RECYCLABILITY OF MODIFIED PS-AQ PULPS AT AROUND KAPPA 30

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Abstract

Bleachable grade pulps prepared from Norwegian softwood spruce chips while simulating possible alternatives of improving pulp yield by application of polysulphide (PS) and anthraquinone (AQ) have been used in this study. The tests for recyclability were performed on hydrogen form pulps. The pulps considered were Iso-Thermal cooking (ITC) kraft pulp which was used as a reference pulp, conventional PS-AQ pulp, soda PS-AQ pulp and kraft PS-AQ pulp. The loss of swelling ability (hornification) of the pulps, was determined by measurement of water retention value (WRV). The results showed that, PS-AQ pulps had similar or slightly better swelling ability after the first cycle of recycling than the reference kraft pulp. That means, hemicellulose retained in the PS-AQ pulps, as a result of yield improvement by the addition of PS and AQ in the cooking liquors, does not appear to induce more hornification on the PS-AQ pulps. The physical properties of handsheet papers made from the pulps, showed similar trend, i.e., either the loss of paper strength of the PS-AQ pulps were similar or less compared to the kraft reference pulp. It was observed that, the loss of fibre swelling takes place more during the process of drying in air than when fibres are exposed to heat after they have been dried.

***Key Words**: PS-AQ, kraft, recycling, WRV and hornification*

INTRODUCTION

The utilization rate of recycled fibre globally is now around 40% [Fjerdingen (1997) and Patrick (1994)]. Before 1980s fibre recycling was a matter of economics but nowdays environmental protection legislation worldwide have necessitated reuse of waste papers [Patrick (1991)]. Furthermore, efforts have been made to increase the pulp cooking yield from the wood during pulping to save the forest, i.e., by minimizing the amount of wood need per tonne of pulp. Use of PS and AQ have gained industrial application as means of improve the pulp yield through carbohydrate stabilization during kraft pulping [Nishijima *et al* (1995), Brooks and Marcinek (1996) and Kleppe (1981)]. Previous studies [Fjerdingen (1997), Berthold and Salmén (1997a), Berthold and Salmén (1997b), Bawden and Kibblewhite (1997), Laine and Stenius (1997), Nazhad and Paszner (1994), Matsuda *et al (1994)*, Paavilainen (1993), Lindström and Carlsson (1982), Scallan and Grignon (1979), Lundberg and Ruvo (1978), Andersson and Fahlin (1956) and Spangenberg (1993)] have looked on how the fibres or paper properties behave on recycling. Several parameters affect the strength of recycled pulp. Among the parameters are pulp never-dried cook yield [Fjerdingen (1997) and Berthold and Salmén (1997a)], the PH or type of counter-ions of acidic groups in virgin pulp [Fjerdingen (1997), Lindström and Carlsson (1982) and Scallan and Grignon 1979)], beating of recycled pulp [Fjerdingen 1997)], drying temperature [Bawden ad Kibblewhite (1997), Nazhad and Paszner (1994), Matsuda *et al* (1994), and Lundberg and Ruvo (1978)], drying stress of the paper [Lundberg and Ruvo (1978)], final dryness of the paper [Lundberg and Ruvo (1978)], and number of drying and rewetting cycles [Spangenberg (1993) and Nazhad and Paszner (1996)].

For chemical pulps, the amount of hemicellulose content affects the mechanical properties of paper [Nazhad and Paszner (1996), Oksanen *et al* (1997), Spiegelberg (1966), and Cao *et al (1997)*]. When paper dries, hornification, i.e., irreversible bonding of cellulose to cellulose making a more crystalline structure takes place [Nazhad and Paszner (1994), Oksanen *et al* (1997), Spiegelberg (1966), and Nazhad and Paszner (1996)]. Because of hornification during drying, recycled chemical fibers have inferior physical properties as they loss their swelling ability [Oksanen *et al* (1997) and Nazhad and Paszner (1996)]. Although hemicellulose was believed to strengthen hornification during drying of chemical pulps, Oksanen *et al* (1997), it has been shown that, by removing part of hemicellulose from chemical pulps the physical properties of the recycled paper deteriorated [Oksanen *et al* (1997) and Spiegelberg (1966)]. Oksanen *et al* (1997), studied paper strength of paper made from pulps whose part of their xylan or glucomannan were selectively removed by enzymes. An increase on sheet density of the never-dried pulps was experienced with hemicellulose removal, but on recycling the pulps, the ones with less hemicellulose (those which hemicellulose were selectively removed) had greatest loss of strength. The improvement of strength of never-dried pulp on hemicellulose removal was attributed to flatten of fibers caused by more cellulose microfibril coalesce. On drying the flatten fibers creates more bonding areas, or in other words increased hornification. Cao *et al* (1997) showed that, increased xylan content had less losses of paper properties on recycling. Spiegelberg (1966), had measured increased hornification by looking on formation of more crystallinity in fibers dried after removing various amounts of hemicellulose. It was revealed that, crystallinity increased because part of cellulose-hemicellulose-cellulose bonds were replaced by cellulose-cellulose bonds.

