

Integrated Master in Chemical Engineering

Method development for analysis of Ray cells content in Thermomechanical Pulp of Spruce

Master Thesis

Developed for the course

Development Project in a Corporate Environment

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Abstract

The world is living a technological revolution, where printing paper has been challenged by modern electronic technologies such as the Internet. In consequence of this technological evolution, there has been a decreasing demand on printing paper. Therefore, to keep the sales level the market share must be potentially increased and this depends on two important factors namely paper quality and price.

At Ortviken Paper Mill large efforts have been done in order to increase product quality, therefore the production of improved newsprint has been increasing compared to standard newsprint. This paper quality type is predominantly printed in heatset web offset printers in which linting is one of the most common causes of complaints. Linting is defined as the removal of loosely bonded particles from the surface of the sheet when paper is in contact with water based ink on the printing press. According to several studies, lint consists of non fibrillated short narrow fibers. Lint particles are generally ray cells, nearly 80% of the total amount of particles. Adding cationic starch has been a temporary solution for linting, but it considerably increases the production costs, therefore it is necessary to bring a new long term solution for it.

Solving the problems requires a deeper knowledge of them, thereby identifying and controlling the amount of linting propensity particles during the papermaking process is inevitable. Even if there are some paper analysis equipments for linting prediction, it is almost impossible to predict it at the paper mill. Proper pulp analysis of the presence and amount of ray cells is therefore indispensable.

A novel method able to determine the amount of ray cells from a pulp sample was developed. The method has as input KajaaniFiberLab individual fiber results, and uses an algorithm based on a decision tree structure to classify the fibers into ray cells and non-ray cells. The results obtained have shown to be in agreement with the theoretical values of ray cells fraction for Norway spruce, nearly 4 %. A small deviation between samples has shown a good accuracy of the method. The results obtained have confirmed the reliability of the method for analysis of ray cells content in TMP of spruce.

Key words:

Ray Cells, Linting, Newsprint, TMP, Thermomechanical pulp, Spruce

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Notation and Glossary

List of Variables

$contributionRC_i$	Contribution of each fraction to the total amount of ray cells (RC)	%
$Deviation_{manual}$	Deviation between ray cells' manual analysis and automatic analysis of ray cells methods	%
$Deviation_{isolation}$	Deviation between ray cells isolation and automatic analysis of ray cells content methods	%
F	Normal distribution	
f	Cumulative normal distribution	
$FalseRC$	Total amount of false ray cells	
$Fraction_{RC}$	Fraction of ray cells in a sample of pulp from the isolation of ray cells method	%
m_{pulp}	Mass of dry pulp	g
m_{RC}	Mass of dry fraction of ray cells	g
n	Number of fibers in the sample	
$npic$	Number of particles with picture associated	
P_{RC}	Fraction of ray cells determined by the automatic analysis of ray cells content method	%
P_{RC1}	Fraction of ray cells in the amount of fibers with picture associated	%
$P_{RCmanual}$	Fraction of ray cells determined by ray cells' manual analysis method	%
P_{TrueRC}	Fraction of true ray cells	%
$P_{FalseRC}$	Fraction of false ray cells	%
RC	Total amount of ray cells	
$RC1$	Amount of ray cells 1	
$RC2$	Amount of ray cells 2	
$RC3$	Amount of ray cells 3	
$TrueRC$	Total amount of true ray cells	
$TrueRC_Ratio$	Fraction of true ray cells in the total amount of ray cells	

Greek letters

$\bar{x}$	Mean
s	Standard deviation

Indexes

i	Index or counter
-----	------------------

List of Acronyms

CCD	Charge-Coupled Device
CDA	Canonical Discriminant Analysis
CLSM	Confocal Laser Scanning Microscopy
CSA	Cross-Sectional Area
CTMP	Chemical Thermomechanical Pulp
CWT	Continuous Wavelet Transform
FE-SEM	Field Emission Scanning Electron Microscopy
FFG	Fin Färsk Gran (Fine Fresh Spruce)
HSWO	Heatset Web Offset
LM	Light Microscopy
LWC	Light-Weight Coated paper
MILDA	Mehalanobis Discriminant Analysis
MWC	Medium-Weight Coated paper
PM5	Paper Machine number 5
SC	Super-Calandered paper
SEM	Scanning Electron Microscopy
TMP	Thermomechanical Pulp
ToF-SIMS	Time-of-flight secondary ion mass spectrometry
VBA	Visual Basic for Applications

1 Introduction and Motivation

The world is living a technological revolution, where printing paper has been challenged by modern electronic technologies such as the Internet. Despite this, paper, a product with more than 2000 years of history, still has an important role as a communication and information medium, as well as in many other areas of our society and there is no doubt that it will continue to have an important place in the future.

Papermaking is a vast, multidisciplinary technology that has expanded tremendously in recent years. Important advances have been made in all the papermaking process, starting from the raw materials control until the end products. The market competitiveness is increasing everyday together with quality standards; therefore to maintain a solid position manufacturers must achieve new innovative printing paper grades with high standard quality levels.

The aim of this thesis is to develop a method for ray cells content analysis in thermomechanical pulp of Spruce. It was also required that the method should only use equipment available at the company.

1.1 Background

1.1.1 Thermomechanical pulp (TMP)

Mechanical pulps and in particular thermomechanical pulps (TMP) are the most important component in the manufacture of printing paper grades (e.g. newsprint, supercalandered (SC) paper, light-weight coated (LWC) and medium-weight coated (MWC) paper), (Fernando and Daniel, 2004).

The pulp is made by heating the chips with pressurised steam and mechanically separating the fibers in a pressurized refiner. The steam pressure is normally 3-5 bar and its temperature correspondingly 140-155 °C (Lönnerberg, 2009).

During the thermomechanical pulping process the chips are fed to a pre-steamer and are steamed with process steam from the refiners. Then a feeding screw moves the chips to a pressurized refiner which mechanically separates the fibers.

The refining process is one of the most important steps of thermomechanical pulping process. Nowadays there are different types of refiner configurations, depending on process requirements, however the main operational principle is the same. The wood chips are fed in-between two parallel refiner discs where at least one of those is rotating. The patterned refiner segments transfer the rotational energy into the pulp. Wood chips are broken down

and due to compressive and shearing forces a wood material suitable for papermaking is developed. Refining is a very complex stage of the process, the conditions inside the refiner are very harsh, the flow phenomena inside the refiner is very complex while two simultaneously phenomena occur, separation and modification of fibers.

The pulp is often refined in two stages with two refiner units, and the refiners are followed by a cyclone to separate the pulp from the process steam (Metso Automation, 2002). However at SCA Ortviken paper mill pulp refining occurs in a single stage with one refiner unit, a second refiner stage happens immediately before the LWC paper machine.

Below in figures 1 and 2 are respectively represented, a diagram of a refiner used at Ortviken, an example of a refiner segment disc and SEM images of wood chips before refining and pulp fiber fragments after being refined.

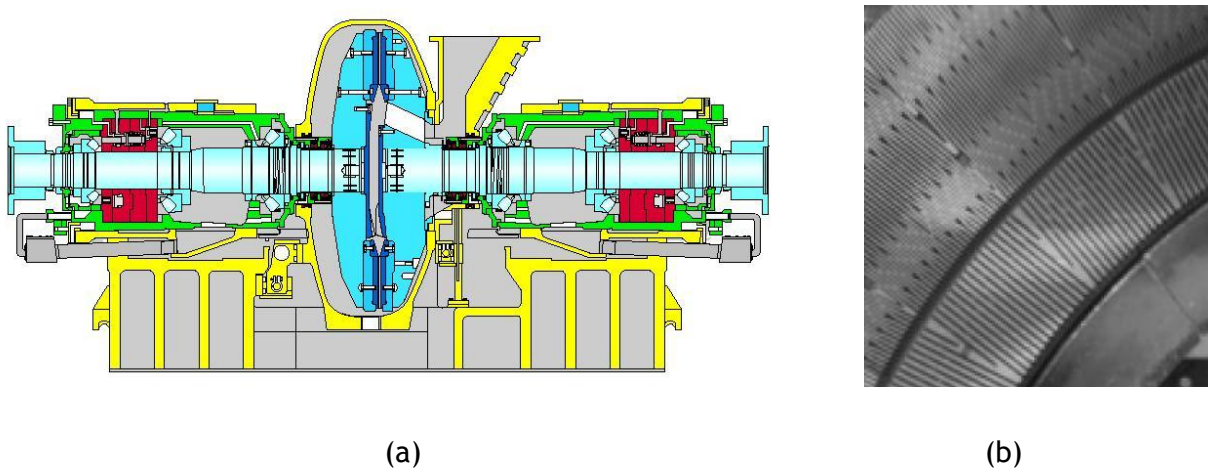


Figure 1 - a) Metso double disc refiner, RGP DD68, used at SCA Ortviken (Courtesy of SCA);
b) Refiner segments (Reyier, 2008).

