MODIFIED POLYSULPHIDE (AQ)-PULPING OF SOFTWOOD

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ABSTRACT

A review of the polysulphide (PS) liquor generation methods has been made. By preparing some PS liquors which simulate future possibilities, pilot scale PS-(AQ) pulping studies were conducted. The studies were carried out in a recently acquired pilot plant which can simulate both conventional cooking techniques as well as the latest pulping developments. Two types of pulps were made, type 1 were liner board grade pulps and type 2 were bleachable grade pulps. Type 1 pulps were made by using up to 20% green PS-liquor in conventional PS-AQ pulping. Type 2 pulps were made by adding all PS in the impregnation stage of ITC simulated cooks and applying either NaOH (Soda liquor) or kraft liquor (WL) as the major alkali source.

The pulping experiments to produce liner board grade pulps showed that, addition of PS-green liquor could save up to 9 % polysulphide cooking liquor (orange liquor). The use of PS-green liquor had a negative influence on the yield increase attained by PS-AQ pulping applying 1% PS and 0.05% AQ. The yield gain was reduced from 4.4% to 3.5% (on od. wood).The paper making properties of the PS-AQ liner board pulps were not significantly influenced by PS-green liquor. All PS-AQ pulps had paper properties very similar to liner board grade kraft pulps. The yield increase by conventional PS-AQ pulping using 1% PS and 0.05% AQ was reduced by 50% when the pulping was extended from kappa number 80 to kappa number 30.

The pulping experiments of making bleachable softwood pulps of kappa number 30 showed that, addition of 2.2% PS (on od. wood) in the impregnation stage of simulated ITC cooks when using soda liquor as the major alkali source, needed an AQ charge of 0.05% to show a yield gain of 3% (on od. wood). Splitting the AQ addition between impregnation stage and cooking stage gave no significant change in yield increase or rate of delignification. By using a kraft liquor instead of Soda liquor as the main alkali source, there was a significant increase in the rate of delignification, but it gave only a minor improvement in pulp yield. The paper making properties of the bleachable grade pulps of kappa number 30 level were all very similar, except for the tearing strength which was somewhat reduced by PS-AQ pulping compared to ITC kraft pulping.

INTRODUCTION

The trend in pulping technology for production of bleachable pulps has during the last decade been to extend kraft pulping to rather low kappa number. This is done through new cooking techniques which employ split white liquor addition. The developments in continuous cooking include: MCC, EMCC, ITC and Lo-Solids Pulping. Modern batch cooking process are: SuperBatch, RDH and Enerbatch. The results have been a considerable reduction in consumption of bleaching chemicals, water pollution load as well as improved heat economy. However, extended cooking has drawbacks like an increased wood consumption and higher loads of organic and inorganic chemicals for the chemical recovery system compared to kraft pulping to conventional kappa numbers.

Polysulphide (PS) pulping with and without anthraquinone (AQ) addition in combination with oxygen delignification could be a possibility to remove drawbacks now experienced by extended cooking [1]. The challenges of applying PS-AQ pulping in kraft mills are first, to find the most efficiency and economical method for generating polysulphide from the sulphide present in the chemical recovery cycle. Secondly, to obtain the highest possible yield gain from the PS. Polysulphide pulping has been practised at the Peterson kraft mill in Moss/Norway since 1973. In 1980, Peterson mill started to use AQ (0.35 kg/ton od. pulp) as well.

At present there are several pulp mills in Japan and one mill in Austria applying PS-pulping on permanent basis. The common PS generation technique in use today is a catalytic air oxidation of the sulphide in the white liquor (WL) by the Moxy process [2] or by the Chioda process [3]. Catalytic air oxidation of green liquor may yield a higher PS content than oxidation of WL [4]. Polysulphide generation technology under development is the Paprican process using oxygen added to a kraft mill caustisizer [5] and the Quantum process with a MnO_2 catalyst in an air feeder reactor for WL [6].

Further enhancement of PS-pulping may happen through the development of new chemical recovery system like the Chemrec recovery process [7, 8], which makes separation of sodium and sulphur possible. Separation of Na_2S and Na_2CO_3 through fractional dissolution of the alkaline smelt [9, 10, 11] or by crystallization of Na_2CO_3 from green liquor [11] will make a high sulphidity liquor very suitable for PS generation.

