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Towards complete impregnation of wood chips with aqueous solutions

Part 4: Effects of front-end modifications in displacement batch kraft pulping

Keywords:
Kraft cooking, penetration, presteaming, bleachability, papermaking potential.

Abstract

The front-end of kraft cooking, i.e. steaming, impregnation and heating, controls the cooking performance in terms of mass transfer, delignification and cooking uniformity. Therefore, it is of vital importance to understand the process mechanisms and optimise the front-end process conditions. This article discusses the effect of applying chip presteaming and higher pressure at the front-end of kraft displacement batch pulping on the cooking performance, bleachability and papermaking potential of the pulp. By applying chip presteaming and higher pressure it is possible to significantly reduce the amount of rejects after the cook and to lower the kappa number at constant cooking conditions. The better delignification uniformity and efficiency can be explained by the more thorough penetration of liquor into the wood chips achieved with the front-end modifications.

According to the results, chip presteaming and higher pressure do not have a significant effect on the bleachability and papermaking potential of pulp. However, better pulp uniformity and the resulting lower pulp rejects content allows higher production of accept pulp and less fibre losses. The lower residual lignin content of the cooked pulp can be used as a trade-off for higher cooking throughput at the same chemical charge and recovery load or reduced bleaching chemical consumption due to lower pulp kappa number.

Tiivistelmä

Sulfaattikeiton alkuvaiheet, höyrytys, impregnointi ja lämpötilan nosto, ovat määräävässä asemassa keiton aineensiirron, delignifioinnin ja keiton tasaisuuden, eli keiton onnistumisen kannalta. Sen tähden on erittäin tärkeää ymmärtää alkuvaiheiden mekanismit ja optimoida prosessiolosuhteet. Tässä artikkelissa käsitellään hakkeen höyrytyksen ja imeytyspaineen vaikutusta syrjäytyseräkeittoon, massan vaalenevuuteen ja paperiteknisiin ominaisuuksiin. Hakkeiden höyrytyksellä ja korkealla imeytyspaineella parannetaan merkittävästi massan delignifioitumista samoilla keitto-olosuhteilla ja vähennetään rejektin määrää keiton jälkeen. Näiden alkuvaiheiden modifikaatioiden avulla keittoliuos penetroituu hakkeisiin paremmin, mikä näkyy delignifioitumisen tasaisuudessa ja tehokkuudessa.

Kokeiden tulosten perusteella hakkeiden höyrytys ja korkea imeytyspaine eivät vaikuttaneet merkittävästi massan vaalenevuuteen eikä paperiteknisiin ominaisuuksiin. Sen sijaan parempi massan tasaisuus eli alempi massan rejektipitoisuus mahdollistaa suuremman akseptimassan tuotannon ja vähentää kuitutappioita. Alempi keiton jälkeinen jäännösligniinipitoisuus eli kappaluku mahdollistaa suuremman keittotuotannon samalla kemikaaliannostasolla ja talteenoton kuormituksella pidettäessä entinen kappataso tai alhaisemman valkaisukemikaalikulutuksen pidettäessä alempi valkaisemattoman massan kappataso.

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Introduction

Movement of reactive chemicals into the core of a wood chip is of great importance in chemical pulping processes, in which effective impregnation is essential. Complete impregnation increases the uniformity of pulping and reduces cooking time /1,2/. On the other hand, incomplete impregnation results in steep delignification gradients inside the chips, causing higher amounts of rejects in the final pulp /3/. Non-uniform pulping can also lead to complications in the performance of downstream operations such as bleaching and papermaking /4/.

One way to make impregnation more complete is to improve the initial liquor penetration. It can be achieved either by optimising conditions, such as pressure, temperature, and time, or by applying some "penetration aid" techniques /5/. Several techniques can be used to reduce the amount of air inside the chip voids /6,7/. Among them, presteaming of the chips under high temperature, due to its simplicity and other advantages, is considered to be the most practical technique for industrial applications. Chip presteaming is an integral part of most continuous kraft cooking processes. However, in batch kraft cooking technology, chip presteaming is not used so effectively. Usually, only slight presteaming of the chips is taking place during the chip packing stage. Efficient presteaming helps to remove a large part of the air present inside wood chips /7-10/, facilitating the penetration of liquor. In addition to its primary effect of air removal, presteaming seems to improve the impregnation by altering the structure of wood capillaries /11-13/.

