained was bleached using the following bleaching sequences; Chlorination, Extraction, Hypochlorite stage 1, Hypochlorite stage 2 (CEHH), Oxygen, Chlorination, Extraction, Hypochlorite stage 1, Hypochlorite stage 2 (OCEHH), Oxygen, Chlorination, Extraction fortified with oxygen and hydrogen peroxide, Hypochlorite stage 1, Hypochlorite stage 2 (OCE_{OP}HH), and Oxygen, Chlorine dioxide, Extraction fortified with hydrogen peroxide, Chlorine dioxide, Hydrogen peroxide (ODE_pDP). The effect of chlorine charge at chlorination stage on brightness and viscosity at hypochlorite stage 1 and 2 and the total Chemical Oxygen Demand (COD) were investigated and reported.

The conventional CEHH which is used by the mill gave maximum brightness of 85% ISO, viscosity 461 cm³/g and COD of 133 kg/t. The elemental chlorine free bleaching sequence (ODE_pDP) gave brightness of 85% ISO, viscosity 596 cm³/g and COD 102 kg/t. OCE_{OP}HH gave brightness of 84% ISO, viscosity 396 cm³/g and COD 118 kg/t. It was concluded and recommended that SPM should strive to go for OCE_{OP}HH bleaching sequence as it is relatively cheaper to implement compared to ODE_pDP. Also, it generates less chlorinated organics reflected as COD compared to CEHH.

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INTRODUCTION

Southern paper mill is the largest paper mill in Tanzania with rated capacity of 60,000 tons of paper per year. The major raw material is wood. Long fibres are obtained from softwood (pine) and short fibres are obtained from hardwood (eucalyptus). During cooking, wood chips are mixed in the ratio of 80% softwood and 20% eucalyptus along with calculated amount of white liquor (NaOH and Na₂S) at elevated temperature of about 170 °C, to produce mixed kraft pulp. This chemical pulp is utilised for making various grades of paper such as sack paper and kraft liners and some of the pulp is bleached with conventional CEHH method to brightness of about 77-78% ISO for making printing and writing papers, also as a reinforcement pulp in newsprint. The maximum brightness attainable by SPM's bleaching sequence is inferior and not good for technical writing and printing grades. Typical brightness for these type of papers is 85% ISO and above, for imported papers brightness reaches up to 90% ISO.

Thus, due to superior properties offered by these imported papers such as brightness, surface strength and printability, most customers are going for imported papers rather than for SPM papers.

THEORETICAL BACKGROUND

Chlorination and Hypochlorite Stages

The conventional multi-stage bleaching sequences involve treatment of pulp with chlorine and its related compounds. These compounds include sodium hypochlorite, NaOCl (H), Chlorine dioxide Cl₂O (D), elemental chlorine gas (C) and alkali extraction (E). Typical combinations are usually CEHH, CEHDED and CEDED.

In bleaching of chemical pulps, one tries to remove lignin left after cooking (residual lignin) and condensed lignin molecules without destroying the pulp strength. Condensed lignin is usually obtained during cooking when the lignin fragments react together to form very stable carbon to carbon bond structures, the phenomena is called "lignin condensation". Bleaching operation also removes colouring materials

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(chromophores, pigments, dyes etc) and thus reducing the light absorption coefficient of pulp.

The reaction of lignin and chlorine during chlorination is primarily an electrophilic attack on the aromatic rings. The point of attack being the ortho and para positions relative to phenol or phenol ether group. The reaction is very fast and can be carried out at room temperature, retention time is usually 30 to 60 minutes. With modern medium consistence technology the temperature can be raised to 50-60 °C and retention time of 15-20 minutes. For unbleached kraft pulps the percentage chlorine charge on pulp is usually 0.22 x kappa number, for sulphite pulps a kappa factor 0.20 is recommended. However, with the current situation of minimizing chlorine utilization, kappa factors of 0.18 and 0.15 for unbleached kraft and sulphite pulps are recommended [1].

The hypochlorite stage is usually regarded as a pulp brightening stage. It involves decolorisation and dissolution of chromophores. Hypochlorite bleaching is very sensitive to pH changes. Below pH of 8-9 serious carbohydrate breakdown occurs resulting to viscosity drop, above pH 10.5 the reaction becomes very slow requiring large retention time [2]. Thus, the required operating range is very small that is 9-10.5. Due to this problem hypochlorite has lost its market and very few modern plants use this chemical for pulp bleaching [1].

The concept of bleaching chemical pulps mainly for printing and writing papers has undergone tremendous changes in the recent years. The great pressure towards these changes is centred on environmental pollution caused by bleaching plants. The chlorination and hypochlorite stages in bleaching have been extensively identified as major sources of polychlorinated phenolic compounds such as dioxin and furan. These compounds have been proved to have genetic effects on marine organisms [3]. These effects thereafter come to human beings. Also, elemental chlorine has been observed to contribute to the ozone layer depletion as it reacts with tropospheric ozone [4].