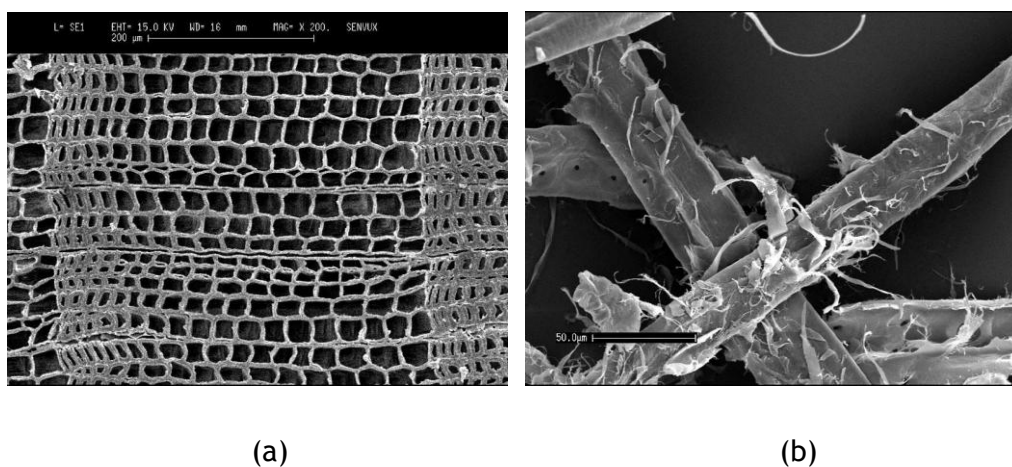


Figure 2 - Scanning electron micrographs of a) a piece of wood seen from above and
b) fibers and fragments of fibers in a thermomechanical pulp (Rundlöf, 2002).

A drawback of this mechanical fiber separation is the high demand of energy, especially when compared with chemical separation applied in the Kraft process. However, the yield is much higher, since more than 95 % of the wood is converted into TMP (Rundlöf, 2002; Lönnberg, 2009; Sjöström, 1993).

The most important raw material parameter in thermomechanical pulp refining is the wood species. Different wood species results in different final pulp properties and also specific energy consumption (Illikainen, 2008). At Ortviken's paper mill, the raw material used for TMP pulping is exclusively Norway spruce and the wood logs are classified as FFG (Fine Fresh Spruce), meaning that the time between harvesting and arriving at the mill is no longer than 3 weeks.

The Norway spruce (*Picea abies* (L.) Karst), belongs to the conifers plant group (cone-bearing seed plants), and has favourable fiber properties, low extractive content and high initial brightness of wood (Illikainen, 2008). These properties make this conifer, one of the most important raw materials for pulp and paper industry, therefore it is both economically and ecologically one of the most important tree species of the Scandinavian countries.

1.1.2 Newsprint Paper

Newsprint is a paper grade manufactured mostly from recycled fiber pulps, mainly in Europe and Asia. However in North America and also at Ortviken's paper mill newsprint is mostly manufactured from fresh fibers mechanical pulp. Newsprint is an uncoated paper and consists of standard and improved newsprint. Standard newsprint is produced exclusively from mechanical pulp. Improved newsprint is produced with mechanical pulp and a small amount of chemical pulp as reinforcement. Mechanical pulp gives paper opacity but it has a tendency to yellow, characteristic of newspapers. Chemical pulp gives paper a larger strength and improves the runnability on the paper machine and the printing press.

The share of chemical pulp has to be kept as low as possible (<10 %) due to its high price, therefore by moving from ground wood to thermomechanical pulp the paper industry has been able to increase the amount of mechanical pulp (Bäckström, 2004; Aittamma, 2007).

The most common technique for printing newspaper is the offset printing. Nowadays almost 99 % of all newsprint is printed with offset. While there are several types of offset printing, the most common for this type of paper is the heatset web offset (HSWO).

In offset printing the ink is not applied directly from the printing plate (or cylinder) to the paper. The ink is first applied to the printing plate and then transferred to a rubber denominated as blanket. The image on the blanket is then transferred to the paper in order to produce the printed product.

During the offset printing with heatset technology structural changes to the paper occur, causing quality disturbances, in the form of gloss reduction in the printed image and an increased surface roughness. Another problem caused by changes in the surface structure of uncoated papers during printing process is that the raised fibers get loose from the paper surface and adhere to the printing rollers. As the printing continues, the fibers accumulate in the image areas causing visible defects in the print. This is a common problem at the printing houses designated as linting by the paper industry (Hoc, 2005).

1.1.3 Linting

Linting is defined as the removal of loosely bonded particles from the surface of the sheet when paper is in contact with water based ink on the printing press (Amiri et al, 2004). It can also be described as a combined effect of the ink film splitting forces and the inter-fibre bonding energy of the paper surface. Fibers are removed from the surface of the paper when the external forces exceed the forces holding the fibres together.

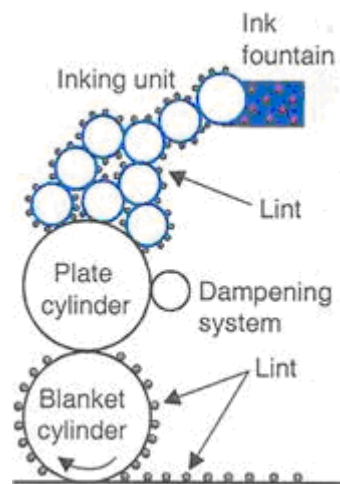


Figure 3 - Linting in the offset printing process (Aittamma, 2007).

As it is possible to see above in figure 3, the detached material adheres to the blanket accumulating on the blanket cylinders surface, what disrupts the transfer of ink from the inking system to the blanket and also when these materials are mixed with sticky ink, they will cause undesired markings on the paper or even tear its surface. This weak bonded material is designated as lint.

The result is a deterioration of print quality to the point where the press must be stopped and cleaned. At the printing houses this is an undesired action, since it has a strong effect on printing efficiency, particularly in high volume printing operations such as daily newspaper production. This has made linting one of the most serious paper-related problems and a

source of frequent complaints that paper manufactures have to deal with (Mangin and Dalphond, 1992). Examples of printing defects due to linting are shown in figures 4 and 5.



Figure 4 - a) Shows printing defects, on the red area, caused by linting. b) Magnification of lint deposits where is possible to see red inked ray cells (Courtesy of SCA).

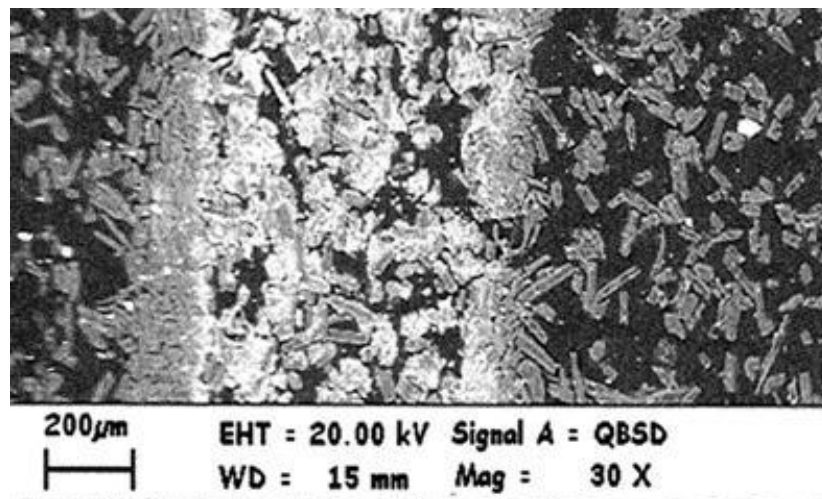


Figure 5 - SEM image of a blanket showing lint deposits (Palovaara, S., Courtesy of SCA).

For around 50 years, both manufactures and printers have been dealing with this linting issue and even its nature has changed, due to print process and paper development, linting is still a problem not solved or fully understood.

The problem can not be regarded solely as a papermaking problem, since linting is influenced by several parameters such as wood composition, pulping, paper machine, calander, printing press, ink, fountain solution, printing plate, blanket and also the printer. Some earlier studies in this field have concluded that many press variables often have a higher influence on paper linting performance than the paper itself. Despite this paper-manufacturers must continually increase paper quality in order to reduce its linting propensity; otherwise it may decrease the sales rates.