Apart from its positive influence on pulp uniformity, chip presteaming and higher pressure might also have negative effects. It is claimed that presteaming could cause changes within the lignin structure and complicate its removal /14,15/. On the other hand, it has been shown that more uniform pulp is easier to bleach and that it requires less bleaching chemicals /16/. It still remains unclear, whether presteaming and better pulping uniformity have any effect on the strength properties of the pulp. Some studies indicate that non-uniform cooking would result in weaker pulp /16-18/. Overcooked fibres present after a heterogeneous cook could reduce the average fibre strength of the pulp. Another study, however, indicated no differences in strength between the uniform and non-uniform pulps /19/.

This article examines the scope for modifying the front-end conditions of kraft displacement batch cooking by increasing the pressure in the hot black liquor stage and presteaming of the chips. The effect of modifications on cooking performance is compared based on the data obtained from two sets of cooks. The effect of presteaming and higher pressure on the bleachability and papermaking potential of the pulp was also examined.

Experimental

Two sets of chip samples were prepared from pine (*Pinus silvestris*) roundwood with different properties. Freshly cut wood with high moisture and low heartwood content was used to prepare the first set of chip samples. The second set of chip samples was prepared from roundwood that had lower moisture and higher heartwood content. The properties of these two pine roundwoods are compared in **Table 1**. Chips for cooking simulations were prepared with an industrial chipper at Kaukas pulp mill, Finland. The chips were classified in the laboratory in accordance with the standard SCAN-CM 40:88. To facilitate the rejects formation during cooking, the over-thick fraction of chips was used in cooks together with accepts, in proportions of 5 % and 95 %, respectively.

The laboratory digester used for pulping was similar to the one described elsewhere /20/. It was used to simulate cooking stages, displacement times, and circulation rates comparable to those of mill-scale liquor displacement kraft batch processes. Two sets of chip samples were cooked according to four scenarios with different front-end conditions. The idea was to examine the effects of presteaming and over-pressure in the hot black liquor stage, while keeping all other cooking conditions constant. The stages of the displacement batch process and the cooking conditions are presented in **Fig. 1**. Cooks made with the chip samples from the second set had a slightly higher alkali charge and sulphidity. The results of the cooking simulations were evaluated based on the yields and amount of rejects as well as the basic pulp properties. In addition, the alkali profiles were monitored by titration of the liquor samples taken throughout the cook.

Table 1. *Properties of pine roundwoods.*

Chips	***Pine (Set 1)***		***Pine (Set 2)***	
Age of the tree, years	70		80	
Diameter of the tree, mm	200		250	
Heartwood fraction, % (volume)	23		53	
	Heartwood	**Sapwood**	**Heartwood**	**Sapwood**
Basic density, g /cm^3	0.405	0.430	0.390	0.410
Dry-matter content, %	70	40	75	45
Moisture content, % on wood	43	150	33	122

To study the effect of front-end conditions on the bleachability and papermaking potential of pulp, samples from the second cooking set were bleached using oxygen delignification and the sequence DEDED with varying chlorine dioxide charges. Oxygen delignification was carried out in a rotating autoclave digester, and bleaching stages in polyethylene bags in a water bath. Bleaching conditions and chemical charges for each stage are shown in **Fig. 2**. Between the bleaching stages, the pulp samples were washed with deionised water by repeating the following procedure three times: thickening to 25 % consistency and dilution to 10 %. Kappa number, viscosity and ISO brightness of the unbleached and bleached pulps were measured according to SCAN standards. Bleached pulp samples of the same brightness (88 % ISO) were refined in a PFI mill (SCAN-C 24:67) at five levels: 0, 500, 1500, 3000, and 6000 revolutions. Preparation of handsheets and testing of paper and pulp properties were based upon SCAN standards, unless otherwise stated. The zero-span tensile strength of the bleached pulp samples was measured with the Pulmac apparatus according to the standard ISO 15361:2000. The results were expressed as a FS number, which is the wet zero-span tensile index corrected to the basis weight of 60 g/m^2. The average fibre length, weighted by length, and the coarseness of the pulp were determined by using the Kajaani 200 fibre analyser.