This study was to a higher degree influenced by the recent

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developments in generating polysulphide from the sulphide in green liquor [4] and the possibility of making elementary sulphur from H_2S in a Claus reactor by using H_2S separated from Chemrec recovery system.

The main objective of the study was to search for ways to improve the efficiency of polysulphide-(AQ) pulping through modification of pulping conditions. This was tried through:

- Replacing part of the PS pulping liquor (orange liquor) by PS-green liquor (GPSL) in conventional polysulphide pulping (batch and simulated continuous cooking) of softwood to higher kappa number (liner board pulps).
- Having all the polysulphide and sulphide added in the impregnation phase of the cook followed by simulated ITC cooking with addition of NaOH (Soda liquor) in the cooking phases of making softwood bleachable pulp grades.

The polysulphide and AQ charges used in this study are chosen according to what could be realised in industrial application.

Table 1. Simulation of conventional pulping- Protocol

Chips steaming	Time	30 min.
	Temperature	110 °C
Impregnation.	% EA	16
	Temperature	100 °C
	Time	30 min.
	Pressure	6 bar
	Liquor ratio	4:1
Heating up to cook temperature.		25 min.
Cooking	Temperature	159 °C
	Pressure	7 bar
Pulp washing.		
Inside digester	1. With hot water at 80 °C	
	2. With warm water at 50°C	
After digester	Soaked once in water at 60°C which was drained before fibrillation with refiner .	
After refiner	1. With water at 60°C	
	2. With water at 20°C.	

Pulp yield .

All the pulp was pressed to about 33 % consistency, fluffed, weighed and stored over night. Two samples were oven dried for moisture and the average was used to calculate the yield.

Kappa number.

The kappa numbers were determined according to SCAN-C1:77 procedure.

EXPERIMENTAL

Pilot Plant Pulping With Modified Polysulphide Liquors

Description of the pilot plant.

The pilot plant digester is similar to that described by Tikka and Kovasin [13]. It is a system which has been designed to simulate both the latest developments in the pulping technologies and the conventional methods. It is possible to simulate batch as well as continuous cookings. The digester system has 28 litres volume and carries a basket of 25 litres which can handle up to 4 kg of oven dried chips. The system has a white liquor tank with injection pumps, and three accumulator vessels. To improve the heat transfer the digester heating system includes an electric heater to heat water which is circulated outside the digester in a jacket and back to the heater. The recirculating cooking liquor is passed through a coil in the digester jacket to bring it to the cooking temperature. Heat transfer to the chips is both from the digester wall and from the cooking liquor. The system is automated through a PLC computer interface. Temperature profile can be controlled automatically or manually. The fast response control program enables to maintain a smooth temperature profile. Most of the operations are performed through the computer PLC interface. By entering a target H-factor a program calculates H-factor from the average temperature of digester top and bottom, and displays the

Table 2. High Yield Pulps - Conventional Cooking

SAMPLE ID.	K9	K11	K13	K14
Liquor type	B/C=90/10	B/C=80/20	B	A
Total % EA charged	16	16	16	16
% S° charged	0.97	1.04	0.91	0
Total S charged*	41.6	44.7	39.0	29.3
%AQ charged	0.05	0.05	0.05	0
H-factor	659	659	653	755
Total EA consumed**	143.7	141.9	143	137.5
Kappa	83	83	80	92.5
% Yield	57.5	57	57.4	55
% Yield at kappa 80	57.0	56.5	57.4	53.0
%△ Yield	4.0	3.5	4.4	0.0

A = Kraft WL, B=PS, C=GPSL, K14= reference,
* =kg S /BDMT wood, **= kg NaOH/BDMT wood,
B/C means volume % of B and C (B+C=100).

attained H-factor as well as time left to reach the target H-factor (H-factors were calculated as for kraft pulping in all cases). Commands like running a pump or heaters or opening valves can be made by clicking on the respective items on the computer screen. The system allows for down-flow recirculation, up-flow recirculation, down-flow displacement or up-flow displacement.

Wood raw material.

Spruce fresh chips obtained from Skogn newsprint mill were used. The wet chips were collected from the conveyor to the TMP pulp machines. The feed normally contains about 10 % of the chips as saw mill. The chips were spread to dry on air while the pulping studies continued. The oven dry weight of the chips had varied from about 51 % to about 91 % when the chips become air dry.

Cooking liquors Used.

