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Towards complete impregnation of wood chips with aqueous solutions

Part 5: Improving uniformity of kraft displacement batch pulping

Keywords:
Pulping uniformity, kraft cooking, displacement batch, front-end conditions, presteaming, FTIR spectroscopy, fibre kappa analyser.

Abstract

Pulping uniformity is critical for improving the performances of cooking and downstream operations such as bleaching and papermaking. In spite of certain success of recent modifications of batch and continuous kraft cooking systems, there is still a lot of room for improving the contemporary cooking systems in order to address problem of heterogeneous pulping. Incomplete penetration and inadequate diffusion of chemicals into wood chips remain the primary reasons for delignification non-uniformity at the scale of a single chip. These factors can be minimized by improved quality of wood chips and optimised cooking conditions. This paper examines the effect of modifying the front-end conditions of kraft displacement batch cooking on pulping uniformity.

Efficient steaming of the chips and application of higher-pressure profile at the front-end of the displacement batch cooking resulted in reductions in amount of rejects and kappa number of the bulk pulp as well as improved uniformity of delignification. In addition, the lower the kappa number level and its variability within the single heartwood and sapwood pine chips were achieved. Application of both modifications simultaneously seemed to provide the biggest effect.

Tiivistelmä

Massan tasaisuus on kriittinen tekijä parannettaessa keittoprosessin ja jatkoprosessien kuten valkaisun ja paperinvalmistuksen toimintaa. Huolimatta viimeaikaisista menestyksekkäistä eräkeiton ja jatkuvan keiton modifikaatiomuutoksista nykyaikaisissa keittoprosesseissa on vielä paljon parantamisen varaa ennenkuin epätasaisen massalaadun ongelmaan päästään kunnolla käsiksi. Yksittäisen hakepalasen mittakaavassa keittokemikaalien epätäydellinen penetroituminen ja puutteellinen diffuusio hakkeeseen ovat ensisijaisena syynä epätasaiseen delignifioitumiseen. Delignifioitumiseen voidaan vaikuttaa hakkeiden laatua parantamalla ja optimoimalla keitto-olosuhteita. Tässä artikkelissa tarkastellaan syrjäytyseräkeiton alkuvaiheen modifioinnin vaikutusta sulfaattimassan tasaisuuteen.

Tehokas hakkeiden höyrytys ja korkea paine keiton alussa laskevat rejektin määrää ja alentavat massan kappalukua sekä parantavat delignifioitumisen tasaisuutta. Modifikaatioiden avulla yksittäisten sydän- ja pintapuuhakkeiden sisällä saavutetaan matalampi kappataso sekä sen pienempi vaihtelevuus. Tulosten perusteella molempien modifikaatioiden yhteiskäytöllä voidaan parhaiten parantaa massan tasaisuutta.

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Introduction

The uniformity of pulping and the ways to improve it have recently attracted a lot of attention. Uniformity of pulping has become a key issue in optimising the performance of digesters. More homogeneous pulping enables shorter cooking times, improved pulp strength /1,2/, lower bleaching costs /3/, and improved environmental performance /4/. On the other hand, heterogeneous pulping may limit the maximum achievable lignin removal /4,5/ and lead to complications in downstream operations, such as bleaching and papermaking /6/. According to current experience, conventional cooking systems do not provide the required uniformity of pulping. Recent modifications of batch and continuous cooking systems have been quite successful in addressing this problem, showing improved results in terms of rejects and pulp strength /7,8/. However, there is still a lot of room for improving contemporary cooking systems to meet the requirements for uniform pulping.

The uniformity of pulping can be considered at two scales: at the macro scale of the digester and at the micro scale of a single chip. The variability at macro scale arises from temperature and active chemical concentration gradients within the digester, from errors in chip moisture determinations, incorrect chemical charges and liquor channelling /6/. Even if all digester variables are kept constant throughout the digester, the pulp can still be non-uniform. Non-uniformity at the chip scale may arise from heterogeneities in the chip itself, incomplete penetration, and inadequate diffusion of chemicals /6,9/. Primary reasons for inefficient penetration and diffusion are oversized chips and improper impregnation conditions.