The strongest impact of the linting phenomenon occurs in offset printing of paper grades composed by mechanical pulps, like newsprint paper. This is related to the low specific surface area of the fibers in mechanical pulps, and consequently their weak bonding potential.

According to several studies, lint consists of non fibrillated short narrow fibers. In general lint particles are ray cells, nearly 80 % of the total, but it can also include fibre fragments, shives and fillers (Amiri et al, 2004; Bouchard, 2001).

In order to minimize the linting problems, paper industry often uses several techniques, such as: increasing the specific energy during the refiner process, increasing the percent of chemical pulp or addition of cationic starches at the wet-end. However all these techniques have a common drawback, they considerably increase production cost.

1.1.4 Fines

Fines is the general term used for small-size solid substances present in papermaking furnishes and suspensions. The fines fraction is usually defined as the part of a fibre suspension that passes through a 200 mesh screen in a Bauer McNett apparatus (Luukko et al., 1997).

Various types of particles can be identified in the fraction containing the fines, e.g., flakes and lamellas, thread-like fibrils, tori from bordered pits and ray cells (Sundberg and Holmbom, 2004). But occasionally fillers and pigments, as well as other small material from paper broke, are also included in the fines concept (Krogerus, 2003). Figure 6 shows a diagram with the most common types of fines.

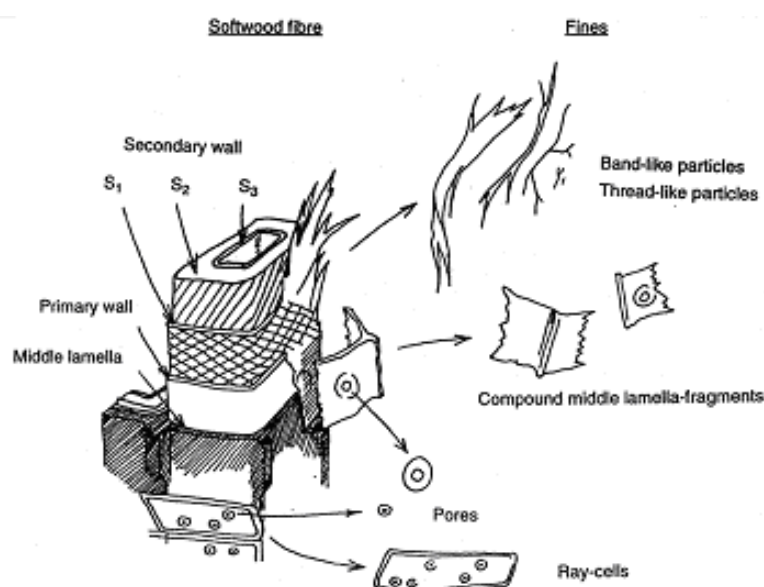


Figure 6 - Most common types of fines (Rundlöf, 2002).

The fines fraction type depends on the refining condition and depending on the end use. Mechanical pulps contain from 25 % to 40 % of fines by mass (Luukko et al., 1997; Ilvessalo-Pfäffli, 1995).

Fines are important for paper production, since they have a special character that gives printing paper its unique combination of high opacity and reasonable strength. It has been shown that flakes increase the light scattering and thereby the opacity, while fibrils increase the strength of the paper (Luukko and Paulapuro, 1999a). However ray cells are known to have a very low bonding ability, and as it was referred before they can cause linting problems.

1.1.5 Ray Cells

Rays are ribbon like structures of short cells (ray cells) extending radially in the stem. Their function is to store and distribute nutrients mainly in the radial direction of the tree. In softwoods rays represent 5-11 % of the volume, and they are mostly one cell wide in the tangential direction, uniseriate rays (figure 7), only rays that have a horizontal resin canal in their centre are several cells wide, fusiform rays (figure 7) (Ilvessalo-Pfäffli, 1995).

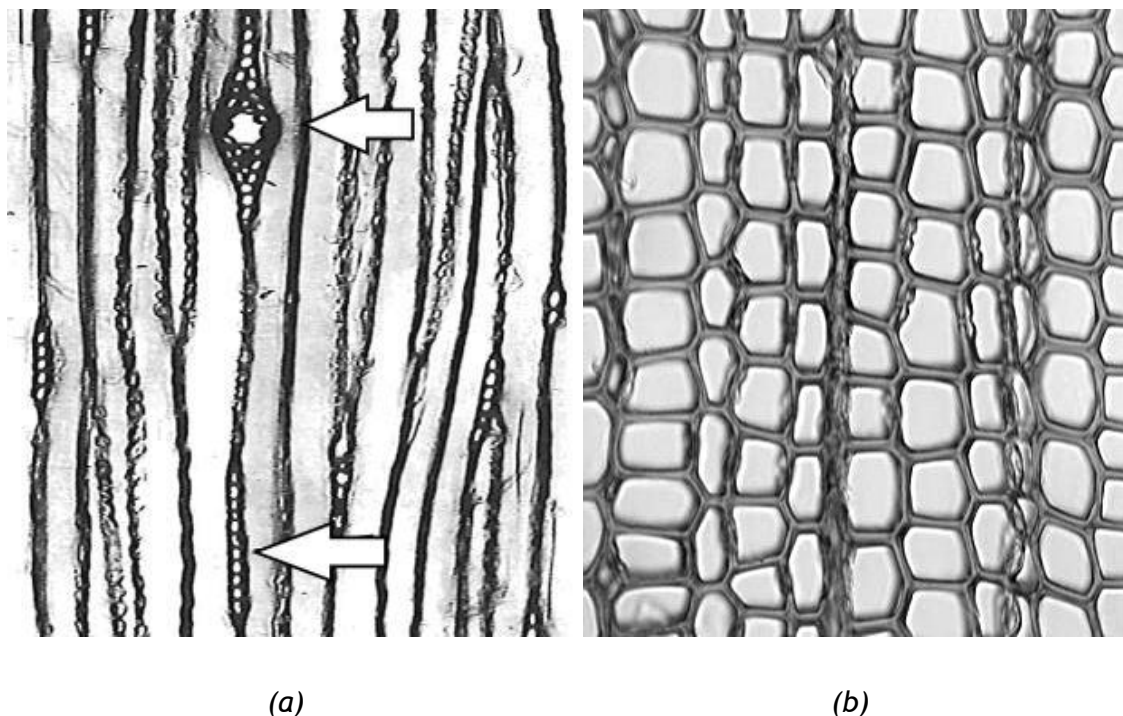


Figure 7 - a) Tangential section of Norway spruce showing the two types of rays, uniseriate and fusiform (down and upper arrow respectively), 100x (Ilvessalo-Pfäffli, 1995).

b) Cross section of tracheids in the earlywood of Norway spruce. In the centre, stretching from bottom to top, a ray can be seen (Modén, C., 2008).

The rays of softwoods are composed either of parenchyma cells alone or parenchyma cells and ray tracheids. Several studies regarding mechanical pulp fines have shown that for TMP samples the fraction of ray cells is mostly parenchyma cells, however small amounts of ray tracheids identified by their bordered pits, can also be seen (Sundberg et al., 2003).

Ray parenchyma cells are rather bricklike, thin-walled and they have simple pits to provide nutrient transport. Ray tracheids are about the same size as the ray parenchyma cells, but they have small bordered pits leading to the other ray cells.

A schematic draw of both ray tracheids and ray parenchyma cells and a SEM image of ray cells fraction in a TMP sample are represented in figures 8 and 9 respectively.



Figure 8 - Schematic representation of Spruce ray cells, a) ray tracheids and b) ray parenchyma cells (Ilvessalo-Pfäffli, 1995).

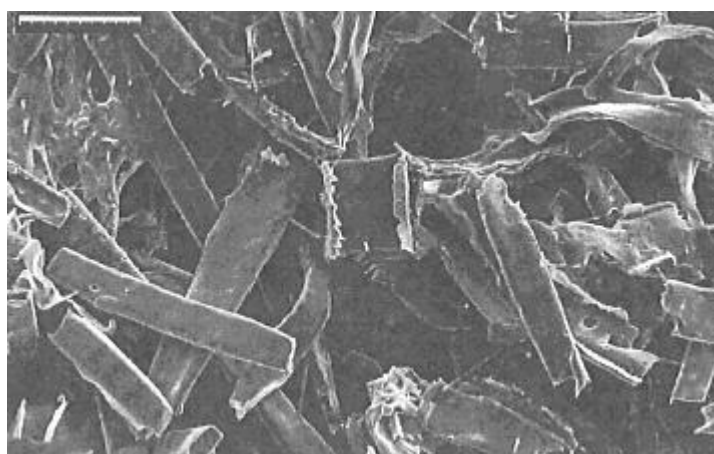


Figure 9 - SEM image of ray Cells fraction from TMP, bar=50 μm (Sundberg et al., 2003).