Results and discussion

Cooking results

The alkali profiles throughout the displacement batch cooks (scenarios A2-D2) are shown in **Fig. 3**. At the beginning of the cook, during the IBL and HBL stages, there is a clear difference between the scenarios with and without chip presteaming. Steaming of the chips prior to cooking resulted in much faster consumption of initial alkali. This can be partially attributed to the fact that steaming of wood chips is usually accompanied by hydrolysis of some carbohydrate components, resulting in liberation of acetic acid. As a result, more alkali is consumed during the initial stages. Residual alkali was almost the same for different cooks.

All basic data from the cooks are listed in **Table 2**. The fibre length and coarseness of the pulp samples do not show any significant differences between the cooking scenarios. However, the amount of rejects and the kappa numbers of the pulps indicate some positive effect of modifying front-end conditions of the cook. **Fig. 4** compares the rejects and screened yields after cooks from two cooking sets. Higher pressure and chip presteaming both drastically reduce the amount of rejects; yet, presteaming seems to be more efficient. Lower amount of rejects can be explained by the more thorough penetration of liquor into the wood chips achieved by the modifications. More complete penetration reduces the volume within the chips that does not get into contact with the chemicals and also shortens diffusion distances. It has also been reported that presteaming of chips through its effect on the wood structure increases the diffusion rate of some ions /21/. The lowest amounts of rejects from the D cooks indicate that higher pressure and chip presteaming can have synergistic effects. The effect of modifications is more pronounced in the second cooking set, for which chips with high heartwood content were used. This can be explained by the fact that pine heartwood is difficult to penetrate by liquors /5/.

***Table 2**. Basic cooking data.*

Scenario	A1	B1	C1	D1	A2	B2	C2	D2
Screened yield, %	48.6	47.9	48.1	48.5	47.5	48.1	47.4	47.1
Rejects, % on wood	1.35	0.83	0.28	0.09	1.80	0.30	0.12	0.15
Kappa number	36.2	35.2	34.0	33.9	34.4	31.3	31.5	29.5
CED viscosity, ml/g	1160	1160	1155	1160	1100	1160	1170	1140
Fibre length, mm	n.d.	n.d.	n.d.	n.d.	2.69	2.64	2.66	2.64
Fibre coarseness, mg/m	n.d.	n.d.	n.d.	n.d.	0.234	0.236	0.239	0.227

Fig. 5 shows that it was possible to reduce the kappa number of the pulp by modifying the front-end of the cook. The lowest kappa number was achieved in the D scenarios, when using both higher pressure and chip presteaming. Similar to the rejects, the effect of modifications on the kappa number of the pulp was more pronounced in the second cooking set. Higher pressure as well as removal of entrapped air during presteaming led to more complete penetration, providing more uniform and easier access for chemicals to fibres present inside the chips. As a result, the delignification degree and uniformity were improved. In addition to improved impregnation, chip presteaming could also affect the structure of the lignin itself. Whether these changes within the lignin structure take place and positively influence the lignin removal efficiency, remains a matter of speculation.

The viscosity values of the pulps from the first cooking set were almost identical (**Table 2**). In the second cooking set, pulp A2 had a much lower viscosity than the other pulps. It is uncertain whether viscosity gives an indication of pulp strength, but it does provide a picture of the depolymerization of the polysaccharides in pulp /22/. The conditions of the A2 cook could cause the more heterogeneous pulping, where reactive chemicals are distributed non-uniformly within the wood chips. As a result of this non-uniformity, more severe depolymerization of some polysaccharide constituents can take place, decreasing the average viscosity of the pulp.

