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The influence of lignin and hemicellulose on some important kraft liner pulp properties

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ABSTRACT

How hemicellulose and lignin affect the mechanical and physical properties of pulp is an important matter when producing a paper product. The tensile stiffness is a key parameter for many pulps, contributing e.g. to stacking performance properties of corrugated boxes. Also hygroexpansion is a central parameter, important for e.g. printing operations. Furthermore, anisotropic hygroexpansion giving high stress concentrations in fibre-fibre joints, is also one of the explanations trying to describe mechano-sorptive creep behaviour, often claimed to be the most critical properties of corrugated board mediums.

The aim of this study is to investigate the influence of lignin and hemicellulose content on the mechanical and physical properties, i.e. tensile, hygroexpansion, fracture toughness and mechano-sorptive creep properties, of pulp sheets. In order to vary the lignin and hemicellulose content as independently as possible to each other in the softwood kraft liner pulp, two selective ways (and combination of these) were used; chlorite delignification and xylanase treatment.

After the treatments, the chemical compositions of the pulps varied between 3-14% Klason lignin, 69-77% cellulose, 16-21 % hemicellulose and 4-7% xylan content. Among these pulps, decreased lignin content and increased cellulose content tend to be positive to all measured parameters except the fracture toughness properties. For these properties only small increases due to increased cellulose and decreased Klason lignin content of the apparent strength index was indicated but, otherwise was the properties not significantly altered by the differences in chemical composition. The xylan content tends to give less pronounced effects on all measured properties.

Keywords: *hemicellulose, xylan, xylanase, lignin, chlorite delignification, tensile stiffness, dimensional stability, hygroexpansion, mechano-sorptive creep, kraft liner, softwood*

INTRODUCTION

Hemicellulose and lignin affect the mechanical and physical properties of pulp. Even though much is known regarding this subject there are also contradictory statements and questions still remaining to be resolved.

The tensile stiffness is a key parameter for many pulps, contributing e.g. to the bending stiffness. The stiffness of individual fibres has been reported to increase with the pulp yield in the chemical pulp range, i.e. up to a kappa number of about 45. Furthermore, a decreased amount of hemicellulose achieved by making the pulp with the prehydrolysis kraft method instead of the ordinary kraft method also increases the stiffness of individual fibres at comparable kappa numbers (Neagu *et al.* 2006). It has been reported that higher hemicellulose content in pulps with same density gives a higher tensile stiffness (Molin and Teder 2002). Bleached pulps have, in line with these results, been observed to have lower tensile stiffness. This could, however, be due to introduction of micro-compressions (De Grâce and Page 1976) and fibre deformations (Page and Seth 1980) in the fibres introduced during washing and dewatering in between the bleaching stages. Contradictory to these results the stiffness of the individual fibres has also been stated to be approximately the same at different hemicellulose content (Horn 1974; Page *et al.* 1977). It is hence not obvious how lignin and hemicellulose content affect the tensile stiffness.

Hygroexpansion is an important parameter for e.g. printing operations but, anisotropic hygroexpansion in fibre-fibre joints giving stress concentrations, is also one explanation trying to describe mechano-sorptive creep behaviour (Alfthan *et al.* 2002). An increase in hemicellulose content, measured as pentosane content, has been stated to give an increased hygroexpansion (George 1958; Brecht *et al.* 1974). The relationship can, however, be different for softwoods than hardwoods for which the hydroexpansion of the later is claimed not to be as clearly dependent on pentosane content (Young and Rowland 1933). Furthermore, additions of guar gum (Swanson 1950) or cationic starch (Laurell Lyne 1994), has not been shown to significantly improve the hygroexpansion. This implies that the location of the hemicelluloses in the fibre wall can be of importance.

Increased lignin content has been reported to give higher hygroexpansion (Brecht *et al.* 1974). A study where NSSC-pulps of Spruce were made by cooking to different yields followed by refining to 55°SR shows that the hygroexpansion is highest at a yield of about 55% (Brecht and Hildenbrand 1960). This corresponds, however, not only to a high lignin content but, also high values of FSP (fibre saturation point) (Scallan and Tigerström 1992; Andreasson *et al.* 2003), WRV (Ohta *et al.* 1986; Andreasson *et al.* 2003) and pore size distribution (Andreasson *et al.* 2003).

Mechano-sorptive creep stiffness is a key parameter for boxes of corrugated board. The performance regarding this parameter of the boxes of corrugated medium and the liner used has been show to correlate (Henriksson *et al.* 2007). Earlier studies utilizing edgewise compression creep tests in cyclic relative humidity environment, showed a higher creep rate for pulps with higher yield and lignin content (Byrd and Koning 1978; Byrd 1984). However, the number of levels of lignin contents was few and a conformation or rejection of these conclusions would be of interest.

The aim of this study was to gain more knowledge about the influence of lignin and hemicellulose content on the mechanical and physical properties of paper pulps. In order to vary the lignin and hemicellulosecontent as independently as possible, two selective ways, chlorite delignification and xylanase treatment and combination of these were used.

EXPERIMENTAL

Instruments

Gas Chromatography - Flame Ionisation Detector, GC-FID was used to analyse the content of different carbohydrate monomers in the pulps. The analyses were preformed on a Hewlett-Packard 6890 with a BPX 70 column (12m, 0.32 μm I.D. and 0.25 μm film thickness). Hygroexpansion measurements were made with an apparatus developed at STFI previously described elsewhere (Salmén *et al.* 1987) consisting of several pairs of rigid and a movable clamps between which the paper samples are fasten. Weights are put on top of the paper strips as the length is recorded with LVDT-sensors. Mechano-sorptive creep measurements were preformed on the apparatus developed at STFI and described previously (Haraldsson *et al.* 1994).

Materials

The pulp used in this study was an unbleached, never dried, softwood (mixed Scots pine, *Pinus sylvestris* and Norway spruce, *Picea abies*) kraft liner pulp with a kappa number of 76, kindly supplied by Smurfit Kappa Kraftliner, Piteå, Sweden. It was carefully washed with de-ionised water and Escher-Wyss beaten in laboratory to about 30 M°SR before use. The xylanase used, Pulpzyme HC, was a mono-component enzyme supplied by Novozymes, Copenhagen, Denmark. All other chemicals were P.A. grade, supplied by commercial suppliers of chemicals and used as received.