In this study an emphasis was placed on establishing the effects of recycling on the paper properties of different PS-AQ pulps of about same kappa number, but with different yield improvements. This was based on the assumption that, different modifications in the way the PS is applied is inevitable in the future kraft pulping mills. The development of new recovery system such as Chemrec recovery process [Stigsson (1994)] which makes separation of sulphur and sodium possible, fractional dissolution of alkaline smelt [Teder (1984), Miziguchi and Naito (1978 & 1982), and Henricson *et al* (1975)] to Na_2S and Na_2CO_3, and crystallization of Na_2CO_3 from green liquor [Henricson *et al* (1975)] will allow production of rich and lean sulphidity liquors. High sulphidity liquors will be very suitable for PS generation. Pulps used in this study were made considering the possibilities for the future PS-AQ pulping. Since the effect of recycling is experienced most during the first cycle [Spangenberg (1993)] the study looked on dried once handsheets only.

EXPERIMENTAL WORK

Wood Raw Material

Industrial softwood spruce fresh chips were used. The chips normally contains about 10 % of the chips as saw mill. The chips were air dried to about 90 % oven dry weight. The different pulps were made in a pilot plant digester as described earlier by Minja *et al* (1997) and Tikka and Kovasin (1994). Both batch and isothermal cooking were simulated with different pulping liquors.

Cooking Liquors Used

Four types of cooking liquors were used in this study. These liquors are described as standard kraft liquor (WL, Liquor A), from a Norwegian mill, Norske Skog Tofte Industrier. Liquor B was prepared by dissolving 7 g/l of sulphur powder in polysulphide liquor made from WL (PS, Liquor B), and Soda liquor (Soda, Liquor C) and a high polysulphide liquor made from sodium sulphide solution (HPS, Liquor D). The WL used was obtained Liquor A. Soda liquor was made by dissolving NaOH in water and liquor D was made by dissolving 22 g of sulphur powder in a litre containing 20 g of Na_2S. The kappa number, yield and other parameters for the four different pulps used in this study are given in Table1.

Table 1. Modified PS-AQ pulps from softwood (spruce)

Sample ID.	Ref. Kraft K36	Conv. PS/AQ K17	Soda-PS/AQ K27	Kraft+PS/AQ K37
Liquor type	A	B	C+D	A+D
Cook type	isothermal	conventional	isothermal	isothermal
Initial S charged*	32.5	46.3	30.2	62.8
% S^o charged	0	1.1	2.2	2.2
% AQ charged	0	0.05	0.05	0.05
H-factor	1410	1312	1866	1414
Total EA consumed**	154.9	163.3	176	173.8
Kappa	32.6	32	31.9	30.1
Yield	48.2	50.1	50.9	51.3
Yield at kappa 30	47.8	49.8	50.5	51.2
%Δ Yield	0	2.0	2.8	3.4

A = Kraft WL, B= PS liquor, C= Soda, D= High polysulphide (HPS)
*= kg S /BDMT wood charged impregnation & after impregnation.
**= kg NaOH/BDMT wood,

Procedure for Testing Recyclability of Pulps

The procedure followed was similar to that by Fjerdingen (1997). In this study only the H-form pulps were prepared. This was due to the fact that H-form pulps have been reported to give a greater decrease in physical properties than sodium form pulps on recycling [Fjerdingen (1997) and Lindström and Carlsson (1982)].