Bleaching results

The conditions for mild oxygen delignification and the $D_0ED_1ED_2$ bleaching sequence were chosen to be in close correspondence with industrial practices (**Fig. 2**). After the oxygen delignification stage, a reduction in the kappa number of 33-37 % was achieved for all pulp samples. To minimise the differences in kappa number between the four pulps, the chlorine dioxide charge based on the Kappa Factor was used in the D_0 stage. Pulp samples taken after the D_0E stages were divided into three fractions and further bleached with the chlorine dioxide charge as a variable. These chlorine dioxide charges were split between the D_1 and D_2 stages in the proportion 2:1. Measured pH values and residual chemicals after each bleaching stage indicate that the sequences were carried out under optimal conditions.

Fig. 6 shows the brightness development of the four pulps as a function of chemical consumption. To reach the full brightness (88 % ISO), 20 kg more of act. Cl had to be used for pulp A2 than for D2 pulp. This difference is important for a pulp producer, when considering the economic and environmental aspects of bleaching. On the other hand, the bleachability diagram (**Fig. 7**) shows no difference between the pulps. Therefore, the differences in chemical consumption were caused solely by differences in the incoming kappa number of the pulps, not by their bleachabilities. Based on this, the presteaming and higher pressure apparently did not have any significant effect on lignin structure.

Papermaking potential

Some properties of the pulp handsheets are compared at five refining levels in **APPENDIX I**. Schopper-Riegler (SR) numbers indicate that the development of dewatering characteristics during PFI refining was very similar for all pulp samples. The light scattering coefficients of refined pulps decrease in a similar manner as a function of sheet density, and when compared at the same sheet density show no difference between the four pulp samples.

The papermaking potential of kraft pulps is largely determined by the quality of the fibres produced in pulping and bleaching operations. Among the important characteristics, physical strength is a unique aspect of kraft pulps, especially for softwood pulp /17/. The strength properties of the four pulps are compared in terms of strength diagram (**Fig. 8**) that is based on the mean values of the tear and tensile indexes. Curves in **Fig. 8** indicate that in the tensile index range between 70 and 100 Nm/g, the strength properties of the pulps are almost the same. A comparison of the tear values at different refining levels through standard analysis of variance and the Tukey test showed that none of the pulps differ significantly.

The tear - tensile data at various degrees of beating are commonly used as a basis when discussing pulp strength /23,24/. However, tensile and tear parameters depend on a number of factors, thus resulting in diverging interpretation /24-26/. Measurement of the Pulmac fibre-quality numbers was suggested as an alternative method for evaluating the response of pulps to beating /27/. **Fig. 9** compares the mean FS numbers of the pulps at different refining levels. Again, the variance analysis and Tukey method for comparing pairs of means showed no significant differences between the pulps.

Table 3. *Reinforcement ability (RA) of pulps.*

Pulp samples	A2	B2	C2	D2
Fibre length, mm	2.50	2.45	2.46	2.43
Coarseness, mg/m	0.232	0.240	0.243	0.232
Tear index, Nm2/kg (at tensile index of 70 Nm/g)	19.1	19.0	19.9	19.3
Reinforcement ability	202	196	201	202
RA = (length/coarseness) x tear index /28/				

Softwood pulp quality can also be evaluated based on so-called "reinforcement ability", which should characterise the papermaking potential better than pulp strength alone /28/. **Table 3** presents the reinforcement ability of the four bleached pulps. These data indicate that the papermaking potential of all pulps is almost the same. One can argue that even if the cooking resulted in differences between the pulps, the subsequent bleaching eliminated them. The biggest threat for elimination of differences between the pulps was the oxygen delignification, which was performed in a rotating autoclave. However, the final pH and temperature profiles indicate that O_2 stages were carried out under optimal conditions with good repeatability. Bleaching stages performed in a laboratory water bath under "ideal" conditions are unlikely to cause any damage to the pulps /23/.