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Oxygen Delignification

Oxygen delignification has gained importance and widely applied in recent years due to the environmental pressure of reducing chlorine compounds. Technical oxygen stage is carried out just before chlorination at relative high consistency in the range of 25-30% in a pressurised reactor at about 6-8 bars. Temperature is usually 90-120°C and retention time of about 30 to 60 minutes. Medium consistency (10-15%) oxygen stage is also possible where by the MC-mixers are used to disperse the oxygen gas throughout the medium consistency pulp. Oxygen charge is normally in the range of 15-30 kg/ton of pulp (1.5-3.0%), however to achieve effective delignification, oxygen must be present in excess during and at the end of the reaction time. Selectivity of oxygen is rather not good as severe carbohydrate degradation can be observed if only oxygen is employed. To avoid this, magnesium compound such as magnesium sulphate, hydroxide or oxide are charged along with the pulp. Magnesium sulphate is basically used and the charge is always in the vicinity of 0.3%. Also, about 2-3% sodium hydroxide is normally charged so as to control the extent of delignification by reacting the phenolic products obtained during the reaction.

Another advantage of using the oxygen stage is that the wash effluent after this stage can be used in the unbleached pulp washing plant and ultimately fired in the recovery boiler. This consequently reduces the waste water load to the environment and also further recovers some solids (wash loss) from unbleached pulp which would disappear if the pulp would be chlorinated.

Global Efforts to Reduce Chlorinated Compounds.

The global efforts have been to avoid or rather minimize utilization of chlorine based compounds in the bleaching plants. Thus, four basic bleaching strategies have been pointed out to be adopted by pulp and paper industries. These include [4]:

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Reduced Chlorine Consumption (RCC)

In this strategy, mills with little ability of rehabilitating or rebuilding the whole bleaching plant are strongly requested to use oxygen delignification stage prior to the bleaching stages. This reduces the kappa number of pulp for about 50% and thus the chemical consumption on the next stages is considerably reduced. Extended delignification by application of chemical additives such as sodium polysulphide and anthraquinone are also recommended.

Elemental Chlorine Free (ECF)

In this case the chlorination and hypochlorite stages are replaced by chlorine dioxide (Cl_2O). Chlorine dioxide is a powerful bleaching agent and produces less dioxin as compared to hypochlorite and elemental chlorine. However, chlorine dioxide is unstable and has to be prepared and used *in situ*. The problem of instability makes chlorine dioxide decompose even at lower atmospheres and thus cause lesser environmental problem such as acid rain than ozone depletion.

Total Chlorine Free (TCF)

This strategy requires no utilization of any chlorine based compounds in the bleaching process. Thus, for the conventional bleaching plants, this requires complete rebuilding of the bleaching plant. Chemicals which are used in this case include: oxygen (O), ozone (Z), chelating agents (Q) and hydrogen peroxide (P). Typical combination could be : OQZ(EOP)(PO)P. Chelating agents such as EDTA and DTPA are frequently used in these bleaching sequences in order to immobilize and deactivate heavy metal ions and thus prevent the decomposition of hydrogen peroxide.

Total Effluent Free (TEF)

For the case of total chlorine free (TCF) bleaching sequences outlined above, pulp washing is carried out countercurrently and the effluent is ultimately sent to chemical recovery whereby sodium is recovered. Here, the total effluent free (TEF) is achieved. However, the main problem here

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is the accumulation of heavy metal ions (such as Mn^{2+} , Fe^{2+} , Cu^{2+} , etc) in the system. These ions, if not taken out, will tremendously reduce the final brightness of pulp and increase unnecessary chemical consumption.

Also, in order to curb the problem of toxic effluent from conventional bleaching plants, some promising work has been done by Bohmer *et al* [5] to recover chemicals from these effluents rather than disposing them. The major problem here is that these effluents are usually very weak, concentrating them needs a lot of energy. Moreover, corrosion offered by these chlorine based compounds is very high and difficult to handle.

EXPERIMENTAL WORK

Preliminary experiments were conducted in autoclaves to find out the H-factor needed for cooking. Thus, softwood chips (pine) and hardwood chips were mixed in the ratio of 80% to 20% by weight and then 100 g (oven dry) of these mixed chips were taken and then charged in four different autoclaves. The autoclaves were first steamed at 2.5 bar to evacuate the air for 30 minutes. 17.5 % active alkali of white liquor was charged (white liquor concentration 120 g/l active alkali, 32.5 % sulphidity). Chips were then impregnated with white liquor using nitrogen gas at 6 bars for 15 minutes and then cooked at 170 °C using different H-factors. From these preliminary results, a large cook was made in a 50 litres digester and the pulp obtained was used for bleaching experiments. Known amount of pulp was mixed with bleaching chemicals and then transferred into the plastic bag. The plastic bag with its content was placed in a constant water bath for specified retention time as indicated in Table 1. Pulp was then washed thoroughly and pressed ready for another stage. Bleaching sequences investigated include: CEHH, OCEHH, OCE_{OP}HH, and ODE_pDP.

All procedures for bleaching and brightness, viscosity and COD (chemical oxygen demand) determination were according to SCAN standards [6]. Table 1 provides the summary for conditions and parameters maintained during bleaching experiments.

RESULTS AND DISCUSSION

The following results were obtained.