About 300 g oven dry weight pulp at a consistency of 30-40 % was disintegrated for 12 minutes using 30 g pulp for each disintegration. The primary fines (-100, i.e, material passing 100 mesh screen) were removed by Bauer McNett fractionation (SCAN-M6;69). The pulp was then treated with 0.05 M HCl at 1% consistency for 2 hrs in order to wash the other ions out of the pulp and form a H-form pulp. The pulp was washed with deionized water until the conductivity of the wash water was about 4 µs/cm. The pulp were beaten in a PFI mill to Schopper-Riegel (°SR) between 22±1 and then disintegrated for 4 min.

A sample was taken for measurement of WRV, which was measured using the proposed SCAN standard procedure [Fjerdingen (1997), Berthold and Salmén (1997), Laine and Stenius (1997), and Lindström and Carlsson (1982)] of 3000 g and 15 minutes. The sample in each sampler was about 0.7 g oven dry (od) pulp. For every measurement 12 parallels were taken and the mean was determined. The handsheet former system was washed with deionized water and the white water tray was half filled with deionized water. The first 18 handsheets were made to create equilibrium of fines in the white water system. About 100 handsheets were made first round. The sheets were dried in an air conditioned room maintained according to SCAN standard (50% RH at 23 °C) over night.

Several hand sheets were used to measure physical properties of paper from virgin pulp. The recycling of air dried hand sheets was done by disintegrating for 4 minutes some of the handsheets to create pulp for making new handsheets and WRV measurement. The rest of the air dried handsheets were heated in an oven with circulating air at 60°C for 2 hrs to simulate the heating effect during drying in paper machines. The sheets from the oven were disintegrated for 4 minutes. The pulp obtained was used for making recycled oven treated handsheets and WRV of the pulp was also determined. In Table 2, PFI beating revolutions as well as °SR values for H-form pulps without -100 fines, and recycled H-form pulps are given.

Table 2. Beating and °SR for the pulps used

Sample Identification	Reference	PS	Soda-PS	Kraft+PS
PFI Rev. H-form pulp no 100 fines	8000	6000	6000	6000
Never dried °SR	21	23	23	22
Dried Once °SR	20	21.5	20.5	19
Dried and post dry treated at 60°C, 2 h. °SR	19	19.5	19.5	18.5

RESULTS AND DISCUSSIONS

The physical properties of the pulps which were used in this study were examined earlier by Minja *et al* (1997) and were found to have minor differences. Water retention value (WRV) measurement is a procedure of measuring the total water uptake by fibres, i.e., fibre swelling ability. The water measured by WRV include bound (non freezing) water, freezing bound water, pore water, and free (surface) water [Berthold and Salmén (1997a)]. As shown in Table 1, the kappa numbers of the pulps used were all around 30. In terms of lignin percentage of pulp, kraft-PS pulp with the highest yield (51.3) and lowest kappa (30.1) will have the least lignin and the reference-kraft pulp with lowest yield (48.2) and highest kappa (32.6) will have the highest lignin content.

Decrease in WRV for the pulps after air drying handsheets and after heat treatment are shown in Figure 1. The pulps have shown a decrease in WRV of about 21-24 % after air drying and of about 25-28 % after heat treatment at 60°C, for 2 h. The reference kraft pulp and kraft-PS pulp had the highest decrease in WRV of about 24% whereas convectional-PS and soda-PS had least reduction in WRV of about 21% due to recycling. Oksanen *et al* (1997), showed that, by removing some xylan or glucomannan, the pulps suffered more

