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Some Mechanical Pulp Fibre Characteristics, Their Process Relationships and Papermaking Significance

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1 INTRODUCTION

Mechanical pulp fibre is the main component in the lower priced segment of printing paper. Besides low cost, the advantages of such fibres are fairly good brightness - especially after bleaching, and good opacity. One main disadvantage is high refining energy consumption, another of rising importance, is the coarse mechanical pulp fibres which cause problems in a market that demands ever increasing paper smoothness.

The details of the mechanical pulping processes have been the subject of numerous studies, in general to elucidate the mechanisms of this process of large economical importance, and more specifically to explore the possibilities for reducing the large amount of energy currently consumed in the refining, and to analyse the gradual changes in the fibre material, during the progressive refining. The problems of the coarse fibre content can be approached both by developing refining methods that reduce the fibre wall thickness, and to change to raw material of lower fibre wall thickness.

In spite of great efforts in the study of mechanical pulping process and fibres, there still are questions that deserve to be looked into, and new useful techniques that may shed light onto the process, the pulp and the final paper product.

In a series of experiments, we have studied various aspects of mechanical pulping processes, as well as the effects on the fibre development during progressive refining. Primarily TMP, but even groundwood fibres have been analysed, and characteristic and significant differences have been quantified. The relationship between coarse mechanical fibres and paper surface smoothness is analysed, especially the roughening effect by coarse surface fibres when subjected to moisture. Inevitably, there will be some insufficiently refined shives and fibre bundles in the paper, acting as web break initiators. The details of the web break mechanisms are of significance for the understanding of web runnability. Today, the recycled mechanical pulp fibres are a main component in many printing paper

grades, and the characteristics of such fibres have important bearing for printing paper quality.

2 MECHANICAL PULPING

Today, there are two main process designs for mechanical pulping, i.e.,

The grinding of wood logs, having two versions, GWP, and pressurized, PGW, Thermomechanical pulping, TMP of wood chips in refiners

For various reasons, like paper strength considerations, raw material utilization, process efficiency and energy utilization (steam recovery), the TMP process has gained a dominant position. The grinding process, especially the PGW version, offers however some quality advantages. It is quite common to separate the steps of the mechanical pulping processes two main sequences (1,2), namely,

- 1) An initial, *fibre separation* stage, where the wood/chips are disintegrated into fibres, and
- 2) A further, *fibre development* stage, when the separated fibres are subjected to further mechanical treatment, removing surface fibre material, thus reducing the fibre wall thickness, flexibilizing the fibres and rendering them more suited as paper structure components.

The two stages are to some extent overlapping. The second is the most complex one. Many studies have been undertaken on either two, and much experimental evidence has been accumulated (1-9). Although both groups are assumed to take place during both mechanical pulping process designs, the detailed fibre treatment, as well as the extent of the fibre development are likely to differ. Detailed knowledge on this point is limited.

No pulping process is perfect with respect to fibre separation. There will always be some not fully defibrated fibre bundles. After paper forming, they will act as break initiators in the paper web, and should be efficiently removed by screening and cleaning. However, a total removal of such imperfect components is impossible.

For either process designs, the produced pulp has to be subjected to a latency treatment in hot water, to release "frozen in" stresses established during the fibre separation and development at high temperature, followed by a rapid cooling. The latency treatment allows the fibres to regain their fairly straight shape of minimum internal stress, that they had in the wood matrix.

One important issue often discussed, is the question of where in the fibre structure the fibre separation rupture most commonly occurs. Having a structure of concentric layers of different morphological and chemical components, the actual rupture surface will have significant bearing on the paper that results from such fibres. An important question which has been less studied, is the typical details of fibre development, for instance, the relationship between fibre cutting and fibre wall delamination and thinning.

3 ANALYSIS OF SOME FIBRE CHARACTERISTICS IN A TMP MILL

3.1 The TMP plant layout and process, for the pulp under study

The fibre material coming from a mechanical pulp mill, will reflect the technical design of that particular mill, and have limited general validity. This should be remembered when judging the results of the present study. Fibre fractions from the TMP plant of a SC magazine paper mill were analysed. The modern (1992) TMP plant has a layout as indicated in Fig. 3.1 (10): Sprout/Bauer TWIN-60 refiners. Refiner stages 1 and 2: 24 MW motors; stage 3: 19 MW; all 1500 rpm. All stages pressurized, 3.6, 3.6 and 2.5 bar resp. Raw material: Norway spruce (Picea Abies). After latency treatment following stage 3, the pulp is screened and cleaned. Reject is thickened to 35% cons., and added to the inject of refiner stage 2; i.e., no separate reject refining. Freeness of the sampled accepted pulp to the paper mill; 26 °CSF. Thus, there are five different fibre populations; i.e., pulp from stage 1, 2 and 3, as well as reject fibres and final, screened pulp for the paper mill. In the fibre analysis, fibres retained on the 48 mesh screen of the BauerMcNett Fractionator were collected for analyses.



Figure 3.1: Process layout of the TMP plant where samples for the study were taken

3.2 Characteristics of the fibre fractions from different refining stages

Table 3.1 presents fibre fractions from refiner stages 1, 2, 3 and the final, screened accept.

Table 3.1:

Fibre fractions in pulp at different stages in the TMP process. The fibres from the +28 mesh and +48 mesh populations were combined and analysed.

Fibre from stage no:	+ 28 mesh fraction	+ 48 mesh fraction	Total fibre fraction
Refiner stage 1	55.5%	16.8%	72%
Refiner stage 2 (screened reject added to feed)	55.7%	15.5%	71%
Refiner stage 3	53.2%	12.8%	66%
Screened accept	40.3%	14.2%	55%

The fibre separation rupture in mechanical pulping does not occur through the middle lamellae material itself (the typical feature in the fibreboard process). The rupture occurs somewhere at the middle lamellae/fibre wall interface, although there seem to be some conflicting opinions on this point (1,5,8).

During fibre separation, the middle lamellae will split from both fibres, or be attached to one of the two. If all the middle lamellae remains on the fibres, defibrated fibres will have 50% coverage.

The continued fibre development then removes surface material from the fibres. If taken in the structural sequence, it is likely to be remains of the middle lamellae, then the primary wall, P and outer secondary wall, S_1 , and layers of the inner secondary wall, the S_2 .

To assess the change in middle lamellae coverage during refining, fibre fraction samples were bromium treated (14,15) (Appendix A). Fig. 3.2 presents a BEI picture of fibres, first refiner stage. Most of the fibres are dark, indicating carbohydrate composition (Appendix A); suggesting P, S₁ or S₂ fibre layers. Other parts are bright, revealing middle lamellae. Middle lamellae patches are located along bordered pits, attached to the fibre by pit material. The rupture edges of the middle lamellae patches across the fibre most often have angles

indicating the fibril angle of the S_1 layers. The fibre surface appears either fully covered, or completely free, confirming the splitting at the lamellae/fibre interface. The covered area fraction is much less than the maximal 50%.

Consisting mainly of hydrophobic lignin, the bonding ability of the middle lamellae is low. Minimizing the middle lamellae coverage of the fibres is important to improve pulp quality. The fibre coverage can be assessed by image analysis of micrographs like Fig. 3.2.



Figure 3.2: Backscatter electron micrograph (BEI) of fibre sample, first refiner stage. Bromium treated. The middle lamellae, rich in lignin, appears bright. Observe edges of the middle lamellae having large angle to the fibre axis, probably reflecting the structure of the S_1 fibre layer.

It can be questioned whether apparent surface coverage gives a correct measure of the real coverage. The fibre cross sections (Appendix A) of fig. 3.3 show that middle lamellae material often is loosely attached to the fibres, not preventing fibre surfaces to establish contact with fibre material. The coverage may be assessed based on fibre cross section images, like Fig. 3.3. Here, the fibre perimeter fraction actually attached to middle lamellae can be estimated.



Figure 3.3: Cross sections of TMP fibres. Middle lamellae is brighter than the fibre wall. Often only loosely attached to the fibre surface. This is clearly seen on the blown up detail at right

Middle lamellae coverage was determined on the fibre populations by both methods, Table 3.2. Only first stage and final pulp readings are listed. Confidence limits were not determined.

Table 3.2:

The fibre surface fraction covered by middle lamellae material. Two methods: 1) assessment of BEI micrographs of fibre surfaces: Area covered by middle lamellae, A_{ML} , as a per percentage of total fibre surface A_{FS} 2) evaluation of SEM Micrographs of fibre cross sections: Perimeter covered by middle lamellae, P_{ML} , as a per percentage of total fibre perimeter, TP.

Pulp type> Surface analysis	TMP fibres, first refiner stage (Freeness 296 °CSF)	TMP fibres, screened pulp (Freeness 26 °CSF)
Fibre wall surface fraction, $A_{\rm ML}/A_{\rm FS}$	24%	15%
Fibre cross section perimeter fraction, P _{ML} /TP	13%	10%

The results of the two methods differ, method 2) yielding the lowest readings. The middle lamellae patches are only partly attached to the fibres. The number ratio of Table 3.2 between the two methods, for first stage and for the screened pulp, is fairly constant. The findings are in the same range as reported by Kibblewhite in (5), but lower than his finding in (11).

The middle lamellae coverage drops during refining. In screened pulp, only some 1/10 to 1/7 of the surface area is covered. Middle lamellae on fibres cannot be important for fibre bonding. The middle lamellae material remains in the stock, acting like filler rather than bonding material.