There were four types of cooking liquors which were used in this study. These liquors are described as standard kraft liquor (WL, Liquor A), polysulphide liquor made from WL (PS, Liquor B), and Soda liquor (Soda, Liquor D). The other liquors were, polysulphide liquor made from kraft green liquor (GPSL, Liquor C) and a high polysulphide liquor made from sodium sulphide solution (HPS, Liquor E). The WL and green liquor (GL) used were obtained from Norske Skog Tofte Industrier.

Liquor B was prepared by dissolving 7 g/l of sulphur powder in Liquor A. Liquor C was made by dissolving 7 g/l of sulphur powder in GL. Liquor E was made by dissolving 22 g of sulphur powder in a liter containing 20 g of Na_2S.

Simulation of conventional pulping.

The steps followed are summarized in Table 1. The cook liquor was pumped from a white liquor tank to the steamed chips by an injection pump. The jacket temperature was maintained at the impregnation temperature of 100 ºC during the injection of the impregnation liquor. The liquor was sprayed on the top of the digester after passing through the coil in the digester jacket. The digester recirculation pump was run in a down-flow recirculation mode. During impregnation a pressure of 6 bars was maintained by nitrogen gas. When the impregnation time was over the system was depressurized to about 3 bars and then the temperature was raised to the cook temperature. Several pulps were made by combination of different liquors as shown in Table 2.

Simulation of ITC pulping.

Simulation of continuous pulping at constant temperature referred as, Iso-Thermal Cooking (ITC), was done in the same pilot plant as described above. The type of the chips used and the steaming procedure were as for conventional pulping simulation. The ITC pulping has four main stages, namely, Impregnation, Concurrent, Countercurrent and Hi-Heat Wash stages.

The impregnation stage was carried out at 110 ºC with recirculation of the cooking liquor in a down flow mode. The effective alkali, EA, charged during impregnation was 14.5 % and the liquor to wood ratio was 3.64. A nitrogen pressure of 6 bars was maintained during the 45 minutes of impregnation. At the end of the impregnation, more liquor amounting to 3.5 % EA was added to the system. The temperature was raised to the cook temperature of 159 ºC within 13 minutes.

The concurrent stage started as the cook temperature was reached. This was a continuous recirculation of the cooking liquor in a down flow mode. At the end of the concurrent stage, extraction of black liquor from the system and injection of fresh cooking liquor started the beginning of countercurrent stage. The liquor injected had a concentration of about 20 g/l as NaOH with an EA of 7.7 %. The time used was the same as for concurrent.

The Hi-heat wash stage, was also an extraction injection process. The time used was 3 times of the Countercurrent time. The liquor used in this stage had a concentration of about 11 g/l as NaOH and with an EA 6.22 %.

At the end of the Hi-heat wash stage, the digester was drained and the pulps were washed twice in the digester as for the conventional cooks. Pulps were then disintegrated and washed by a water jet at a pressure of 3 bars. The pulps were dewatered in a water press to about 37% consistency. The yield and the kappa number of the pulps were determined as explained in Table 1.

RESULTS AND DISCUSSION

Effect on Yield

Higher kappa pulps.

In Table 2 results for higher kappa pulps from modified polysulphide liquors are given. The use of PS liquor containing 0.91 % PS and 0.05% AQ, K13, had a 4.4 % increase in pulp yield compared to the reference, K14. The use of 10 and 20 % GPSL implies a saving of about 4 and 9 % PS liquor usage and an increase of the polysulphide sulphur charge by about 6.6 and 14 % respectively. The yield at kappa 80 shows a decrease in yield with increased use of GPSL (K9 and K11) as compared to the cook with PS liquor, K13. The decrease in yield with increased charge of GPSL is probably caused by increased buffering effect of Na_2CO_3. The yield increase with PS/AQ is similar to that obtained on an industrial scale study [14] in which a charge of 1.2 % PS and 0.05 % AQ had a 4.6 % increase in yield around kappa 60. Prasad et al [15] obtained a 5 % increase in yield at kappa 95 with a 1% PS and 0.04% AQ.

Bleachable grade pulps.