Chlorite delignification

Prior to the treatment the pulp was disintegration with 10 000 revolutions and de-watered on a Büchner-funnel with a steel wire. The filtrate was re-circulated one time to retain fines. The chlorite delignification was then performed using plastic bags in water baths. Three different charges of sodium chlorite were used (based on previous experiments (Ahlgren and Goring 1970; Wedin *et al.* 2005)), where 0.3g to every B.D. gram of pulp corresponds to 100% chlorite. Chlorite delignification with charges of 100%, 50% and 20% (i.e. 0.3, 0.15 and 0.06 g sodium chlorite to every BD gram pulp) were made. The delignifications were preformed in a 0.2 mol/dm^3 sodium acetate pH 4.7 buffer at a consistency of 2.3%. After 1 hour at 70°C in the water bath, the pulps were dewatered on a Büchner-funnel and carefully washed with de-ionized water.

Xylanase treatment

The xylanase treatment was performed on pulp pre-treated in same way as made in the chlorite delignifications and in addition to treatments of reference pulp; also pulp chlorite delignified with a charge of 50% was treated. The treatments were performed in water baths using plastic bags and two different charges of enzymes were used, 500μl and 5μl to every BD gram pulp (equal to 500 and 5 XEU/BD g pulp respectively, according to the activity given by the supplier). The treatments were preformed in a 20 mmol/dm^3 pH 7 phosphate buffer (Na_2HPO_4/NaH_2PO_4) during 2 hours, at 60°C and 3% consistency. After the treatments, the pulps were dewatered on a Büchner-funnel and carefully washed with de-ionized water.

Sheet forming and mechanical/physical testing

Standard methods were used when possible. Schopper Riegler values were determined according to ISO 5267-1:1999. Tap water was used for the stock preparation. In order to be able to compare pulps of similar °SR-values one xylanase treated pulp was PFI-beaten according to ISO 5264-2:2002 after the treatment.

The pulps were disintegrated in tap water according to ISO 5263:1997. Sheets were prepared in a Rapid Köthen equipment according to ISO 5269-2:1998. The sheets were dried at 93°C for 20 minutes and then stored in a climate room for a minimum of 24 hours at 50 %RH and 23°C prior to testing. Grammage and thickness was determined according to ISO 536:1995 and ISO 534:2005 respectively (the thickness was measured on one sheets for all pulps). Tensile strength, tensile stiffness, strain at break and tensile energy absorption were measured according to ISO 1924-3 and fracture toughness according to SCAN P77:95.

The test pieces used both in the hygroexpansion tests and the mechano.sorptive creep tests were cycled without load between 50 and 90%RH at least four times prior to testing in order to release dried in stresses. Hygroexpansion of the sheets was measured according to ISO 8226-1:1994. The test span between the clamps was 120mm, the width of the test piece 15mm and the difference in length was measured between 33 %RH and 66 %RH at 23°C. Mechanosorptive-creep tests were preformed on test pieces with a width of 25mm. The strain values at certain tensile loads were detected after three humidity cycles, starting and ending at 50% RH and reaching up to 90 %RH.

Klason lignin and Carbohydrate analysis

Klason lignin and carbohydrate analysis were performed on the test pieces used in the mechanical testing. The pieces were destructed in a kitchen mixer until wool-like materials were obtained and about 300 mg of each pulp was used for each sample and at least two samples were prepared from each pulp. Klason lignin analysis were preformed using sulphuric acid hydrolyzes at 120°C according to Tappi standard T222-om02.

The mono-saccharides in the acidic filtrate from Klason lignin determination were reduced and acetylated according to a method utilizing 1-methylimidazole as catalyst originally established by Blakeney et al. (1983) but further developed by Englyst and Cummings (1984) and carried out as described by Theander and Westerlund (1986). The samples were thereafter analysed by means of GC-FID.

The mono-carbohydrates contents were re-calculated to polysaccharides according to equation 1-3 valid for coniferous woods and pulps thereof.

$$\text{cellulose} = \text{glucose} - \frac{\text{mannose}}{3.5} \qquad \text{(eq. 1)}$$

$$\text{glucomannan} = \text{mannose} + \frac{\text{mannose}}{3.5} \qquad \text{(eq. 2)}$$

$$\text{xylan} = \text{xylose} \qquad \text{(eq. 3)}$$

Equation 1 and 2 are based on a number of studies on glucomannans from different coniferous woods resulting in ratios between glucose and mannose between 1:3 to 1:4 (Lindberg and Meier 1957).

RESULTS AND DISCUSSION

The pulps

The aim of the study was to compare pulps with different lignin and xylan content, achieved by means of chlorite delignification and xylanase treatment of the same original kraft liner pulp, at about the same °SR values and sheet densities. As seen in Table 2, the °SR-values and sheet densities are similar, while there are differences in lignin and xylan content.

Table 2: Chemical composition, dewatering resistance and sheet densities of the pulps.

Pulp	Klason lignin [%]	Xylan [%]	Hemi-cellulose [%]	Cellulose [%]	Drainage resistance [°SR]	Sheet Density [kg/m^3]
SKK ref	13.3	5.5	17.2	69.5	15.8	640
SKK 20%	12.7	5.6	18.0	69.3	15.0	640
SKK 50%	7.9	6.4	19.2	72.9	15.5	700
SKK 100%	3.1	6.2	20.6	76.3	17.0	740
SKK 5XEU	13.0	4.8	16.8	70.2	15.0	640
SKK 500XEU*	13.9	3.8	15.9	70.2	14.0*	720
SKK 50%5XEU	7.4	5.4	20.8	71.8	-	700
SKK 50%500XEU	7.5	4.2	18.4	74.1	-	720

* after 2000 PFI-revolutions

Tensile properties

Tensile properties are essential for many paper products and one of the most important properties for kraft liner is tensile stiffness. The effect of the chemical composition on the tensile stiffness is shown in Figure 1.