Unfortunately, not all requirements for uniform pulping can be met. However, some conditions for production of more homogeneous pulp can be fulfilled or strongly influenced. One approach is to improve the wood chip quality either by efficient screening to produce uniform thin chips without knots and reaction wood /10-12/, or by applying an innovative chipping technique /13/. Another approach involves application of chip pre-treatments and optimisation of conditions at the front-end of the cook, aiming for improved penetration and efficient diffusion /14-17/.

The current study is part of a larger research effort undertaken at the Helsinki University of Technology, dealing with the process of liquid penetration into wood chips, its modelling, and optimisation of process conditions. The present article examines the scope for improving the uniformity of pulping by modifying the front-end conditions of kraft displacement batch cooking. The effects of chip presteaming and increased pressure in the hot black liquor stage on the uniformity of delignification are examined with reflection FTIR spectroscopy and a technique that uses fluorescence staining of fibres.

Experimental

Wood chips

The chips for cooking simulations were prepared from pine (*Pinus silvestris*) roundwood with the properties shown in **Table 1**. Chipping was done 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 formation of rejects during cooking, the over-thick fraction of chips was used in cooks together with accepts, in proportions of 5 % and 95 %, respectively. In addition, handmade chips were prepared from heartwood and sapwood portions of the same pine roundwood. The handmade chips were cut into exact dimensions: length (longitudinal direction in wood) – 34 mm, width (tangential direction in wood) – 14 mm, and thickness (radial direction in wood) – 8 mm.

***Table 1**. Properties of pine roundwood.*

Age of the tree, years	80	
Diameter of the tree, mm	250	
Heartwood fraction, % (volume)	53	
	Heartwood	**Sapwood**
Basic density, g /cm^3	0.390	0.410
Dry-matter content, %	75	45
Moisture content, % on wood	33	122

Cooking trials

Cooking experiments were performed at the Helsinki University of Technology by using a laboratory digester similar to the one described elsewhere /18/. This system is enabled to simulate cooking stages, displacement times, and circulation rates comparable to those of mill-scale kraft displacement batch processes. The chip samples were cooked in accordance to four scenarios with different front-end conditions, while other cooking conditions were kept constant. Scenario A resembled the front-end conditions used in some contemporary industrial kraft displacement batch systems. Three other cooking scenarios included certain modifications, namely chip presteaming, a higher-pressure profile, or both. The stages of the displacement batch process and the cooking conditions are presented in **Fig. 1**. After cooking and washing, the pulp samples were screened on a flat screen with 0.15 mm slots. The cooking results were evaluated based on the yield and amount of rejects as well as pulp properties. The kappa number and CED viscosity of the cooked pulps were measured in accordance to the standards SCAN-C 1:77 and SCAN-CM 15:88.

A small wire basket containing several handmade heartwood and sapwood chips was placed in the middle of the chip column before each cook. To wash out the residual chemicals after the cooking, the basket with handmade chips was placed in a large vessel with deionised water and kept there at room temperature for 24 hours. After that, the chips were gently removed from the basket and frozen at -20 °C for further studies.

Measurement of pulping uniformity

A number of different approaches can be used for evaluating the uniformity of pulping. Basic cooking data, such as rejects and kappa number may give some indication of the uniformity of pulping. It is also possible to mircotome the cooked chips and determine the lignin content of the chip sections by a wet-lab method, such as chlorine number /2/. In addition, Fourier-transform infrared (FTIR) spectroscopy /19-21/ and UV spectroscopy /22,23/ can be used as non-destructive techniques for estimating the amount of lignin in pulp fibres or within cooked chips. Variability in lignin content can also be measured at the fibre level, for example, by a density gradient column that takes advantage of the fact that lignin and cellulose have different densities /21/, or by a technique that uses fluorescence of Acridine Orange (AO) stained fibres for measuring their kappa number /24/.

In the current study, the effect of front-end conditions on the uniformity of delignification was examined through two approaches. In the first approach, the kappa numbers of single fibres and their distribution were studied. A pulp sample from every cooking scenario was analysed by using the flow-through fluorescence image analyser at the University of Washington. A detailed description of the apparatus and method is given in /25/. Kappa distributions, or the percentage of fibres versus kappa, were obtained from five replicate measurements.