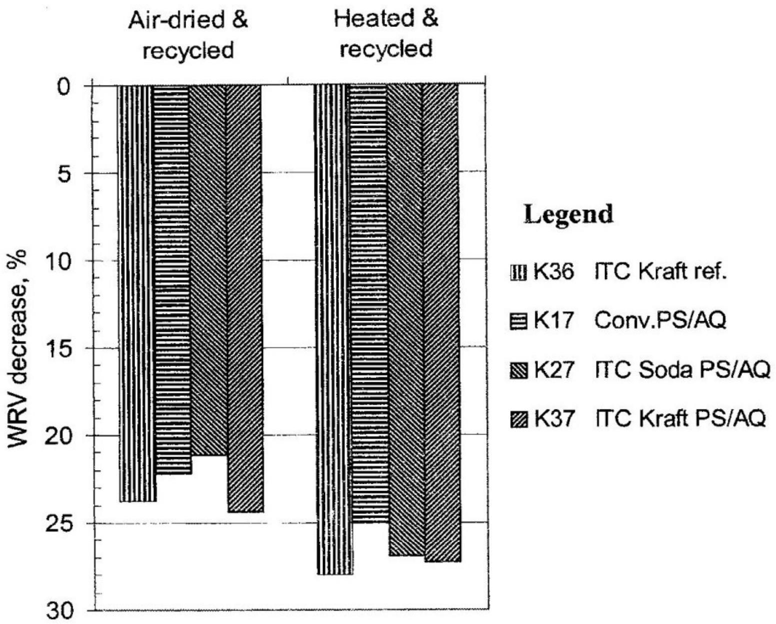

Figure1. The decrease in water retention value (WRV) of recycled pulps compared to their respective undried H-form pulps

loss in WRV. Fjerdingen (1997), had worked with a pulp of kappa number 35 and a yield of 47 %. His results showed a reduction in WRV of 24% for handsheets dried in air conditioned room and 28 % for sheets from an experimental paper machine. The results obtained by Fjerdingen (1997), are similar to results obtained in this study as the heating of the handsheets in oven seemed to simulate the heat treatment received in the paper machine. Effect of heating on the WRV is small compared to the effect of drying, i.e., a further change of about 3-4 % during heat treatment as compared to 21-24% during drying in air. Although the temperature used was not as high as the one encountered in the paper machine, the 2 hour treatment time used was considered long enough to educe the heating effect experience for a shorter time in paper machine. Earlier results by Lundberg and Ruvo (1978), who studied the effect of temperature by drying at 20, 60 and 120°C had shown a much smaller further reduction in WRV when comparing drying at 20 and 120°C. It can therefore be concluded that, swelling ability of pulp is decreased (or hornification increases) more by drying than by heating. Matsuda *et al* (1994), used X-ray diffraction technique to look for a change from non-crystalline to crystalline structure in the cellulose chain during heat treatment of paper. They did not observe any such change and therefore they concluded that the decrease in WRV during thermal treatment was caused by formation of hydrogen bonds in non-crystalline parts of cellulose and hemicellulose. Although pulps with higher hemicellulose may have better WRV, the small differences obtained are not sufficient to make conclusions about the pulps without looking on the sheet properties.

The effect of recycling on paper properties are shown in Figures 2 to 7 as % decrease/increase compared to the properties of the papers made of H-form virgin pulp. The tensile index showed a reduction of between 12-17 % among the different pulps, when the hand sheets were dried in air and about 19-24% after a post air drying heat treatment at 60°C for 2 h (Figure 2). The reference-kraft pulp which has lowest yield showed the greatest

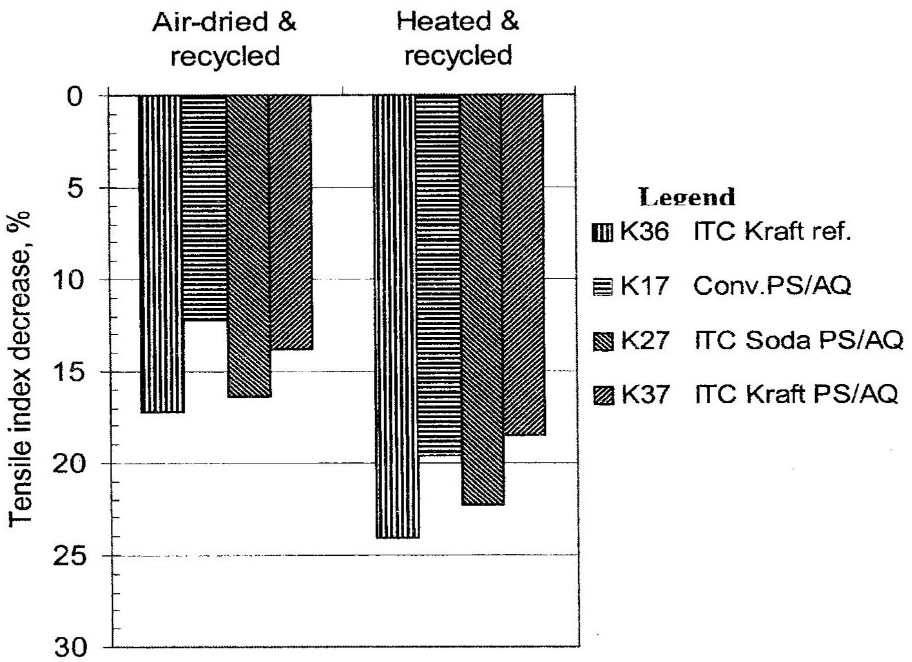

Figure 2. The decrease in tensile index of the papers made from recycled pulps compared to papers made from respective undried H-from pulps.

