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Deliverable D2.83

The effect of using a novel cooking technique on the mechanical physical properties of these pulps compared with industrial grade pulps.

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Abstract

One of the main obstacles with kraft liner boards is poor performance at variation in relative humidity due to the so called mechano-sorptive creep effect. In earlier studies tensile stiffness and hygroexpansion have been shown to be important parameters affecting this property.

Both tensile stiffness and hygroexpansion is considerably affected by the fibre form of the pulp fibres. Ordinary production of kraft liner pulp involves in-line refining in order to separate the fibres at high kappa numbers. This creates pulps containing fibres with more dislocations and high curl. However, by using a novel cooking technique the defibration point can be moved to higher kappa numbers and the in-line refining avoided.

In this study, mill pulps and laboratory pulps made with this novel cooking technique, were compared at approximately same levels of kappa numbers. The results of the unbeaten and slightly beaten pulps, where the PFI-beating has not straightened out the fibres again, show that mill pulps have lower tensile stiffness and higher hygroexpansion compared to the pulps produced by the novel cooking technique. Mechano-sorptive creep behaviour is also indicated to be improved pulps produced with the novel cooking technique.

Keywords: *high kappa number pulp, kraftliner pulp, softwood, mechano-sorptive creep, hygroexpansion, dimensional stability tensile properties, SCT*

Introduction

Corrugated board materials are with a share of the total world paper production of about 30 percent (FAO 1998) an important category of paper to develop. One of the main obstacles in usage of corrugated board boxes is the bad performance when subjected to variations in relative humidity. This is due to the so called mechano-sorptive or accelerated creep behaviour. This behaviour of boxes has been shown to correlate with mechano-sorptive creep behaviour of kraft liner (Henriksson *et al.* 2007), and to reduce this creep while retaining suitable fracture toughness performance is a challenge.

Tensile stiffness and hygroexpansion have in earlier studies been shown to be important parameters when predicting mechano-sorptive creep behaviour (Antonsson *et al.* 2008; Antonsson *et al.* Manuscript) and this also has a theoretical background (Alfthan *et al.* 2002). The fibre form is one important factor when trying to increase tensile stiffness (Page and Seth 1980) and decrease hygroexpansion (Salmén *et al.* 1987).

In ordinary kraft liner pulp production in-line refining is utilized in order to separate the fibres at high kappa numbers, i.e. a high pulp yield. This treatment makes the pulp contain more fibres with dislocations and high curl. By using a novel cooking technique concept, moving the defibration point to higher kappa numbers, previously described (Karlström and Lindström Manuscript), it is possible to avoid the use of in-line refining.

This study aims at evaluating the possible benefits of using this novel cooking technique, regarding key properties of the kraftliner such as tensile, compression, fracture toughness, hygroexpansion and mechano-sorptive creep properties.

Materials and Methods

Industrial softwood kraft pulps

Softwood pulps with kappa numbers of 73 and 67 were kindly supplied by SCA, Munksund, Sweden and Smurfit Kappa Kraftliner, Piteå, Sweden respectively. All pulps were made of softwood mixture of Pine, *Pinus Sylvestris*, and Spruce, *Picea Abies*. The pulps were carefully washed with deionised water until the wash water had conductivity below 5.5 µS/cm and thereafter screened in a water jet NAF defibrator (Nordiska Armatur Fabriken) followed by sceening over a 32 mesh wire. After each operation the dry content of the pulps was increased by means of centrifugation.

Novel Technique laboratory softwood kraft pulps

The raw material used for the Novel technique kraft pulps was softwood chips of industrial grade kindly supplied by Smurfit Kappa Kraftliner, Piteå, Sweden. The chip mixture consisted of up to 80 % Pine, *Pinus Sylvestris*, and the remaining part of Spruce, *Picea Abies*. The chips were air-dried and sorted by hand to remove chips with bark and knots as well as chips with thickness above 8 mm and less than 2 mm.