Effect of H-factor on Kappa number

The effect of H-factor on kappa number is presented in Fig.1. It can be noted that as the H-factor increases the kappa number decreases. This is theoretically true, since an increase in H-factor at a constant set cooking temperature such as 170°C, means an increase in cooking time. Increased delignification time will consequently result to increased extent of delignification and thus low kappa number. Thus, one could argue that, cooking at higher H-factors and hence to very low kappa numbers is desirable. However, the risk of destroying pulp strength and viscosity increases, which is unwanted. Hence a compromise between low kappa number and higher pulp strength has to be made and the H-factor will be directly defined. This compromise will primarily depend on the type of raw material (wood or non-wood), pulping process (sulphite, sulphate or modified processes) and end use of the pulp.

Table 1: Parameters maintained during bleaching experiments.

Stage	Chemical charge, wt. %	Temp. °C	Retention time, hr	Consistency %	Final pH
O	NaOH: 2.8 MgSO ₄ : 0.5 Oxygen: 2.0	100	1	12	10.5
C	Chlorine : 0.22k for CEHH.	25	0.5	3	1.5
E	NaOH: 2.8	70	2	12	10
E _{OP}	NaOH: 2.8 Oxygen: 1.5 H ₂ O ₂ : 0.5	70	1	12	10
H1	NaOCl: 2.2	40	3	12	10.5
H2	NaOCl: 1.1	40	3	12	10.5

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Since the target for kappa number at SPM is 30 ± 3 , then Fig.1 suggests that H-factor of 2400 would be sufficient as it gave pulp of kappa number 29 and yield of 48%. However, pulp made in a 50 litres digester at NTH using these preliminary results gave a kappa number of 35 and yield of 48% which is acceptable for further processes such as bleaching.

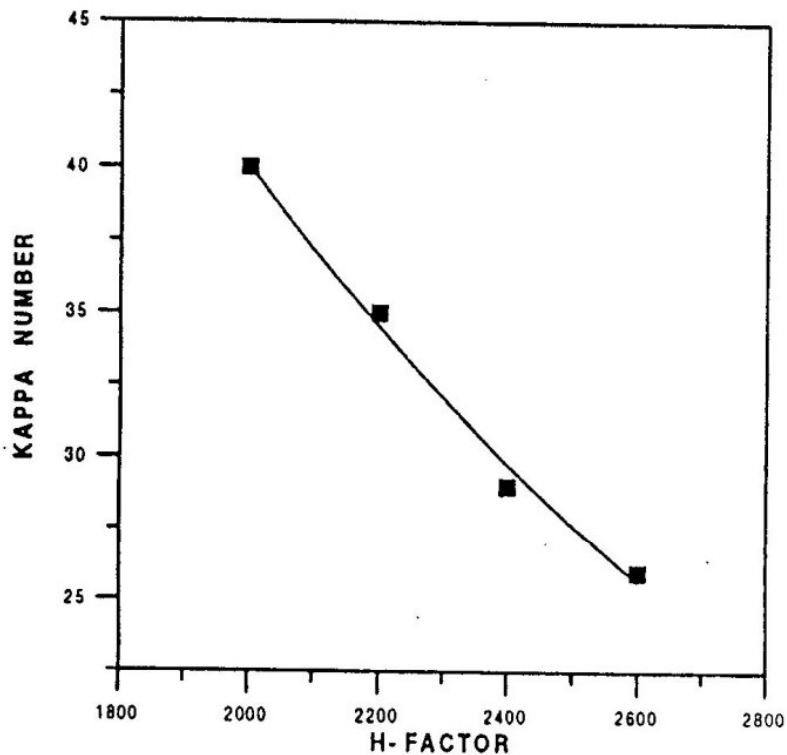


Fig. 1 Kappa number versus H-Factor for mixed cook

Effect of sodium hydroxide charge on Kappa number and viscosity during oxygen delignification

Fig. 2 shows the effect of sodium hydroxide charge on the kappa number and viscosity during oxygen delignification. It can be noted that both kappa number and viscosity decrease gradually as the amount of NaOH increases. To explain these trends, one has to revisit the mechanism for oxygen delignification. Oxygen breaks down the lignin molecule through

formation of free radicals whereby acidic and phenolic groups are formed and they are instantaneously neutralised with NaOH. Thus the trend for kappa number depicted in Fig. 2 suggests that the higher the amount of NaOH charge, the more neutralisation reactions occur and thus the more lignin dissolution, which is reflected by decrease in kappa number. However, lignin dissolution will always occur along with carbohydrate breakdown. Apart from addition of magnesium sulphate to control the rate of carbohydrate breakdown, some cellulose and hemicellulose will be dissolved out which results to decrease in viscosity as noted in Fig.2.

