Na_2SO_3/Na_2CO_3 PULPING OF SISAL FIBRES: EFFECTS OF ADDITION OF $MgCl_2$ AND SOAKING OF THE FIBRES IN THE COOKING LIQUOR

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ABSTRACT

Effects of addition of $MgCl_2$ and soaking of the fibres in the cooking liquor for Na_2SO_3/Na_2CO_3 pulping of sisal fibres were investigated. Addition of 6% w/w $MgCl_2$ based on oven dry weight of fibres gave a pulp yield improvement of about 4% compared to pulping without $MgCl_2$. Also, a Kappa number of 16.47, viscosity of 13.26 [mPa.s] and brightness of 78.4% [ISO] were obtained. After beating the pulp, tear index of 19.04 [Nm^2/kg], burst index of 6.41 [$kPam^2/g$] and tensile index of 75.30 [Nm/g] were observed. These values are slightly higher than those obtained for pulping without $MgCl_2$. On soaking the fibres in the cooking liquor for 21 days, pulp viscosity increased to 24.52 [mPa.s], Kappa number decreased to 12.64, and pulp brightness was reduced to 62.10% [ISO].

INTRODUCTION

Sisal fibres are hard fibres extracted from the leaves of *Agave Sisalana*. The plant belongs to the family of monocotyledonous fibre producing plants called *Agavacea*. In this study an attempt has been made to produce chemical pulp from the sisal fibres. This pulp can be used in paper making or as a reinforcement pulp in making various paper qualities or paper boards from wood pulp.

Conventionally, the basic raw material for paper making is wood, but due to increasing consumption of wood, a critical problem of deforestation is emerging world wide. Thus, in an attempt to arrest this situation, serious measures are being taken to minimize wood consumption. In Paper making for instance, non-wood pulping is currently being seriously revisited (Casey, 1980; Goyal, 1988). Pulping of sisal fibres as suggested in this paper also aims at reducing the total dependence on wood in paper making.

Sisal fibres are mainly used in the manufacture of ropes, twines, padding, cordage, sacks and carpets. However, after the invention of synthetic fibres these items could also be made using synthetic fibres such as nylon and poly- propylene. This consequently led to the fall in prices of the natural sisal fibres in the world market. Thus, the present attempt of utilizing sisal fibres in paper making gives an alternative way of marketing the sisal fibres.

THEORY

Pulping

The primary purpose of pulping is to liberate the fibre from the fibrous raw material. The secondary purpose is to impart some physical properties to the fibre required for its ultimate end use. Plant fibres which are cellulosic in nature are usually cemented together with an amorphous, highly branched, three dimensional phenolic polymer called lignin. Lignin has a general effect of inhibiting the fibre from water absorption and fibre swelling (Casey, 1980). This makes the fibre less responsive to mechanical treatment or refining. Thus, in the context of paper making lignin is unwanted material which has to be removed as far as possible.

In order to obtain good strength properties of pulp, the removal of lignin from the fibre matrix should be carried out carefully and selectively, leaving cellulose and hemicellulose unaffected. After selective removal of lignin from the fibrous material, the resulting product is called "pulp" which is regarded as an intermediate product in paper making.

Basically, there are three types of pulping processes, namely; mechanical pulping, chemical pulping and a combination of the two which is also referred to as semi-chemical pulping.

In mechanical pulping, fibres are brushed out from the surface of the fibrous material by the grits of a grinding stone. In this case lignin is practically not removed, but softened as the temperature at the grinding zone reaches about 150 °C (Casey, 1980). High yields are usualiy obtained but the pulp is of very poor strength. In chemical pulping, lignin is attacked carefully using suitable pulping chemicals, and thus splitting up the lignin molecule into small fragments which are eventually removed along with black liquor. Pulp yields are usually low but the pulp obtained is of very good strength. In semi-chemical pulping, the fibrous material is partially treated with pulping chemicals followed by mechanical refining. The pulp obtained has moderate strength and yields are higher compared to chemical pulping.

Conventional chemical pulping processes include: sulphite process and sulphate or kraft process. The sulphite process utilizes a solution of sulphur dioxide in water or sodium sulphite. The sulphite process utilizes a solution of sodium hydroxide, sodium sulphide and sodium carbonate. Chemical pulping always gives high quality pulp as far as pulp strength is concerned.