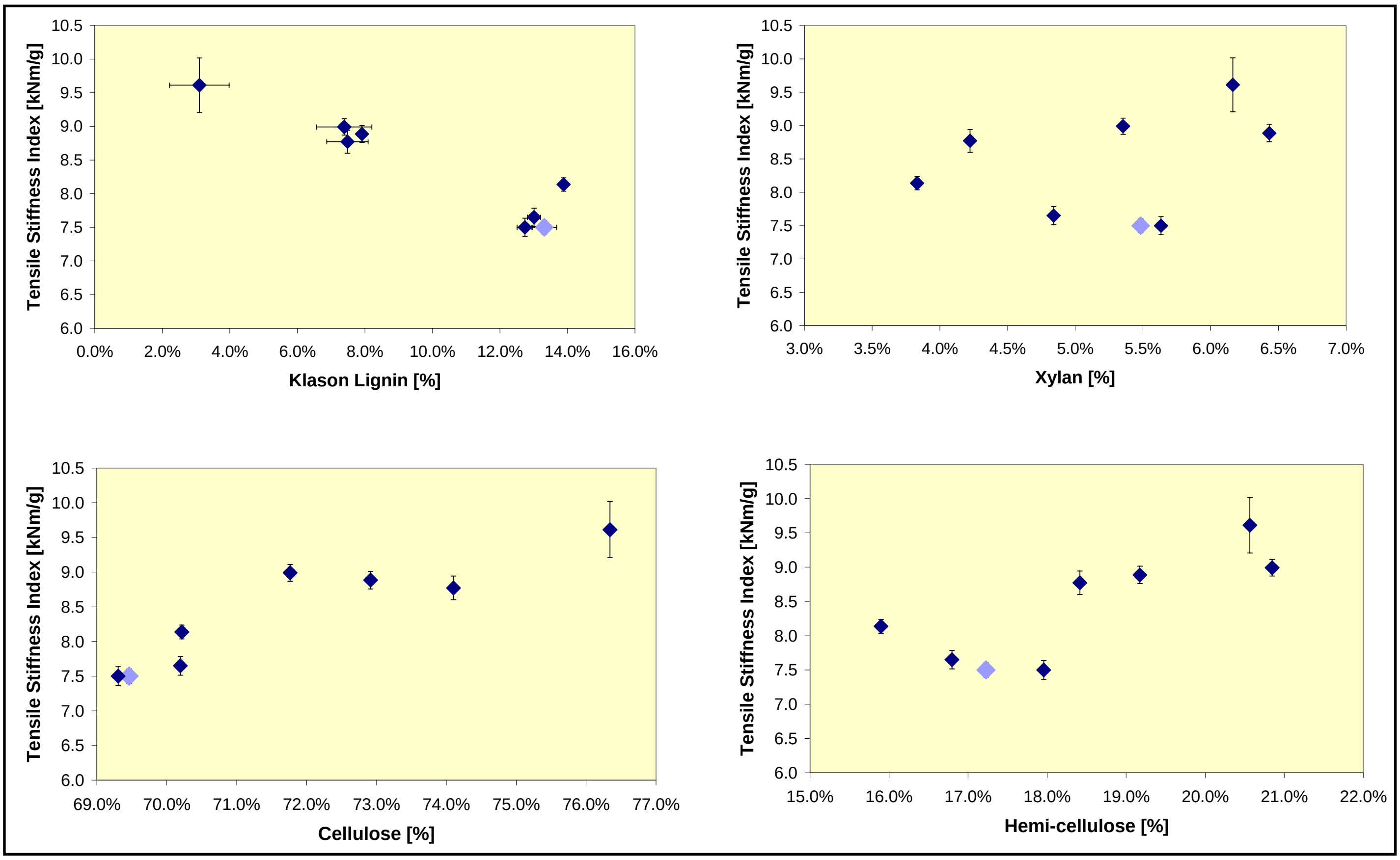

Figure 1: The effects on the tensile stiffness of the Klason lignin, hemicellulose, xylan and cellulose content. Error bars indicate a 95% confidence level and the light blue dot shows the untreated kraft liner reference pulp.

As the Klason lignin content is increased the tensile stiffness is decreased and when the content of hemicelluloses and cellulose is lowered the tensile stiffness is also decreasing. The xylan content, on the other hand, has no significant impact on the tensile stiffness among the pulps investigated in this study.

The conclusions in the literature saying that a higher yield is beneficial for tensile stiffness is not valid for these pulps and the reason why bleached pulps may have lower tensile stiffness is probably not due to lower lignin content but more likely due to other reasons such as different number of charges, partial removal of hemicelluloses as well as fibre deformations of different kinds.

The tensile strength, strain at break and tensile energy absorption, follows the same tendencies as the tensile stiffness does with regards to the chemical composition of the pulps as seen in Figure 2, 3 and 4 respectively.