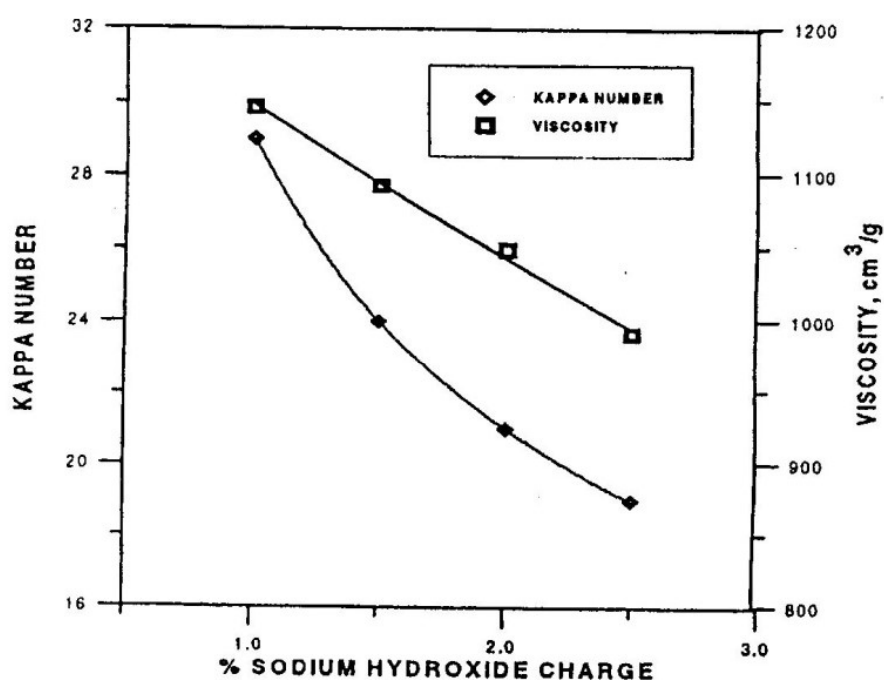


Fig. 2 Effect of sodium hydroxide charge on Kappa number and viscosity during oxygen delignification

Effect of chlorine charge at chlorination stage on brightness and viscosity at hypo 1 stage

The effect of chlorine charge at the chlorination stage on brightness and viscosity are shown in Fig.3 for OCEHH (normal extraction). From Fig.3, it is clear that as the amount of chlorine charge increases, pulp

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brightness increases and viscosity decreases. The reason for these trends could be as follows; high amount of chlorine charged at the chlorination stage results to more dissolution of lignin and chromophores at this stage. Hence, the active chlorine charged at hypo stage will be utilized for dissolving away the residual lignin and coloured compounds. For low amount of chlorine charge at C stage, the active chlorine charged at H1 stage will be used for dissolution of large amount of residual lignin and chromophoric groups in the pulp. Thus, high amount of chlorine charged at C stage will ultimately result to higher brightness and low viscosity compared to that with low amount of chlorine charge at the C stage.

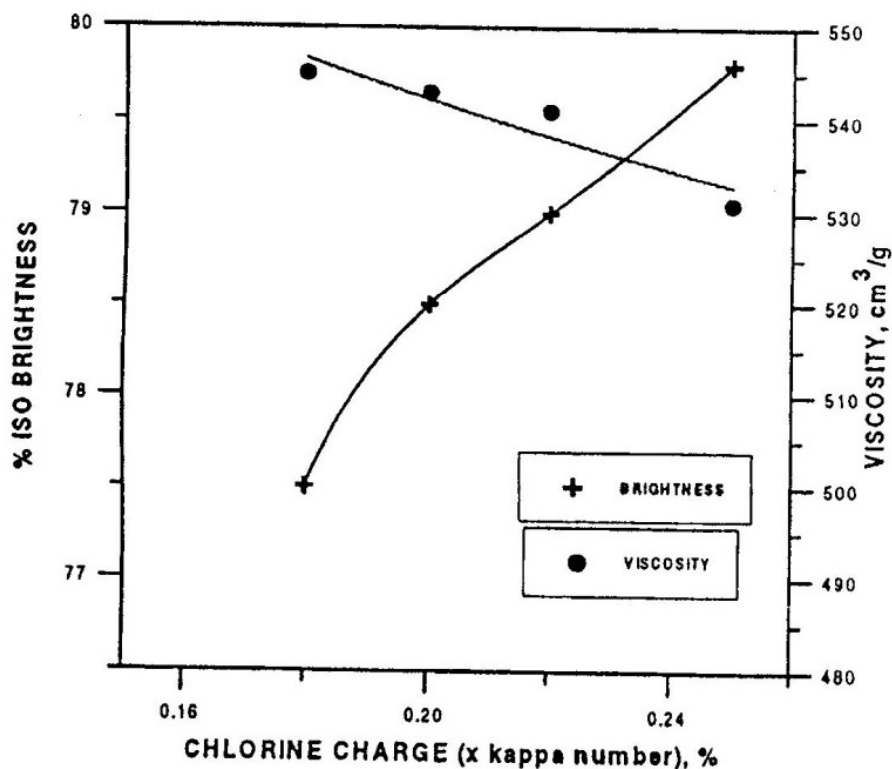


Fig. 3 Effect of chlorine charge at C stage on brightness and viscosity after hypo 1 sate, normal extraction

Fig.4 shows results for pulp with E_{OP} extraction. It is observed that the same trend depicted above is also noted here. The reason could be the

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same as explained above. Further observations reveal that pulp with E_{OP} extraction has relatively higher brightness at H1 compared to that with normal extraction. This indicates that considerable amount of lignin and coloured compounds are removed at the E_{OP} stage and thus pushing the brightness on the upper limit compared to normal extraction. For instance, percentage chlorine charge of 0.22k (recommended in literature) gave brightness of 83.2 % ISO and 79.0% ISO at H1 for E_{OP} and normal extraction respectively. This reflects that E_{OP} is better than normal extraction for brightness improvement. However, the viscosity of the E_{OP} pulp is less than that for normal extraction (470 and 541 cm^3/g respectively). This is mainly due to the selectivity of oxygen whereby some carbohydrate degradation occurs whenever oxygen is used.