It is surprising that fibre coverage is only cut in half during the refining from 296 to 26 °CSF resp. At the same time there is a significant drop in fibre wall thickness. An widely spread idea of having a sequence of successive fibre wall "strippings" of the concentric fibre layers; for the whole fibre population, appears unrealistic.

Similar estimations of middle lamellae coverage on other fibre populations have confirmed the findings of Table 3.2.

3.3 The fibre wall cross section characteristics, and development during refining

The fibre wall thickness varies around the wood cell's cross section; by a factor of 2 or more. The wall is thicker at the cells' corners. This is observed in pulped fibres, especially mechanical ones. Our methods assess total cross sectional area of the fibre, as well as fibre perimeter, allowing calculation of the true average fibre wall thickness.

Some changes of the fibre characteristics during refining were quantified, based on image analyses of SEM micrographs, BEI mode (Appendix A, B).

Table 3.3:

	Number of fibres	Cross section wall area Α, μm²	Fibre perimeter P, µm	Form circle 4πA/P ²	Fibre wall thickness
Refiner stage 1	632	219	94	0.60	2.66μm σ = 1.16
Refiner stage 2	305	214	94	0.54	2.55μm σ = 1.15
Refiner stage 3	364	193	88	0.55	2.47μm σ = 1.10
Screened pulp	983	177	87	0.64	2.23μm σ = 1.04
Screen rejects	292	182	89	0.55	2.29μm σ = 1.23

Fibre characteristics as determined by the image analyses of TMP fibre cross sections.

The fibre wall thickness is consistently reduced, as has been found in other studies. Here, a total of some 16% average fibre wall thickness reduction was found from first stage to screened pulp. At the same time, the middle lamellae fibre coverage was only cut in half (Table 3.2). Refining thus certainly does not remove outer fibre layers uniformly, for the whole population.

Will the fibre development stage of refining tend to remove specifically coarse, or thin walled fibres? The variation within each population, of the fibre wall thickness offers some

clue, Table 3.2. Comparing wall thickness averages and standard deviation indicate that the fibres within a population are fairly uniformly treated, regardless the fibre wall thickness. There is no indication that coarse or slim fibres are selectively eliminated.

3.4 The cross sectional shape of the fibres during refining

Fibre cross sectional shapes were assessed by "Form circle", FC (Appendix B) which is useful to assess fibre collapse. Cf. Table 3.3.

Fig. 3.4 depicts FC readings as a function of fibre wall thickness, for the fibres after the third refiner stage, as well as that of the screened and latency treated pulp. Two interesting points:

- Fibre collapse (low FC reading) is more pronounced for thin walled fibres

- Latency treatment increases the FC (reduces fibre collapse). The increase is most pronounced for the much collapsed thin walled fibres.



Figure 3.4: Form circle (FC) for the fibres 1) after third refining, and 2) after latency treatment and screening. Most pronounced fibre collapse (low FC) for thin walled fibres. The latency treatment raises the FC, i.e., the fibres regain more of the original tubular shape of the wood cells. (From Table 3.3)

Fig. 3.5 depicts fibre perimeter as a function of fibre wall thickness for the same two fibre populations. Perimeter length is fairly independent of fibre wall thickness, although the slight minimum for fibre walls of $3 - 4 \mu m$ thickness has repeatedly been found.



Figure 3.5: Fibre perimeter as a function of fibre wall thickness. Fibres after third refiner stage, and after latency treatment and screening. The perimeter is fairly uniform, with a possible slight minimum for wall thicknesses in the $3 - 4 \mu m$ thickness range. From Table 3.3.

3.5 The reduction of the cell wall thickness during the fibre development stage

The gradual reduction of cell wall thickness is essential in TMP refining. Several conceivable mechanisms may cause this reduction, like fibre wall densification, removal of the outer fibre layers, or selective crushing of coarse fibres. Most published studies (8-12) have conclude that removal of outer fibre material is the main cause for fibre wall thickness reduction. This is even the conclusion here.

The location of the typical fibre wall splitting during fibre development may be anywhere in the layered fibre structure. The literature offers diverging views. The main options for splitting appears to be (P and S_1 are assumed to be too thin to split):

- 1) Between the middle lamellae and the primary wall, P
- 2) Between P and the outer secondary wall S_1
- 3) Between S_1 and S_2
- 4) Through the S_2

One may always find examples of all the listed options. Quantitative conclusions, characterising a fibre population as a whole, therefore cannot be given.

Middle lamellae material is generally separated from the fibres during the initial defibration.

In the middle lamellae separation from the fibres, the rupture may occur according to all alternatives 1) - 4). In published studies, 1) and 2) appear to be the most common assumption. Based on detailed surface study of a large number of fibres, using SEM, SEI and BEI modes, we conclude that 3) and even more 4) are most frequent, already at fibre separation, and certainly during fibre development. This is in accordance with Kibblewhite (5), however not supported in many more recent studies. Often, fibre topography wrongly indicates P or S₁ surface, and only very close inspection reveals the typical axial fibrillar structure of the S₂ layer.

The fibre development then mainly includes consistent removal of S_2 material; producing thin, flexible fibril sheets of high fibre bonding capacity; along with a gradual reduction of the fibre wall thickness, as has been found in other studies. The 16% reduction of average fibre wall thickness, creates a large amount of bonding effective S_2 fines.

Table 3.4, lists fibre fractions from the first refiner stage and screened pulp, as well as their average cross-sectional fibre wall cross sectional areas. The fibre weight fraction was found to drop from 72% to 55% during the refining. From the numbers in table 3.4 it can be concluded, that the reduction in fibre wall thickness accounted for more than 80% of this reduction, indicating that only some 4% of the number of long fibres after stage 1 were removed (cut) during the continued refining.

TABLE 3.4

Fibre weight fraction, and fibre cross sectional area during refining. The reduction in fibre cross section thus accounts for (19,2/23,5), i.e., 81% of the reduction of the fibre weight fraction.

	First refiner stage	Screened pulp	Relative reduction
Fibres, weight per cent of solids	72	55	23,6%
Average fibre wall cross sectional area, µm²	219	177	19,2%

Fig. 3.6 depicts the fibre wall thickness distribution for the same two fibre populations. The shapes of the two distributions support the conclusion that the main effect of the fibre development is a gradual fibre wall reduction, probably by surface stripping, in a fairly uniform fashion for all the fibre wall thickness groups.



Fibre wall thickness distribution

Figure 3.6: Fibre wall thickness distribution after first refiner stage, and for the final, latency treated pulp. The reduction in fibre wall thickness is reflected over the whole wall thickness range

4 FIBRE SPLITTING - A SIGNIFICANT FIBRE TREATMENT EFFECT IN MECHANICAL PULPING

4.1 Fibre cross sectional shape and printing surface roughness

Practically all mechanical pulp based paper grades are used for printing purposes. For such paper, the surface smoothness will be critical. As the requirement for improved print image quality is ever increasing, so is the demand for paper surface smoothness. As will be further analysed in section 6 of this paper, the coarse mechanical pulp fibres represent special problems for paper smoothness. Such fibres tend not to collapse spontaneously, and if forced to collapse by calendering etc., they will decollapse upon moistening [33], causing a roughening of the paper surface.

Papers made from SGW and PGW pulps are found to have superior printability characteristics to TMP based paper (16-18). The fibre properties are different when comparing TMP-pulps with SGW and PGW pulps. Groundwood fibres have been reported to have higher coarseness, and shorter length than TMP fibres (19).

As the paper surface roughening effect is related to the coarse long fibres, one way of reducing the problem will be to lower the long fibre content, or reducing the fibre length. This is not very attractive, as it will reduce paper strength. As explained in section 3.5, there will be a general reduction in fibre wall thickness during refining, however no specific reduction of the coarse fibres. The question then is whether it will be possible to maintain the fibre length, and reduce the fibres' tendency to decollapse afther moistening in other ways.

Tuooinen and Liimatainen (56) found that the fibrillation of the fibre fraction of groudwood pulps was bether than that of refiner pulps. A fibre with longitudinal cracks in the S_2 structure is likely to have a less circular cross section than an intact fibre. The extent of fibre cracks and splitting may therefore be significant for the surface roughness. When inspecting mechanical pulp fibres in microscope, such longitudinal cracks are often observed, especially for groundwood grades, and primarily at the fibres' ends. Parallel cracks here may change the fibre ends into a besom-like shape. If so, and one has a fibre where its length is retained, the structure is effective for fibre bonding, and the cross section less prone for fibre decollapse.

As the fibre splitting may be significant for papermaking properties, it was decided to conduct a study exploring the degree of splitting of the long fibre fraction for a series of mechanical pulps and look into its relevance for some paper properties.

4.2 Quantification of fibre splitting degree

Included in this study were two stone groundwood (SGW1&2), two pressurized groundwood (PGW1&2) and two thermomechanical pulps, (TMP1&2) from Norway spruce. Details on the pulps and the methods applied are given in Table C.1 (Appendix C).

Samples from the long fibre fractions of the pulps were dyed and inspected in light microscope. The lengths of splits of cracked fibres were quantified and related to total fibre length, as explained in Appendix C. Even some other fibre characteristics were assessed. Table 4.1 presents some results.

TABLE 4.1 Measured fibre length, fibre width and fibre split for various mechanical pulp grades and fractions. The results are presented together with their respective 95 % confidence interval limits.