Ray cells represent a mass fraction of 4-7 % for most of the coniferous species used for mechanical pulping, around 4.7 % for Norway spruce (Ilvessalo-Pfäffli, 1995; Beaulieu and Shallhorn, 1993; Bouchard, 2001).

Ray cells are easily recognized by its characteristic bricklike shape and therefore very low aspect ratio. They have width similar to normal fibers but the length can be ten times smaller than an average fiber.

A study of characterization of mechanical pulp fines from Norway spruce revealed that ray cells have length of 70-160 μm and a width of 15-25 μm . It is possible to find longer ray cells with lengths of 90-350 μm , this is more common in CTMP but small fractions can also be found

in TMP. This might be due to the chemical treatment in CTMP pulping, which causes less damage to ray cells (Sundberg et al., 2003; Amiri et al, 2004).

It has been shown that ray cells' chemical composition is different from fibers. Ray cells have less cellulose and they contain approximately 42 % of lignin as compared to regular fibers with a lignin content of 27 % (Bouchard, 2001, Sundberg et al., 2003).

Thus they have a nutrient transport and storage function ray cells have higher resin content, it may comprise 10 - 40 % of the dry weight for ray parenchyma cells (Back, 2002).

It has been suggested that the morphological shape and the very low surface area together with a low hydrogen bonding, related to high resin content, are responsible for a weak bonding ability of ray cells to fibres (Sudarno, A., 2006; Wood et al., 2000). Therefore they are easily dislodged from the paper surface, being the source of linting problems.

1.2 Project Contributions

In consequence of technological evolution, there has been a decreasing demand on printing paper. Therefore to keep the sales level the market share must be potentially increased and this depends on two important factors namely paper quality and price.

At Ortviken Paper Mill large efforts have been done in order to increase product quality, thereby the production of improved newsprint has been increasing compared to standard newsprint. This quality is predominantly printed in heatset web offset printers for which linting is one of the most common causes of complaints. Adding cationic starch has been a temporary solution for linting, but it considerably increases the production costs, therefore it is necessary to bring a new long term solution for it.

Solving the problems requires a deeper knowledge of them, thereby identifying and controlling the amount of linting propensity particles during the papermaking process is inevitable. Even there are some paper analysis equipments for linting prediction, it is almost impossible to predict it at the paper mill. Proper pulp analysis of the presence and amount of ray cells is therefore indispensable. Thereby this thesis brings a novel method with the capacity of predicting the amount of ray cells in a pulp sample.

1.3 Thesis structure

This thesis consists of 7 chapters. Chapter 1 includes a brief introduction to the topic and a description of the motivation for this study. Chapter 2 comprises a short literature review with focus on identification and analysis of fines. Chapter 3 presents a detailed description of the experimental procedures together with the equipment used for the present method development. In chapter 4 are presented the results obtain during the experimental

procedures. In chapter 5 are discussed the results. Chapter 6 comprises a summary of the conclusions obtained during the project. Chapter 7 includes a personal evaluation of the project together with recommendations for future work.

2 State of the Art

As the aim of the project is to identify a specific group of fines, in thermomechanical pulp, it is interesting to introduce what has been discovered in this area.

In order to organize all the information, the state of the art was divided into three groups: acoustic, chemical and image analysis.

2.1 Acoustic analysis

Björk and Danielsson (2004) have developed a method to extract fibre length distributions from acoustic data recorded on pulp streaming from a TMP refiner. The method is based on a Continuous Wavelet Transform (CWT) and post-treatment with transformed data. The measurements were performed using an accelerometer on the refiner blow-line during a full-scale trial with a double disc refiner. The post-treatment, for calculation of the pseudo fiber length classes was done with Wavelab and Matlab software (Björk and Danielsson, 2004).

In another article published by the same authors in 2007, was concluded that, considering each fiber as a beam and using beam theory and reasonable dimension and material data, the Eigen-frequency for fibers would be some kHz to hundreds of MHz, which corresponds to roughly 3 mm to 0.01 mm of fiber length. It was also demonstrated that there was a good agreement between Björk and Danielsson results and Fiber-Master results (Björk and Danielsson, 2004; Björk and Danielsson, 2007).

This method has the advantage of being suitable for on-line implementation, however this requires an improvement of the acoustic signal quality. Although the first article was published six years ago, to our knowledge, no further investigations have been done in this area. However implementing an acoustic measurement requires substantial engineering and method development, a work that can take several months up to years with an experienced research group and the likelihood of succeeding is extremely low. Other drawbacks of using these acoustic measurements are related to the variety of results that are possible to obtain with it, as it was said before, is only possible to obtain the fiber length and this single criteria is not enough to characterize and classify the parenchyma cells in the pulp.

2.2 Chemical analysis

According to bibliographic research done, it has not been yet published any studies concerning the relation between the presence of a certain chemical component and fibres distribution in pulp. Although some studies in wood and mill water effluents have been done.

Tokareva *et al.* published an article, where they use a novel method for labelling of anionic groups on wood. The authors use metal ions as markers and their patterns of attachment to anionic groups were assessed by time-of-flight secondary ion mass spectrometry (ToF-SIMS). Sr^{2+} was found to be the best marker, and ToF-SIMS imaging of Sr^{2+} -labelled anionic groups revealed abundant anionic groups ion bordered pit tori and ray parenchyma cell walls in spruce wood (Tokareva *et al.*, 2010).

Despite the importance of this study to the knowledge of spruce wood morphology in respect to presence and distribution of anionic groups, it is not bringing any advantage to this thesis, because some important questions are still unknown. For example if there are any specific anionic groups from parenchyma cells, or if there is any pattern that can be used to characterize the parenchyma cells in a sample of pulp. However, in the future, it may be interesting to study if there are any specific anionic groups or some charge pattern characteristic from parenchyma cells.

Serreqi *et al.* developed a method to determine the concentration of resin in TMP and CTMP pulp mill effluent samples. In this method the authors determine a suitable marker for the total content of resin, and concentration of the marker was correlated with concentration of resin. According to the results, abietic acid is the best single marker for total resin content in this effluent samples (Serreqi *et al.*, 2000). It is well known that mostly of the resin is located inside ray cells, therefore it can be interesting to study the amount of resin in the pulp and relate it with the amount of ray cells (Krogerus, 2003; Ilvessalo-Pfäffli, 1995). Although a study of this type requires several months for data collecting and also there are no certainties if any correlation between the concentration of resin in the effluent and the amount of parenchyma cells exists or with the linting propensity of the paper.

2.3 Image analysis

Since 1988, several microscopy techniques have been utilised for pulp fibre assessment, including light microscopy (LM) (Kibblewhite and Bailey, 1988), confocal laser scanning microscopy (CLSM) (Jang *et al.*, 1991) and scanning electron microscopy (SEM) (Reme and Helle, 2002). However, in these techniques, fines classification is usually performed manually, requiring large efforts from an operator and is thus time-consuming, which increases production costs. Therefore automatic image analysis routines are required for optimising the characterisation of pulp fibres (Chinga-Carrasco *et al.*, 2009).

Luukko *et al.* in 1997, developed a method that uses a standard CCD camera, a light microscope and an image analysis program. The processing principle is based on the finding that fibrils generally are brighter than other particles. Therefore in this method the particles

are classified into fibril and non-fibril material, where the output is the apparent fibril portion in per cent of total fines mass (Luukko *et al.*, 1997).

This method is an approximation, because some lamellas may appear bright, and some long fibrils may look dark, however in 1999 other studies have shown that the method gives a correct indication (Luukko and Paulapuro, 1999; Krogerus *et al.*, 2002a).

The method developed by Luukko *et al.*, was further improved by Metso Field Systems Inc and started to be commercialized in 2002 with the name of kajaaniFiberLab. The equipment has length measurement optics (with Laser and Xenon light sources), that detects the presence of a fiber and monitors its passage through the capillary. When the fiber reaches the centre, the Xenon lamp flashes and the length and cross-sectional cameras simultaneously capture an image of the same fiber. The output of the equipment can be different formats of fiber images (e.g. binary form), or the image can be processed by the software, and results such as fiber length, fiber width, curl, cell wall thickness, cross sectional area and fiber volume index are calculated (Oikarinen and Rakkolainen, 2002). With a trial version and confidential property of Metso field Systems Inc, of this image analysis software is also possible to identify the amount of ray cells among. The programme identifies particles with a rectangular shape (brick-like), which is typical from ray cells, and computes their portion in terms of total mass percent of fines (Ilvessalo-Pfäffli, 1995; Krogerus *et al.*, 2002b). Also here the value has to be regarded as an “apparent” value, because its significance has not been explored sufficiently, and the ray cells shape criteria are very strict. It means that, only totally intact ray cells are counted, and also other fines with similar shape can be counted (Krogerus *et al.*, 2002a).