The used laboratory digester had a cooking vessel volume of 16 dm^3 with forced circulation and flow rate of 15 dm^3/min. Air-dried chips corresponding to 1 kg of BD chips were pre-steamed at 15 bars for 5 min followed by impregnation at liquor-to-wood ratio 7:1 for 2h at 110^{o}C. The impregnation liquor consisted mainly of black liquors from similar cooks adjusted with fresh white liquor. The initial effective alkali was 17.5% corresponding to 25 g/dm^3 NaOH and an initial sulphidity of 47%. The temperature was increased by 1^{o}C/min between 70^{o}C and 110^{o}C. After the impregnation 5 dm^3 of impregnation liquor was withdrawn and 2 dm^3 of fresh white liquor was added increasing the alkali concentration to 35 g/dm^3 NaOH and resulting in a liquor-to-wood ratio 4:1. Between 110^{o}C and the desired cooking temperature the temperature was increased by 1^{o}C/min and held constant for 4 h. After 2 h the liquor-to-wood ratio was reduced to 3:1 by withdrawal of 1 dm^3 of black liquor. In Table 1 the additional cooking parameters are specified. Residual hydroxide ion concentration and hydrogen sulphide ion concentration were determined according to SCAN N 33:94 and SCAN N 34:96 respectively.

Table 1 Cooking parameters of Novel Technique pulps.

Pulp	Cooking Temp (^{o}C)	H-factor	Yield (%)	$[OH^-]_{res}$ (mol/dm^3) after impregnation	after 4h cook
Novel Tech 60	138	220	51.6	0.242	0.433
Novel Tech 75	135	165	53.2	0.224	0.448

After cooking the chips were washed with a flow of deionised water of 1.8 dm^3/min for 12-15 h. The novel technique with the long impregnation and more even alkali profile moves the defibration point towards higher kappa numbers and after each cook a small portion is subjected to water jet NAF defibrator in order to establish the need of disintegration prior to water jet defibration. The NT 60 pulp was only defibrated with the water jet NAF defibrator while the NT 75 pulp vas disintegrated for 50 000 revolutions in portions corresponding to 30g - 40 g dry pulp in 2 dm^3 deionised water before addition to the NAF water jet defibrator. The pulps were centrifuged to 25% - 30 % dry content and the total yield was determined. The pulps were screened in the same way as the industrial pulps over a 32 mesh wire.

Pulp analysis

Kappa numbers were determined in duplicates for the screened pulps according to ISO 302:2004 with 3 min disintegration time. Kappa number results are presented in Table 2.

Table 2. Kappa numbers of pulps compared in this study.

Pulp	Description	Kappa Number	
			CI 95%
Novel Tech 60	Novel Technique Lab	60	0.7
Novel Tech 75	Novel Technique Lab	75	0.7
Mill 67	Industrial Smurfit Kappa Kraftliner	67	0.5
Mill 73	Industrial SCA Munksund	73	0.7

Sheet preparation

PFI-beating, with beating degrees of 0, 1000, 3000 and 6000 revolutions, was performed according to ISO 5264-2:2002. Prior to sheet forming, the pulps were disintegrated for 30 000 revolutions according to ISO 5263-1:2004 in deionised water with addition of sodium chloride to obtain a concentration of 10 mmol/dm^3, i.e. a conductivity of approximately 2.5 µS/cm. Rapid Köthen sheets with a grammage of about 120g/m^2 was prepared according to ISO 5269-2:2004 with deionised water using an apparatus from Paper Testing Instruments, Austria. The prepared sheets were dried under restrained conditions at 93°C for 20 minutes.

Paper testing

Grammage and thickness were measured according to ISO 536:1995 and ISO 534:2005 respectively. A stack of eight sheets were measured instead of the standard value. Short span compression test, SCT, tensile properties and fracture toughness properties were determined according to ISO 9895:1989, ISO 1924-3 and SCAN P77:95 respectively. Fracture toughness was measured according to SCAN P77:95 with the exception that 6 instead of 20 notched test pieces were tested.

The hygroexpansion coefficient was measured between 33%RH and 66%RH according to ISO 8226-1:1994 using equipment developed at STFI-Packforsk consisting of 30 clamps and 30 freely movable clamps with a gap between the clamps of 100 mm where 30 paper strips were placed independently in a horizontal position. The test pieces were precycled between 50%RH and 90%RH three times before the measurements to release dried in stresses. Isocyclic creep stiffness tests, evaluating mechano-sorptive creep properties, were preformed on test pieces with a width of 25mm on an apparatus previously described elsewhere (Haraldsson *et al.* 1994). 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.