In the second approach for examining the uniformity of pulping, the kappa number distribution within the cooked handmade chips was assessed based on FTIR measurements. Three heartwood and three sapwood chips from each cooking trial were chosen for the analyses. The chips were cut across the thickness dimension, as shown in **Fig. 2**. As a result, three test-pieces with corresponding surfaces of 0 mm, 2 mm, and 4 mm depth were prepared from each chip. The chips were cut along the same annual ring. Heartwood chips were cut through the earlywood region of the annual ring. In the case of sapwood chips, it was not clear whether cutting was done through earlywood or latewood regions of the annual ring. Before analysis, the test pieces were thawed and dried in an oven at 105 °C for 24 hours. Infrared (IR) spectra were measured from fifteen points selected on the surfaces of each prepared test piece (**Fig. 2**). Nine points were selected along the chip length dimension, from the middle to the edge of the piece, with a step of two millimetres. Six other points were selected along the chip width dimension, from the middle to the edge of the piece, with a step of one millimetre.

The IR spectra were recorded using FTIR spectroscopy at the reflection mode of operation. A Bio-Rad FTS 6000 spectrometer equipped with a UMA 500 microscope fitted with an MCT liquid nitrogen-cooled detector was used. The spectra were measured in the region between 4000 and 700 cm^{-1}. The spectral resolution was 8 cm^{-1} and 100 scans per sample were accumulated prior to Fourier transformation. Analysis area was 100 x 100 μm.

Calibration and FTIR data analyses

Infrared spectra were calibrated to traditional wet-lab analysis of kappa number. Three chip samples were cooked under conditions corresponding to scenario B (**Fig. 1**) but with different H-factors. Pulps with kappa numbers of 36.7, 65.8, and 85.7 were produced using H-factors of 650, 300, and 200, respectively. Laboratory sheets prepared from the pulp samples were dried in an oven at 105 °C for 24 hours.

Twenty IR spectra were recorded from each of the pulp sheets. The spectra were baseline-corrected at 3800, 1850 and 930 cm^{-1}. The spectral region between 1520 cm^{-1} and 1200 cm^{-1} was used for calibration. Multivariate calibration was done with PLS modelling. PLS stands for projection to latent structures by means of partial least squares analysis. To enhance the predictive power of the calibration model, the spectral data were pre-processed by using the orthogonal signal correction (OSC) method prior to data analysis. PLS modelling was carried out by Simca 7.01-P software, yielding a four-component model with $R^2X = 0.96$, $R^2Y = 0.98$, and $Q^2 = 0.97$. The overall model statistics point to a workable model of sound explanatory and predictive powers. The first component is the most important (**Fig. 3**), accounting for more than 75 % of the modelled variations. The PLS loading spectrum of the first component indicates that spectral information of great relevance for modelling the kappa number can be found in the regions of six peaks. These principal peaks can be assigned to the lignin structures as follows /26/: 1512 cm^{-1} and 1423 cm^{-1} - aromatic skeletal vibrations, 1462 cm^{-1} and 1370 cm^{-1} - C-H deformations, 1270 cm^{-1} and 1226 cm^{-1} - guaiacyl ring breathing with CO-stretching. The agreement between the observed and predicted kappa numbers is quite good as shown in **Fig. 4**.

The spectral data recorded from the prepared test pieces were pre-processed in a similar way as the spectra from the calibration set. Then, the kappa number was predicted based on spectral data, using the established calibration model. All these steps were performed with the Simca 7.01-P software package.

Results and discussion

Cooking results

Basic data from four cooks are listed in **Table 2**. The amount of rejects and the kappa numbers of the pulps indicate that the front-end modifications have a positive effect on the uniformity of pulping. Higher pressure and chip presteaming both drastically reduce the amount of rejects; yet, presteaming seems to be more efficient. Also, the average kappa number of the pulp was significantly reduced by the application of either chip presteaming or higher pressure during the hot black liquor stage. The lowest kappa number achieved after D-cook pulp indicates that higher pressure and steaming may have a synergistic influence on the performance of the cooking.