Fraction	Pulp grade	Average length (mm)	Average width (µm)	Fibre split (%)
+ 28	TMP1	$2,20 \pm 0,06$	34 ± 1	4 ± 1
mesh	SGW1	$2,05 \pm 0,05$	35 ± 1	27 ± 5
	TMP2	$2,30 \pm 0,05$	31 ± 2	10 ± 2
+ 48 mesh	SGW2	$1,73 \pm 0,04$	35 ± 3	46 ± 5
	PGW 1	$1,96 \pm 0,05$	32 ± 2	30 ± 5
	PGW 2	$1,85 \pm 0,06$	33 ± 2 -	39 ± 5

There is a distinct difference between TMP and groundwood pulps (including PGW). When all the +48-fraction fibres are considered, a much higher split fraction is found than when only the +28-mesh fraction fibres are measured. This is reasonable as fibres that are partly damaged during grinding or refining may have a tendency to be reduced in size moving them from the+28 into the +48 mesh fraction. Figure 4.1 shows a SEM-micrograph of fibres from the SGW-pulp. The fibres are split and damaged in their longitudinal direction, especially at the fibre ends. Also, there are fibres with fibre wall cracks going parallel to the fibril axis in the S2-layer. In some cases these cracks result in an α -helix like structure.



Figure 4.1: SEM-micrograph of a typical SGW-fibre. (Magnification $100 \times$) The fibre is taken from the SGW1 pulp grade. A clear split can be observed in the end of the fibre.

There is a characteristic difference between the TMP on the one hand, and the various GWP fibres with respect to fibre wall cracks. It is not our intention to try to give a full analysis as to the basis for the difference in pulp fibre characteristics, a few comments may however be justified. The differences have to be related to the TMP and GWP processes. In the GWP process, the fibres are always subjected to forces perpendicularly to their axis. In the TMP refining, the fibres are subjected to defibrating forces in a much more random way.

In both processes, the lignin is softened by the high temperature. This plasticization of the middle lamellae in addition to repeated stresses and relaxations on the wood substance will release the fibres. The grinding process involves the fast passage by the stone's very small, individual grains over the log's surface, scraping over parts of the fibres. The fibres will often not lie completely parallel to the grinding surface. They will thus often be released from the log by one of the ends.

Figure 4.2 depicts the fibre split degree vs. fibre width for the +28-fractions of TMP- and SGW-pulps. There is a linear relationship for the SGW-pulp. The linear relationship even holds for the TMP, however of less significance. With increasing fibre width, it seems obvious that the fibre surface exposed to the grinding stone increases, subsequently resulting in larger splits. Similar trends were not found for the +48-mesh fractions. In the +28-fraction the fibres are split in the ends, whereas parts of the fibre remained undamaged. The +48-fraction contains many fibre fragments peeled off from other fibres. No correlation was found between fibre length and fibre split for any of the pulp grades investigated.



Figure 4.2: Plot of fibre split versus fibre width. The plot is given for the +28 mesh fraction of pulp grades TMP1 and SGW1.

5 SOME EFFECTS ON PAPER PROPERTIES BY FIBRE DIMENSIONS OF MECHANICAL PULPS

5.1 General

The morphology of the native wood fibre and the specific mechanical pulping process will determine the fibre morphology in the derived mechanical pulp. Large variation in tracheid dimensions can be observed between individual trees within the same species and also within different parts of a single trunk. Such variations in fibre morphology will strongly affect important paper properties like tensile strength, surface smoothness and gloss.

This part of the study is focusing on the quantitative effect of fibre dimensions on various properties of standard handsheets. The focus is thus on the effect of fibre dimensions and not the significance of the process.

To assess the quantitative effect of fibre dimensions on various paper properties, a backward stepwise linear regression analysis was applied.

Fibre length and coarseness obviously affect strength properties of paper (21,22). Fibre coarseness results from a combination of fibre diameter and fibre wall thickness. There still are questions with respect to the individual effects of these parameters. Based on the results from tested handsheets, the effects of the single fibre characteristics are explored. Test methods is described in Appendix C.

It is important to keep in mind that the tested paper is standard laboratory sheets formed using recycled white water (grammage 60 g/m^2 , 100 mesh wire). Changing the procedure of sheet forming and consolidation, e.g. different pressing or calendering, will change the relationship. The models and constants are thus only valid for this specific data set.

5.2 Tensile strength

The statistical model used to assess the effect of fibre dimensions on tensile strength is given in Appendix C. All the variables in the model are statistically significant on 95 % confidence level, and the model explains 92 % of the variation in tensile strength index between different thermomechanical pulps. The thermomechanical pulps used in this analysis had freeness values in the range 56 - 520 ml CSF.

$$T_{i} = -424 + 28.6L - 33.5WT + 350(PF/PL) + 2.66(BM50/100) + 2.32(BM100/200) + 1.46(BM-200) + \epsilon_{i}$$

$$R^{2} = 0.92$$
(5.1)

The effects of some of the independent variables on tensile strength are illustrated as component plots in Figure 5.1, and 5.2.

A component plot is equivalent to a residual plot around the plane spanned out by a specific independent variable, like fibre length in Figure 5.1. One can use such a plot to judge the relative magnitude of the residuals with respect to the explanatory power of a specific variable. A component plot illustrates well the effect of each independent variable.



Figure 5.1: Component effect of length-weighted average fibre length on tensile strength.

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Figure 5.1 depicts the effect of fibre length on tensile strength. As expected, increasing fibre length raise tensile strength. Even fibre wall thickness has significant effect on tensile strength (Figure 5.2). Tensile strength is highest for thin-walled fibres. Thick-walled fibres have less surface area available for bonding compared to thin-walled fibres. Thin-walled fibres also collapse more easily, thus allowing more bonding than thick-walled fibres. The tensile strength can thus be expected to decrease with increasing fibre wall thickness. For constant fibre wall thickness, the ratio between fibre perimeter and lumen perimeter is an indirect measure of fibre width. This ratio will be larger for narrow fibres than wider ones.



Component effect, Tensile index (kNm/kg)

Figure 5.2: Component effect of average fibre wall thickness on tensile strength.

For constant fibre wall thickness, the ratio between fibre perimeter and lumen perimeter is an indirect measure of fibre width. For a given wall thickness, narrow fibres will have a larger fibre-to-lumen perimeter ratio, than wider fibres. From the model, it is evident that a large ratio between fibre perimeter and lumen perimeter will raise tensile strength. This indicates that at a given wall thickness narrow fibres will yield better tensile strength. At a given wall thickness, there will be a larger number of narrow fibres to carry load in the sheet, than will wider fibres. This means that long, narrow and thin-walled fibres have the highest potential in terms of tensile strength.

Tensile strength is also very much affected by the amount of material in the middle and especially the fines fraction. The material in the BMcN 50/100 mesh, 100/200 mesh and the -200 mesh fractions contribute to better fibre bonding, thus improving tensile strength. Increasing fines content raise bonding and tensile strength. It is important to realise that factors not included in the model like the degree of fibrillation and the conformability of the fibres also can have a significant effect.

5.3 Smoothness

As has already been stated, surface smoothness is a basic requirement for printing paper. The smoothness is primarily dependent on the fibre properties and treatments which the paper has been subjected to. The coarseness of the fibres, and the amount and quality of fines are factors having strong effects on surface smoothness (see, for example, 23). Calendering raises surface smoothness, and this is achieved principally by lumen collapse (24). However, the extent of lumen collapse depends on the cross-sectional fibre dimensions (25).

The effect of fibre wall thickness on surface smoothness measured as Parker Print Surf (PPS) was studied. The thermomechanical pulps had freenesses in the range 56-230 ml CSF. The laboratory sheets made with recycled white water were calendered, 2 nips in a soft nip (100 kN/m, 10 m/min, 130 \circ C, 93 D Shore). The sheet densities varied between 825 and 941 kg/m³.

The statistical model used to explain the variation in Parker Print Surf is presented in Appendix C:

$$PPS_i = 4.08 + 0.292WT - 0.122(BM50/100) -0.111(BM100/200) - 0.0498(BM-200) + \epsilon_i$$
(5.2)

 $R^2 = 0.97$

All the variables are statistically significant on the 95 % confidence level, and the model explains 97 % of the variation in PPS readings between the different pulps. Surface smoothness is raised by finer particles in the surface structure. The PPS reading is thus decreasing with increasing BMcN 50/100, 100/200 and the -200 fractions. Fines thus contributes to a smoother surface. Surface smoothness is also very much influenced by fibre wall thickness. As can be observed from Figure 5.3, the PPS reading is increasing with increasing fibre wall thickness when all other independent variables in the model are kept constant. Thick-walled fibres are more rigid and resist collapse more than do thin-walled fibres.



Figure 5.3: Component effect of average fibre wall thickness on PPS.

To further investigate the effect of fibre wall thickness on the paper surface characteristics, the surface topography was characterised in a Confocal Laser Scanning Microscope (CLSM). To quantitatively describe the surface topography the arithmetic mean deviation, R_a , is used.

 R_a is the arithmetic mean deviation of areas of all profile values of the surface profile. The surface topography was measured on sheets calendered one nip in a soft nip calender (100 kN/m, 10 m/min, 130 °C, 93 D Shore). The sheet densities varied from 791 to 868 kg/m³. This approach allows assessment of the impact of fibre wall thickness for sheets of varying density. Since sheet density is expected to have a significant effect on surface smoothness it is important to compare the effect of fibre wall thickness at a constant sheet density. This is taken into account in the statistical model.