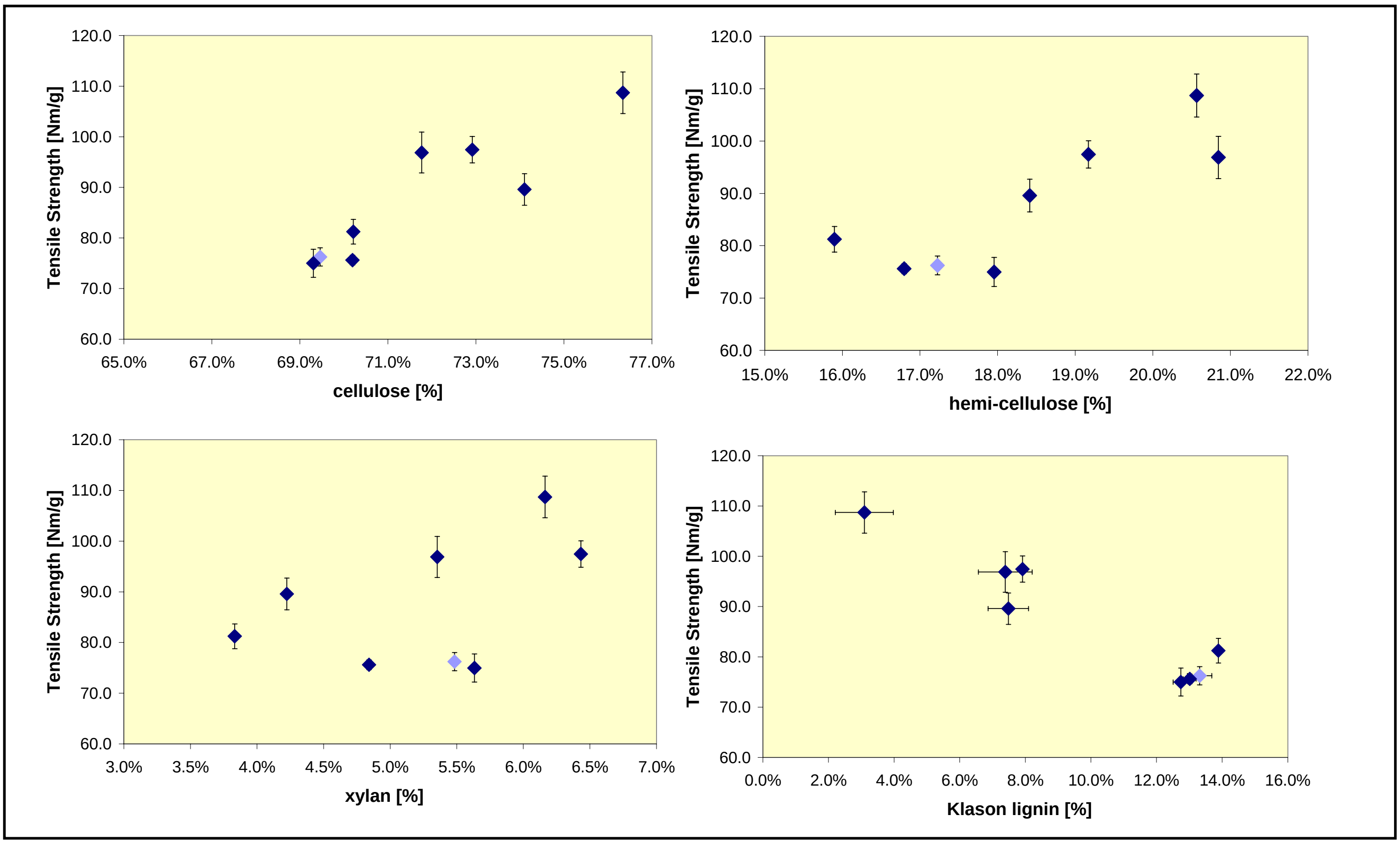

Figure 2: The tensile strength follows the same tendencies as the tensile stiffness as the chemical composition is altered. Error bars indicate 95% confidence level and the light blue dot shows the untreated kraft liner reference pulp.

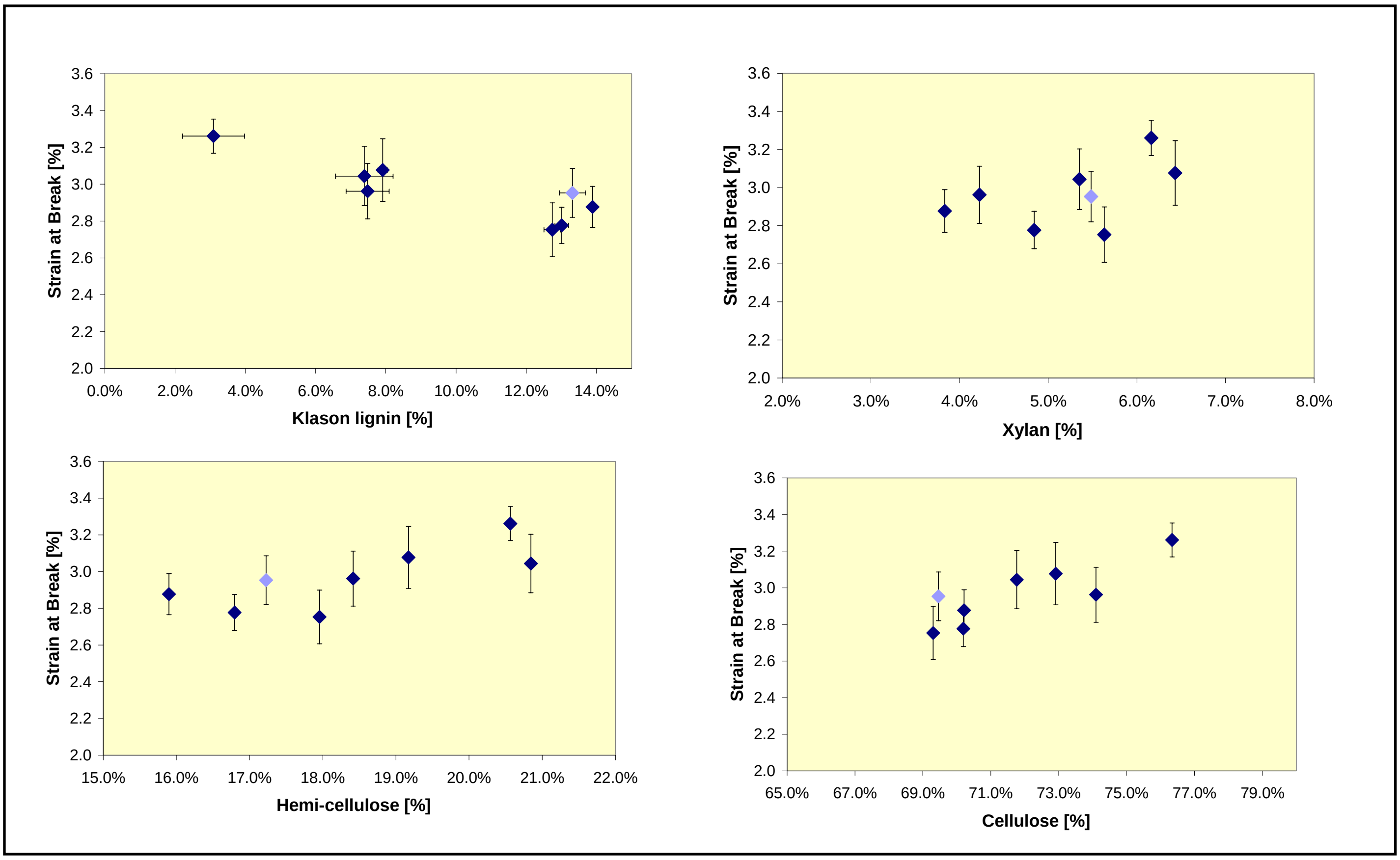

Figure 3: The strain at break follows the same tendency as the tensile stiffness and tensile strength index. Error bars indicate 95% confidence level and the light blue dot shows the untreated kraft liner reference pulp.