reduction in tensile strength both after air drying and after heating (Figure 2). Kraft-PS pulp, with highest yield, had the least reduction in tensile strength after heat treatment. The results are similar with that observed by Oksanen *et al* (1997) and Cao *et al* (1997) who have shown less reduction of tensile strength with increasing hemicellulose content in pulp.

Nazhad and Paszner (1994), had looked on effect of temperature on paper recycling. They found that, the process of water removal (drying) caused more damage than heating and consequently concluded that, the main cause for change in paper properties was due to drying and not heat treatment. Like in the WRV measurements, the decrease in tensile index occurred more during drying than during the period of heat treatment.

The reduction in apparent density, was less severe as compared to reduction in tensile index (Figure 3). The changes were between 6.5-8% variation between the pulps on air drying and up to 9-10% reduction on further heat treatment. For air dried sheets only, both

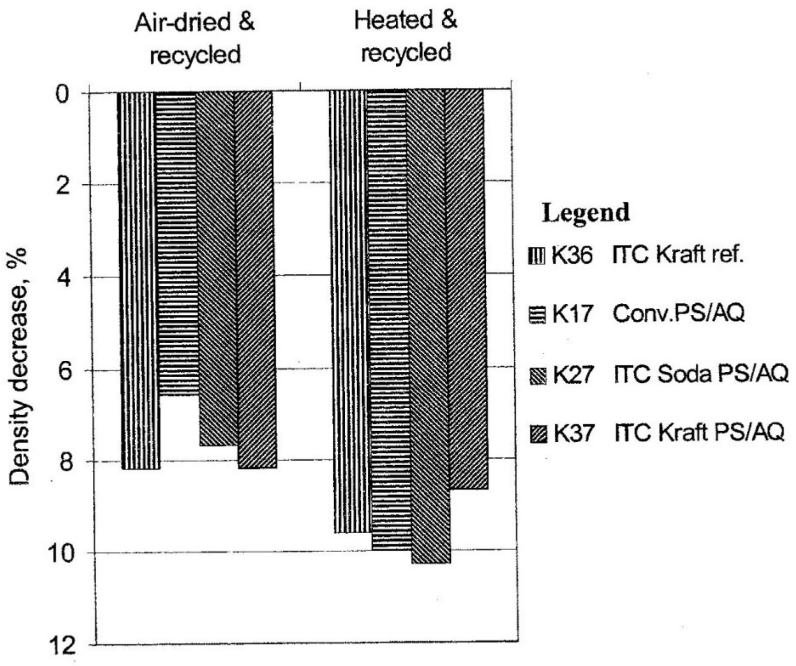

Figure 3. The % decrease in apparent paper density of papers made from recycled pulps compared to that of papers made from the respective undried H-form pulps

reference and kraft-PS had slightly higher loss of apparent density, but after heat treatment conventional PS and soda-PS attained more loss in apparent density. It has been shown that, the apparent density is one of the least changed physical property [Spangenberg (1993)]. Other findings by Fjerdingen (1997) have shown 11% and 12% decrease in apparent density when recycling hand sheets dried at 23°C and papers from experimental paper machine respectively.

The most affected paper property was tensile energy absorption (TEA), where an average decrease of 33 % and 41% were observed for the two cases of air and post air heat treatment respectively (Figure 4). The reference kraft pulp showed the greatest loss both on air drying and after heat treatment. The conventional-PS pulp showed the least loss on TEA both after drying on air and after heat treatment. Fjerdingen (1997), reported a loss of 37% both for