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

Scenario	A	B	C	D
Screened yield, %	47.5	48.1	47.4	47.1
Rejects, % on wood	1.80	0.30	0.12	0.15
Kappa number	34.4	31.3	31.5	29.5
CED viscosity, ml/g	1100	1160	1170	1140

The effect of modifications can be primarily explained by the improved penetration of liquor into the wood chips during initial cooking stages /15/. More thorough penetration reduces the volume within the chips that does not get into contact with chemicals, and shortens diffusion distances. It has also been shown that presteaming of chips may increase the diffusion rate of some ions /27/. These phenomena lead to more uniform distribution of chemicals within the chip when high cooking temperatures are reached. As a result, delignification reactions proceed more uniformly within the wood chips, resulting in less rejects and a lower kappa number for the final pulp.

Fibre kappa distributions

Kappa distributions of the pulp samples from cooking scenarios A and B are compared in **Fig. 5**. Cooking with a higher-pressure profile improves the uniformity of pulp. This can be concluded from the narrower kappa distribution and higher percentage of fibres at average kappa. Also, the high-kappa tail present in the pulp sample from scenario A is not seen in the pulp sample from scenario B. The same result is obtained when using chip presteaming but keeping the low-pressure profile of cooking front-end (**Fig. 6**). Actually, the kappa distributions of pulp samples from scenarios B and C are very similar.

***Table 3**. Statistics on fibre kappa distributions.*

Pulp sample *(scenario)*	Measured Kappa	1st Moment	2nd Moment	3rd Moment
A	34.4	34.45	39.81	1834.14
B	31.3	31.31	33.85	1240.48
C	31.5	31.42	34.25	1275.06
D	29.5	29.51	31.75	1086.76

Kappa distributions of the pulp samples from cooking scenarios D and B are compared in **Fig. 7**. The D sample has a slightly narrower distribution and higher percentage of fibres at average kappa. This indicates that chip presteaming improves pulp uniformity even when the chips are cooked with the

higher-pressure profile, but the effect is less significant than in case of cooking with the lower-pressure profile. The statistics on the fibre kappa distributions given in **Table 3** clearly show that both chip presteaming and higher pressure result in more homogeneous pulping, and that the best pulp uniformity is achieved with scenario D.

FTIR measurements

The data obtained from the FTIR measurements are summarized in **Fig. 8** and **Fig. 9**. The average kappa numbers predicted from all FTIR measurements made with the handmade chips from the corresponding cooks are shown in **Fig. 8**. Scenario A resulted in the highest average kappa for both heartwood and sapwood chips. The chips from scenario D seem to have the lowest average kappa number after cooking, while the values for scenarios B and C occupy the intermediate positions. These results show a similar trend as the bulk kappa numbers of the pulps (**Table 2**). It can be noticed, however, that there are significant differences in kappa number values shown in **Table 2** and **Fig. 8**. The reason for this is that **Table 2** presents the kappa numbers of the bulk pulps produced from the industrial chips, while **Fig. 8** presents the average kappa numbers within the handmade chips predicted based on the FTIR measurements.

Fig. 9 compares standard deviations in predicted kappa numbers within the handmade chips, which can be seen as a factor indicating the uniformity of delignification. When considering heartwood chips, there is a gradual improvement from scenario A to scenario D. For sapwood chips, the highest standard deviation corresponds to scenario A. There is no significant difference between the values for scenarios B, C and D. It has been shown /15/ that because of the small initial air content in pine sapwood chips it is enough to apply either slight presteaming of the chips or high pressure to achieve close to complete penetration. This could be the reason why the application of both chip presteaming and the higher-pressure profile (scenario D) did not further improve the uniformity of delignification of sapwood chips.

The main idea behind the FTIR measurements was not to get accurate quantitative data on lignin contents, but rather to produce a qualitative measure of pulping uniformity. Accordingly, the kappa numbers assessed from the FTIR measurements were used to draw delignification profiles for the handmade chips. **Appendix I** shows kappa number profiles within heartwood and sapwood chips for cooking scenarios A and D. The points on the graphs correspond to the average kappa obtained from the measurements on three different chips. Of course, the kappa number prediction based on the IR spectra may contain an error. Sample preparation for FTIR measurements could also affect the results, especially in the case of sapwood chips, for which the cutting pattern was not very accurate. However, these profiles can be assumed to give a certain indication of the lignin distribution within the cooked chips.