Pulps cooked with various combination of pulping liquors to Bleachable kappa numbers are given in Table 3. These include a conventional cook, K17, and a series of ITC cooks. The ITC cooks included a study of spilt AQ charge. It has been found [14] about 70% of the 0.05% AQ charged was found in the cooking liquor. This means during extraction, most of the AQ will be removed from the digester. The split was therefore divided into 50% (0.025 %AQ) added with impregnation liquor and 50% added slowly with the countercurrent liquor charge, K30. Another cook, K27, had all the AQ added during the impregnation. The results showed similar extent of delignification and yield. A higher consumption of alkali was obtained with the AQ split. Griffin et al [16] had three cases of AQ addition in an MCC process, all in the impregnation, all in the concurrent, and a split between the impregnation and concurrent stages. Their result had indicated a lowest yield at kappa 30 when all the AQ was added in the concurrent stage. It is therefore essential to have some of the AQ added with the impregnation liquor.

Table 3. Bleachable grade pulps- ITC and conventional

Sample ID.	K17	K23	K27	K28	K30	K36	K37
Liquor type	B	D+E	D+E	A+E	D+E	A	A+E
Cook type	conv.	ITC	ITC	ITC	ITC	ITC	ITC
Initial S charged*	46.3	30.2	30.2	61.4	30.2	32.5	62.8
% S^o charged	1.1	2.2	2.2	2.2	2.2	0	2.2
% AQ charged	0.05	0	0.05	0	0.025+	0	0.05
H-factor	1312	1840	1866	1852	1858	1410	1414
Total EA consumed**	163	189	176	190	184	155	174
Kappa	32	43.1	31.9	25	32	32.6	30.1
Yield	50.1	50.1	50.9	48.6	51.2	48.2	51.25
Yield at kappa 30	49.8	48.0	50.6	49.4	50.9	47.8	51.30
%△ Yield	2.00	0.19	2.77	1.64	3.10	0.00	3.50

D= Soda, E= HPS (E= 1 %EA), **= kg NaOH/BDMT wood,

*= kg S /BDMT wood charged impregnation & after impregnation.

+= split feed of 0.025% AQ to impregnation & Countercurrent stages.

Using Soda liquor as source of major alkali without AQ, K23 had about 18 units of kappa number higher than using WL as major source of alkali, K28, at same H-factors. With AQ added in the Soda, K27, the extent of delignification approaches that of WL with better yield. In cook K37, with kraft replacing the Soda liquor as source of alkali, compared to cook K27, it was possible to lower the H-factor by about 450 to achieve the same extent of delignification.

When the ITC cooking yields are compared to the reference ITC kraft cook, K36, the increase in yields are in the following order, Kraft-AQ+HPS>Soda-AQ+HPS>kraft+HPS>Soda+HPS≈kraft. The addition of 2.2 % PS during impregnation gave a yield increase of 3.5% with Kraft-AQ+HPS, 2.8 % with Soda-AQ +HPS, and 1.6% with kraft+HPS, compared to ITC kraft at kappa 30. An MCC pulping study [16] with 0.1% AQ and 0.97% PS had shown yield increase at kappa 30 of 2.3 % with AQ/PS, 1.6 % with AQ and 1% with PS. Jiang [17] added 2 and 3 %PS in EMCC pulping of southern pine with kraft liquor and reported 2 and 3 % increase in yield respectively.

Sulphide and Alkali Profiles - Bleachable Grade

The concentration of hydrogen sulphide in the cooking liquor at various stages of the cook are given in Figure 1. The values were measured as sulphur concentration and were determined by potentiometric titration using the Wilson method [18]. There was a slight increase in sulphide concentration in the Soda+HPS cook, K23, although no sulphide was added after impregnation. There is also an increase with WL+HPS cook, K28, as compared to the WL cook, K36. The increase is caused by the decomposition of polysulphide to sulphide and thiosulphate [19]. The results shows that, with ITC pulping using Soda+HPS, K23, as compared to WL+HPS, K28, and kraft WL cook, K36, we have a much lower sulphide profile in the Soda+HPS. Comparing the values to that for a conventional cooking with PS-AQ, K17, the ITC cooks have lower sulphide at the end of the cook. The effective alkali profile of the same cooks is shown in Figure 2. The alkali profile was similar for all the ITC cooks (only two are shown for clarity). At the end of the cook we have a higher residual alkali for the ITC cooks than the conventional cook. The result shows the usual phenomena of higher initial effect alkali and lower residual alkali for conventional pulping.

Physical Properties

Higher kappa pulps.

Figures 3 and 4 show some of the physical properties of the pulps shown in Table 2. In Figure 3, a relationship between tear index and apparent density of the pulps shows no significant deference between the pulps. Development of strength for the liner board grade with beating is shown in Figure 4 as tensile stiffness index as a function of beating revolutions in a PFI mill. The results shows no significant different. A study [14] on sack paper properties from PS/AQ pulps have also indicated no significant change in paper strength.