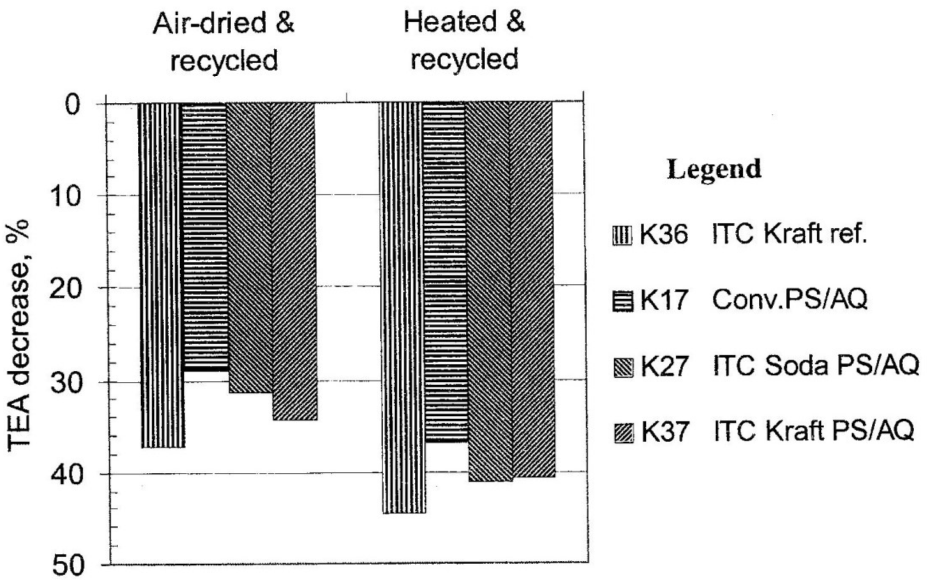

Figure 4. The decrease in TEA of the papers made from recycled pulps compared to that of papers made from the respective undried H-form pulps

papers from experimental paper machine and hand sheets made papers. It seems both hemicellulose composition of the pulp and the type of the cook influence paper properties during recycling. For example, for the TEA, the reference-kraft pulp with the lowest yield, had highest loss of TEA. But kraft-PS pulp, with the highest yield lost more TEA on recycling than conventional-PS pulp which had less yield. The differences may be accounted by a difference in xylan and glucomannan contents of the pulps as has been shown by Oksanen *et al* (1997). Conventional cooking, where no cooking liquors are extracted during the cook, allows precipitation of xylan dissolved in the black liquor to the pulp.

Figure 5 shows the effect of recycling on the pulps' tensile stiffness index. The tensile stiffness is defined as, the initial slope of the load-elongation curve of a paper strip with a

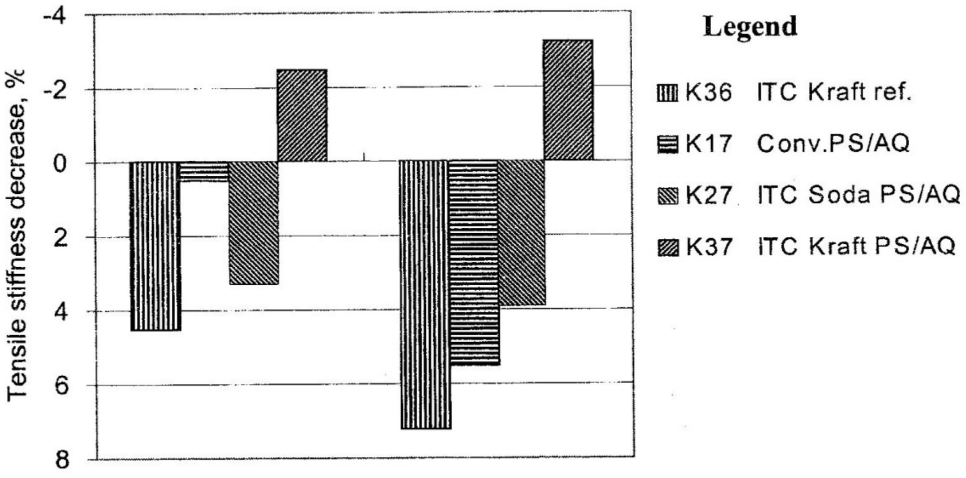

Figure 5. The decrease/increase in the tensile stiffness of the paper made from recycled pulps compared to paper made from the respective undried H- form pulps