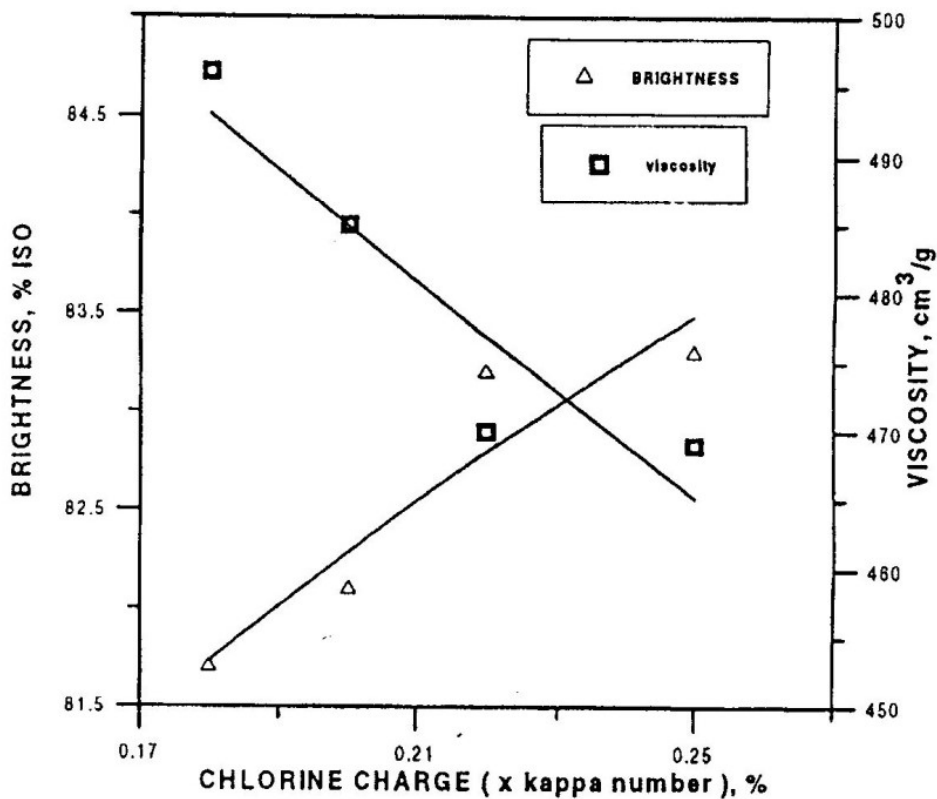


Fig. 4: Effect of chlorine charge at C stage on brightness and viscosity after 1 stage, E_{OP} extraction

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EFFECT OF CHLORINE CHARGE AT THE CHLORINATION STAGE ON BRIGHTNESS AND VISCOSITY AT HYPO 2 STAGE

The effects of chlorine charge at C stage on final brightness and viscosity can be seen from Figs. 5 and 6. For chlorine charge of 0.22k %, Fig.5 gives 81.5% ISO , 442 cm³/g and Fig.6 gives 83.8% ISO, 405 cm³/g. It is clear that both brightness and viscosity are on the advantage side as they are higher than that obtained by SPM (77-78 % ISO). The loss in final viscosity between that of Fig. 5 and that of Fig.6 may be due to oxygen treatment in the second case. However, it is anticipated that pulp with E_{OP} extraction has relatively higher viscosity than that of normal extraction due to viscosity stabilization caused by addition of peroxide. The difference in final brightness for Figs. 5 and 6 is due to the different extraction process used. This reflects the importance of E_{OP} with respect to brightness gain.

It is important to observe that there is a significant viscosity drop from hypo 1 to hypo 2 for both of the pulp samples, whereas the brightness gain is only in the range of 2-3% ISO. This is due to the fact that hypochlorite bleach usually results to very serious carbohydrate degradation.

The effect is more serious when the pH is not controlled closely. The small brightness gain reveals that, increase in brightness especially above 80% ISO becomes more and more expensive. Large amount of chemicals are being used with very little increase in brightness and considerable loss in viscosity. Thus in some cases such as OCE_{OP}HH, the hypo 2 stage is uneconomical. Also, a significant loss in final viscosity is observed as chlorine charge at C stage is increased at a relatively very low brightness increase at final stage. This suggests that charging more than 0.22k % chlorine is undesirable.