Bleachable grade pulps.

The effect of PS and AQ on the strength properties for the Bleachable grade pulps is shown in Figure 5. In this graph tear as a function of apparent density is found to give a linear relationship. Both the conventional PS-AQ, K17, and ITC Soda-AQ+HPS, K27, pulps had same tear strength and about 5 to 10 % decrease in tear compared to the reference ITC kraft cook, K36, at higher density and lower density respectively. A slight decrease in tear strength of the same magnitude caused by addition of PS/AQ has also been reported [20].

In Figure 6, beatability of the bleachable grade pulps is shown by plotting TEA as function of PFI rev. In this case no significant different was obtained between the ITC kraft pulp, K36, conventional PS-AQ pulp, K17, and the Soda-AQ+HPS pulp, K27.

CONCLUSIONS

Liner Board Grade Pulps

Addition of PS-green liquor corresponding to 10 and 20 % of the total cooking liquor charge could save 4 and 9 % of the total caustisized cooking liquor when PS-AQ pulping of softwood to kappa number 80.

However, the addition of PS-green liquor had a negative effect on the yield increase attained by PS-AQ pulping with 0.05 % AQ and 1 % PS (on od. wood). The yield increase compared to a conventional kraft cooking at kappa 80 was decreased from 4.4 to 3.5% (on od. wood) by addition of 20% PS -green liquor.

The PS-green liquor addition had no significant influence on the rate of delignification at a given effective alkali charge. Compared to conventional kraft pulping the rate of delignification was increased by PS-AQ pulping at equal effective alkali charge. By cooking to the same H-factor the kappa numbers of the PS-AQ liner grade pulps would be more than 10 units lower than that of kraft cook.

The beatability as well as the tensile stiffness and tear strength of the PS-AQ pulps with and without PS-green liquor were very similar. Compared to higher kappa number conventional kraft pulp there was no significant change in the paper properties of the PS-AQ pulps.

Bleachable Grade Pulps

Simulation of ITC pulping experiments to kappa 30 with 2.2 % PS (on od. wood) added in the impregnation stage and using NaOH (Soda liquor) or kraft WL as the major alkali source gave results as follows:

- With Soda liquor as major alkali source, it was necessary to add 0.05% AQ to attain a significant yield increase (about 3 % on od. wood) compared to ITC kraft at kappa 30.
- A 50% split AQ charge between impregnation and counter-current stage gave similar delignification rate and yield compared to addition of all the AQ in the impregnation stage.
- Compared to ITC-kraft pulping, conventional PS-AQ pulping with 1% PS and 0.05% AQ improved the yield by about 2 % (on od. wood) at kappa number 30 . The EA alkali consumption in the conventional PS-AQ was about 1% (as NaOH on od. wood) higher.
- The yield increase by conventional PS-AQ pulping over conventional kraft pulping was reduced from about 4.4 % to 2 % (on od. wood) by lowering the kappa number from 80 to 30.
- The physical properties of the conventional PS-AQ pulp (△yield=2%), ITC PS-Soda-AQ pulp (△yield=3%), and the ITC kraft pulp (all around kappa 30 level) were similar in beatability and TEA strength. The tearing strength of the ITC kraft pulps was however slightly higher than that of the PS-AQ pulps.
- ITC PS-AQ pulping consumed more effective alkali than ITC-kraft pulping to kappa number 30, i.e., 17.5 - 18.5% as NaOH compared to 15.5 - 16% as NaOH.
- Comparing ITC kraft +PS-AQ and Soda+PS-AQ, with WL replacing Soda liquor as the major source of alkali, the H-factor could be reduced from 1860 to 1415 for production of a 30 kappa number pulp. This can be explained by the greatly increased sulphidity of the cooking liquor. The yield increase was however minor, from about 3% to 3.5 % (on od. wood).