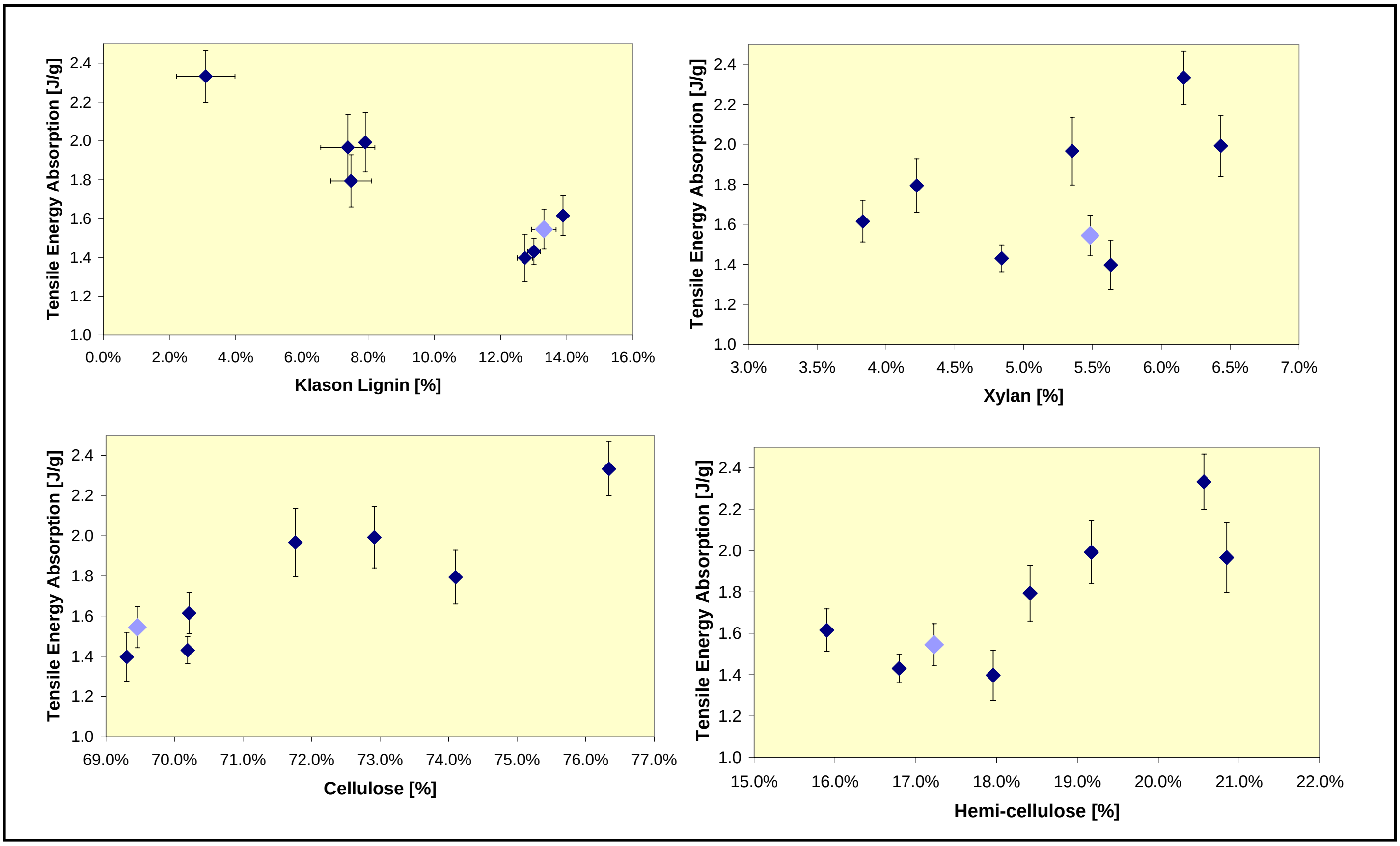

Figure 4: Tensile energy absorption is increased by a lower Klason lignin content and increased hemicellulose or cellulose content. Error bars indicate 95% confidence level and the light blue dot shows the untreated kraft liner reference pulp.

These higher values of the tensile properties, obtained for pulps with lower lignin content and higher cellulose and hemicellulose content, is in accordance with previous results found in literature, e.g. those of Annergren et al. (1963) and Molin and Teder (2002).

Fracture toughness properties

The results show not as clear relationships between apparent strength or apparent strain at break and the chemical composition of the pulps. However, the differences in chemical composition are not large in this matrix and the number of levels are low (fracture toughness has not been measured on SKK 100%, SKK 50%5XEU and SKK 50%500XEU). Still, there are indications that pulps with higher cellulose content and lower lignin content have higher values of apparent strength index while the apparent strain at break is more or less independent on the chemical composition as seen in Figure 5 and 6.