Summary

By modifying the front-end conditions of a displacement batch cook by applying higher pressure and chip presteaming, it was possible to significantly reduce the amount of rejects and decrease the kappa number of the pulp. At the same time, fibre length, coarseness and viscosity of the pulp samples did not show any significant differences between the cooking scenarios. The lower amount of rejects and lower pulp kappa number can be attributed to more thorough penetration of liquor into the wood chips achieved by the modifications made. More complete penetration reduces the volume within the chips that does not get into contact with chemicals and also shortens diffusion distances. As a result, the delignification degree and uniformity are improved. Presteaming seems to be a more efficient way to achieve these gains. Still, the higher pressure and chip presteaming can have synergistic effects.

The results indicate that chip presteaming and applied higher pressure did not affect the bleachability of the pulp. However, they resulted in much lower consumption of bleaching chemicals. Differences in chemical consumption were caused solely by the variability of the incoming kappa number. Properties measured from the pulp handsheets indicate no differences between the fully bleached pulps. Assuming "ideal" and repeatable conditions in laboratory bleaching, the cooking modifications apparently did not have any significant effect on the papermaking potential of the pulp.

Acknowledgements

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Figures

Cooking set	Scenarios	No 1: A1	B1	C1	D1	No 2: A2	B2	C2	D2
FRONT END: *Chip charge*									
FRONT END: *Chip steaming*	Steaming time, min	0	0	30	30	0	0	30	30
	Steaming temperature, °C	-	-	105	105	-	-	105	105
FRONT END: *IBL stage - warm black liquor impregnation*	Time (IBL), min	20	20	20	20	20	20	20	20
	Temperature (IBL), °C	80	80	80	80	80	80	80	80
	Over-pressure (IBL), bar	2	2	2	2	2	2	2	2
FRONT END: *HBL stage - hot black liquor treatment*	Time (HBL), min	30	30	30	30	30	30	30	30
	Temperature (HBL), °C	155	155	155	155	155	155	155	155
	Over-pressure (HBL), bar	5	9	5	9	5	9	5	9
Hot liquor charge	WL charge (EA), %	16	16	16	16	17	17	17	17
	Sulphidity, %	35	35	35	35	40	40	40	40
Heating-up	Heating-up time, min	20	20	20	20	20	20	20	20
	Circulation, l/min	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5
Cooking time	Cooking temperature, °C	170	170	170	170	170	170	170	170
	H - factor	890	890	890	890	890	890	890	890
Terminal displacement									

Fig. 1. *Scenarios for displacement batch cooks.*

Pulp → O_2 → D_0 → E → D_1 → E → D_2

	O_2	D_0	E	D_1	E	D_2
Duration, minutes	60	60	120	180	90	120
Consistency, %	10	10	10	10	10	10
Temperature, C	90	60	70	70	70	70
Chemical charge						
- ClO_2, % as act.Cl	-	KF : 0.20	-	Variable	-	Variable
- NaOH, % on pulp	1.5	-	2.0	0.3	1.3	0.3
- Mg, % on pulp	0.1	-	-	-	-	-
- O_2 pressure, bar	5	-	-	-	-	-

Fig. 2. *Conditions for oxygen delignification and bleaching stages, (KF – Kappa Factor).*