Results and discussion

Brightness

A higher brightness is of importance also when considering kraft liner pulps due to the fact that products such as white top liner are increasing in production volume. White top liner is liner board with a top layer of bleached pulp. If the brightness of the unbleached pulp is increased less bleached pulp is required to achieve a high brightness on the board. The brightness results of the pulps investigated are presented in Figure 1. Pulps produced with the novel cooking technique have higher brightness. This technique utilizes higher alkaline concentrations compared to ordinary mill cooking and increased alkaline concentrations have been shown to give pulps of higher brightness (Axelsson and Lindström 2004). Pulps having a higher kappa number also naturally have lower brightness due to the fact that different lignin structures contribute as the main source of chromophores in pulps.

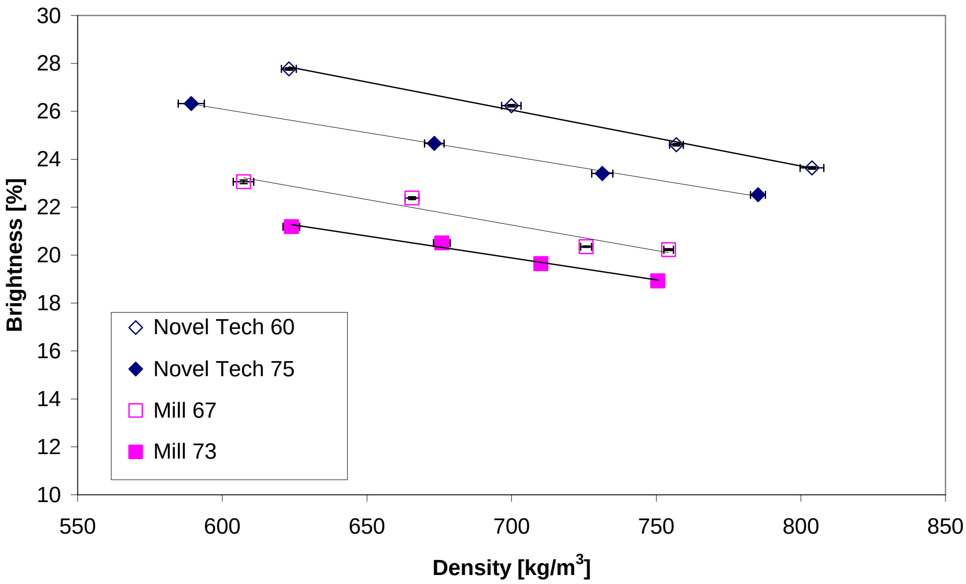

Figure 1: The brightness is higher for the pulps produced with the novel cooking technique and for pulps with lower kappa number. Error bars indicate a confidence interval of 95%.

Tensile and compression properties

The tensile properties of the different pulps are rather similar as shown in Figure 2. However, the Mill 73 pulp has lower tensile stiffness and higher strain at break at lower densities indicating more curly fibres that are straightened out after more PFI-beating. The pulp cooked with the novel technique to kappa number 60, i.e. with the lowest lignin content, has the highest tensile stiffness. This is in accordance with a previous study showing that lower lignin content gives higher tensile stiffness (Antonsson *et al.* 2008).