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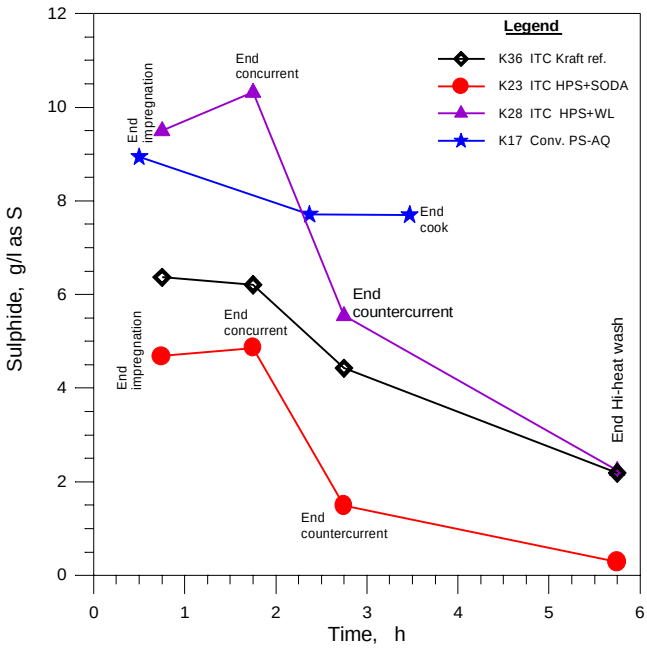