The applied regression model used is:

$$R_{ai} = 12.3 + 2.13WT - 0.00268\rho + \epsilon_i$$

$$R^2 = 0.90$$
(5.3)



Figure 5.4: Component effect of average fibre wall thickness on R_a.

All the variables are statistically significant on 95 % confidence level, and the model explains 90 % of the variation. The effect of fibre wall thickness is illustrated in Figure 5.4. The R_a is increasing with increased fibre wall thickness. Thus, at a given sheet density thickwalled cause a rougher surface than thin-walled and narrow fibres.

5.4 Gloss

Paper gloss is highly dependent on the surface properties of the sheet. A large variation in gloss between different thermomechanical pulps was found after calendering. This variation was assumed to be caused both by the differences in fibre dimensions, and the amount of finer particles. The TMP's used in this analysis had freeness in the range 56-230 ml CSF. Handsheets made with recycled white water were calendered 2 nips in a soft nip calender (100 kN/m, 10 m/min, 130 °C, 93 D Shore). The sheet densities varied between 856 and 941 kg/m³. Gloss was measured using 75° incidence and reflection angle.

The regression model used to explain the variation in gloss between the pulps is:

$$G_i = 14.9 - 3.82WT + 1.07(BM50/100) + 0.762(BM100/200) + 0.361(BM-200) + \epsilon_i$$
(5.4)

$$R^2 = 0.78$$

All the variables in the model are statistically significant on 95 % confidence level, and the model explains 78 % of the variation between pulps. As can be seen from the model, increasing amount of finer particles and fines, causing a denser and smoother sheet, will raise paper gloss. Fibre wall thickness also has a significant impact on gloss. As can be seen from Figure 5.5, thin-walled fibres promote higher gloss than do thick-walled fibres. Thin-walled fibres collapse easier in calendering and is thus expected to give a smoother surface yielding higher gloss than thick-walled fibres.



Figure 5.5: Component effect of fibre wall thickness on gloss.

6 PAPER ROUGHNESS

6.1 General Effect of Fibre Wall Thickness and Fibre Type on Moisture Induced Roughening

Converting processes like pigment coating and offset printing involve the addition of water to the paper surface. It is well known that the paper surface becomes rougher during such processes (26,27). In the future, water based inks may become more common. This may cause surface roughening even in gravure printing (28,29). Paper with mechanical pulp content tends to roughen much more by exposure to water, or even to moist air, than does paper from purely chemical pulps (24,26,30).

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It can be concluded that reducing the surface roughening tendency is a key issue if the use of mechanical pulp fibres is to be expanded to higher quality paper grades. The problem of surface roughening of the mechanical pulp paper thus deserves serious attention. A number of studies have investigated the effect of different paper making conditions on moisture-induced roughening (31), but there are still questions as to the basic mechanisms (32). To reduce the problem, the underlying mechanisms have to be thoroughly understood.

6.2 Some mechanisms affecting the moisture induced roughening

The objectives of the present part of the study were to:

- study the effect of coarse fibre content and fines on paper surface smoothness after moistening
- visualise and quantify the details of fibre decollapse

when calendered paper containing mechanical pulp fibres is subjected to moistening.

To take a closer look at the water-induced roughening mechanisms, we chose to study the change in cross-sectional shape of the fibres in commercial SC paper. Commercial SC paper was chosen because:

- calendering has caused pressure-induced stresses which may cause severe roughening by moistening
- it is a commercial paper of direct industrial importance
- it contains both chemical and mechanical pulp fibres, and it is of interest to visualise the different behaviour of the two fibre grades in situ
- commercial papers are more uniform than hand sheets
- we espouse that many of the mechanisms involved during moistening of paper containing mechanical pulp will be the same both for uncoated and for coated paper

Several investigators [24,26,33] have stated that moisture-induced roughening is much more pronounced for paper containing mechanical pulp than for paper consisting of solely chemical pulp. It is therefore of interest to distinguish between the mechanical and the chemical pulp fibres in the same paper to visualise the different behaviour of the pulp types in situ. This was achieved by treating SC paper samples with bromine gas after moistening treatment and prior to preparation for SEM analysis, see Appendix A.

A SEM micrograph of bromine treated, uncalendered SC sample is shown in Fig. 6.1. There is a pronounced difference between the chemical and the mechanical pulps in the extent of fibre collapse that has occurred on the paper machine. Almost all chemical fibres are completely collapsed, while many mechanical fibres are not. This uncalendered paper sample clearly demonstrates the difference in the tendency of the two types of fibres to collapse during sheet forming, pressing and drying.

Fig. 6.2 shows a similar cross-section of the same SC paper after calendering. When comparing Figs. 6.1 and 6.2, the effect of calendering is striking. The calendering process has caused a severe compression of the paper, and nearly all of the mechanical pulp fibres have collapsed. The number and size of pores in the paper have decreased considerably and the paper surface has become very smooth. Fig. 6.3 presents two SEM micrographs of the same calendered SC paper as in Fig. 6.2, moistened in two different ways: Fig. 6.3, top: A sample having been wetted by soaking into water for 2 s, with subsequent blotting and air drying. Fig. 6.3, bottom: A sample having been moistened by exposure to humid air, 97% RH for 24 h.

Wetting by soaking the sample into water is a rather extreme treatment, but was chosen because:

- liquid water is applied
- distinct reactions is achieved showing the reaction potential

Moistening by humid air was chosen to achieve:

- a lower level of moistening than soaking into water
- a method of moistening which is not influenced by sizing and surface roughness
- a uniform moistening at a level which is well defined and reproducible.

Both of the moistened papers show increased pore size and surface roughness, compared to the unmoistened SC paper of Fig. 6.2. A general increase in lumen opening is observed for most of the mechanical pulp fibres (light fibres) in the paper wetted by water. Thus, the wetting causes the compressed mechanical fibres to regain some of the lumen opening lost by the calendering process, making the fibres more tube-like. The collapsed fibres decollapse. This reaction is even observed, however less pronounced for the paper moistened by humid air. The cross-sections of the chemical pulp fibres (dark) on the other hand, appear not to be affected by water, not even for the paper sample soaked in water.



Figure 6.1: SEM micrograph of a cross section of commercial SC paper sampled before the super calender. The paper sample was subjected to bromine gas before preparing for SEM analysis. Some mechanical pulp fines are marked M, and some chemical pulp fibres are marked C.



Figure 6.2: SEM micrograph of a cross section of commercial SC paper. The paper sample was subjected to bromine gas before preparing for SEM analysis.



Figure 6.3: SEM micrographs of a cross section of moistened commercial SC papers. The samples were subjected to bromine gas before preparing for SEM analysis.

Top: Moistened by soaking into water for 2 s, free air drying at 50 % RH 23 °C. Bottom: Moistened by exposure to humid air, 97 % RH for 24 h, free air drying at 50 % RH 23 °C

6.3 Quantifying the Effect of Wetting; Influence of Fibre Wall Thickness

The overall fibre cross-sections and lumen openings were measured on SEM micrographs of paper cross-sections (Appendix A). Our findings from measurements of uncalendered, calendered and moistened SC papers are depicted in Fig. 6.4. The graphs present the lumen opening as a function of fibre wall thickness. By comparing Fig. 6.1 and 6.2 it can be seen that the calendering process compresses many of the mechanical pulp fibres, thus causing a large reduction in average lumen opening. Fig. 6.4 confirms this and indicates a considerable reduction in lumen opening regardless of the fibre wall thickness. Upon moistening, the fibres decollapse, confer Fig. 6.2 and 6.3 Again Fig. 6.4 confirm the findings from the SEM micrographs and shows correlation between:

- the level of moistening and the extent of decollapse.
- fibre wall thickness and the extent of decollapse.

However, some effect of the calendering compression remains even for the sample soaked into water.



Figure 6.4: Effect of calendering and level of moistening in commercial SC paper. Number of fibres measured is indicated above the bars.

The relative recovery of lumen opening and paper thickness is listed in Table 6.1. The degree of decollapse is some 40% when moistening by humid air of 97% RH, and some 77% when wetting by water for 2 s. The paper thickness is measured by conventional caliper test equipment, and the relative paper thickness increases are 12 and 63% respectively. There is a relatively large difference between the findings of the two measuring methods for the paper moistened by humid air. This large difference may indicate that the relative increase in lumen opening is larger than the relative increase in cross-section of inter fibre pores for the paper moistened by humid air.

It was expected that the relative recovery of lumen opening would depend on the fibre wall thickness. This seems to be the case for the paper wetted in water, however for paper moistened by humid air there is no dependence on the fibre wall thickness (Table 6.1).

Print quality is highly dependent on paper smoothness, i.e. the nominal height differences in the paper surface. Fig. 6.4 shows that for paper wetted by water, the fibres with thickest walls have the largest nominal change in lumen opening, some 4 μ m, and thus may give the greatest contribution to the moisture-induced surface roughening. To put this lumen expansion into perspective, one may consider the total thickness of the SC paper. The thickness of the finished SC paper was some 50 μ m, evaluated in conventional caliper test equipment. By averaging the local cross-section line lengths measured on SEM micrographs, a local thickness of only 43 μ m was found (33). The expansion of one single fibre may thus cause a local paper thickness increase by 10%. In this particular case, a total paper cross-section length of some 2.3 mm was inspected and 14 fibres having wall thickness of 4 μ m were found, i.e., one per 160 μ m. Assuming the fairly few fibres to be representative, it means that a straight line on the paper will cross 60 of the coarse fibres per cm, representing a pronounced roughening mechanism indeed.