Let us first consider the heartwood graphs. In cooking scenario A, the middle part of the chip (4 mm deep) is clearly undercooked. It is interesting to note that there is a gradual transition along the chip length from the edge kappa number of 40 to the undercooked regions with kappa numbers over 90. At the same time, this transition is very abrupt along the chip width. At a point, which is only 2 mm from the chip edge, the kappa number is already near 90. This may reflect the limitations of mass transfer in the tangential direction in softwoods. The “2 mm deep” chip layer shows similar trends as the core layer, but within the lower kappa number region of 60-70. There is also a clear gradient of kappa numbers in the radial direction of the wood chip, emphasising the importance of chip thickness for achieving the uniform pulping. In cooking scenario D, the uniformity of delignification of heartwood chips is drastically improved. The “2 mm deep” chip layer has a uniform kappa number profile within the region of 40-50, which is very close to the kappa level of the chip surface. Still, some gradient is present within the middle (“4 mm deep”) layer of the chip. However, the undercooked region is much narrower than in scenario A, and the kappa number level of this region is lower.

For sapwood chips, a similar behaviour can be observed. Cooking scenario A resulted in heterogeneous delignification of the sapwood chips, with clear kappa number gradients taking place in longitudinal and radial directions. Wide undercooked regions with a relatively high kappa level of 80 are present in the middle of the chip. Application of presteaming and the higher-pressure profile (scenario D) provided more homogeneous delignification with practically no gradients in longitudinal direction. A lower kappa number level was achieved through the whole chip. Still, a slight gradient was present along the thickness dimension of the chip. The effect of pulping modifications on the homogeneity of pulping within the sapwood chips was not as pronounced as in heartwood chips.

Summary

Presteaming of chips and application of a higher-pressure profile at the front-end of displacement batch cooking improved the uniformity of pulping. As a result, the amount of rejects and the kappa number of the bulk pulp were significantly reduced. The positive effect of modifications on the uniformity of pulp samples is seen from the fibre kappa distributions. Application of both modifications simultaneously produced the most uniform pulp.

Interesting results were obtained from the examination of kappa distributions inside the handmade chips with the FTIR technique. Cooking with front-end conditions corresponding to those of many contemporary kraft displacement batch systems (scenario A) resulted in very heterogeneous delignification profiles within both heartwood and sapwood chips. Clear gradients in kappa number take place in the longitudinal and radial directions of the wood chips. The middle part of the chips contains large undercooked regions with high level of kappa numbers. Application of presteaming and a higher-pressure profile reduced the kappa number level, and minimized the gradients and variability of the kappa number within the chips.

Acknowledgements

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Figures

FRONT END		Scenarios	A	B	C	D
	Chip charge					
	Chip steaming	Steaming time, min	0	0	30	30
		Steaming temperature, °C	-	-	105	105
	IBL stage - warm black liquor impregnation	Time (IBL), min	20	20	20	20
		Temperature (IBL), °C	80	80	80	80
		Over-pressure (IBL), bar	2	2	2	2
	HBL stage - hot black liquor treatment	Time (HBL), min	30	30	30	30
		Temperature (HBL), °C	155	155	155	155
		Over-pressure (HBL), bar	5	9	5	9
	Hot liquor charge	WL charge (EA), %	17	17	17	17
		Sulphidity, %	40	40	40	40
	Heating-up	Heating-up time, min	20	20	20	20
		Circulation, l/min	3.5	3.5	3.5	3.5
	Cooking time	Cooking temperature, °C	170	170	170	170
		H - factor	890	890	890	890
	Terminal displacement					