In chemical pulping many factors may affect the rate of delignification. However, the most important factors are: concentration of the cooking chemicals, fibre to chemical ratio (also called bath ratio), cooking temperature, cooking time, catalyst (or additive), and soaking of the fibrous material in cooking liquor. This paper reports only on the effect of the presence of pulping additive i.e $MgCl_2$ and soaking of the sisal fibres in cooking liquor before cooking commences. The effect of other parameters are currently being studied and will be subsequently published.

Pulp Quality Parameters

To evaluate the pulp quality, following conventional pulp quality parameters have been used:

Pulp yield: The pulp yield can be defined as indicated in equation 1.

$$\%yield = \frac{o.d\ wt\ of\ pulp\ obtained}{o.d\ wt\ of\ fibres\ charged} x100 \tag{1}$$

Kappa number: This is an empirical measure of the degree of delignification and it reflects the amount of residual lignin in pulp. It is an important property in assessing the extent of lignin removal. For chemical pulps and semi-chemical pulps of high yields up to 70%, equations 2 and 3 can be used to evaluate the Kappa number as follows (BSI,1982):

$$\log K = \log(\frac{P}{W}) + 9.3x10^{-4}(P - 50) \tag{2}$$

where:

$$P = \frac{(V_1 - V_2)xC}{0.1} \tag{3}$$

Kappa number is always corrected for the reaction temperature, as follows (BSI,1982):

$$K_c = K[1 + 0.013(25 - \theta)] \tag{4}$$

For kraft or sulphate pulps residual lignin can be estimated using equation 5 below (Britt, 1988)

$$\%lignin\ content = 0.147xK_c \tag{5}$$

Freeness: This is also an empirical measure of degree of fibrillation. It indicates the amount of fines in the pulp sample. It is a very important property in determining the strength properties and pulp drainage on the paper machine.

Brightness: Brightness is a measure of spectral reflectivity of pulp sample. High quality printing papers usually require pulp with very high brightness.

Viscosity: Pulp viscosity indicates the amount of cellulose present and hence it predicts the nature of selective removal of lignin. The higher the pulp viscosity the better is the pulp quality and strength properties.

Strength properties: Strength properties are usually reported as strength index which is defined as a reading in that particular strength property divided by the grammage of the hand sheet under investigation. Strength properties investigated are : tensile, burst and tear.

EXPERIMENTAL

Sisal fibre obtained from sisal estates were chopped into small sizes of about 3 -5 cm. The moisture content was then determined. Cooking chemical was prepared by dissolving 95 g of solid Na_2SO_3 and Na_2CO_3 (each of purity: 96 % w/w) at a ratio of 7:1 with distilled water and making up to one litre. Na_2SO_3/Na_2CO_3 was used as a pulping chemical with the aim of maintaining the good features of the sulphite and sulphate process. These good features include: high brightness and less odour for sulphite process, strong pulp and efficient chemical recovery for sulphate process. Weighed amount of fibres were charged into four autoclaves each 0.85 litres together with calculated amount of cooking chemical at a fibre to chemical ratio of 1:6. (based on oven dry weight of fibres). The autoclaves were mounted on a shaft and then inserted in a 32.0 litres steam chest, the shaft was coupled with a variable speed motor rotating at 28 rpm. The experimental set up is as indicated in Fig. 1 (Ntalikwa, 1994). Saturated steam at 10 bar was used as a heating medium, cooking temperature was maintained at 168 °C and cooking period, 6 hours. Cooking was carried out with increasing amount of $MgCl_2$ addition. Some of the fibres were soaked in the cooking chemical for a considerable period of time and then cooked with fresh chemical. The pulp obtained was subjected to evaluation of pulp quality parameters discussed in 2.2, according to standard TAPPI testing methods [TAPPI, 1980]. The spent liquor was analyzed for pH, total dissolved solids, residual Na_2CO_3 and residual Na_2SO_3.

RESULTS AND DISCUSSION

Fig. 2 shows the effect of the amount of $MgCl_2$ added on the yield and Kappa number of the resulting pulp. As the amount of $MgCl_2$ increases, pulp yield increases slightly whereas Kappa number remains constant in the range of 1% to 8 % $MgCl_2$. However, the Kappa number shows a decreasing trend when $MgCl_2$ added is above 8%. This observation suggests that the catalytic effect of $MgCl_2$ in sisal fibre pulping is not noticeable at low additions of about 1-8%. Thus, in this range $MgCl_2$ acts as a pulping additive and not as a catalyst.