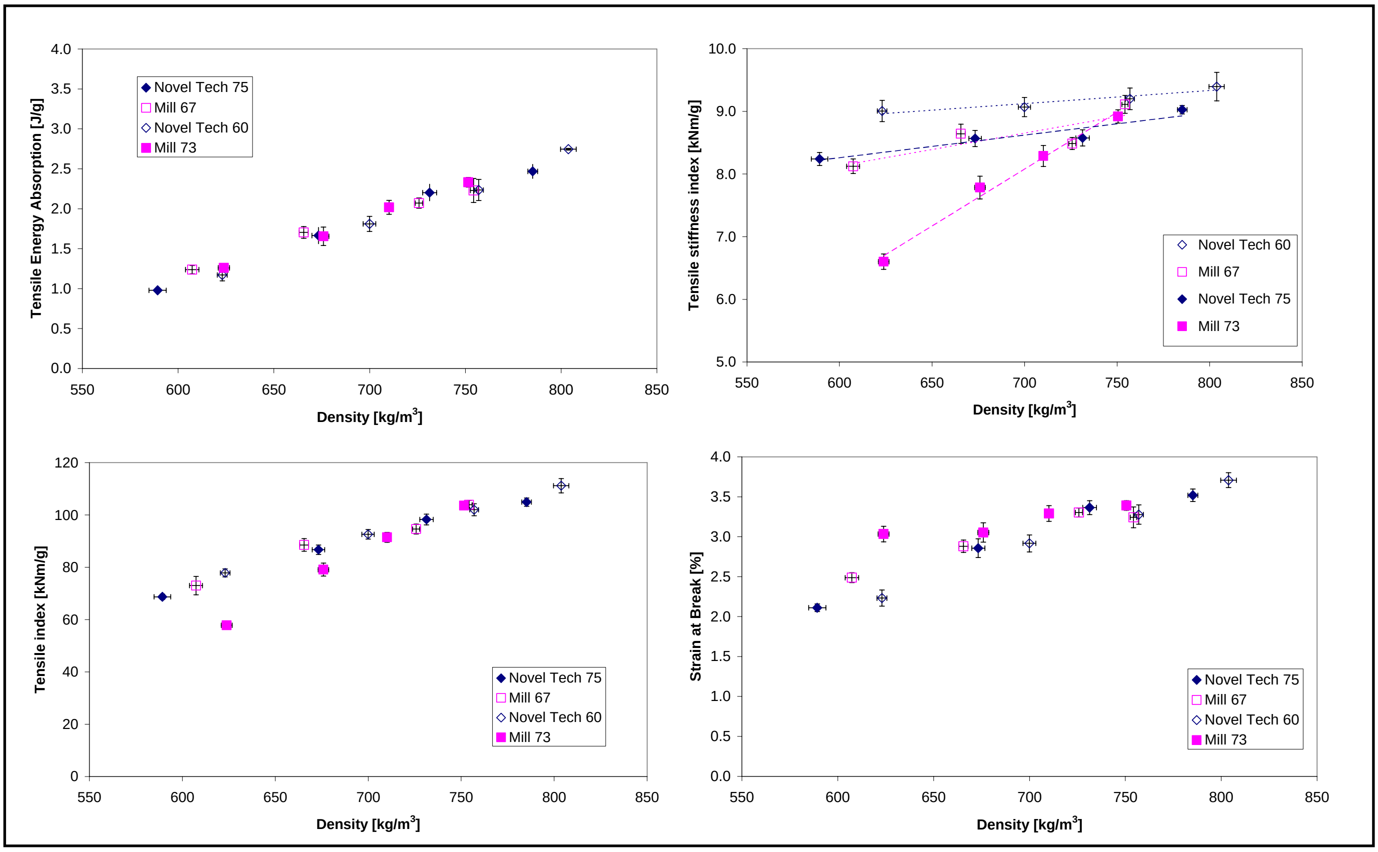

Figure 2: The tensile properties of the different pulps. Error bars indicate a confidence interval of 95%.

The SCT- values of the pulps are quite similar as shown in Figure 3. This is not expected since an increased tensile stiffness usually also results in higher SCT-values (Fellers and Henriksson 2006).