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

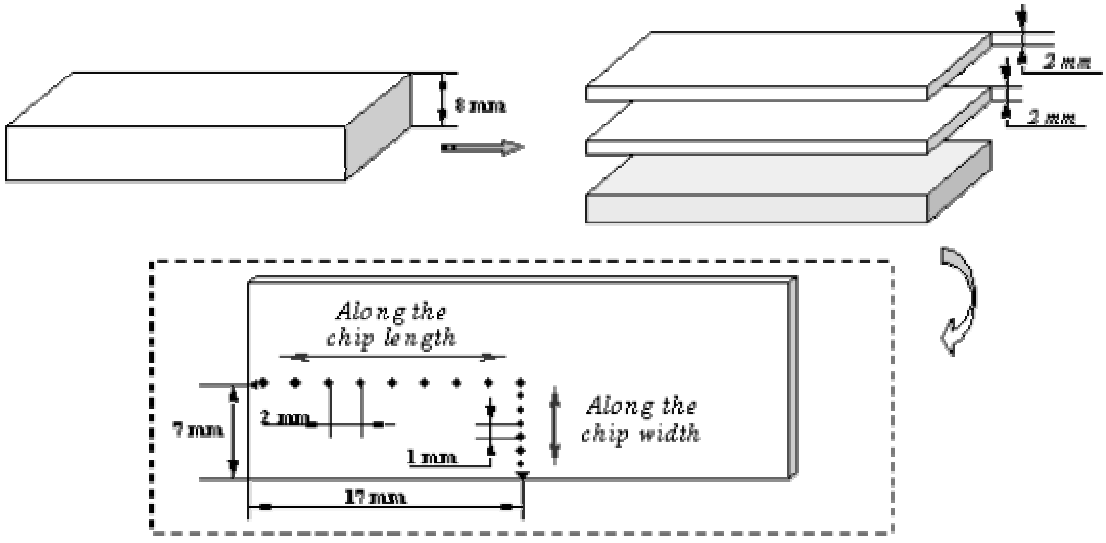

***Fig. 2**. Preparation of test-pieces and selection of the points for FTIR analysis.*

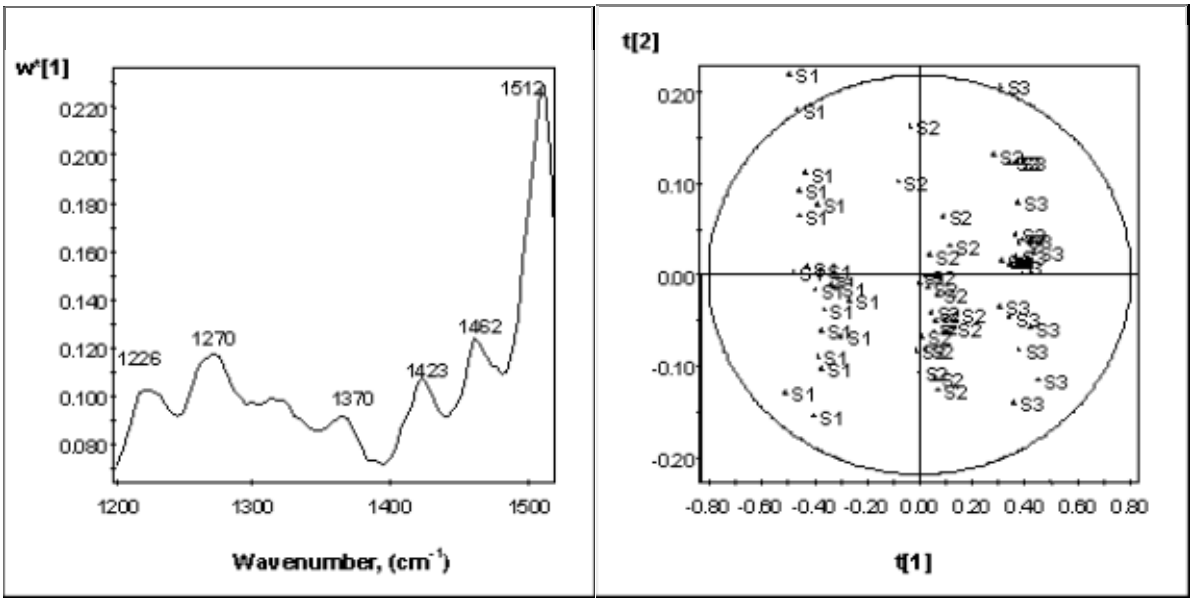

Fig. 3. *PLS loading spectrum of the first component and PLS t_1/t_2 score plot of the calibration data set.*