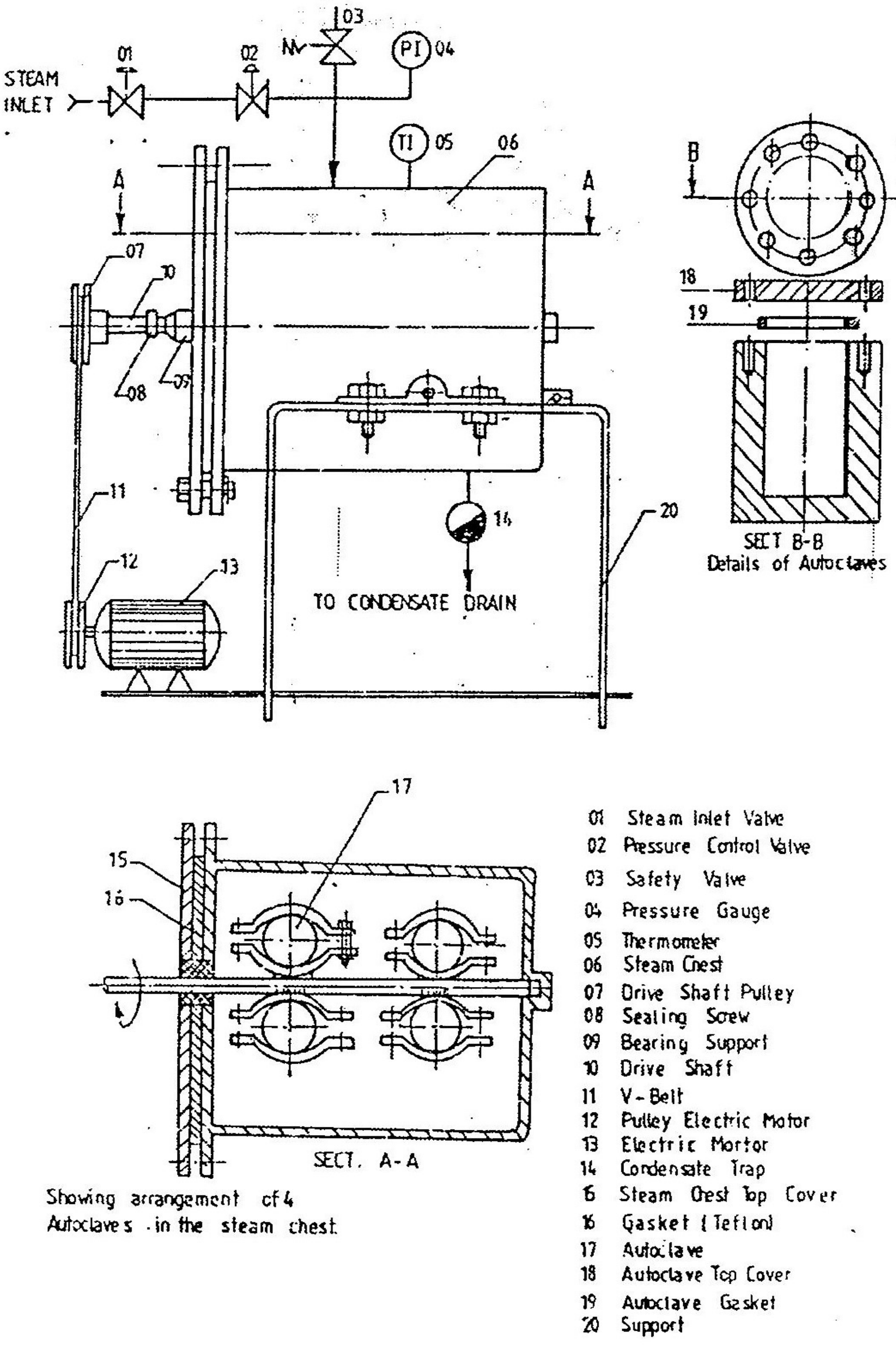

Fig.1 Experimental set up

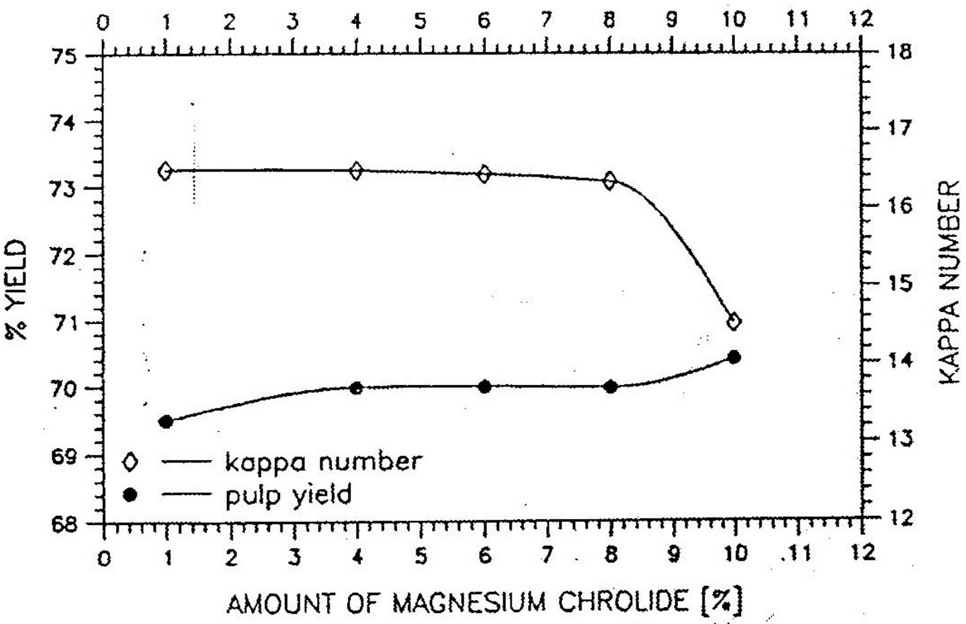

Fig. 2: Pulp yield and Kappa number versus amount of $MgCl_2$

The catalytic effect which is revealed in the range above 8% $MgCl_2$ has a negative effect on brightness and viscosity as depicted in Fig. 3. While the effect on brightness is significant, it is less pronounced on viscosity. Brightness increases with an increasing amount of $MgCl_2$ and reaches a maximum of 78.4% ISO when about 6% w/w $MgCl_2$ has been added. A further addition of $MgCl_2$ results into a decrease in brightness. Also, pulp viscosity remains constant in the range of 1-6%, while further addition of $MgCl_2$ results into a slight decrease in viscosity. Thus, from Fig.3 above 6% w/w $MgCl_2$ brightness and viscosity starts decreasing. This phenomena might be caused by excess $MgCl_2$ which probably reacts further with extractives or lignin derivatives to produce compounds with low spectral reflectivity resulting in lowering of brightness. Also, a slight cellulose degradation starts to occur in this range which results in a slight decrease in viscosity. Since one would like to maintain a high pulp yield as well as high pulp brightness the 6% w/w $MgCl_2$ is taken as the maximum concentration.

Fig. 4 presents the effect of soaking the fibres in cooking liquor on Kappa number and pulp yield. It can be seen from this Figure that, the Kappa number decreases significantly with increasing soaking period. Likewise, pulp yield increases slightly with an increase in soaking period. The increase in both yield and Kappa number suggest that, a slow delignification