Fig. 1. Sulphide concentration Vs Cooking time

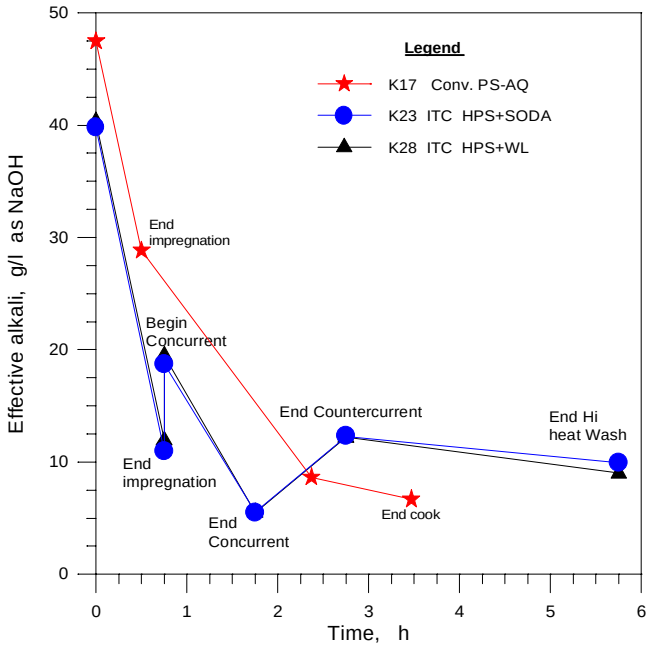

Fig. 2. EA concentration Vs Cooking time

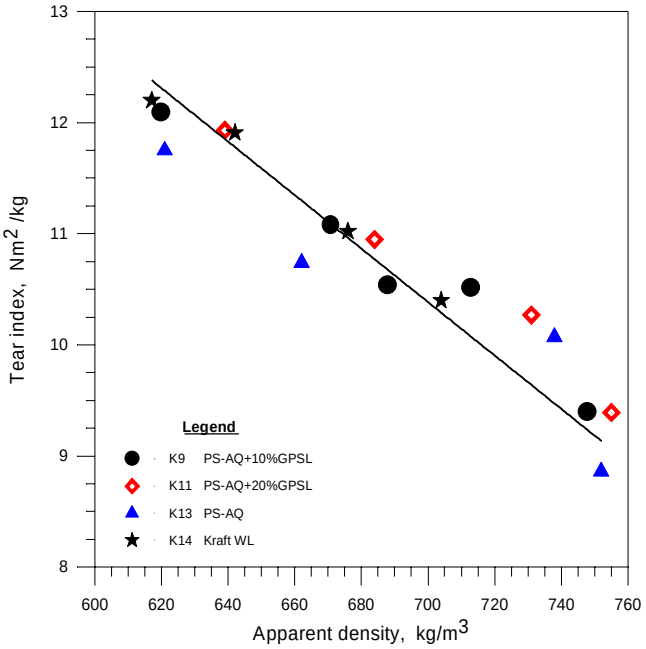

Fig. 3. Tear Vs Apparent density for higher yield pulps

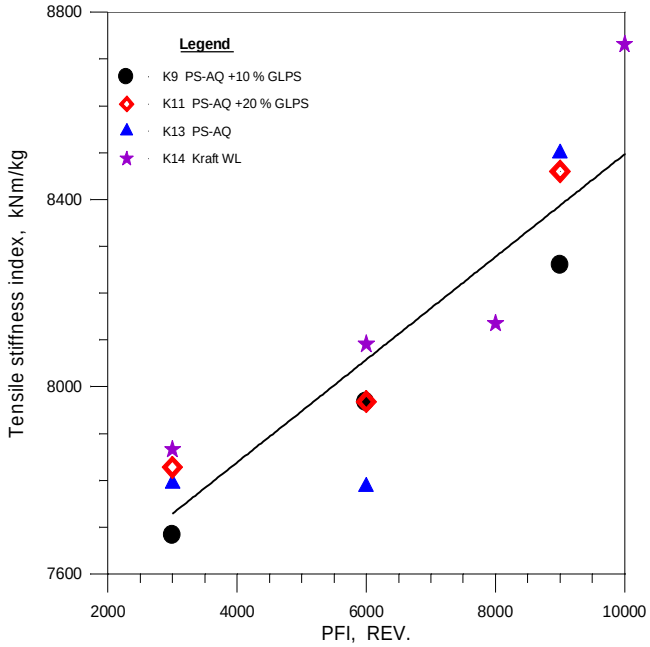

Fig. 4. Tensile stiffness index Vs PFI revolutions for the higher yield pulps.

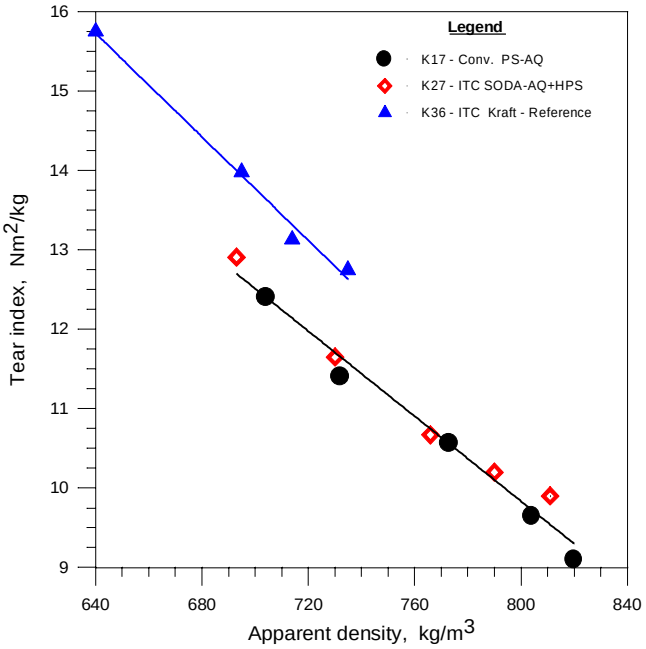

Fig. 5. Tear Vs Apparent density for bleachable pulps

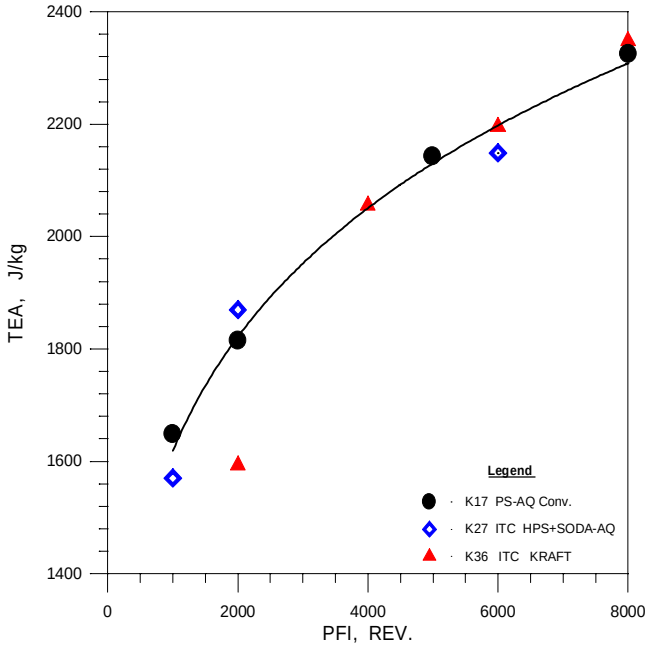

Fig. 6. TEA Vs PFI beating revolutions for the bleachable grade pulps.