Because thick-walled fibres have the largest nominal change in lumen opening upon moistening, it seems likely that the roughening tendency of paper may be reduced by modifying the pulping process so that the amount of whole, thick-walled mechanical pulp fibres in the stock is reduced. Another possibility seems to be a specific mechanical or chemical treatment of the thick-walled fibres in the pulping process

	Relative	recovery of lu	men openii	ng⁴	Paper thickness recovery ^b
	Fibre wall	thickness inte	rval	Average	
	0 - 2 μm	2 - 4 µm	4 - 6 µm		
	%	%	%	%	%
Moistened by humid air					
97 % RH, 24 h	42	40	38	40	12
Wetted by water, 2 s	72	66	91	77	63

Table 6.1 Recovery of lumen opening and paper thickness

The relative recovery of lumen opening is the increase in lumen opening after moistening as percent of the reduction of the lumen opening by the calendering compression.

² Paper thickness recovery is the increase in paper thickness as percent of the reduction in paper thickness caused by the calendering.

6.4 Effect of fibre splitting on the surface roughening tendency upon moistening

In section 4, the phenomenon of fibre splitting was explored. Here, this phenomenon will be related to the fibre's surface roughening effect on papers. Gane and Hooper (18) report increased roughening of TMP base sheets upon coating compared to SGW and PGW.

It is reasonable to believe that structural differences in the fibres will cause at least parts of the observed variances in surface smoothness. Skowronski (24) made the following statement: "Because of the presence of intra fibre stresses, the surface changes caused by calendering are not permanent. These stresses can be released by water during coating. Fibres show a tendency to recover their original uncollapsed shape." Such a decollapsing behaviour was also found by Forseth (34). If one proceeds along this train of thoughts, it seems likely to assume that the fibres' resistance to being compressed upon calendering influences their ability to recover their original shape upon moistening. TMP fibres are known to be relatively stiff, and fibrillated only to a minor extent (35). As showed in section 4, SGW and PGW fibres are to a large degree split in the longitudinal direction. It seems obvious that damages in the fibre walls reduce the fibres' mechanical resistance to compression, and a probable effect could be less roughening of the paper surface on sheets made from these particular pulp grades.

Included in this study were one stone groundwood (SGW2), two pressurized groundwood (PGW1&2) and one thermomechanical pulp, (TMP2). Details on the pulps are given in Appendix D and the methods applied is described in Appendix A and C. In order to cultivate

the effects of fibre structural differences, the fines were removed by fractionation. All papers were made from the +48 mesh fraction of the pulps investigated. By choosing such an approach, the retention on the paper wire was very close to 100 %, and the composition of the papers was known.

As a measure of paper roughness, Parker Print Surf was utilised. The paper roughness was measured on laboratory sheets both before and after calendering, and after subsequent moistening. The results are presented in Figure 6.5, with roughness measured on the wire side of the laboratory sheets, at a clamp pressure of 1.0 MPa.



Figure 6.5: The roughness of laboratory sheets (PPS, 1.0 MPa, wire side) presented for various mechanical pulp grades both before and after calendering, and after subsequent moistening.

The roughness was also measured on the top side, and similar trends were discovered there but with offset values. The changes in roughness (absolute values) when the base papers are subjected to calendering and moistening are shown in Figure 6.6. The untreated base papers made from TMP are significantly rougher than the base papers made from the various groundwood grades. It seems likely that this could be caused by the larger extent of splitting in the SGW and PGW fibres, as shown earlier in this report. Large degrees of splitting may give the fibres a better ability to conform to the surrounding surfaces.



Figure 6.6: Changes in roughness (absolute values) when the laboratory sheets are subjected to calendering and moistening.

After calendering, the differences in roughness among the various pulp grades are to a certain extent equalled out. The reductions in PPS upon calendering are between 6.0 and 4.7 μ m for paper made from TMP2 and SGW2, respectively. The calendering compresses the rather stiff TMP-fibres more than the damaged groundwood fibres. The damaged parts of the groundwood fibres are already flattened to some degree, and have a less potential to be further compressed. Tensions are introduced to the fibres, as explained by Skowronski (24). It seems likely that larger tensions are introduced to the TMP-based sheets, than the tensions introduced to the SGW and PGW based sheets. These stresses should, according to Skowronski (24), partly be released upon moistening.

The calendered laboratory sheets were subjected to moisture in the same manner as described in section 6.2, except that the time of exposure to liquid water was 20 s. Increases in roughness (absolute values) between 5.1 and 4.1 µm are observed when moistening with water for paper made from TMP2 and SGW2, respectively. This indicates that larger stress relaxations take place in the TMP based sheets upon moistening, than in the SGW and PGW based papers. A consequence of the last statement is that the degree of roughening of the paper surface depends on the amount of out of plane tensions present in the paper, which in turn depend on the mechanical properties, shape and general conditions of the fibres before calendering. As the roughness is not totally restored, the effect is only partially reversible.

When calendered papers were subjected to humid air, the same effects were observed, however much less pronounced. The observed increases in roughness upon moistening were between 2.4 μ m (TMP2) and 2.0 μ m (SGW2, PGW2). The same reasoning as above can be utilised to explain the experienced differences between the groundwood pulp grades and the TMP pulp.

The density of the paper could be expected to correlate well with the measured roughness values. This is also what is observed. Figure 6.7 shows absolute changes in density upon calendering, and upon moistening. The decrease in density from the calendered state is less for the SGW and PGW based papers (431- 435 kg/m³) than what is the case for TMP based papers (486 kg/m³).



Figure 6.7: Changes in density (absolute values) when the laboratory sheets are subjected to calendering and moistening.

The fibre splitting as percentage of total fibre length was measured, and is reported in Table 4.1. A correlation between this variable and the degree of roughening upon moistening was found. This is shown in Figure 6.8, where the change in PPS on the wire side upon moistening with water is plotted against the extent of fibre splitting for the four pulp grades investigated. The PPS was measured at three different clamp pressures. The increase in PPS upon moistening is linearly decreasing with extent of fibre splitting. This correlation is a direct implication of what has been discussed above, that split fibre parts will contribute very little to the roughening of the paper surface upon moistening, due to lack of ability to restore their original cross-sectional shape.



Figure 6.8: Plot of moisture induced roughening (PPS) on the wire side versus extent of fibre splitting.

7 FIBRE SHIVES IN WOOD CONTAINING PAPER

7.1 General

In the defibration process, all fibres should be separated. On the other hand, it is desirable to limit the amount of energy input, both for energy cost reasons, and to avoid excessive mechanical damage on the fibres. It is statistically unavoidable that some fibre boundles will pass the refining without being fully defibrated. To take care of such component, the pulp passes through a complex screening and cleaning system. Though refining and screening technology have improved the later years, there are still significant amounts of shives in the finished newsprint. Shives act as crack inducing weak spots and thus reduce paper strength (36-42). The newsprint paper studied here contains some 10 possible crack inducing shives pr square metre, which is too few to be detected in standard laboratory strength tests, however more than enough to be important from a runnability point of view. In this study shives themselves and the variation in the paper properties around the shives are studied to better understand which is the important paper variables governing paper rupture in converting and printing presses.

7.2 A Study on Crack Inducing Fibre Structure

The experimental setup is explained in Appendix C. The fracture load and strain of the sheets studied are shown in Table 7.1. As can be seen the fracture force does not vary much, but the fracture strain is depending on humidity due to the dependence of paper stiffness on humidity.

	25% RH	50% RH	75% RH	75% RH
Tensile index (kNm /kg)	35.5	35.5	30.5	32.9
Fracture strain (%)	0.54	0.54	0.64	0.71

Table 7.1: Fracture load and strain for the different climates.

The shive length distribution and crack length distribution were measured in an image analysis program on light microscope images magnified 50x. The shives counted are those which gave rise to a crack in the paper during straining. Both shive and crack lengths are manually transposed to the paper CD. The shive length distribution is given in Fig. 7.1.



Figure 7.1: Shive length distribution in the examined newsprint sheets.

Similar distributions were measured for crack lengths. By subtracting the shive length from the crack length for each crack for the different climates three new crack growth distributions were made. The crack has grown significantly (95% level) more for the paper strained at 75% RH compared to paper strained at 25% RH. The paper strained at 50% RH falls in between this values. The correlation between crack growth and humidity is given in Fig. 7.2.



Figure 7.2: Average crack growth as a function of humidity.

As the paper strained at 75% RH has a much larger irreversible deformation, compared to the others it is reasonable that the crack growth also is larger here. It is then important to note that although the cracks grow more, the fracture stress is not significantly reduced. This implies that the fracture resistance of the paper is increasing with moisture as earlier reported (43, 44).

Choi (45) found that the paper strain was influenced by a crack in an area at the crack tips corresponding to ca 35 % of the crack length. Thus to investigate the base weight influence on crack growth, the base weight of a 1 x 1 mm square at each end of the cracks was measured from β -radiograms. Three of the four fracture initiating areas show a lower base weight than the average (Fig. 7.3).



Base weight, g/m 2

Figure 7.3: Base weight at crack tips and fracture initiating spots

The SEM images of the paper occasionally show chemical pulp fibres when they lie in the paper surface or they are left as unbroken fibres in the rupture area. Figure 7.4 shows an unbroken chemical fibre in the middle of the shive area which initiated the final rupture. This is quite surprising as this initiating areas are characterized by almost total fibre rupture (brittle fracture). This indicates that the chemical fibres may stay unbroken behind the crack front in the fracture initiating spot, thus reducing the stress intensity at the crack front.