Hirn and Bauer in 2006, made a review of image analysis methods to evaluate fiber properties. This review shows that a considerable effort has been put on both, academic research as well as commercial development of image analysis based fiber characterization methods. Nevertheless for fiber surface chemistry respectively pulp swelling and resolving behaviour no commercial analyzers are on the market yet (Hirn and Bauer, 2006).

Later on, in 2009, Chinga-Carrasco *et al.* introduced a shape description approach as a step for performing an automatic processing of fibre cross-sectional images. The automatic processing consists of a shape description approach, where fibres that did not have a predefined shape requirement were rejected and removed. The authors classify fibres into several clusters, where each cluster may be post-processed differently; and that it may be advantageous to divide a fibre population into intact and split fibres (Chinga-Carrasco *et al.*, 2009). This approach is valuable to assessing the spatial distribution of wood pulp fibres, however this is a generic method that classifies fibres according fibre morphology, and it not classifies/quantifies a specific group of fibres.

In 2010 Chinga-Carrasco *et al.* published an article that describes a new approach, where FE-SEM and relevant computerized image analysis are applied to quantify the fibre wall characteristics. Details such as surface roughness and texture of the fibre are quantified objectively. The image processing and analysis is fully automated and implemented as an ImageJ plug in and the method consists in applying a gradient analysis for quantifying fibre surface structures. The gradient analysis is based on Sobel operators and is performed in the middle area of the images, and with it is possible to measure the anisotropy and the azimuthal orientation of a certain fiber.

This approach offers a novel characterisation of surface structures, moving forward from performing subjective evaluations to performing objective quantifications of wood pulp fibres surfaces (Chinga-Carrasco *et al.*, 2010).

Still in 2010, Yamakawa and Ching-Carrasco compared two statistical approaches for automatic classification of fibre shapes. Open-source software, ImageJ was applied for image processing analysis and the fibres were classified into three groups by the number of lumens. In order to classify the fibres into several groups, two discriminant analyses methods were used, Canonical Discriminant Analysis (CDA) and Mehalanobis Discriminant Analysis (MILDA) (Yamakawa and Chinga-Carrasco, 2010). This approach can not be used for parenchyma cells classification, because there are several fines with similar shape and also it will not identify broken cells, nevertheless it may be interesting to combine this method with others.

In 2002 Krogerus *et al.* said that determination of ray cells by image analysis is an interesting option that has to be further studied (Krogerus *et al.*, 2002b). And, to our knowledge, no studies have been published so far concerning determination of the amount of ray cell in the pulp.

3 Materials and Methods

This chapter presents a detailed description of the experimental procedures and equipment used during this project.

3.1 Samples

The pulp samples used in this project were unbleached Norway spruce thermomechanical pulps (TMP), obtained from Ortviken paper mill. TMP samples were collected at the TMP mill and at 2 different places of PM5 (paper machine number 5) production line and named according to its locations as Standpipe TMP84, PM5 TMP Chest and PM5 Machine Chest. Standpipe TMP84 comprises pulp collected at the TMP mill, immediately after the refining and screening process. PM5 TMP chest and Machine chest refers to samples collected at the end of the pulp line right before the paper machine. Pulp samples details and a scheme of the collected locations are presented in appendix 1.

Fines samples, extracted from PM5 machine chest pulp samples with a Bauer McNett apparatus, according to SCAN-CM 6:05 test method and considering as fines particles passing through a 200 mesh and being retained at a 635 mesh (P200/R635), were also used in this project. More detailed information about the Bauer McNett apparatus together with fines extraction procedure available in appendix 3.

3.2 Microscopic analysis method

This method was the first approach to the entire project; the initial idea was to quantify ray cells manually using an optical microscope and after it analyze exactly the same sample in the kajaaniFiberLab. The aim was to compare both results and determine the accuracy of kajaaniFiberLab for measuring ray cells in other pulp analysis.

After an initial process of getting familiar with the microscope, ray cells and other fines elements, the microscopic analysis was done according to the following steps: a PM5 machine chest fines sample was used; 2 drops of the sample were added to a microscope slide together with 5 drops of water. Using a spatula the sample was homogeneously scattered on the slide and left to dry in a hot plate for 5-10 min. The samples were then visualized at the microscope using 200x magnification. For this magnification the microscopic image diameter corresponds to 1 mm in the slide and since it has a width of 25 mm the sample was scanned 25 times according to figure 10 scheme.

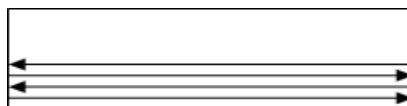


Figure 10 - Microscopic slide scan scheme.

During the scan procedure, ray cells were counted, using shape, size, wall thickness, Piceoid pits presence and resin content as selectivity criteria. In order to be considered as a ray cell, fines must have a brick-like shape, small length when compared to average fibers, thick cell walls, Piceoid pits and high resin content. Broken ray cells were also considered during the selection method. For further description of ray cells see section 1.1.5. Figure 11 shows the different characteristics of ray cells.

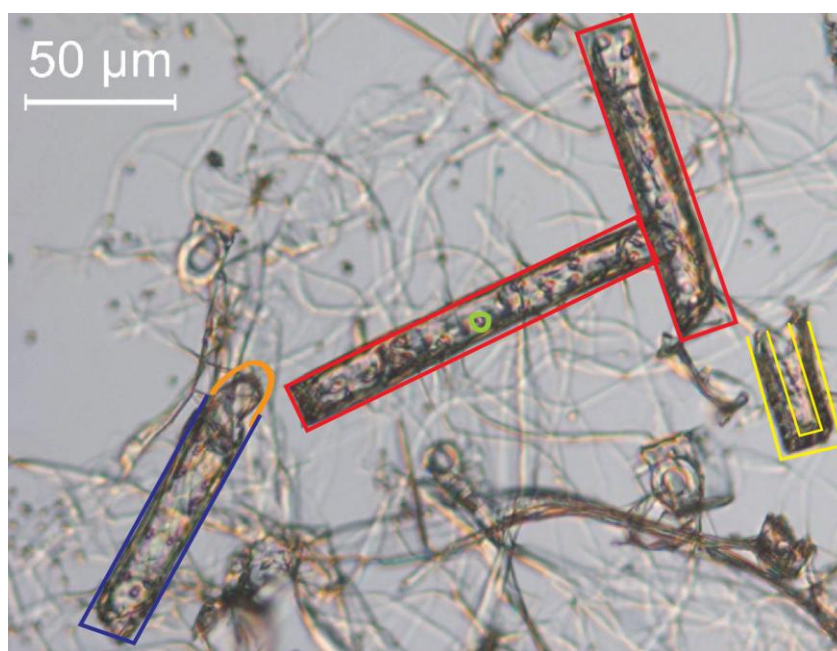


Figure 11-Optical microscope photography using differential interference contrast technique.

Red - unbroken ray cells with characteristic brick-like shape; Yellow - Ray cell with thick cell walls; Blue - Broken ray cell; Orange - Resin dropping from the cell interior; Green - Piceoid pits.

3.3 KajaaniFiberLab analysis method

The aim of this procedure was to determine if the analyzer was able to calculate relevant parameters for ray cell selection as well as to do a deeper study of pulp fiber morphological properties and size distributions.

The equipment used is a kajaaniFiberLab, developed by Metso automation, equipped with two CCD cameras and version 4 of FiberLab software; equipment details are available in appendix 4

A beaker containing the sample is placed in the sample unit. After the start command the analyzer mixes and dilutes the sample to the correct consistency for measurement. The sample is then sucked in, so that it flows through the capillary. Length measurement optics (with Laser and Xenon light sources) monitors its passage through the capillary. When the fiber reaches the centre, the Xenon lamp flashes, the two cameras, length and cross-sectional cameras simultaneously capture an image of the same fiber. The cameras have different field of view and different resolution. The length camera has a resolution of 10 μm . The cross-section camera has a resolution of 0.15 μm and a smaller field of view that in most cases is too small to see the full fibre length. The image is then processed by the software, and results such as fiber length, fiber width, curl, cell wall thickness, cross sectional area and fiber volume index are calculated. Singular fiber pictures from both cameras are also an output of the equipment. However for cross-section camera the pictures saved are not linked to any kind of measurement information. Examples of kajaaniFiberLab output are available in appendix 4.