given basis weight [Lobben (1975) and Giertz (1964)]. The results show a most decrease in tensile stiffness for the reference kraft pulp and less decrease by conventional-PS and soda-PS pulps, but a slight increase for kraft-PS pulp. The average of 4 and 6.5% decrease in stiffness after air drying and post air heating respectively is smaller compared to that observed by Fjerdingen (1997) who obtained a 9% decrease for air dried handsheets and 23% for papers from experimental paper machine. An increase in stiffness during recycling of paper has also been reported by Spangenberg (1993). A higher tensile stiffness may lead to lower percentage elongation to rupture. The rupture elongation of the pulps are shown in Figure 6. The decrease in rupture elongation for the reference kraft pulp is almost the same to the kraft-PS pulp, but kraft-PS pulp had an increase in tensile stiffness while the reference-kraft pulp had the largest loss of tensile stiffness. Both conventional-PS pulp and soda-PS pulp had less loss of both tensile stiffness index and percentage rupture elongation compared to the reference-kraft pulp.

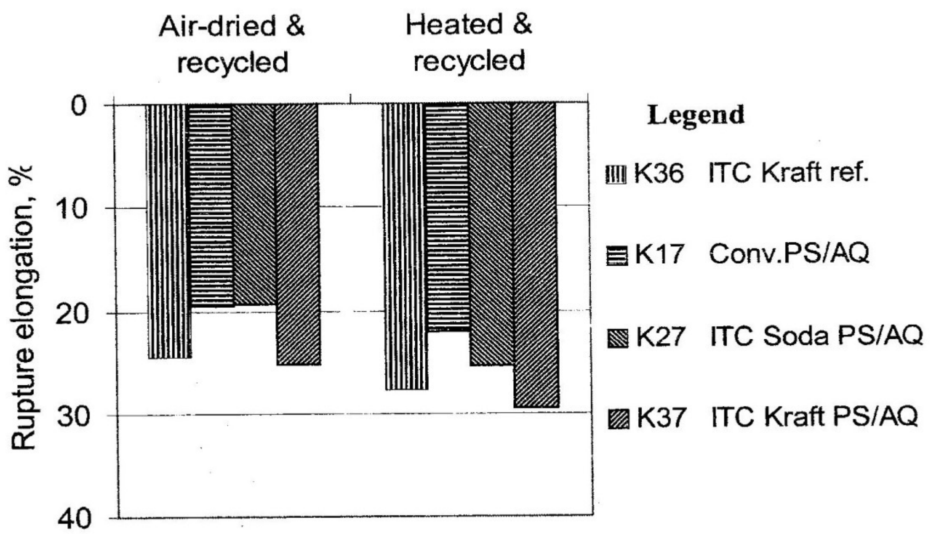

Figure 6. The decrease in rupture elongation of paper made from recycled pulps compared to paper made from the respective undried H-form pulps

Figure 7 shows effect of recycling on tear index. There was about 24% increase in tear after air drying and up to about 35% after post air drying. The increase in tear has been reported

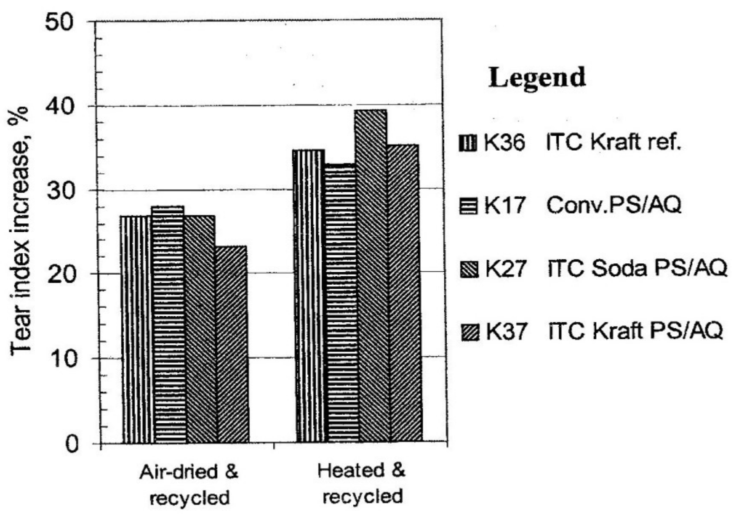

Figure 7. The increase in tear index of the papers made from recycled pulps compared to that of the papers made from the respective undried H-form pulps

by Nazhad and Paszner (1994), Fjerdingen (1997) and Spangenberg (1993). Figure 8 shows the development of strength through beating of the pulps measured as a function of increasing apparent paper density. In other words, it shows the development of fibre bonding (increased apparent paper density) as a result of pulp beating to various °SR, leading to a decreased tear