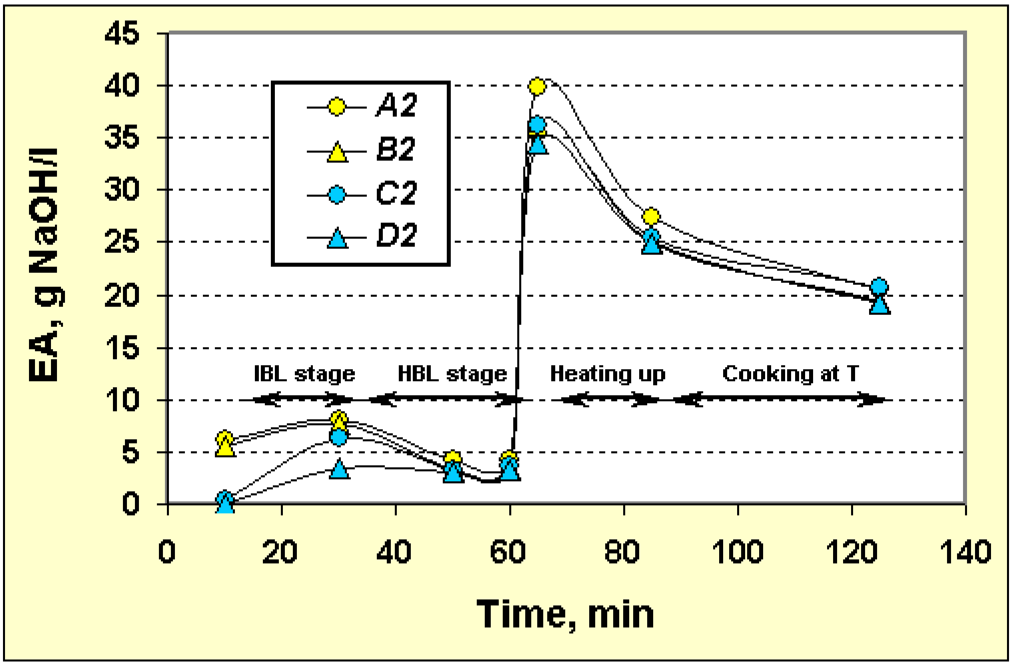

Fig. 3. *Alkali profiles throughout the cooks.*

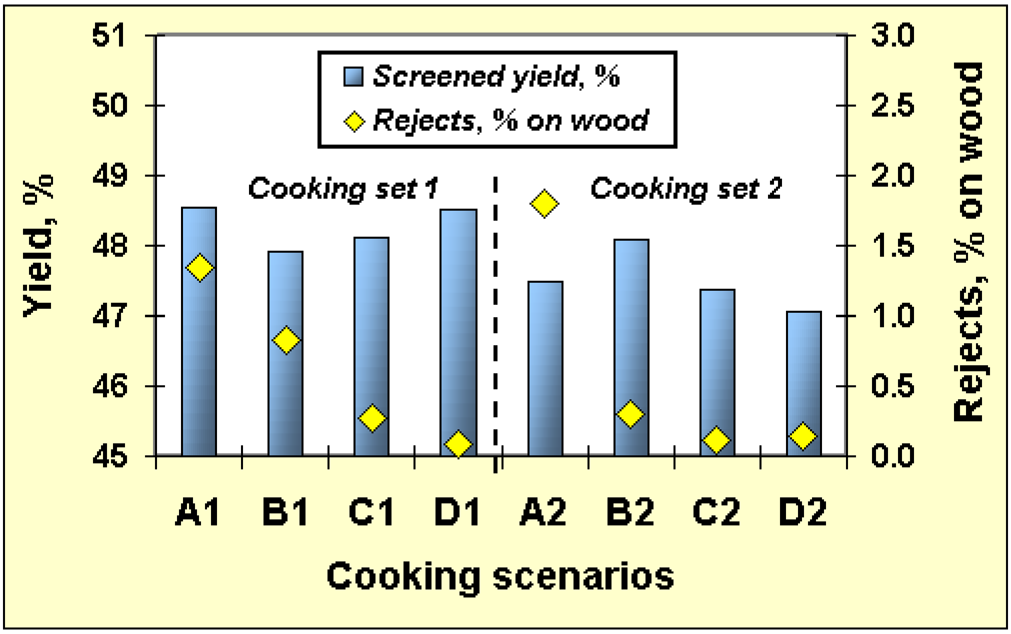

Fig. 4. *Yields and rejects from the cooks.*

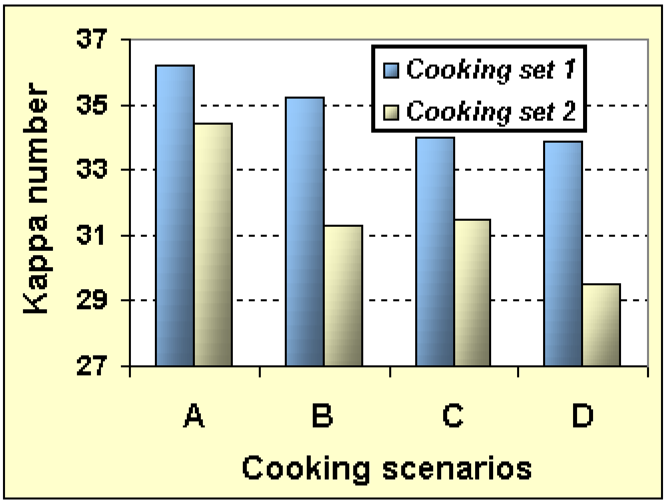

Fig. 5. *Kappa number of the pulps.*

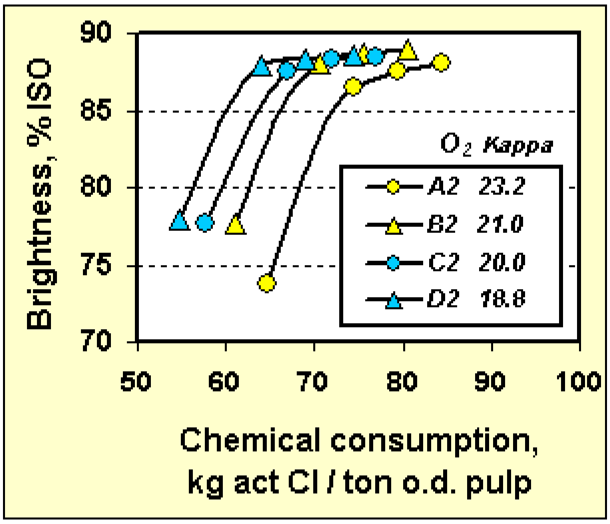

Fig. 6. *Brightness vs. chemical consumption.*

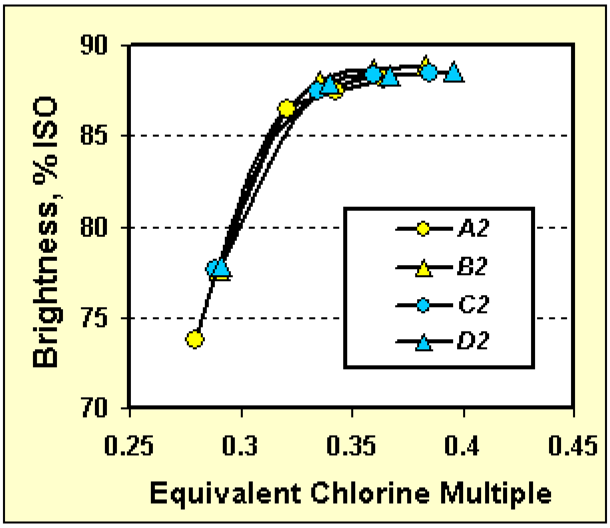

Fig. 7. *Bleachability of pulps.*

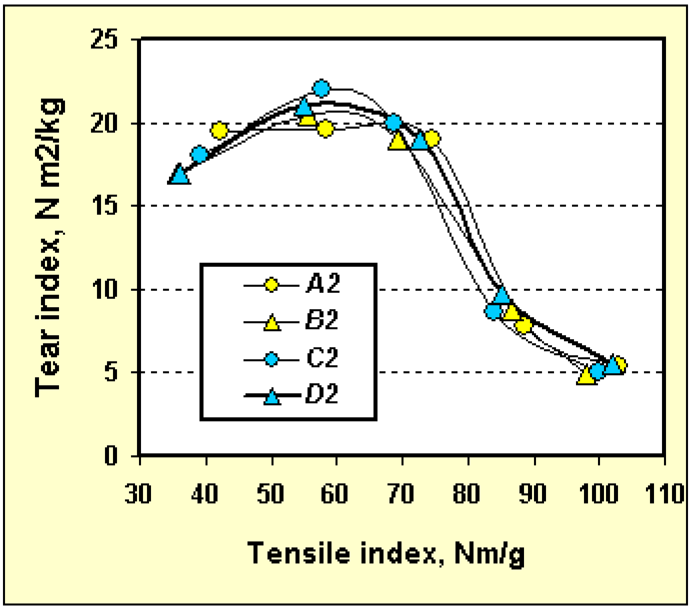

Fig. 8. *Strength diagram for the bleached pulps.*

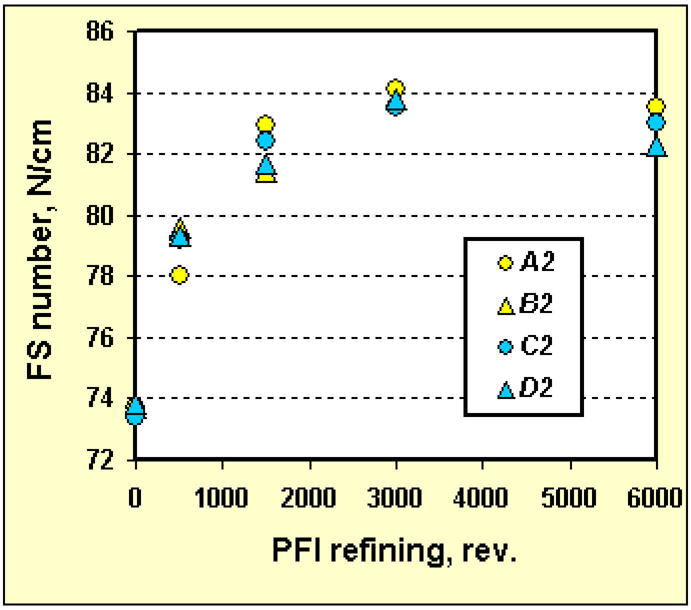

Fig. 9. *Pulmac FS number as a function of refining.*