KajaaniFiberLab is an equipment developed for fiber analysis, therefore it has some limitations concerning fines analysis. With FiberLab the minimum particle size that can be measured is 2 pixels, which corresponds to approximately 20 μm , this is due to camera resolution. However with this camera no fine particles with length smaller than 0.2 mm are measured. This is related to FiberLab measurement procedure, the fiber length is measured with the length camera and the width is measured from a magnified part of it with the cross-section camera. Fine particles do not always stabilize in the correct position to be measured by the cross-section camera, remaining outside the view of the camera; thereby no width measurements are performed for particles smaller than 0.2 mm in fiber length. Another limitation of the equipment is related to the software, picture with poor quality are not saved, even if the picture is taken and the measurements are done.

3.3.1 Samples

For pulp samples preparation, the amount of wet pulp needed in order to prepare a solution with a concentration of 0.1 g of dry pulp/L was determined according to appendix 2. The weighted pulp was then disintegrated using a manual disintegrator delivered with the analyser. Pulp samples were diluted with water at a concentration of 0.1 g of dry pulp/L and samples of 50 mL were poured into the analyzer cups. A concentration of 0.1 g dry pulp/L assures that FiberLab will be able to dilute the sample until the desired consistency (0.015-0.025 % *cs*) in order to obtain a fiber count of 40-100 fibers per second.

For fines samples preparation, the fines obtained from the Bauer McNett were diluted in order to obtain a fiber count of about 90 fibers per second.

If the fiber count is higher than 100 fibers per second the measurement is automatically interrupted and the sample is diluted again. Procedure details are available in appendix 4.

3.3.2 Image analysis

Image analysis procedure comprises a manual analysis of all the pictures collected with kajaaniFiberLab length camera.

Two samples from each pulp type: Standpipe TMP84, PM5 TMP Chest and PM5 Machine Chest and two fines samples extracted from a PM5 Machine Chest pulp sample were analysed. Per each sample analyzed FiberLab stored an average of approximately 2000 pictures. These pictures were manually analyzed in an ordinary image visualization program and pictures of ray cells were selected with the following criteria: it must have a brick-like shape, smaller length when compared to average fibers, thick cell walls, Piceoid pits and resin content. Piceoid pits were identified, when present, by small black dots and resin by brighter areas. Broken ray cells were also considered during the selection method. Figure 12 shows examples of FiberLab pictures of particles that can be identified as ray cells.

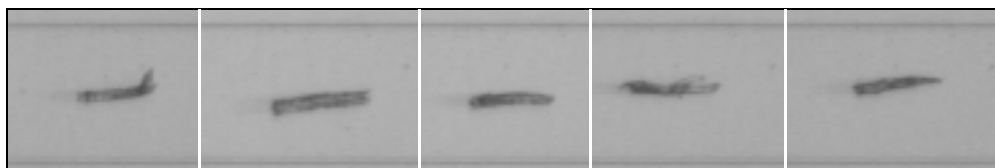


Figure 12 - Examples of kajaaniFiberLab pictures of ray Cells.

3.3.3 Data analysis

Data analysis consists of a detailed statistic evaluation of the morphological properties of ray cells and remaining particles, obtained with kajaaniFiberLab per each type of pulp.

The data analysis procedure comprises a statistical analysis of a large set of objects divided in ray cells and non-ray cells. For each parameter particle size histograms were determined using Microsoft Excel Analysis ToolPak add-in and using 11 discrete intervals (BINs). Sample mean, $\bar{x}$ and standard deviation, s were also calculated with Microsoft Excel Analysis ToolPak add-in using equations 3.1 and 3.2.

$$\bar{x} = \sum xP(x) \quad (3.1)$$

Where $\bar{x}$ is the mean and $P(x)$ the probability of finding a certain value x .

$$s = \sqrt{\frac{\sum (x - \bar{x})^2}{n - 1}} \quad (3.2)$$

Where s is the standard deviation and n the total number of objects.

A normal distribution was adjusted to the histogram by changing mean and standard deviation values. The cumulative normal distribution was also calculated, both calculations were done using excel formula NORMDIST that calculates the normal distribution and the cumulative normal distributions with equations 3.3 and 3.4 respectively.

$$f(x, \bar{x}, s) = \frac{1}{\sqrt{2\pi}s} e^{-\left(\frac{(x-\bar{x})^2}{2s^2}\right)} \quad (3.3)$$

$$F(x, \bar{x}, s) = \int_{-\infty}^x \frac{1}{\sqrt{2\pi}s} e^{-\left(\frac{(x-\bar{x})^2}{2s^2}\right)} dx \quad (3.4)$$

3.4 Ray Cells isolation

The main purpose with ray cells isolation was to obtain an estimation of the real amount of ray cells in a pulp sample and compare these results with the developed method results, in order to validate the algorithm.

Ray cells' isolation laboratory procedure used is based on two articles about chemical characterization of mechanical pulp fines published by Sundberg, A. *et al.* (Sundberg *et al.*, 2003; Sundberg and Holmbom, 2004). Some modifications were done to Sundberg's procedure, mainly caused by material availability together with the final purpose of this project. The Dynamic Drainage Jar (DDJ), was replaced by the Bauer McNett because it was not available at the laboratory, it also has been shown that there is a good linear correlation between fines content given by both apparatus (correlation available at appendix 5) (Krogerus *et al.*, 2002a). The settling times were also modified because there were only 1 L and 500 mL cylinder tubes available at the laboratory therefore it was impossible to dilute the sample in order to reach Sundberg's procedure consistencies.

TMP samples from PM5 Machine Chest and PM5 TMP chest were used, details about the samples available in appendix 1.

Extracted TMP samples were suspended in 3 L of water at a concentration of 3-4 g dry pulp/L. The pulp was then filtered in the Bauer McNett apparatus equipped with a 200-mesh wire to remove the fibers. The fines passing through a 200-mesh wire and retained at a 635-mesh wire (P200/R635), were collected in a filter paper at the bottom of the tank. The fines were removed from the filter paper with water and diluted in cylinder tubes of 1 L and 500 mL. Then the samples were left to settle for two days in a cold room at 4 °C. The supernatant was rejected and the sediment was again diluted in the same cylinder tubes and let to settle for

3 hours at 4 °C. 1 mL samples of sediment and supernatant were collected and analysed at the microscope (check 1). The supernatant was rejected and the sediment was again diluted in 1 L and 500 mL cylinder tubes and left to settle at 4 °C for 12 hours. The supernatant was rejected and the sediment was again diluted with water in the same cylinder tubes and left to settle for 1 hour at 4 °C. Samples of 1 mL from the sediment and supernatant were collected and analysed at the microscope (check 2). Supernatant was removed and the sediment was diluted and left to settle during 30 min at room temperature. 1 mL samples of both sediment and supernatant were collected and analysed at the microscope (check 3). During the microscope verifications it was considered accepted when the amount of other particles seen in each fraction was estimated to be smaller than 20-30 %. If the fraction of ray cells still is lower than 70-80 % it is recommend diluting the sample and let it to settle for 10 min at room temperature, this procedure should be repeated until the desired fraction is obtained. Bellow in figure 13 is shown a scheme of the procedure used.