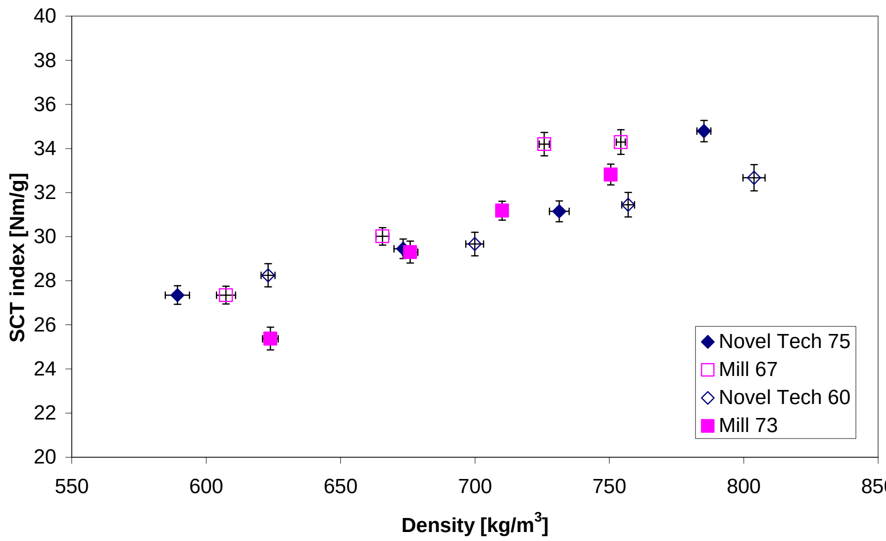

Figure 3: The short span compression tests show similar performance of the pulps. Error bars indicate a confidence interval of 95%.

Fracture toughness properties

The fracture toughness properties, i.e. apparent strength index and apparent strain at break is similar for the pulps even though the mill pulps have somewhat lower apparent strength index values as shown in Figure 4. This may be due to more straight fibres in these pulps, creating a network of more activated fibre-fibre joints that more easily withstand the high stresses at the crack edges. Interesting to note is also that the apparent strain at break is very little changed due to beating and higher sheet densities.

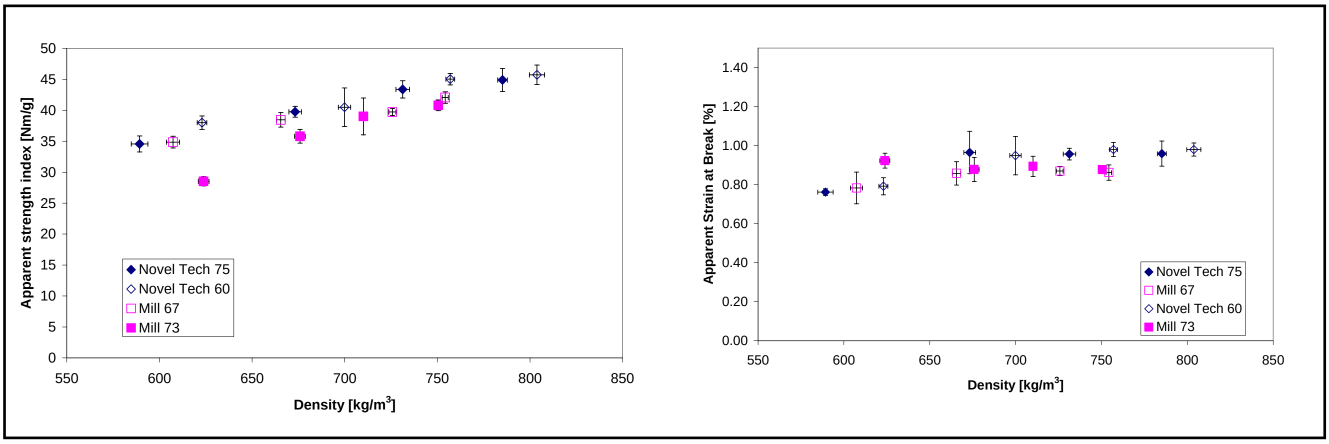

Figure 4: The fracture toughness properties, i.e. apparent strength index and apparent strain at break is similar for the pulps even though the mill pulps have somewhat lower apparent strength index values. Error bars indicate a confidence interval of 95%.

Hygroexpansion and isocyclic creep stiffness

Hygroexpansion coefficient is generally increased with the densities as shown in Figure 5. However, for the Mill 73 pulp the straightening of pulp fibres achieved by the PFI-beating will initially not increase hygroexpansion but, on the contrary slightly decrease it. The initial values are on the other hand higher for this pulp.