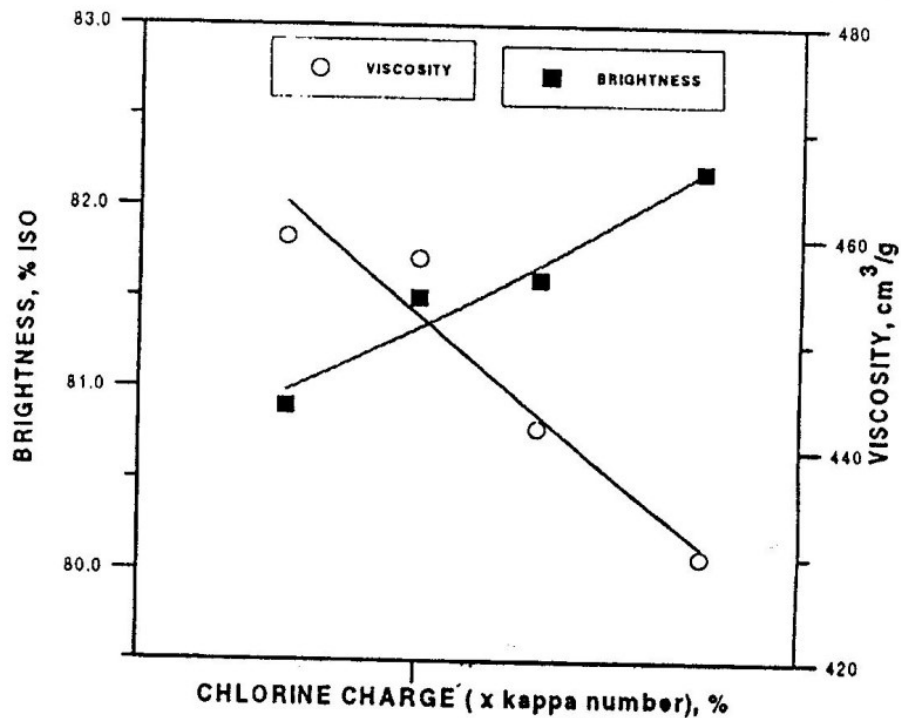


Fig. 5 Effect of chlorine charge at C stage on brightness and viscosity after hypo 2 stage, normal extraction

The final results for CEHH are shown in Table 2. Final brightness of about 85% ISO and viscosity of 461 cm³/g were obtained which are better than the oxygen delignified pulps. SPM uses a kappa factor of 0.18 (i.e 0.18k % active chlorine) at C stage and final brightness obtained is around 77-78% ISO. This work suggests that it is possible to increase the brightness to about 85% ISO with only CEHH if chlorine charge at the C stage is raised to 0.22k. To fulfil this achievement the process parameters such as temperature, pH, chemical charge, consistency, etc, has to be monitored closely. As a possible solution to brightness problem at SPM, an overview of all bleaching parameters at the mill and maintaining them during bleaching process will be advantageous.

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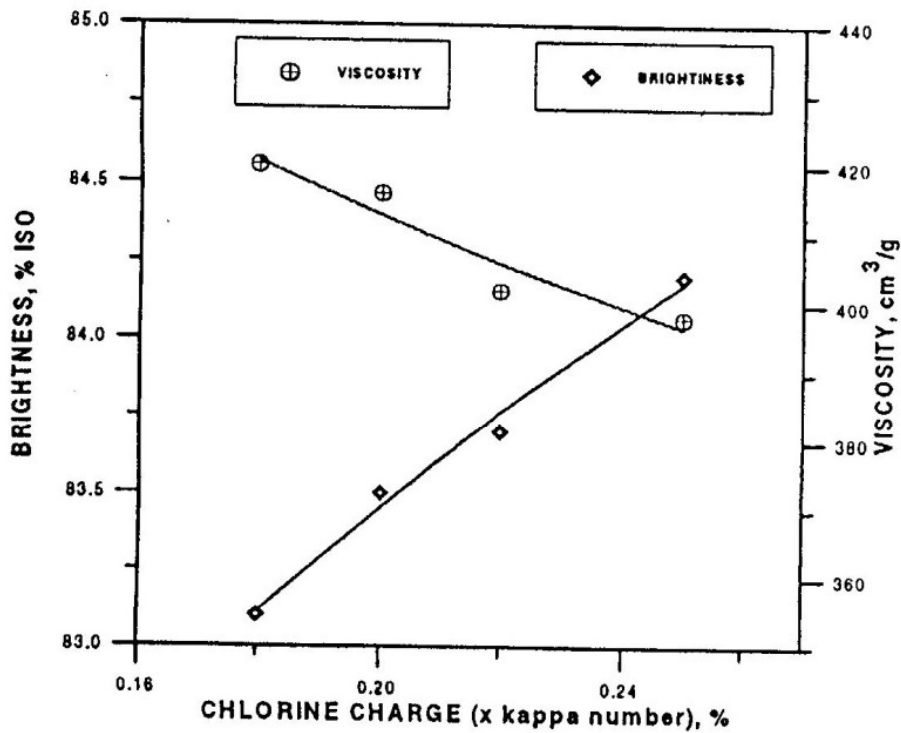


Fig. 6 Effect of chlorine charge at C stage on brightness and viscosity after hypo 2 stage, E_{OP} extraction