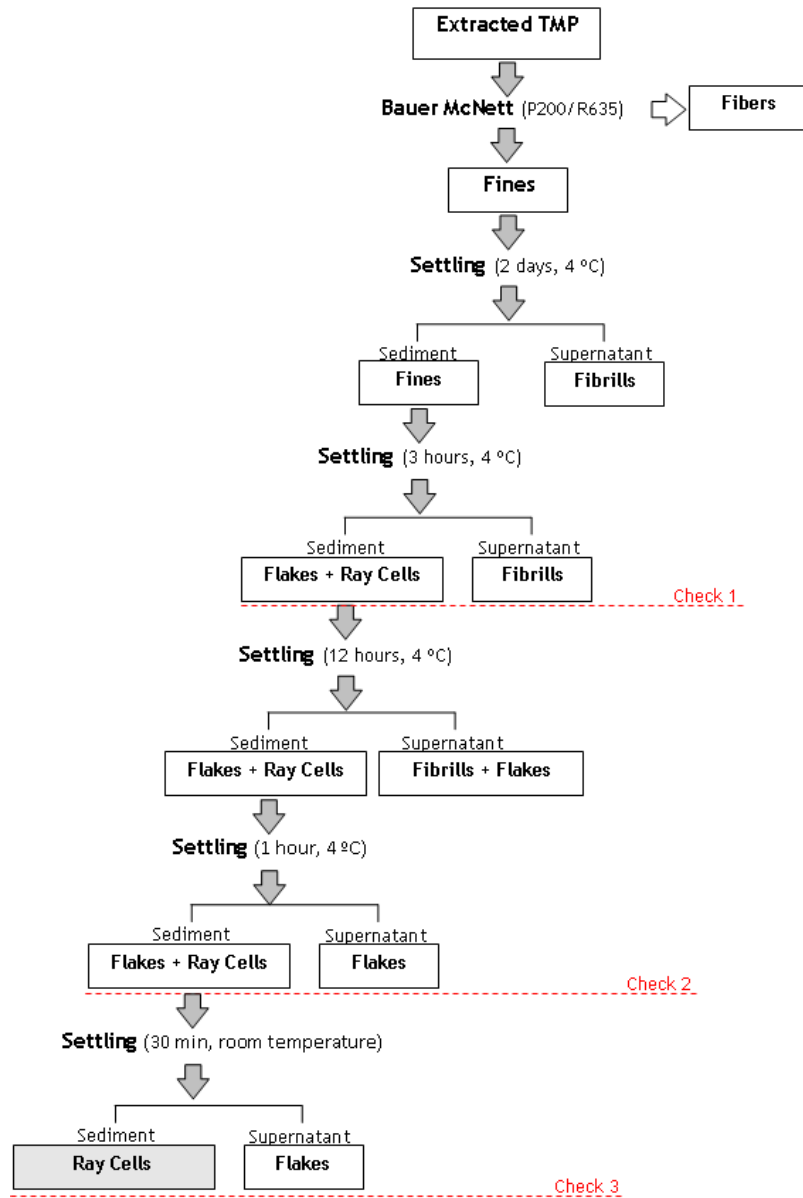


Figure 13 - Scheme of the laboratory procedure used for isolation of ray cells.

Completed the isolation of ray cells, flakes, fibrils and ray cells fractions were analysed in the FiberLab. Then the ray cells fractions were dried, following the procedures reported in appendix 2. The percentage of ray cells in the pulp was calculated with equation 3.5, where $Fraction_{RC}$ is the fraction of ray cells in the sample of pulp in %, m_{RC} is the dry mass in grammes (g) of ray cells from the isolation of ray cells procedure, m_{pulp} is the mass in grammes (g) of dry pulp added at the beginning of ray cells' isolation process.

$$Fraction_{RC} = \frac{m_{RC}}{m_{pulp}} \times 100 \quad (3.5)$$

3.5 Automatic analysis of ray cells content

This subchapter describes the procedures taken for development of an algorithm for ray cells quantification in a sample of TMP of spruce.

The algorithm does an automatic grouping of objects according to similarity properties having as input a set of data from kajaaniFiberLab individual fibre morphological analysis. The objects are clustered in two groups namely ray cells and non-ray cells, and the classification is based in a decision tree data mining technique.

The Decision tree, also known as classification tree, is one of the most popular classification methods in data mining. A decision tree takes as input an object or a situation described by a set of properties, and outputs a binary decision. This is an iterative process of splitting the data into partitions, classifying it into several clusters, known as binary recursive partitioning (Russel and Norvig, 1995). Figure 14 shows a diagram of a decision tree example, where a set of objects are submitted to certain condition and the algorithm gives a binary response.

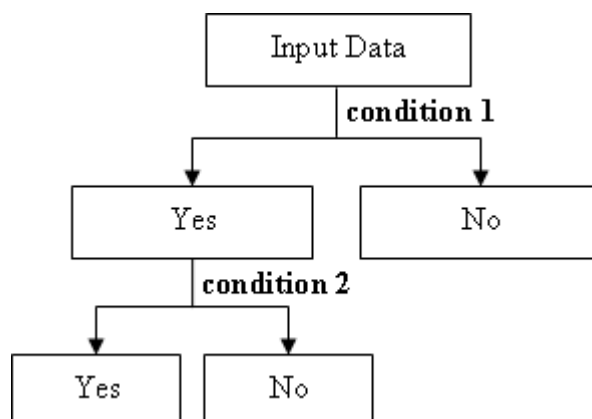


Figure 14 - Decision tree example.

The algorithm was implemented using Microsofts' VBA - Visual Basic for Applications, and runs using Microsoft Excel.

3.5.1 Algorithm evolution

Solving a cluster analysis problem depends on several important steps. The first two steps are reported in section 3.3 of this report, and comprise a manual classification of kajaaniFiberLab pictures into ray cells and non-ray cells and a posterior statistic analysis of ray cells and non-ray cells morphological parameters. The most relevant morphological parameters for the separation were then selected. Where the selection criteria is supported by the differentiation capacity, where a good parameter must show significant differences between both clusters.

Developed the general structure of the decision tree, the criterion of grouping was defined. Thereby numerical conditions were applied to each morphological parameter. These numerical conditions were based on statistical information provided by section 3.3.3 procedure. Both decision tree structure and decision conditions were defined in order to select ray cells among the amount of particles analysed.

Version 1 of ray cells' quantification algorithm has as input a set of kajaaniFiberLab data, from a pulp sample analyse of single fiber properties. The first decision is to separate the data into particles with pictures and particles without pictures. Then all the particles having pictures are divided by length size, into a cluster of smaller or equal to 0.5 mm and a cluster of particles bigger than 0.5 mm. Particles smaller or equal to 0.5 mm are classified according to curl, and divided into a group of particles with curl equal to zero and another group with curl different from zero. Then particles having curl equal to zero are grouped into fibrillation equal to zero and different to zero and finally particles having fibrillation equal to zero, are divided in two groups depending on cell width. Particles having width between 25 and 50 μm are classified as ray cells. Thereby according to this algorithm, a single particle must have a picture associated to it, length smaller or equal to 0.5 mm, curl and fibrillation equal to zero and a cell width between 25 and 50 μm in order to be considered as a ray cell. During the decision process the amount of ray cells is determined and at the end, the algorithm calculates the percent of ray cells, according to equation 3.6, where P_RC1 is the percentage of ray cells and $RC1$ is the amount of ray cells calculated by the algorithm (version 1) and $npic$ is the number of particles with picture associated. A list with picture file names for all particles classified as ray cells is also reported.

$$P_RC1 = \frac{RC1}{npic} \times 100 \quad (3.6)$$

Finally the calculated percentage of ray cells is compared with the percent of ray cells determined by the manual selection of ray cells and the deviation between both is calculated according to equation 3.7. Where P_RC_{manual} is the percentage of ray cells determined by the manual selection of ray cells, P_RC1 is the percentage of ray cells determined by version 1 of the algorithm and $Deviation_{manual}$ is the deviation between the algorithm and the manual image analysis.

$$Deviation_{manual} = |P_RC1 - P_RC_{manual}| \quad (3.7)$$

The deviation calculation is only performed if there is available information regarding ray cells' manual selection, in this case only samples refereed before in section 3.3.3.3.21 were analysed. A diagram of version 1 of the algorithm is available in appendix 6 - figure 29.

In order to increase clustering efficiency, the numerical conditional limits of each morphological parameter were optimized. The optimization was done for each type of pulp: PM5 Machine chest, PM5 TMP Chest and Standpipe TMP84 pulp samples. KajaaniFiberLab results of two samples from each type of pulp were used and several trials with different combinations of morphological parameters and numerical limits were taken with version 1 of the algorithm. The results of each trial were plotted, and the numerical limits were analyzed and selected in order to minimize the deviation between both methods.

Version 1 of the algorithm was upgraded and named as version 2. This version, apart from all the calculations reported before, determines the amount of true and false ray cells, where a true ray cell is considered as a particle selected in both methods, manual and automatic, and a false ray cell is a particle selected as a ray cell by the algorithm and identified as a non-ray cell particle by the manual selection. Determination of true and false ray cells was also performed manually in order to check algorithm accuracy. Percentage of true and false ray cells are calculated with equations 3.8 and 3.9 respectively.

$$P_TrueRC = \frac{TrueRC}{npic} \times 100 \quad (3.8)$$

$$P_FalseRC = \frac{FalseRC}{npic} \times 100 \quad (3.9)$$

Where, P_TrueRC is the percentage of true ray cells, $TrueRC$ is the number of true ray cells in the sample, $P_FalseRC$ is the percentage of false ray cells, $FalseRC$ is the number of false ray cells in the sample and $npic$ the number of particles with picture analyzed. A diagram of version 2 is available in appendix 6 - figure 30.