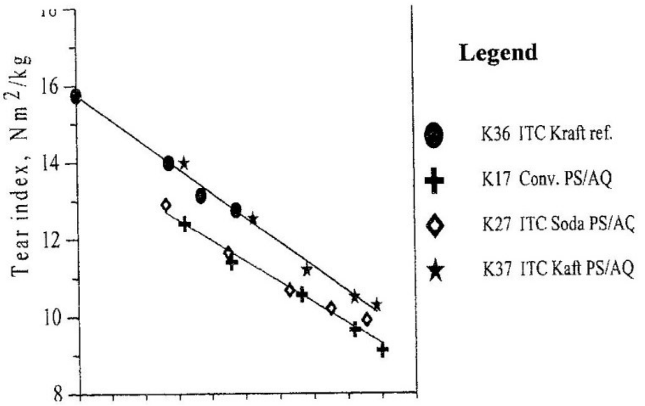

Figure 8. Tear index as a function of apparent paper density for pulp grades made from various cooking liquors to around kappa number 30

strength. Figure 9 shows the effect of recycling on pulps which were beaten and recycled without beating. The figure refers to apparent densities of the four pulps at three stages, i.e., never dried, air dried and heat treated. Recycling of fibres lead to the distortion on fibre bonding resulting into a decreased paper apparent density and increased tear strength. It is interesting to note that, the high tear at a given apparent density for the reference-kraft pulp and kraft-PS before recycling were not changed by recycling.

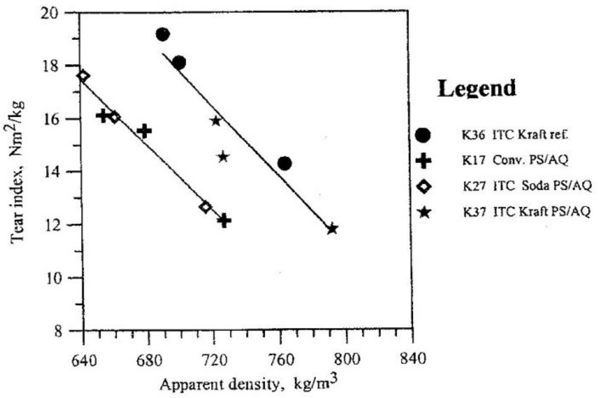

Figure 9. Increase in tear index as function of decreasing apparent paper density caused by recycling the H-form pulps after air drying and then after a combination of air drying and heat treatment.

CONCLUSIONS

The use of PS and AQ have shown an improvement on the re-swelling or less hornification of the pulps. The paper properties have indicated some improvement when pulping with PS-AQ as compared to reference-kraft pulp as follows:

- The reference kraft pulp showed the highest loss in WRV both after air drying and heating. Hornification (loss of fiber swelling) is more caused by drying than by heating.
- Tensile strength loss was most experienced by the reference-kraft pulp while kraft-PS had the least loss of tensile strength. Soda-PS though with higher yield than conventional-PS pulp had higher loss of tensile strength than conventional-PS pulp.
- The loss of apparent density was of less magnitude with soda-PS showing the highest loss after heat treatment.
- Tensile energy absorption, TEA, was the most affected paper property. The reference-kraft pulp showed the highest loss of TEA both on air drying and after heating. Conventional-PS pulp showed the least loss of TEA on both cases of air drying and heating.
- Kraft-PS pulp showed increased tensile stiffness index while the reference-kraft and other pulps showed a decrease in tensile stiffness index. The reference-kraft pulp showed the greatest loss.
- All pulps showed increased tear with soda-PS having the most increase after heating.
- The study has shown some improvement in the recycling properties when comparing PS or PS-AQ pulps with conventional kraft pulp. Thus, the benefit of improved pulp yield which is obtained by addition of PS or PS-AQ in kraft cooking does not bring about detrimental effects on the recycling properties of the pulps.

NOMECLACTURE

AQ	anthraquinone
C-PS	Conventional Polysulphide
BDMT	born dry metric tonne
ITC	Iso-Thermal Cooking
PS	polysulphide
HPS	high polysulphide liquor
PS+S	polysulphide soda cooking
WL	white liquor
WRV	water retention value
°SR	Schopper-Riegel

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