(FS number - zero-span tensile index corrected to the basis weight of 60 g/m^2)

APPENDIX I. Properties of the bleached pulps.

	Beating at PFI mill				
	0 revs	500 revs	1500 revs	3000 revs	6000 revs
Pulp - A2					
SR number	14	15	17	21	33
Sheet density, kg/ m^3	579	596	656	685	726
Light scattering coefficient, m^2/kg	26.0	24.6	21.4	18.8	16.0
Tensile index, Nm/ g	42.1	58.4	74.6	88.6	103.0
Tear index, N m^2/kg	19.5	19.6	19.0	7.8	5.4
FS number	73.7	78.0	82.9	84.1	83.5
Pulp - B2					
SR number	14	15	17	21	33
Sheet density, kg/ m^3	551	602	663	683	733
Light scattering coefficient, m^2/kg	26.9	24.4	20.5	18.8	16.2
Tensile index, Nm/ g	35.7	55.6	69.3	86.9	98.0
Tear index, N m^2/kg	17.0	20.4	19.0	8.8	4.9
FS number	73.7	79.6	81.4	83.8	82.3
Pulp – C2					
SR number	13	15	16	20	32
Sheet density, kg/ m^3	574	618	665	682	726
Light scattering coefficient, m^2/kg	25.6	22.9	19.7	18.4	15.7
Tensile index, Nm/ g	39.2	57.9	68.9	83.9	100.0
Tear index, N m^2/kg	18.0	22.0	20.0	8.6	5.1
FS number	73.4	79.2	82.4	83.5	83.0
Pulp - D2					
SR number	14	15	17	21	33
Sheet density, kg/ m^3	542	593	671	684	727
Light scattering coefficient, m^2/kg	27.5	24.3	19.8	18.6	16.1
Tensile index, Nm/ g	36.2	54.9	72.6	85.2	102.0
Tear index, N m^2/kg	17.0	21.0	19.0	9.7	5.5
FS number	73.8	79.3	81.7	83.8	82.3