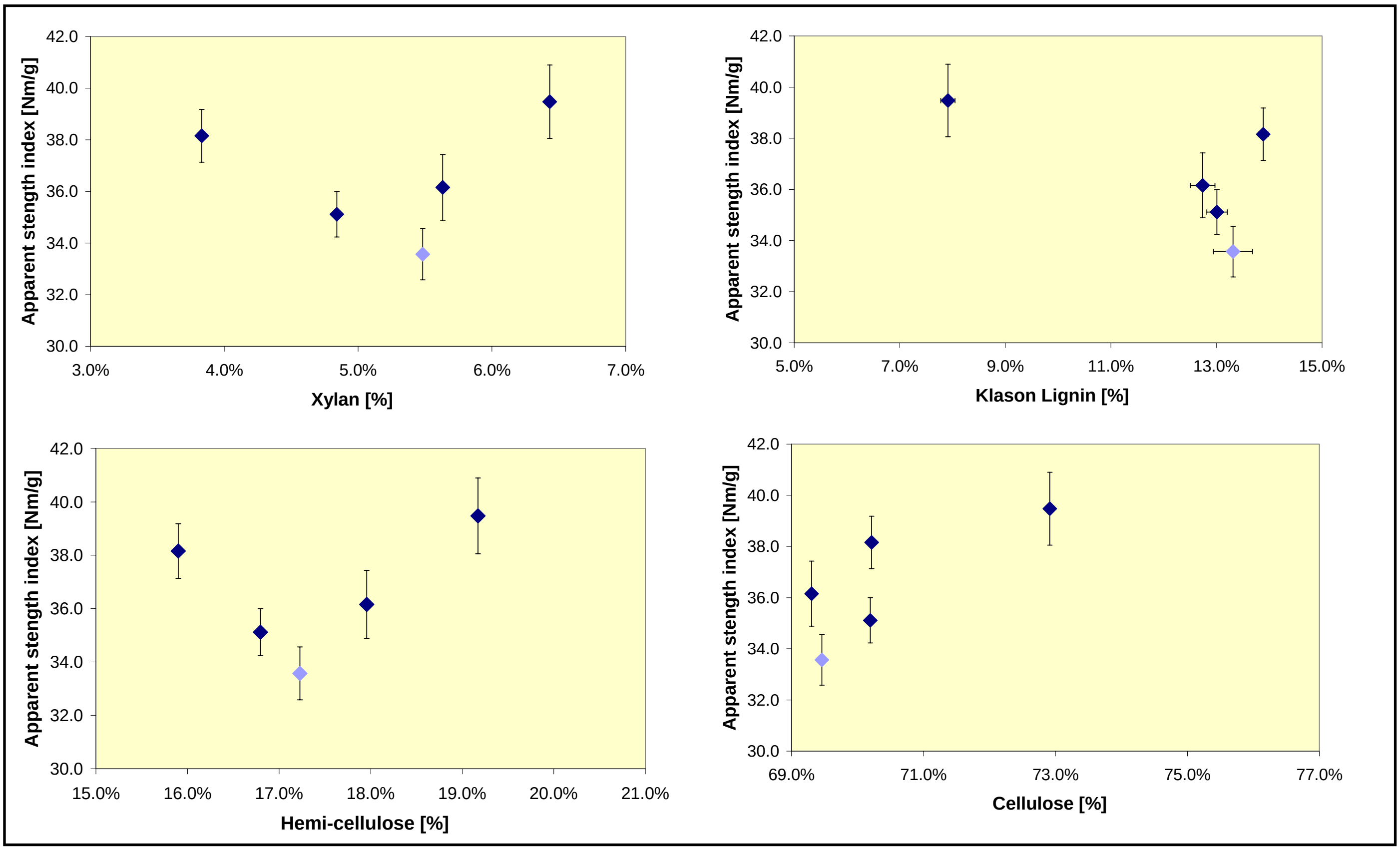

Figure 5: There are indications that apparent strength index is increased with higher cellulose and lower lignin contents. Error bars indicate 95% confidence level and the light blue dot is the untreated kraft liner reference pulp.

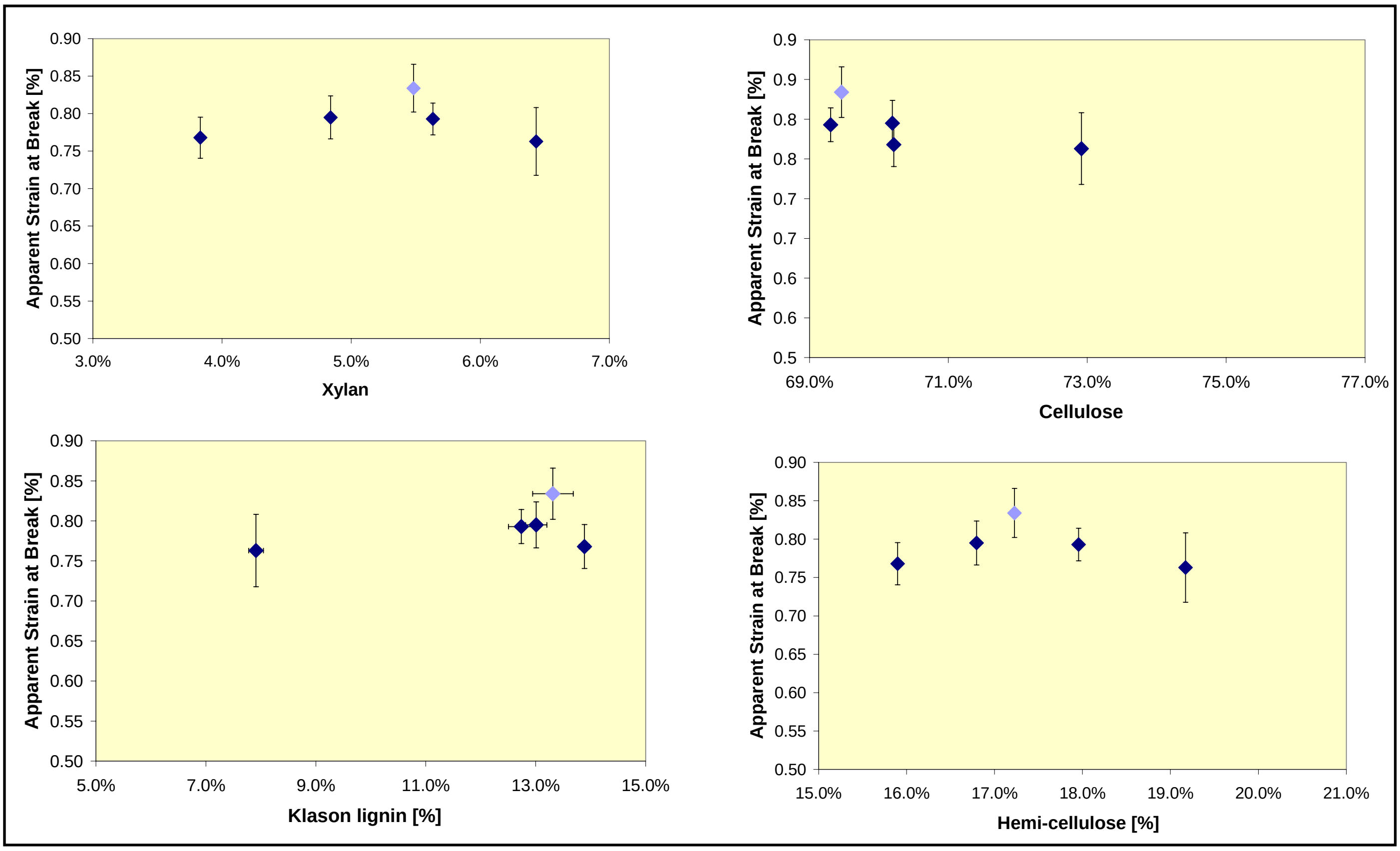

Figure 6: There are no clear correlations between chemical composition of the pulps and apparent strain at break. Error bars indicate 95% confidence level and the light blue dot shows the untreated kraft liner reference pulp.

Hygroexpansion

Increased hemicellulose and cellulose contents give a low hygroexpansion as seen in Figure 7. Consequently high lignin content has the opposite effect. Xylan content large impact on hygroexpansion values among these pulps.