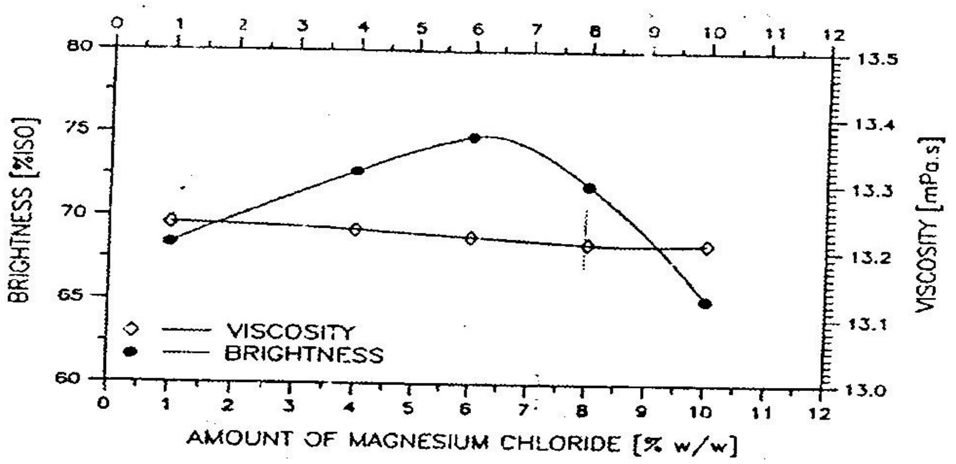

Fig. 3: Brightness and viscosity versus amount of $MgCl_2$

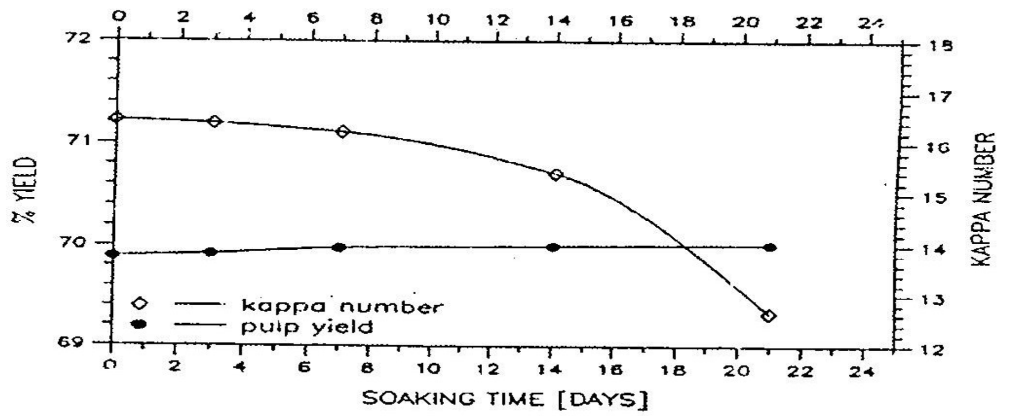

Fig. 4: Pulp yield and Kappa number versus soaking time

reaction does exist even at room temperature. Fig. 5 shows a considerable increase in pulp viscosity whereas pulp brightness decreases with increasing soaking period. This predicts that there is lignin softening effect at room temperature, which is highly selective and dominant whereas cellulose and hemicellulose degradation reactions are not noticeable. This softened lignin can be removed easily on pulping resulting to higher pulp viscosity. The reduction in brightness could be due to prolonged period of contact between fibres and liquor.

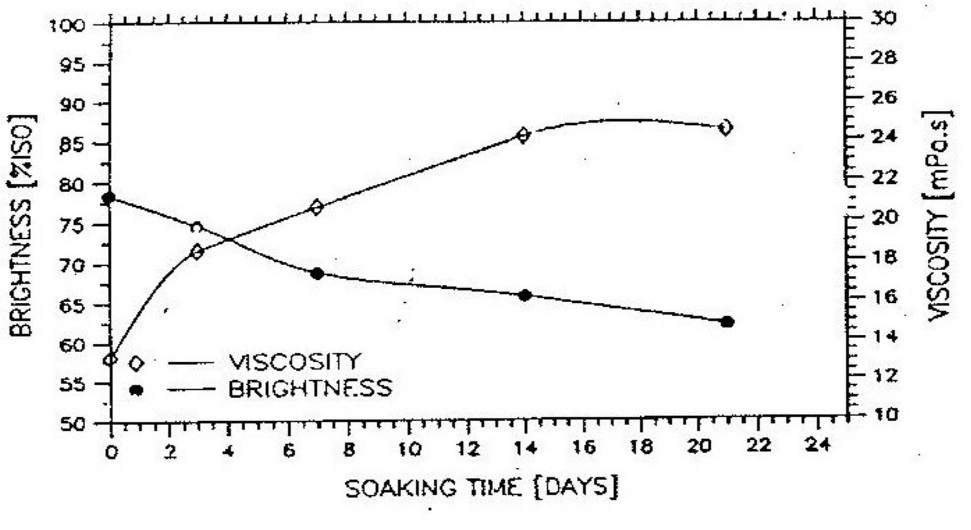

Fig. 5: Brightness and viscosity versus soaking time

Fig. 6 shows the strength properties without addition of magnesium chloride, whereas Fig. 7 shows the same when 6% $MgCl_2$ has been added. It can be noted that maximum strength indices in Fig. 7 are higher than those in Fig. 6. This suggests that addition of $MgCl_2$ also improves the strength properties of pulp. Figures 6 and 7 can be well compared with Fig. 8, which is for pine kraft pulp obtained from Souther Paper Mills (SPM). Further more maximum strength indices revealed in Figures 6 and 7 are slightly higher than those in Fig. 8, this suggests that sisal pulp is rather superior compared to wood pulp. Spent liquor analysis is presented in Table 1.