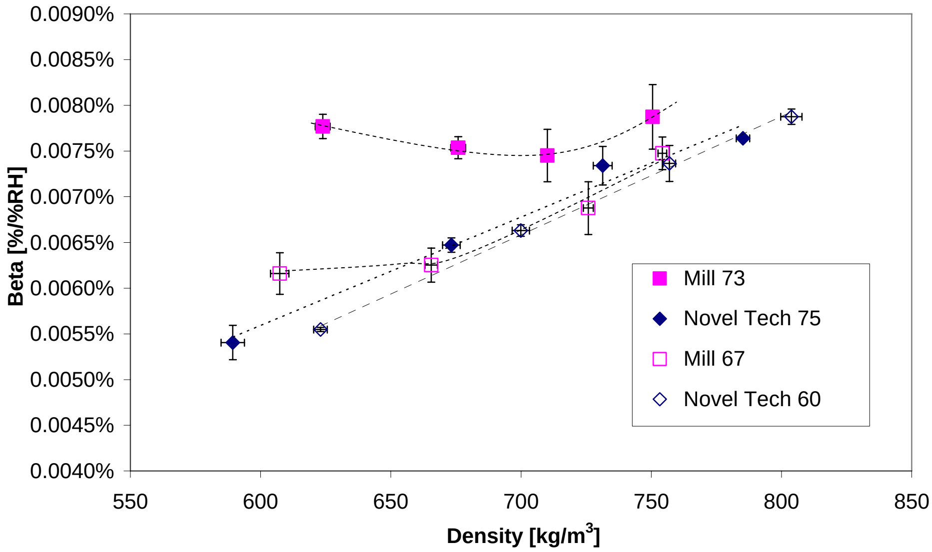

Figure 5: The hygroexpansion coefficent, based on changes in relative humidity, is somewhat higher for the mill pulps, especially at lower densities. Error bars indicate a confidence interval of 95%.

When hygroexpansion is compared with tensile stiffness values as in Figure 6, this effect is even more explicit shown and more beating of the mill pulps give larger improvements of the stiffness with slight deterioration of the hygroexpansion properties.

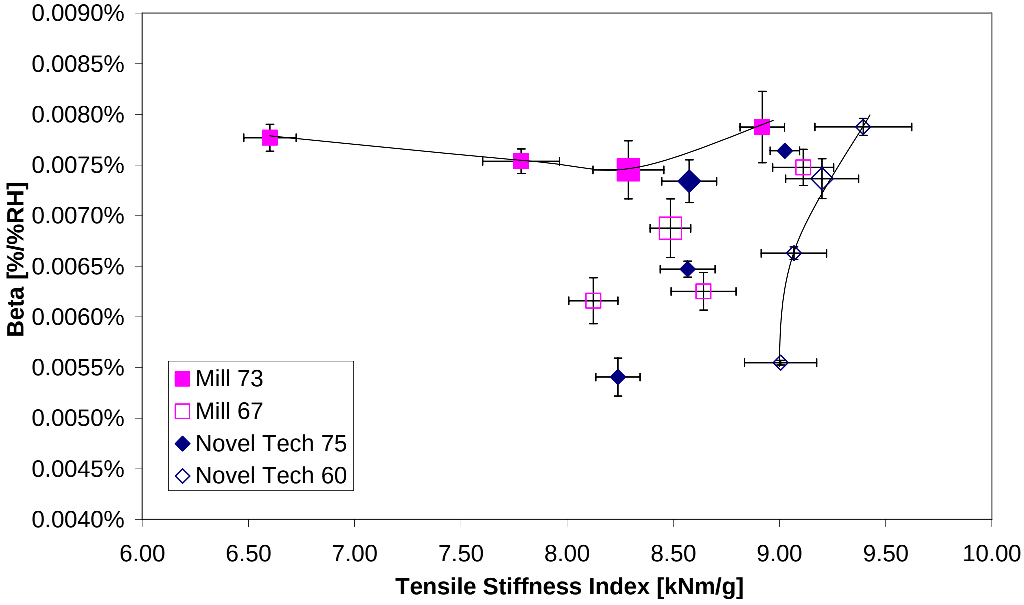

Figure 6: The relationship between tensile stiffness and the hygroexpansion coefficient suggest that mill pulps and the pulps with higher kappa number will perform worse in isocyclic creep stiffness. The pulps beaten with 3000 revolutions are marked with larger dots. Error bars indicate a confidence interval of 95%.