Table 2: Results for CEH₁ and OD₁E_PD₂P

Bleaching process	% Cl ₂ charge	HYPO 1		HYPO 2		TOTAL COD, kg/t
		Bright. % ISO	Viscosity, cm ³ /g	Bright. % ISO	Viscosity, cm ³ /g	
CEH ₁ H ₂	0.22k at C	76.1	583	84.8	461	133.1
OD ₁ E _P D ₂ P	0.3k at D ₁	-	-	85.3 at P stage	596 at P stage	101.5

Rate of chlorine consumption at hypo 1 and hypo 2

Fig.7 shows the rate of chlorine consumption at hypo 1 and hypo 2 stages for $OCE_{OP}HH$. It can easily be seen that most of the chlorine charge is consumed in the first 30 minutes and the rest is consumed with very small reaction rate. Final residual chlorine for the E_{OP} extraction was 0.13 (5.9% of original charge) at H 1 and 0.14 (12.7% of the original charge) at H 2. Theoretically, it is required that at least 5-10% of the original chemical charge should be left at H stage to avoid brightness reversion [1,3]. Thus, retention time of 3 hours at hypo 1 and hypo 2 for E_{OP} type of extraction is justified.

Effect of chlorine charge at C stage on total COD

Fig.8 indicates the effect of active chlorine charge at C stage on the total COD of the effluent stream for both OCE_{HH} and $OCE_{OP}HH$. It can be seen that the total COD increases with the increase of chlorine charge. This indicates that application of high amount of chlorine results to high amount of chlorinated organics and adsorbed organic halides (AOX) which eventually constitute to an increased amount of COD in the waste water stream.

Modern plants with reduced amount of chlorine and waste water treatment plants are capable of reducing the total COD to the range of 30-20 kg/t [7]. The amount of COD values observed in Fig.8 are tremendously higher than that indicated above. The elemental chlorine free bleach sequence carried out in this work i.e $OD_1E_P D_2P$ gave a total COD discharge of about 102 kg/t, this value is relatively small compared to values observed in Fig.8. The conventional CE_{HH} according to SPM's bleaching style gave a total COD of 133 kg/t (Table 1.). This shows that if chlorine based bleaching has to continue at SPM, then treatment of the bleach effluent to reduce the total COD is necessary.

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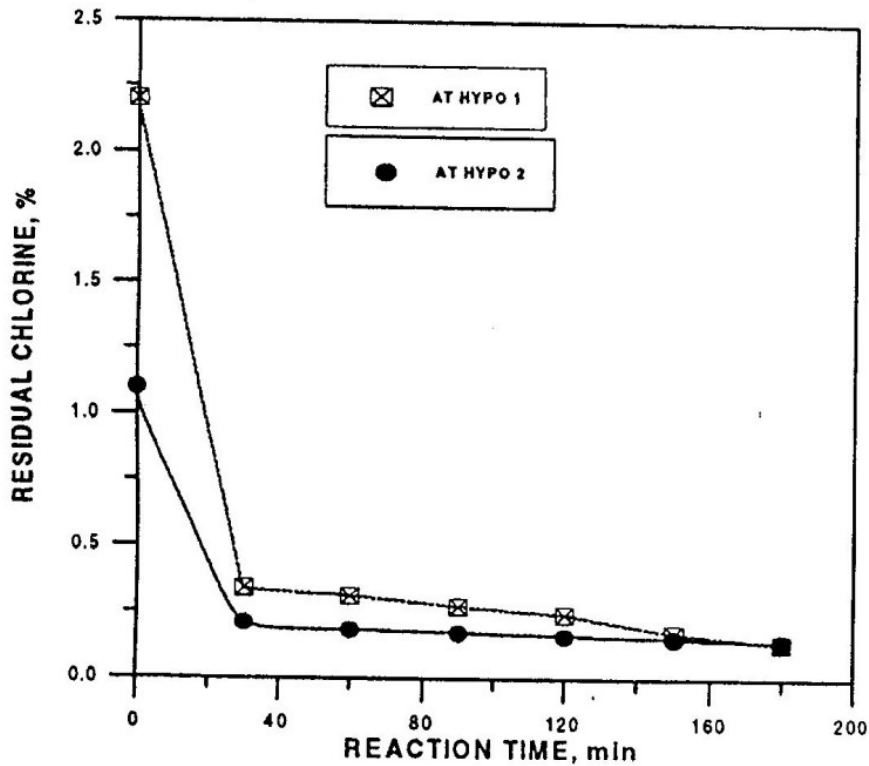


Fig. 7 Rate of chlorine consumption at hypo 1 and hypo 2 for OCE_{OP}HH bleaching sequence

Elemental chlorine free bleaching

The results for elemental chlorine free bleaching sequence (OD₁EPD₂P) are presented in Table.2. Brightness of 85% ISO was obtained with very good viscosity of 596 cm³/g and minimum COD of 102 kg/t. This reveals the superiority of chlorine dioxide and hydrogen peroxide in the bleaching operations. However, as stated earlier, these chemicals are very expensive. On the other hand, pulp with 85% ISO brightness qualifies for making technical grade of writing and printing papers. These paper grades will certainly attract more customers and raise SPM'S sales revenue. Thus, to SPM, this is the typical technology that has to be considered in her future investment plans so as to solve the problem of getting high brightness at low pollution to the environment.