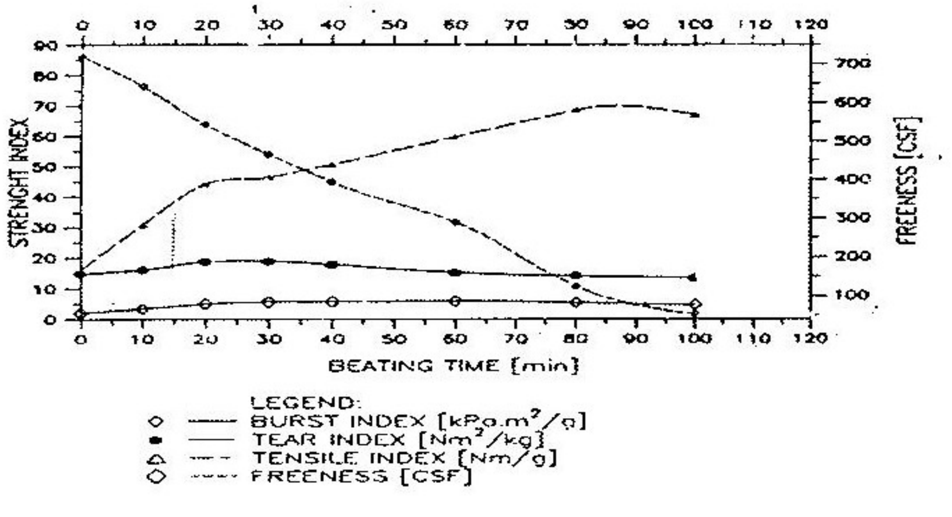

Fig. 6: Strength properties and freeness versus beating time

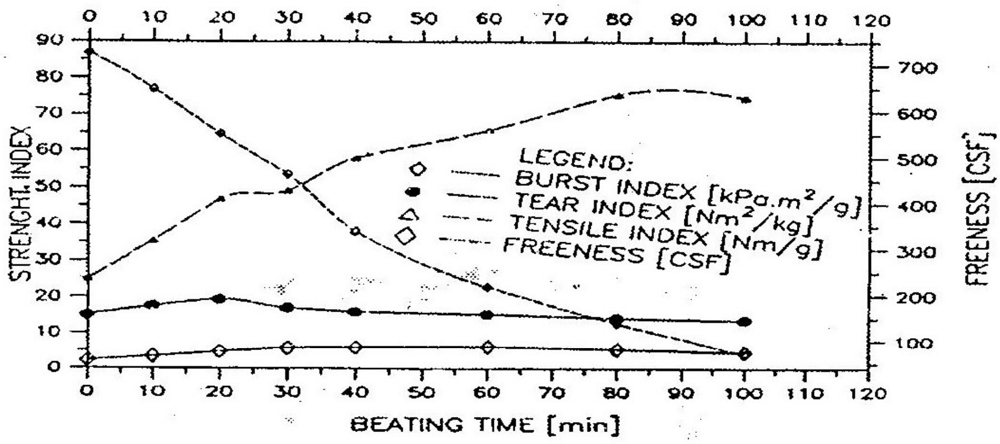

Fig. 7: Strength properties and freeness versus beating time

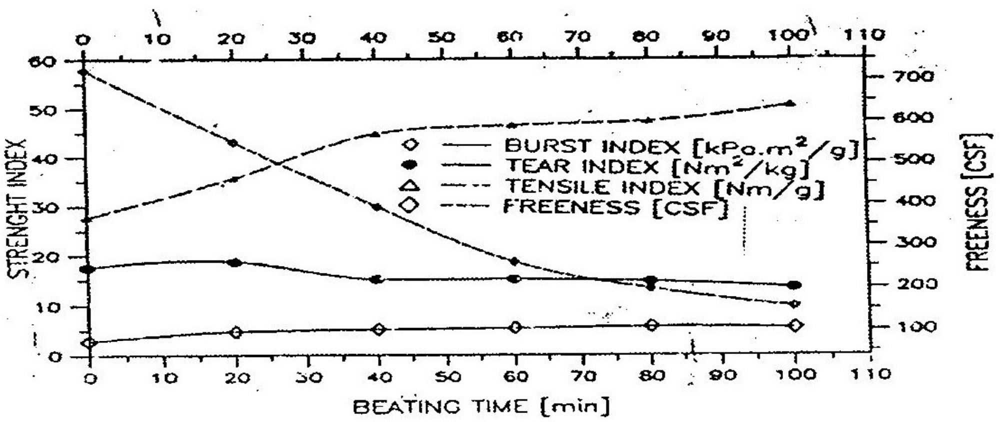

Fig. 8: Strength properties and freeness versus beating time for bleached kraft pulp from SPM