Figure 7.4: Unbroken chemical fibre in the fracture initiating shive area.

The density and thickness of the shives vary widely. The combined effect of shives and calandering has been shown to reduce paper strength significantly (41,46). The fibres over and under thick and dense shives are subjected to a severe deformation and cutting (Fig. 7.5) in the calandering nip. The fibre cutting and deformation can be seen by studying the glossy paper surface over the shive, the shive cross section and the fact that there are almost no unbroken fibres in the fracture zone at the fracture initiating shive. It is obvious that fibres damaged in the calender will crack first and appear as an out of plane buckle at low stress, however depending on the toughness of the surrounding paper these do not have to be the cracks initiating the final fracture.



Figure 7.5: Fibres over a shive broken in the calendering process.

8 THE EFFECT OF RECYCLING ON FIBRE CROSS-SECTIONAL PROPERTIES

The effects of recycling on pulp properties have been thoroughly examined the last 20-30 years. It is a well known fact that fibre properties change upon recycling. This is a main concern when recycled fibres are used in commercial grades. Fibres from different pulp grades are changed differently upon recycling. Fibres from mechanical pulps will experience a progressive flattening and flexibilisation, thus giving a denser sheet (47). Chemical pulps have most lignin components removed, and consist almost purely of carbohydrates. When recycled, these fibres experience a reduction in intrinsic fibre properties such as swelling potential, bonding capacity and flexibility (48). The mechanism behind the reduction of swelling potensial is assumed to be irreversible bonding of hydroxyl groups on cellulose chains in the cell wall (49).

The repulping subject the fibres to mechanical forces that can alter their cross-sectional shapes. This will in many cases cause an increase in the degree of collapse (47,50), and thus an improvement of the bonding properties.

Both the transverse fibre dimensions and fibre morphology are factors affecting the properties of paper. In this part of the study, fibre fractions of warious pulp grades were recycled in the laboratory; mechanical (TMP2&3, SGW2, PGW1&2), semimechanical (NSSC1) and chemical (sulphite, SI1 and sulphate, SA1). Details on the pulps and the methods applied are given in Table C1 (Appendix C). The objective of this work was to gain broader insight on how fibres' cross-sectional shapes are altered upon recycling, and also how fibres from pulps with different backgrounds are affected differently.

Fibre cross-sectional parametres were assessed before and after recycling. The results of the measurements are presented in Table 8.1 together with the respective 95 % confidence interval limits.

	Pulp	Fibre wall	Lumen	Dmax	Dmin	Aspect	Form	Outer	Lumen	Fibre wall	Collapse
	Flade	arca (µiii)	arca (µiii)	(µ11)	(μπ)	14110	CHEE	penineter (µm)	penineter (µm)	(nickness (µm)	index
NSSC	New fibres	105 ± 2	48±2	17.7 ± 0.2	11.2 ± 0.2	0.65 ± 0.01	0.75 ± 0.01	49 ± 1	28 ± 1	2.46 ± 0.03	0.35 ± 0.01
	Recycled	108 ± 3	49 ± 2	18.0 ± 0.3	11.3 ± 0.3	0.65 ± 0.01	0.73 ± 0.01	50 ± 1	28 ± 1	2.45 ± 0.04	0.35 ± 0.01
тмра	New fibres	268 + 6	187 + 8	369+06	168+03	0.48 ± 0.01	0.59 + 0.01	00.2	72.2	216.006	0.60 - 0.01
	Requested	255 + 6	192 + 0	353.04	170.02	0.48 ± 0.01	0.59 ± 0.01	90 I 2	12 = 2	5.15 ± 0.06	0.60 ± 0.01
	Recycled	200 ± 0	104 1 9	55.5 ± 0.0	17.0 ± 0.5	0.50 ± 0.01	0.00 ± 0.01	95±2	69±2	3.08 ± 0.06	0.56 ± 0.01
SGW	New fibres	181 + 13	194 + 29	30+2	167+09	0.58 + 0.03	0.66 ± 0.02	87 + 1	50 + 4	250+017	0.42 + 0.02
	Pervoled	176 + 11	102 + 25	21 + 2	162+08	0.50 ± 0.03	0.60 ± 0.02	02 2 4	39±4	2.30 ± 0.17	0.42 ± 0.05
	Recycled	1/0 ± 11	1)) + 20	5112	10.2 ± 0.8	0.5410.05	0.05 ± 0.02	6J I 4	03 ± 4	2.57 ± 0.15	0.40 ± 0.05
TMP2	New fibres	150 ± 11	171 ± 20	31 + 2	147+07	0.40 + 0.02	0.60 + 0.02	82.1.4	64 . 4	216 - 014	0.50 . 0.02
TIVE 2	New indies	1.59 ± 11	1/1 ± 20	31 1 2	14.7 ± 0.7	0.49 ± 0.02	0.00 ± 0.02	83 ± 4	04±4	2.16 ± 0.14	0.50 ± 0.03
	Recycled	101 ± 12	162 ± 20	32 ± 2	14.2 ± 0.7	0.46 ± 0.02	0.56 ± 0.02	85 ± 4	64 ± 4	2.11 ± 0.15	0.54 ± 0.03
DOW	Name Change	170 . 11	200 . 27	22 . 1	162.00	0.52 . 0.02	0.41 0.00				
rGw.	i new nores	1/0±11	209±2/	32±1	10.3 ± 0.8	0.53 ± 0.02	0.61 ± 0.02	80 ± 4	66 ± 4	2.22 ± 0.15	0.47 ± 0.03
	Recycled	158 ± 10	157 ± 21	30 ± 1	14.1 ± 0.7	0.50 ± 0.02	0.60 ± 0.02	79 ± 3	58 ± 3	2.21 ± 0.12	0.50 ± 0.03
PGW2	New fibres	165 ± 10	164 ± 20	30 ± 1	15.0 ± 0.7	0.51 ± 0.02	0.61 ± 0.02	81 ± 3	61 ± 3	2.30 ± 0.13	0.50 ± 0.03
	Recycled	156 ± 11	142 ± 18	29 ± 1	13.7 ± 0.7	0.49 ± 0.02	0.60 ± 0.02	78 ± 3	56 ± 4	2.24 ± 0.13	0.50 ± 0.03
SII	New fibres	182 ± 7	105 ± 11	34 ± I	13.4 ± 0.5	0.43 ± 0.01	0.49 ± 0.02	89 ± 3	59 ± 3	2.38 ± 0.09	0.62 ± 0.02
	Recycled	105 ± 11	43 ± 3	34 ± 1	10.8 ± 0.4	0.35 ± 0.02	0.41 ± 0.02	85 ± 2	51 ± 3	2.32 ± 0.09	0.79 ± 0.02
SA1	New fibres	169 ± 6	171 ± 16	38 ± 1	13.8 ± 0.5	0.38 ± 0.02	0.48 ± 0.02	96 ± 3	76 ± 3	1.98 ± 0.09	0.70 ± 0.02
	Recycled	148 ± 5	94±11	38 ± 1	10.5 ± 0.4	0.30 ± 0.01	0.30 ± 0.02	92 ± 3	69 ± 3	1.81 ± 0.07	0.83 ± 0.01

Table 8.1 Cross-sectional parametres (average value for each population) before and after recycling, together with their respective 95 % confidence interval limits.

The results show that the mechanical pulp fibres remain unaffected or are somewhat flattened by the recycling treatment. The fibres from the pulp grades SGW2, TMP2 and PGW1 are significantly flattened, experiencing reductions in aspect ratio in the range 6.0 to 6.9 %. All mechanical pulp fibres seem to get a reduction in fibre wall thickness, however significant changes only in the case of TMP3 and SGW2. Fibres from the pulp grades SGW2, TMP2 and PGW1&2 were subjected to calendering before being recycled. In the case of NSSC1 and TMP3, the fibres were not subjected to this treatment. It appears that repulping alone does not affect the mechanical fibres' cross-sectional shape significantly. When the fibres are subjected to rougher mechanical treatment, such as calendering, it seems that the fibres are flattened. The results suggest that the tensions introduced to the fibres in the calendering process are not completely relaxed during rewetting. The two chemical pulps investigated, SI1 and SA1, are to a much larger extent affected by recycling. Reductions in form circle by 16 and 21 % are observed, for SI1 and SA1 respectively. The collapse index show even larger changes, of 27 and 19 %, respectively. The largest fibre diameter is not significantly changed upon recycling for any pulp grade, while the smallest diameter

decreases for all pulps where a flattening is observed. As the largest fibre diameter is unaffected, the changes in cross-sectional shape are dependent on the decrease in fibre thickness. For SA1, the fibre wall thickness decreased upon recycling. However, in the case of S11, no significant change was found. Similar results have been achieved by Bawden and Kibblewhite (49), who found that dried and rewetted kraft fibres were more collapsed than the corresponding new fibres. They reported a decrease in fibre wall thickness, a decrease in smallest diameter, and no changes in largest diameter.

In Figure 8.1 frequency distributions for form circle before and after recycling are given for some of the pulp grades investigated. Upon recycling the distributions are clearly skewed to the left for some pulp grades. Among the mechanical pulp fibres, this can be observed for fibres from the SGW2 and PGW1 pulps. The form circle distributions are to a larger extent skewed to the left for the chemical pulps investigated. In all cases, this is a consequence of the flattening of the fibres.