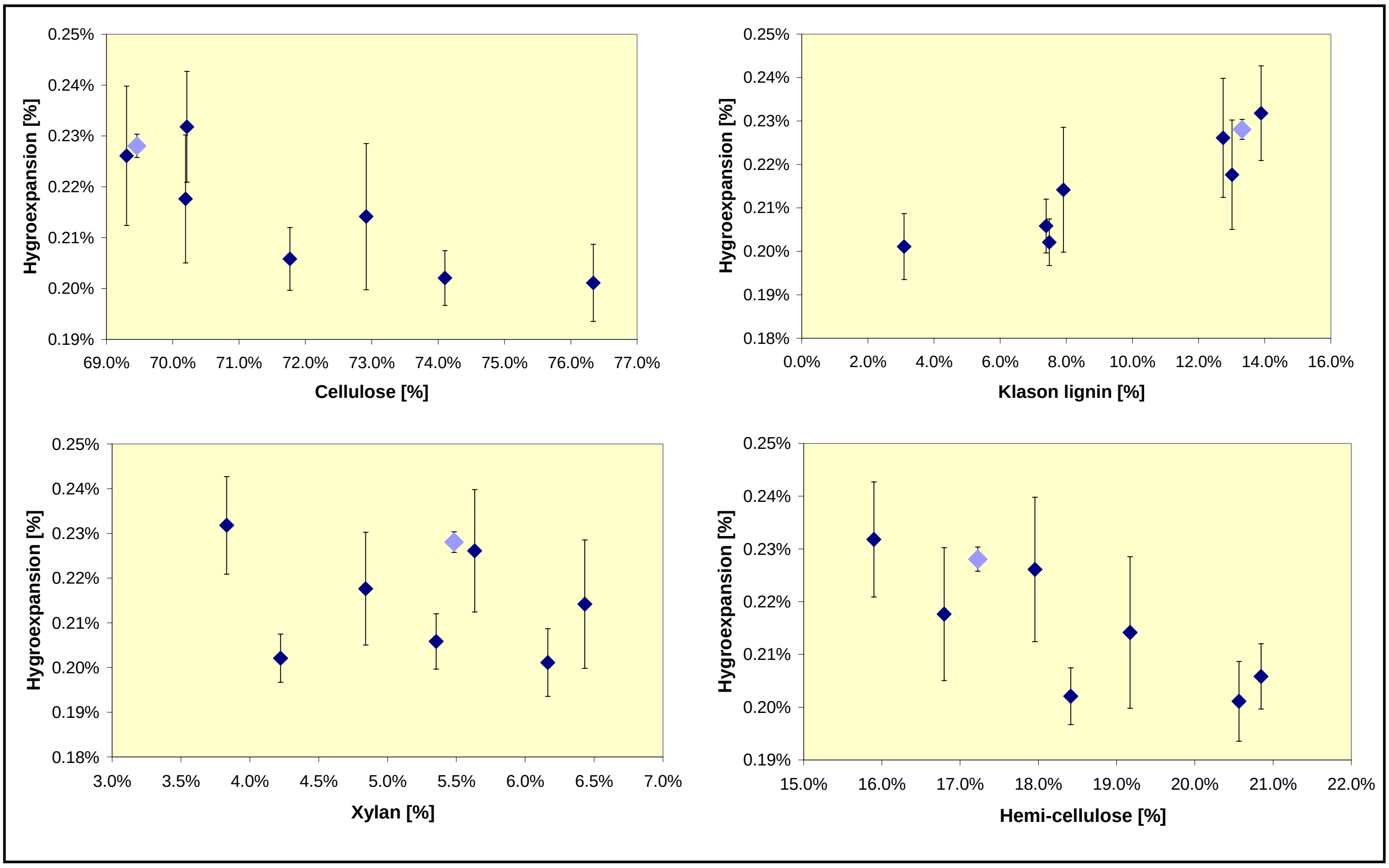

Figure 7: Increased hemicellulose and cellulose content as well as a decreased Klason lignin content give lower hygroexpansion. Xylan has no large impact on the hygroexpansion. Error bars indicate 95% confidence level and the light blue dot is the untreated kraft liner reference pulp.

Cellulose, mainly located in crystalline form building up the fibril aggregates in the fibres, is to large extent inaccessible to water and hold the fibres together also in a wet state. Hence, it is reasonable that increased cellulose content gives decreased hygroexpansion. That increased hemicellulose contents give the same effects is more difficult to explain but, may be due to that hemicellulose to large extent is crystalline glucomannan.

The effect of auto-cross-linking in lignin rich fibres that can decrease hygroexpansion (Antonsson *et al.* Manuscript) shows among these pulps not to be as important as the effect of increased amorphous Klason lignin maybe given raise to increased moisture uptake. Furthermore, it has been shown that differences in yield gives pulp with different pore size distribution (Andreasson *et al.* 2003) and this may also influence the results.

Mechano-sorptive creep
When considering the theories trying to explain mechano-sorptive creep, e.g. the theory of Alftan et al. (2002), it can be possible to link hygroexpansion of the paper and the mechano-sorptive creep. Of natural reasons, the stiffness at constant humidity and varying relative humidity ought to be possible to link as well. When the isocyclic creep stiffness and the hygroexpansion coefficent tensile stiffness ratio is plotted against each other it is also possible to observe that a relationship exist between these parameters, even though the deviations are large, as seen in Figure 8.