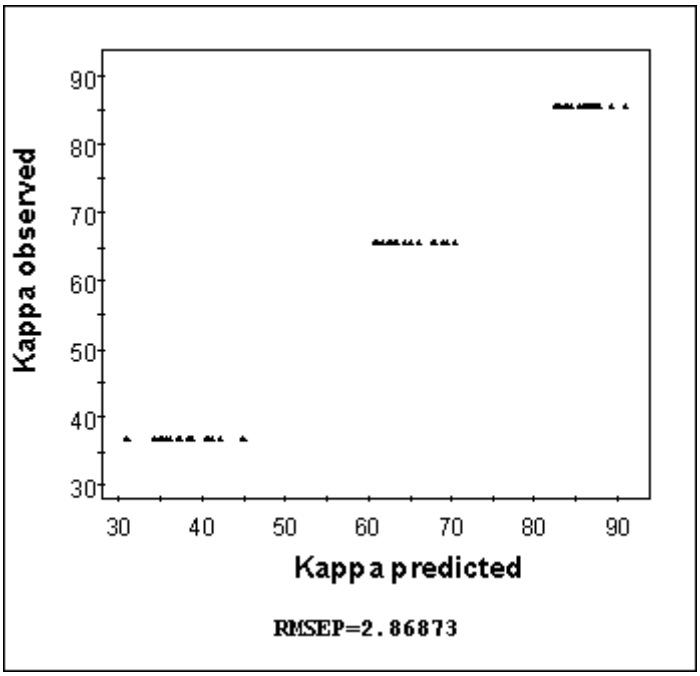

Fig. 4. *Relationship between observed and predicted kappa number.*

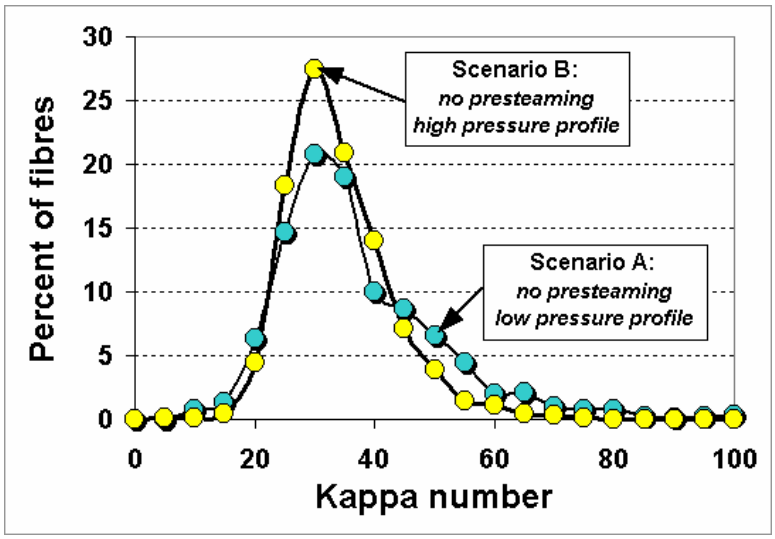

***Fig. 5**. Fibre kappa distributions: scenario B vs. scenario A.*

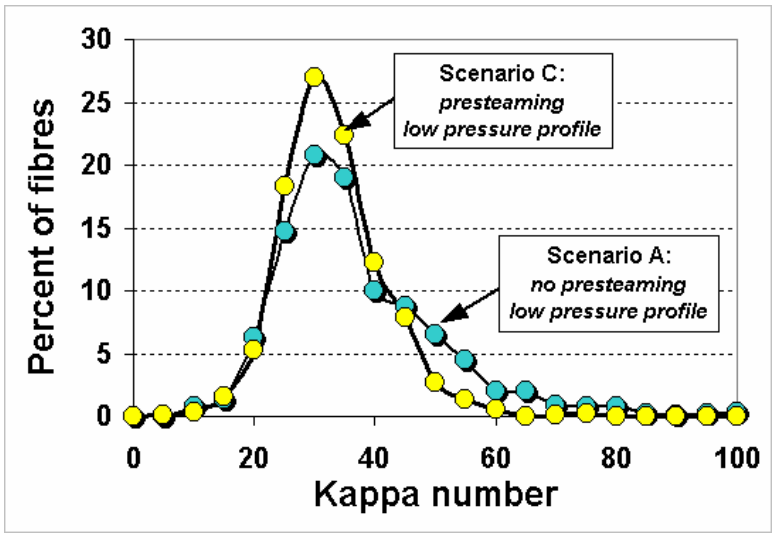

***Fig. 6**. Fibre kappa distributions: scenario C vs. scenario A.*

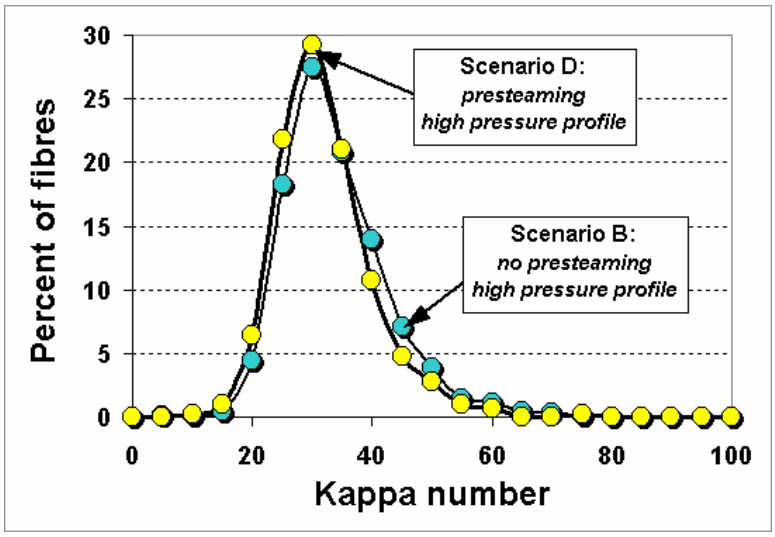