Figure 8.1: Frequency distributions for form circle before and after recycling.



Figure 8.1.C: SI1.

Figure 8.2 shows the form circle plotted versus fibre wall thickness for some of the pulps investigated. These plots show that the form circle decreases when the fibre wall thickness is reduced. This tendency is more pronounced for the chemical fibres than in the case of the mechanical fibres. The results further show that there is a reduction in form circle upon recycling of chemical pulp fibres for all fibre wall thicknesses.

Mechanical and chemical pulp fibres behave differently upon recycling. The chemical pulp fibres, consisting mostly of carbohydrates (cellulose and hemicellulose) can be assumed to experience a contraction of the fibre wall, due to irreversible hydroxyl group bonding between fibrils (e.g. 49). Most chemical fibres collapse completely upon drying. It seems likely that the reduction in swelling ability prevents the fibres from completely restoring their original cross-sectional shape. It is an established theory that a different mechanism is valid when mechanical pulp fibres are recycled. These fibres are produced in high yields,



Figure 8.2: Plots of form circle versus fibre wall thickness, before and after recycling

leaving much lignin in the cell wall structure (51). Presence of ligno-hemicellulose gel in the fibre wall prevents direct contact between cellulosic surfaces during drying. The swelling ability of high yield fibres is only to a minimal extent affected by recycling. The results show clear differences in behaviour between mechanical and chemical fibres, if subjected to the same recycling treatment (without calendering). Chemical pulp fibres are significantly flattened, while the mechanical pulp fibres seem unaffected. However, when mechanical fibres are calendered before recycling, a flattening can be observed.

Both mechanical and chemical pulp fibres experience reductions in fibre wall thickness when recycled. Two different mechanisms for these reductions can be suggested:

- Reduction due to contraction of the fibre wall.
- Reduction due to mechanical wear.

From the discussion above, the contraction mechanism seems to be valid for the chemical pulp fibres, while the reduction in fibre wall thickness of mechanical pulp fibres could be explained by mechanical wear in the repulping process.

APPENDIX A: Experimental methods applied in several chapters

A.1 Preparation and characterisation of fibre cross-sections

The fibres to be characterised were separated and collected using the BMcN classifier. To secure fibre cross sections cut perpendicular to the fibre length direction, the fibres have to be parallelized. This was done by diluting the fibres in water and draining them through a special grid equipped with several parallel slots (The main principle has been developed at STFI). The slots were about 1 mm apart. No or only a light suction was applied in order to avoid possible collapse of fibres. After the drainage the fibres are in bundles of parallel fibres. The fibre bundles were freeze dried to prevent drying induced changes in fibre cross-section and prepared for SEM-analysis as described below.

SEM binary micrographs of magnification 250 were prepared. These micrographs did not include only whole undamaged fibres even though the fibres were fractionated and parallelized. Thus the pictures had to be edited prior to the automatic image analysis, which means that small fibre fragments, heavily damaged fibres and fibres clearly cut diagonally to the fibre axis were removed. Fibres with minor damages in the fibre wall were "repaired", and fibres lying together were separated.

Between 300 and 500 fibres from each sample were measured with the automatic image analysis system. The average values changed only moderately by further increase of the number of fibres measured. The following cross-sectional properties of freeze dried fibres were measured in this investigation: Fibre wall thickness, fibre width, fibre wall area, outer and lumen perimeter, lumen area, cross-sectional shape, and degree of collapse.

A.2 Sample preparation for cross-section SEM analysis

The paper or fibre samples were dried at 103 °C for 2 h., mounted in a plastic mould and embedded in epoxy resin. The epoxy was cured, grounded, polished and cleansed to give a smooth surface (10,52).

A.3 Moistening of paper samples

The paper samples were moistened in the following ways:

- 1 Subjected to moist air, 97% RH, 20 °C for 24 h.
- 2 Soaked in distilled water for some 2 or 20 s. The excess of water was wiped of by blotters.

After the moistening treatment, the samples were re-conditioned at standard conditions, $23^{\circ}C$ 50 % RH.

A.4 Distinguishing between Mechanical and Chemical Pulp Fibres in Paper

To distinguish between mechanical and chemical pulp fibres in a paper samples the samples were subjected to bromine gas from excess fluid bromine in a closed vessel for 120 minutes. Bromine reacts with the lignin, causing mechanical pulp fibres to appear lighter than chemical pulp fibres in the SEM, BEI (15).

A.5 Measuring the lumen opening in paper

To quantify the fibre cross-sectional characteristics of mechanical pulp fibres, SEM micrographs of paper cross-sections of magnification 1600 were prepared from the samples. Only fibres having a full perimeter were accepted. The fibre thickness and the lumen opening of single fibres were quantified, perpendicular to the direction of the largest lumen width, at a point judged to have fibre wall of average thickness. The fibre wall thickness was then calculated by subtracting the lumen opening from the measured fibre thickness and divided by 2. Fibres cut more or less parallel to the fibre length direction were measured in the z-direction of the paper to prevent overestimation of the fibre wall thickness.

In commercial paper the main fibre orientation is in the MD and the fibres are located in planes of the paper. When cutting a commercial paper in CD, many fibres are cut roughly perpendicular to the fibre length direction. Some fibres are however cut at an angle to the fibre length direction. By considering the uniformity of fibre wall thickness in the paper cross-section it is possible to evaluate the cutting angle in a coarse way. For the skewely cut fibres, the fibre wall thickness appear larger in the CD than in the z-direction of the paper. To prevent an overestimation of the fibre wall thickness of such inclined cut fibres, all measurements were performed in the z-direction of the paper.

APPENDIX B: Evaluation of cross-sectional fibre characteristics

The single fibre average fibre wall thickness (FWT) was calculated according to Equation B.1:

$$FWT = \frac{P - \sqrt{P^2 - 4\pi A_w}}{2\pi} \qquad B.1$$

594

where P is the outer perimeter, and A_w is the fibre wall area. The equation is based on the assumption that the fibre can be approximated by a cylinder with the same perimeter and fibre wall area as the real measured fibre.

The cross-sectional shape was evaluated in two different ways, termed form circle (FC) and aspect ratio (AR) (10):

$$FC = \frac{4\pi A}{P^2}$$
B.2

$$AR = \frac{D_{\min}}{D_{\max}}$$
B.3

where A is the cross-sectional area of the fibre including the lumen area, and D_{min} and D_{max} is defined as minimum and maximum distances between parallel tangents to the cross-sections (47).

The collapsability was evaluated with a method similar to (12).

Collapse Index =
$$1 - \frac{LA}{LA_0}$$
 B.4

where LA is the real lumen cross-sectional area, and LA_0 is the lumen cross-sectional area of the same fibre in an completely uncollapsed condition. Jang et al. (12) assumed a rectangular cross-section for the fibre in its uncollapsed condition. In this investigation we assume a circular shape for the uncollapsed fibre.

Mechanical pulp fibres have little tendency to spontaneous collapse during forming and drying of commercial SC. All chemical fibres on the other hand collapse completely during this process. During the calendering process however, the mechanical pulp fibres are

compressed to a collapsed state. The fibres remain collapsed, but with pressure-induced stresses in the fibre walls. By introducing moisture to the cell wall, the mechanical pulp fibres will recover some of the tubular shape they had before calendering. The degree of decollapse increases with increased level of moistening and fibre wall thickness. Moistening by humid air was found to cause some 40% average recovery of lumen opening while wetting by water gave some 77% average recovery. The chemical pulp fibres did not show any tendency to such decollapse.

APPENDIX C: Experimental procedure unique for the different chapters

Chap 4. Fibre splitting

The pulps investigated (TMP1, TMP2, SGW1, SGW2, PGW1, PGW2) were fractionated in a BMcN Fractionator, and the fibres not passing the 48 mesh wire were collected except for the pulp grades TMP1 and SGW1, where the + 28 mesh fraction was collected. To assess the fibre splitting the fibres were dyed in a saturated solution of Chlorazol Black at 100°C for 10 min, and washed in deionized water. The dyed fibres were transferred from a fibre suspension (deionized water) to glass-slides with the aid of a drop tube, and separated from each other with a needle. Upon drying capillary forces attached the fibres to the glass surface. The fibres were studied in a light microscope (50 x magnification) connected to a projector unit. The fibre projections of approximately 150 fibres were transferred to a tracing paper. The fibre length, fibre width and split length were measured on each fibre by hand. The "fibre split" is characterised as the average split length of the fibre population in percent of the fibre length.

Chap. 5 Effects of fibre dimensions on some paper properties

Various wood samples of Norway spruce were refined by the thermomechanical pulping process. The refining was carried out in a 36 " Sunds Bauer double disc refiner in PFI's pilot plant. TMP was produced in one single stage using specific energies of 1000, 1500 and 2000 kWh/t, 2.5 kg/min production rate, 20 % outlet consistency and 120 s preheating time.