The pulps beaten with 3000 revolutions, marked as larger dots in Figure 6, were used for the isocyclic creep measurements. However, as indicated in Figure 6, the beating degree of the Novel Cooking Technique pulps is probably not optimised for high mechano-sorptive creep behaviour. Considerable lower beating degrees is enough to yield a high tensile stiffness and this also further reduces the need of electricity to produce the board (by avoiding in-line refining the major reduction is achieved).

As the pulps beaten with 3000 PFI-revolutions were compared in isocyclic creep stiffness in tension after three humidity cycles, the mill pulp had lower values as seen in Figure 7. However, the numbers of measurements are small, four to five test pieces were tested on each pulp, and the deviations large, i.e. the values are more preliminary indications than exact highly reliable values.

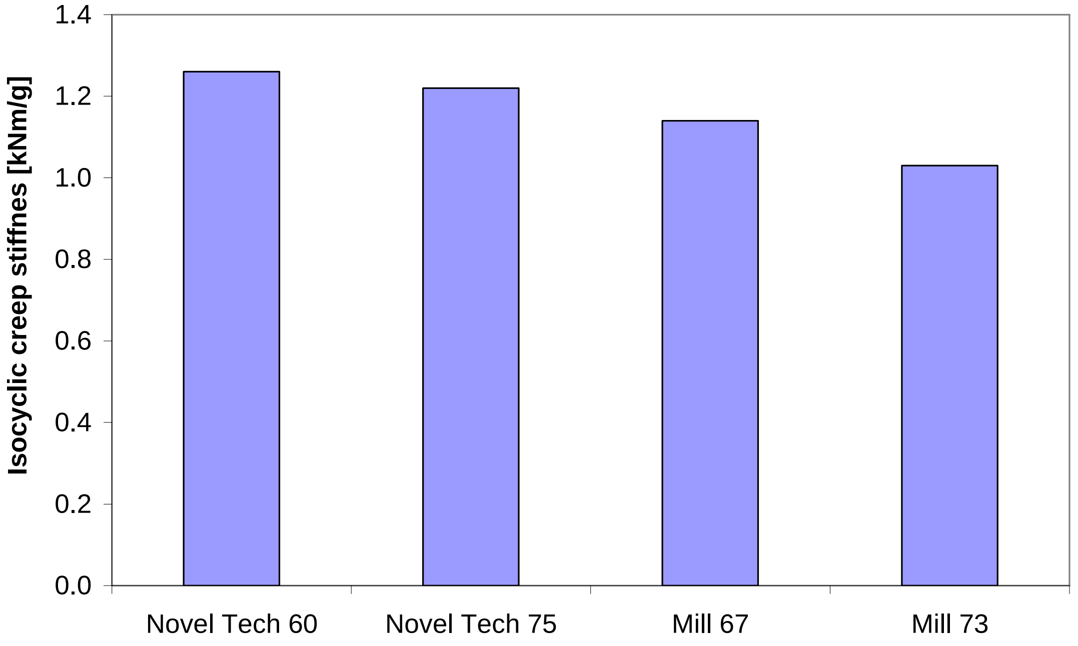

Figure 7: The isocyclic creep stiffness values in tension after three humidity cycles made for pulps beaten with 3000 PFI-revolutions on a minimum of four test pieces. The values are somewhat lower for the mill pulps.

Conclusions

The pulps produced in laboratory by means of a novel cooking technique have higher tensile stiffness and lower hygroexpansion at lower densities, probably due to the fact that in-line refining of these pulps can be avoided, making the pulp fibres less curly. It could also be concluded that higher kappa number of the pulp give a lower tensile stiffness and somewhat higher hygroexpansion.

Acknowledgements

Access to the testing equipment at STFI-Packforsk; the hygroexpansion tester, the fracture tester with Fracture lab, the creep apparatus and the climate chamber, Troëdssonska Klimatkammaren, is gratefully acknowledged. Petri Mäkelä and Magnus Gimåker are gratefully acknowledged for assistance with some measurements. Smurfit Kappa Kraftliner, Piteå is gratefully acknowledged for supplying pulp and chips. SCA Munksund is acknowledged for supplying pulp. The SustainPack Program has financially supported the study.

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