***Fig. 7**. Fibre kappa distributions: scenario D vs. scenario B.*

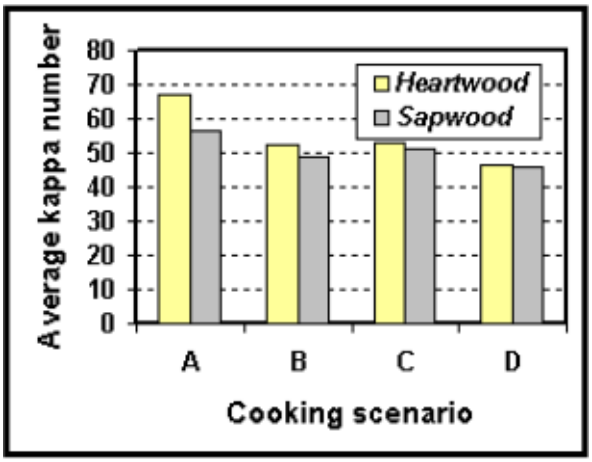

Fig. 8. *Average kappa number. (Calculated from all FTIR data)*

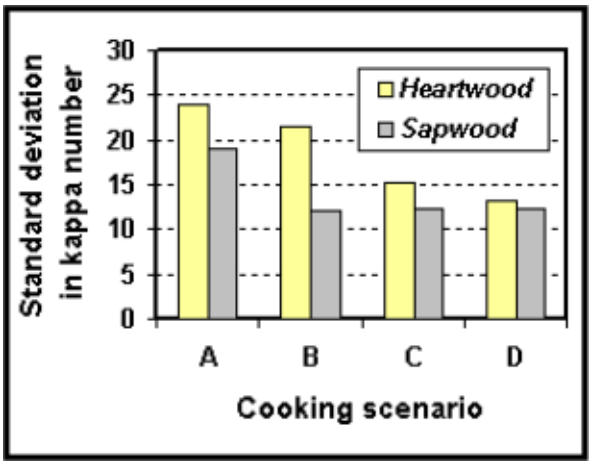

Fig. 9. *Standard deviation in kappa number within the handmade chips.*
(Calculated from all FTIR data)

APPENDIX I. Delignification profiles within the handmade chips.