The cross-sectional fibre dimensions in the fibre fraction (BMcN +50 mesh) were measured by our standard procedure described in Appendix A. Fibre length was measured in a Kajaani FS-100 fibre analyser. All the results in chapter 5 are based on laboratory sheets made with recycled white water. Tensile index and Parker Print Surf were measured using the SCAN standards P67.93 and P76.95 respectively. Gloss was measured using 75° angle of incident light. Detailed surface topography of paper was measured using Confocal Laser Scanning Microscopy (CLSM). The vertical height distribution gives per definition the number of area elements that is placed at a given height above a chosen reference plane, relative to the total number of area elements. All area elements have the equal projection $\Delta x \Delta y$ in the xy-plane ($\Delta x = \Delta y = 1.9 \mu m$). The approach used is to record a stack of reflected light intensity images (256 images) of the paper surface, each a fixed distance Δz from the next one (53). Thus one has the 3D distribution of light reflected at the surface. To define the position of the paper surface one may use the distribution of the light intensity in the z-direction for each xy-position by calculating the centre of mass in z-direction of the first intensity spot (53). The distribution one can calculate various parameters describing the surface topography of the sheet. In this study the arithmetic mean deviation of areas of all profile values of the surface profile, R_a (Equation C.1), is used.

$$R_{a} = \frac{1}{l \cdot b} \int_{0}^{1} \int_{0}^{b} |z(x,y) - \overline{z_{xy}}| dx dy$$
 C1

To assess the quantitative effect of fibre dimensions on various paper properties backward stepwise linear regression analysis is used. This search procedure develops sequentially the best subset of variables to be included in the final model. The search procedure develops a sequence of regression models, at each step deleting one independent variable which is not statistically significant. It finally arrives at a subset of only statistically significant variables best describing the variation in a specific paper property like tensile strength. 95 % confidence level is used.

Chap 6. Paper roughness

Effect of fibre splitting on the degree of surface roughening upon moistening

Standard laboratory sheets (SCAN-C 26:76) were made from the +48 mesh fraction of each pulp grade. The sheets were calendered in a laboratory super calender, 6 nips, 140 kN/m, with roll temperatures 75 and 90 °C on the paper cylinder and metal cylinder, respectively. The sheets were turned between each nip, so that each side of the paper was exposed three times to each cylinder. After conditioning at standard conditions, 23 °C, 50% RH, the sheets were subjected to moisture some by direct contact with deionized water in 20 seconds and others by exposure to humid air of 97 % humidity for 24 hours, see further details above. The

sheet density, roughness (PPS) and light scattering coefficient were measured for each pulp grade both before and after calendering, and after subsequent moistening. Paper crosssections from the same steps were embedded in epoxy as described above.

Chap. 7 Shives affecting newsprint strength

1.8 m x 0.8 m sheets of commercial newsprint was strained slowly to fracture at respectively 25, 50 and 75% RH in an apparatus developed at PFI in Oslo (54). Shives, cracks, hair, holes etc. were identified as an out of plane buckle as the paper was strained (36). After rupture these areas, and the fracture initiating spot were cut out. The length of the shive (if any) and crack causing the out of plane buckle was measured in a light microscope. A 2.5 cm x 3 cm rectangle of each area was examined as a β -radiogram (37) to determine the local basis weight in the close vicinity to the crack ends.

To distinguish reinforcement pulp from the mechanical pulp, the paper samples were treated with bromine gas as described in Appendix A prior to surface examination of the samples in SEM, BEI mode. The paper cross-sections were also studied in SEM as described in Appendix A. The dimensions and density of the shives and the amount of chemical reinforcement fibres close to the crack were determined.

Chap. 8 The effect of recycling on fibres cross-sectional properties

The pulp grades NSSC1, TMP3 and BS11 were recycled according to the procedure given by Korpela [55]. The remaining pulp grades, except SA1, were repulped from the calendered laboratory sheets described in section 6.4. The pulp grade SA1 was recycled as paper made on an experimental paper machine. Fibre cross-section samples were made according to the procedure given in appendix A.1. and A.2., and cross-sectional parameters were measured by image analysis on SEM-micrographs as described in appendix A.1.

Paper characterisation

The paper thickness was measured according to SCAN-P 7:75. Paper roughness was measured with the Parker Print-Surf roughness tester.

APPENDIX D: Characterization of the pulps investigated

Code	Pulp grade	Raw material	Freeness (ml CSF)	Coarseness (mg/m)	Fibre fraction examined
TMPI	Thermo mechanical pulp	Spruce	35	-	+ 28 mesh
TMP2	Thermo mechanical pulp	Spruce	35	-	+ 48 mesh
TMP3	Thermo mechanical pulp peroxide bleached	Mainly spruce, some pine	63.5	0.249	+ 28 mesh
NSSC1	Neutral semi chemical sulphite pulp, 82 % yield	Mainly birch, some eucalyptus	688	0.124	+ 28 mesh
SGW1	Stone ground wood	Spruce	35	-	+ 28 mesh
SGW2	Stone ground wood	Spruce	35	-	+ 48 mesh
PGW1	Pressure ground wood	Spruce	85	-	+ 48 mesh
PGW2	Pressure groundwood	Spruce	35	-	+ 48 mesh
BSI	TCF-bleached bisulphite pulp, unbeaten	80 % spruce 20 % pine	-	-	+ 28 mesh
SA1	laboratory pulped sulphate pulp, unbleached, 48 % yield, beaten	Spruce	29 °SR	-	+48 mesh

Table D1 Characterization of the pulps investigated

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Symbols

 A_{FS} = Total fibre surface area in an image A_{ML} = Fibre area covered by middle lamellae in an image BEI = Backscattered electron image BM50/100 = mass fraction in the Bauer McNett 50/100 fraction (%) BM100/200 = mass fraction in the Bauer McNett 100/200 fraction (%)BM-200 = mass fraction in the Bauer McNett -200 fraction (%) D_{min} = Minimum fibre diameter D_{max} =Maximum fibre diameter FC = Form Circle FD = average fibre diameter (μ m) FS = Form Shape G = paper gloss (%)L = length-weighted average fibre length (mm) P = Primary fibre wall P_{ML} = Fibre perimeter covered by middle lamellae (µm) PF/PL = average ratio between fibre perimeter and lumen perimeter $PPS = Parker Print Surf (\mu m)$ R_a = arithmetic mean deviation (µm) RH = Relative Humidity $S_1 =$ Outer secondary fibre wall $S_2 =$ Inner secondary fibre wall SEI = Secondary Electron Image T = tensile strength (Nm/g)TP = Total fibre perimeter (μm) WT = average fibre wall thickness (μm) $\epsilon_i = \text{statistical error}$ ρ = apparent sheet density (kg/m³)

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Transcription of Discussion

Some Mechanical Pulp Fibre Characteristics, their Process Relationships and Papermaking Significance

Øyvind W Gregersen, Norwegian University of Science and Technology

Dr Gary Baum, Vice President - Research, ISPT, USA

In talking about fibre development you indicated that there were four fractions that you saw as where the split might occur in the fibre cell wall. These were between the middle lamella and primary wall, the primary wall and S1 layer, between S1 and S2 and within the S2 layer itself. - Do you have any kind of estimate as to the fractions of each of those possible fracture points?

Øyvind Gregersen

That depends very much on refining conditions, temperature and pressure - what we want to achieve is always a splitting within the S2 layer, of course if you increase temperature the lignin in the middle lamella will soften. This will increase the degree of splitting in the middle lamella. Your question is not easy to answer, the fraction of each of those fracture points are highly dependent on process conditions. Thus, the fibre quality is different between different mills. This might make these kind of studies very helpful to develop the process in the different mills.

John F Waterhouse, Senior Associate Scientist, IPST, USA

What are the implications of the fibre shape change which occurs on recycling, and secondly, is it possible to reverse the shape change that you see through say, refining or other treatments?

Øyvind Gregersen

For the shape change during recycling of mechanical fibres the major thing is the flattening of the fibres. The major implication is that you get higher density in your recycled sheets and also increased smoothness. I have also seen higher strength reported. These changes are very desirable and we do not want to reverse those at all.

Professor Gunter Gerischer, University of Stellenbosch, South Africa

How far do the calendering techniques have an effect on the flattening of the fibres or secondary fibres? Have you investigated that?

Øyvind Gregersen.

We have not investigated that - of course calendering is very important for the flattening. If you try to study uncalendered recycled fibre shapes then the flattening is much less pronounced. We have not investigated the difference between different calendering techniques.

Dr Kari Ebeling, Director, UPM Kymmene Group, Finland

You stated in the presentation that the fibres which had more splitting had better resistance to roughening upon rewetting and that this was due to some kind of capability in them to store absorbed energy. Could the reason be that the split fibres have more contact area and the splitting has opened up more layers which make stronger bonds?

Øyvind Gregersen

It is just a hypothesis - when undamaged, unsplit fibres are compressed in the calender the lignin will be soft and the entire fibre is compressed. The cellulose microfibrils are deformed and locked in this form when the lignin is cooling and passing back through its glass transition temperature and stiffens the structure. So what we have got now is a compressed fibre with internal stresses between the stiff lignin matrix and the cellulose microfibrils. When this is moistened then the lignin will soften again and the fibre will regain its circular shape. We think when we have split fibres the capability of the cellulosic microfibrils is much less for storing this elastic energy which is the driving force of the decollapse . We have not confirmed this experimentally.

Ron Crotogino, Director of Research, Paprican, Canada

You have shown an increase in tensile strength with fibre length and with decreasing fibre wall thickness - presumably to make a strong mechanical pulp to maximise the fibre length and minimise the wall thickness - how do I do that?

Øyvind Gregersen

That is the question which people dealing with breading or cloning wood are dealing with. You can influence this to a certain degree by the refining process and influence the fibre properties in the right direction. My point is that high fibre length and thin fibre walls are the ideal conditions for high tensile strength. We can now measure whether our attempts to improve our process are going towards our goal or not but I do not have a solution to make a perfect raw material or pulp.