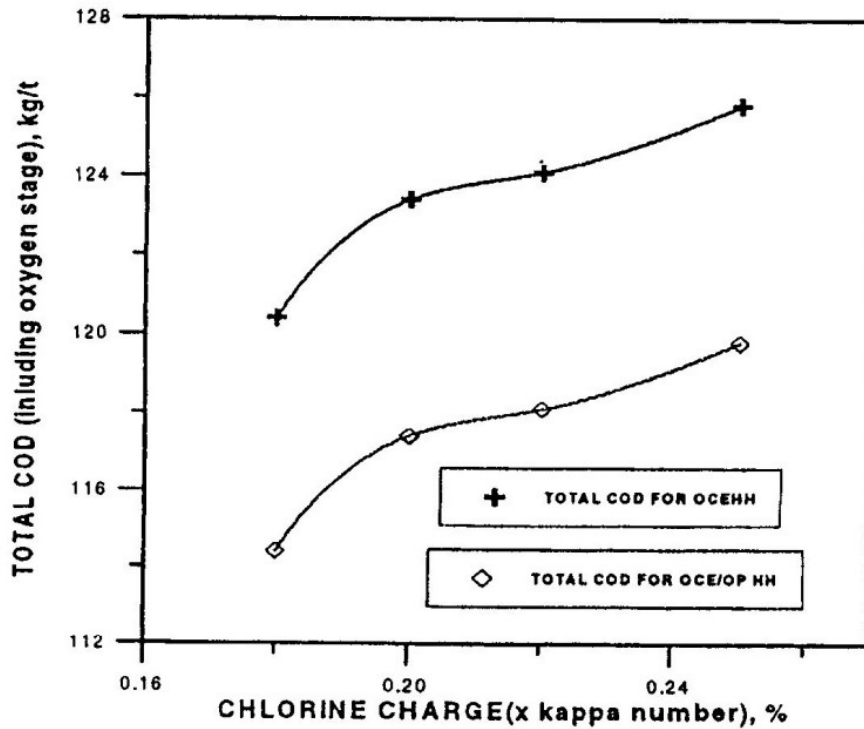


Fig. 8 Effect of chlorine charge on total COD for OCEHH and OCE_{OP}HH sequences

CONCLUSIONS AND RECOMMENDATIONS

Based on this work following conclusions are made:

It is possible to bleach the SPM mixed cook pulp (80% pine and 20% eucalyptus) to brightness in the range of 80-85% ISO using the existing CEHH bleach plant with the same bleaching style provided that chlorine charge at C stage is increased to 0.22k. Reduction of COD for the bleach plant effluent from SPM is an important aspect which the mill has to consider. This may pressurise the mill to go for oxygen delignification in future due to strong international and national regulations on plant

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effluent.

Oxygen delignification reduces the kappa number to almost 40%. This reduction results to low active chlorine consumption on the next stages. Thus, it is recommended that SPM should also think about adopting this technology so as to minimize water pollution caused by chlorine based bleach effluent. The Typical bleach sequence which can be adopted with relatively low capital cost is OCE_{OP}H. This will give a brightness of about 83% ISO. Care has to be taken to avoid carbohydrate degradation by addition of MgSO₄ at all places where oxygen is involved.

If SPM wants to go for fully bleached pulp or paper (i.e greater than 90% ISO) grades then, oxygen, chlorine dioxide and hydrogen peroxide have to be used. The use of these chemicals result to reduced amount of chlorinated organics. However, today the general tendency of modern mills is to bleach using environmentally friendly chemicals i.e oxygen, ozone and hydrogen peroxide so as avoid water pollution. Adoption of this type of technology involves heavy investment, however, long term plans may be put forward to acquire this technology.

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ABBREVIATIONS

AOX:	Adsorbable Organic Halides
CEHH:	Chlorination, Extraction, Hypo stage 1, Hypo stage 2.
COD:	Chemical Oxygen Demand
DTPA:	Diethylene Triamine Pentaacetic Acid
ECF:	Elemental Chlorine Free
EDTA:	Ethylene Diamine Tetraacetic Acid
ISO:	International Standard Organisation

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k:	Kappa number
MC:	Medium Consistency
NORAD:	Norwegian Agency for Development Cooperation
NTH:	Norwegian Technical High School (University)
OCEHH:	Oxygen, Chlorination, Extraction, Hypochlorite stage 1, Hypochlorite stage 2.
OCE _{Op} HH:	Oxygen, Chlorination, Extraction fortified with oxygen and Hydrogen peroxide, Hypochlorite stage 1, Hypochlorite stage 2.
ODE _p DP:	Oxygen, Chlorine Dioxide, Extraction fortified with Hydrogen peroxide, Chlorine Dioxide, Hydrogen Peroxide.
OQZ(EOP)(PO)P:	Oxygen, Chelating agent, Ozone, Extraction fortified with Oxygen and Hydrogen Peroxide, Pressurised Oxygen and Peroxide, normal Peroxide stage.
RCC:	Reduced Chlorine Consumption
SCAN:	Scandinavian Standards Association
SPM:	Southern Paper Mill
TCF:	Total Chlorine Free
TEF:	Total Effluent Free

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