Table 1: Analysis results for spent liquor (black liquor)

Parameter	Value	Goyal et al (1988)
PH	7.10	9.30
Total solids	4.56 %	8.42%
Residual Na_2CO_3	0.85 g/l	0.048 g/l
Residual Na_2SO_3	2.41	-

Jackson (1990) recommends a pH range of 6.5-8.5 for effluent stream to water bodies. The pH reported above suggest that the black liquor meets this requirement. The total solids and pH obtained in this work are lower compared to those obtained by Goyal et al, (1988) from soda pulping of rice straw. However, residual alkali reported by Goyal et al (1988) is lower than that obtained in this work, which may be attributed to the different pulping methods used (i.e soda pulping and Na_2SO_3/Na_2CO_3 pulping).

CONCLUSION

From this study, following conclusions can be made: Pulp brightness increases with increasing amount of $MgCl_2$ added to a maximum of 6% w/w $MgCl_2$, and further addition, brightness decreases. The pulp yield increases slightly, whereas the Kappa number remains constant, when 1-8% w/w $MgCl_2$ is added. Above 8% w/w $MgCl_2$ the Kappa number starts falling. Since one would like to maintain high yield and high brightness a 6% w/w $MgCl_2$ is the best concentration. Soaking of fibres in the cooking liquor increases the pulp viscosity, decreases the Kappa number and increases slightly the pulp yield. However, brightness decreases significantly with an increase in soaking period. This suggests that good pulp properties can be obtained by soaking the fibres in cooking liquor before pulping. The observed decrease in brightness can be resolved by bleaching the pulp using chlorine-free bleach chemicals.

RECOMMENDATION

In order to improve the pulp quality properties and conserve the environment, further studies centred on chlorine free pulping additives have to be done.

NOMENCLATURE

C = Concentration of sodium thiosulphate used in Kappa number determination, mol/l
P = Volume of $KMnO_4$ consumed during Kappa number determination, ml
V_1 = Volume of sodium thiosulphate consumed in blank test, ml
V_2 = Volume of sodium thiosulphate consumed in Kappa number determination, ml

W	=	Oven dry weight of pulp sample, g
θ	=	Reaction temperature in Kappa number determination, °C
K	=	Kappa number
K_c	=	Corrected Kappa number

Abbreviations

BKP	=	Bleached Kraft Pulp
BSI	=	British Standards Institution
CPE	=	Chemical and Process Engineering
CSF	=	Canadian Standard of Freeness
ISO	=	International Standards Organisation
o.d	=	Oven dry weight
SPM	=	Southern Paper Mills
TAPPI	=	Technical Association of Pulp and Paper Industries
UDSM	=	University of Dar es Salaam
wt.	=	Weight

REFERENCES

1. J.P. Casey, Pulp and Paper, Inter-science Publishers, New York. 3rd. ed., 1990
2. Goyal et al, " Pulping of Rice Straw with Soda and Soda Anthraquinone Processes", University of Rookee, U.P. India. 1988.
3. British Standards for Determination of Kappa number (degree of delignification), BSI, London, 1982.
4. K.W. Britt, Hand book of Pulp and Paper Technology, CBS Publishers and contributors, New Delhi, 2nd. ed. 1984.
5. J.W. Ntalikwa, Investigation of Production of Good Quality Paper Pulp from Sisal Fibres, M.Sc. Thesis, CPE, UDSM., 1994
6. TAPPI, Technical Association of Pulp and Paper Industries, Testing Methods, Technology Park, Atlanta, Georgia, 1980.
7. A.T. Jackson, Process Engineering Biotechnology, Open University Press, Buckingham, U.K., 1990
8. J.F. Manfred, The Science of non-wood fibre in pulp and paper making, UNIDO, Viena, Austria, 1988.

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