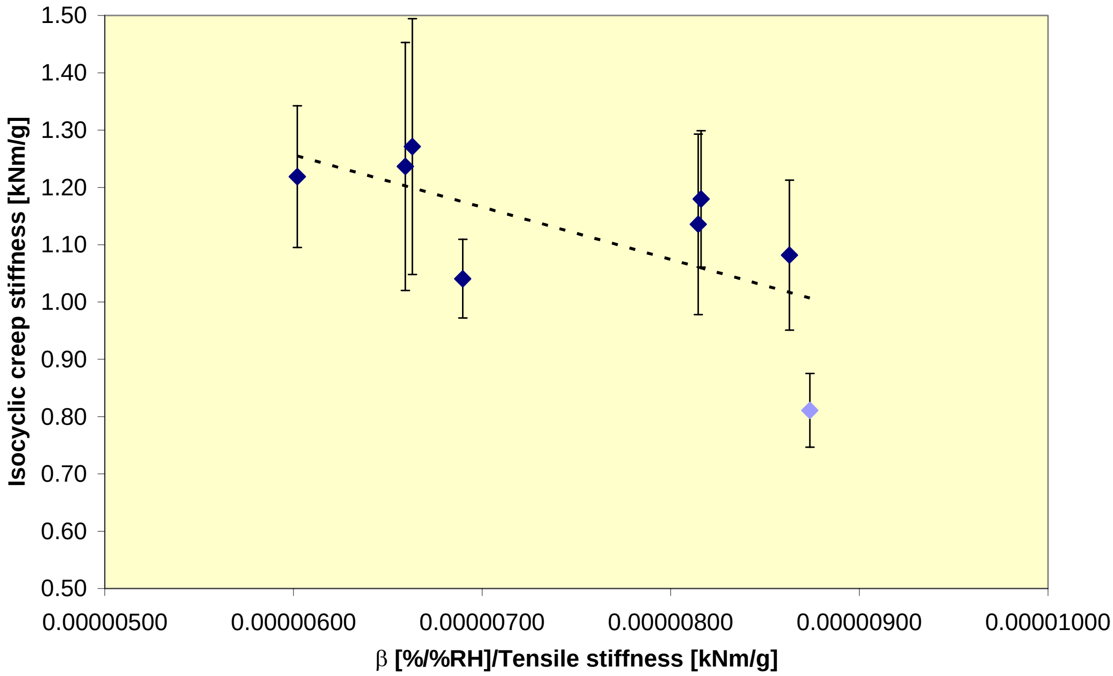

Figure 8: Isocyclic creep stiffness (in tension after 3 humidity cycles) shows a trend with the ratio between hygroexpansion coefficient tensile stiffness ratio. Error bars indicate a 95% confidence interval and the light blue dot is the untreated kraft liner reference pulp.

As seen in Figure 9, the relationships between isocyclic creep stiffness and chemical composition of the pulp sheets are not very clear, partly due to high deviations in the creep measurements. However, it is a tendency that higher cellulose content and lower Klason lignin content results in higher isocyclic creep stiffness.

That the Klason lignin and cellulose content influences the isocyclic creep stiffness in this ways is also reasonable, since lignin nor contribute to fibre or fibre-fibre joint strength, nor to a decreased hygroexpansion while cellulose rich pulps shows both increased stiffness and lower hygroexpansion. This is also in accordance with Byrd and Koning (1978) and Byrd (1984), that have presented results indicating that pulps with lower yield and lower lignin content generally results in lower creep rates in cyclic humidities.

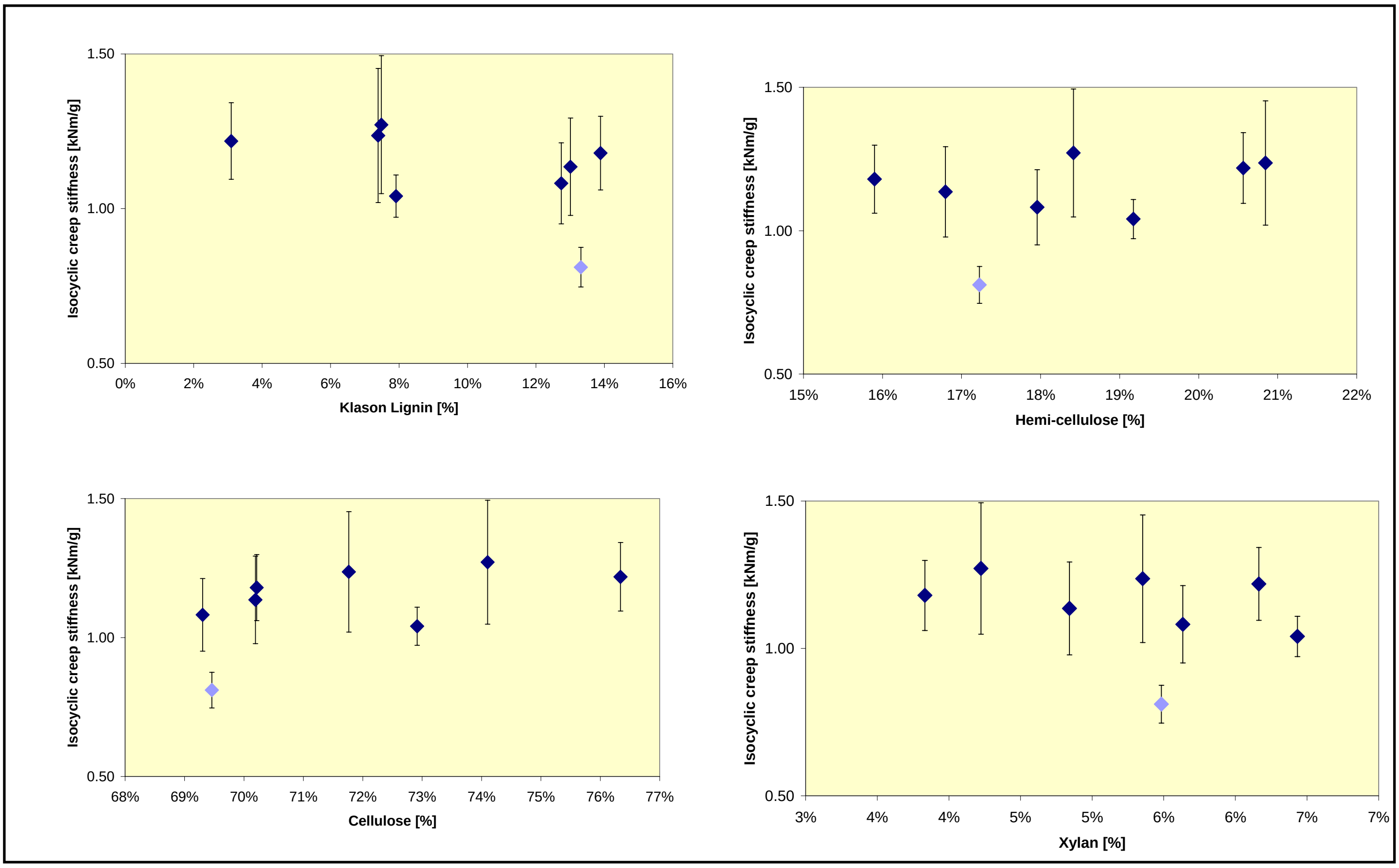

Figure 9: Isocyclic creep stiffness (in tension after 3 humidity cycles) as a function of chemical composition. It is a tendency, although not very clear, that higher cellulose content and lower Klason lignin content results in higher isocyclic creep stiffness. Error bars indicate 95% confidence level. The light blue dot is the untreated kraft liner reference pulp.

CONCLUSIONS

Among the pulps investigated, lignin tend to have a negative effect on the key properties; tensile stiffness, hygroexpansion and mechano-sorptive creep. Increased cellulose content has the opposite effect while xylan content tends to give less pronounced effects on the measured properties. Furhermore, a trend between isocyclic creep stiffness and the ratio between hygroexpansion coefficient and tensile stiffness was observed in the data.

A high yield makes the pulp cheaper per ton but results in a less attractive pulp for production of kraft liner that have desired properties such as high tensile stiffness, low hygroexpansion and mechano-sorptivt creep.

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