Cell width numerical limits were optimized. For this optimization, the same samples as for the prior optimization were used. Several trials with different numerical limits of parameter width were calculated with version 2 of the algorithm. Per each trial the ratio of true ray cells in the total amount of ray cells was calculated with equation 3.10.

$$TrueRC_Ratio = \frac{TrueRC}{RC} \quad (3.10)$$

Where, $TrueRC_Ratio$ is the fraction of true ray cells ($TrueRC$) in the total amount of ray cells (RC).

Results of the deviation, percentage of true ray cells and true ray cells ratio of each sample were plotted and analyzed. The limits selection criterion is based on a minimization of the

deviation, and a maximization of the percentage and ratio of true ray cells. Following these criteria new width limits were defined.

Version 2 was upgraded, and now version 3 of the algorithm adds the determination of ray Cells from the amount of fibers/particles without picture. The calculation procedure for particles without associated picture is exactly the same as for particles with picture. The calculated amount of ray cells is defined as ray cells 1 ($RC1$) from particles with picture, and as ray cells 2 ($RC2$) from particles without picture. The total number of ray cells (RC) corresponds to the addition of both fractions, $RC1$ and $RC2$ (equation 3.11). A diagram of version 3 is available in appendix 6 - figure 31.

$$RC = RC1 + RC2 \quad (3.11)$$

The last version of the algorithm designated by version 4 adds a third calculation of the amount of ray cells designated as ray cells 3 ($RC3$). As it is presented in section 3.3 kajaaniFiberLab has some limitations, thus fiber pictures with poor quality and with fiber length smaller than 0.2 mm are not saved, and for particles with fiber length smaller than 0.2 mm no cell width calculations are performed. Regarding these limitations, and the fact that ray cells can have very small fiber lengths, This third calculation, determines the amount of ray cells from particles without picture and with a fiber length smaller than 0,2 mm. Three calculations are now performed by the algorithm, equations 3.12, 3.13 and 3.14 show the characteristics that a particle should have in order to be considered as a ray cell per each calculation, ray cells 1, 2 and 3 respectively.

$$RC1 = \{Picture; Length \leq 0.5; Curl = 0; Fibrillation = 0; 24.5 \leq Width \leq 50\} \quad (3.12)$$

$$RC2 = \{0.2 \leq Length \leq 0.5; Curl = 0; Fibrillation = 0; 24.5 \leq Width \leq 50\} \quad (3.13)$$

$$RC3 = \{Length \leq 0.2; Curl = 0; Fibrillation = 0\} \quad (3.14)$$

The total amount of ray cells (c) corresponds now to the addition of $RC1$, $RC2$ and $RC3$ (equation 3.15).

$$RC = RC1 + RC2 + RC3 \quad (3.15)$$

The total fraction of ray cells, P_{RC} is calculated with equation 3.16. Where RC is the total amount of ray cells and n is the total number of fibers in the sample.

$$P_{RC} = \frac{RC}{n} \times 100 \quad (3.16)$$

A diagram of version 4 of the algorithm is available in figure 15.

The contribution of ray cells 1, 2 and 3 for the total amount of ray cells was studied; for that several pulp samples were analyzed in kajaaniFiberLab and the respective data files executed by the algorithm.

The contribution of each fraction was calculated with equation 3.17 for all the samples analyzed and the average of them were determined per each summand.

$$contributionRCi = \frac{RCi}{RC} \times 100 \quad (3.17)$$

Where RCi is the number of ray cells i ($i=1, 2$ or 3), RC is the total amount of ray cells in the sample and $contributionRCi$ is the contribution in percentage of ray cells i ($i=1, 2$ or 3) to the total amount of ray cells.

The results of the amount of ray cells obtained with the last version of the algorithm were for each pulp sample compared with the results obtained from the isolation of ray cells method. The deviation between both methods was calculated with equation 3.18.

$$Deviation_{isolation} = |P_RC - P_RC_{isolation}| \quad (3.18)$$

Where, P_RC is the total percentage of ray cells determined with the algorithm, $P_RC_{isolation}$ is the percentage of ray cells calculated from ray cells' isolation method and $Deviation_{isolation}$ is the deviation between both methods (automatic analysis and ray cells isolation).

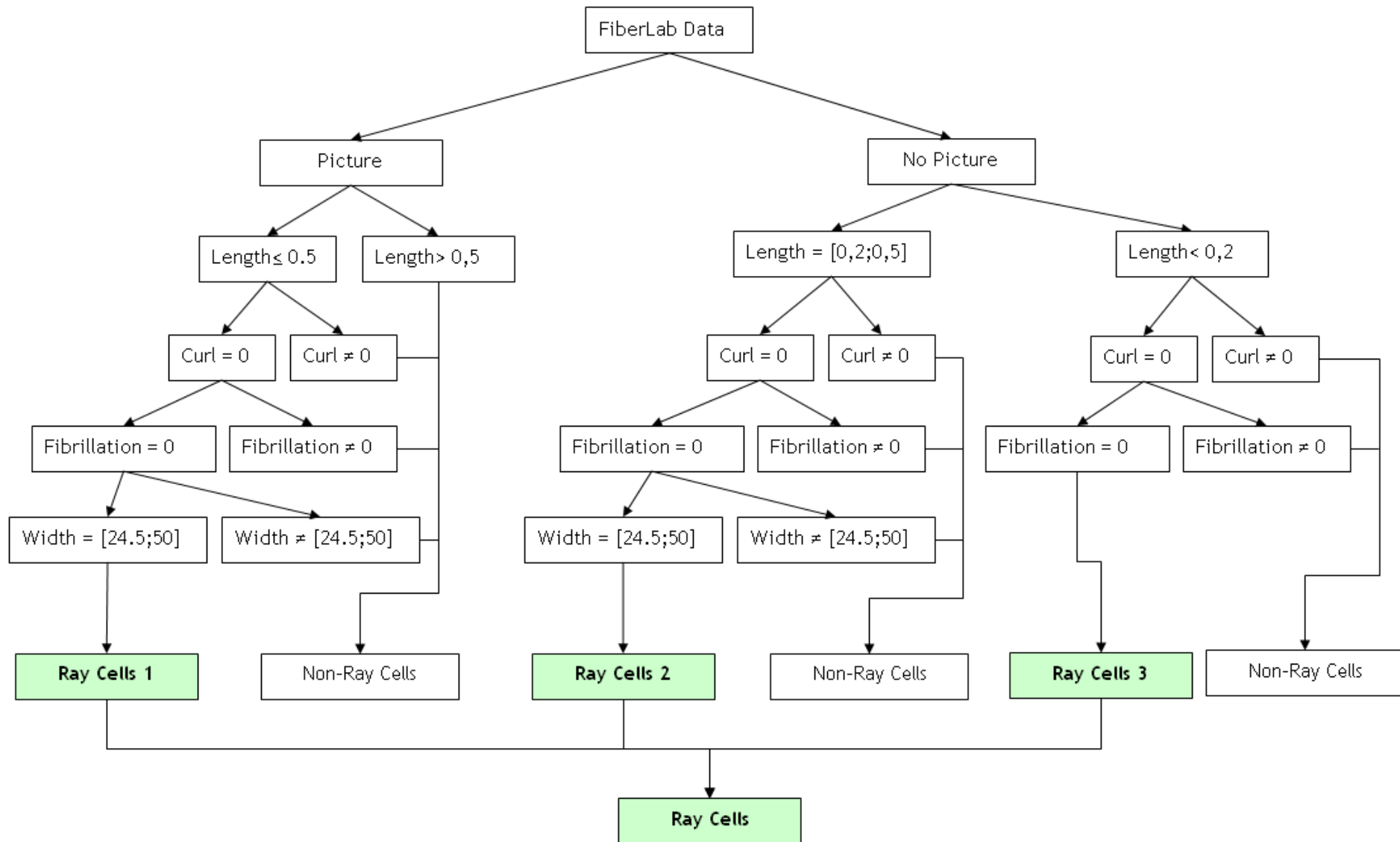


Figure 15 -Representative diagram of ray cells automatic quantification algorithm - version 4.

4 Results

This chapter presents the results obtained with the procedures reported in chapter 3. The results are divided according to the method used, microscopic analysis method, FiberLab analysis method, ray cells isolation procedure and automatic analysis of ray cells content.

4.1 Microscopic analysis method

For the microscopic analysis method no relevant results were obtained. It was possible to quantify ray cells in a microscopic glass sample, however with this technique it was not possible to determine its fraction, due to the higher amount of particles that were overlapped. Another drawback with this technique is that it is time consuming, being necessary approximately 4 hours to analyze each sample.

Due to a very low consistency of the sample extracted from the microscopic glass it was impossible to analyze it in the FiberLab.

4.2 FiberLab analysis method

Figures 16 and 17 shows examples of particles selected as ray cells and non-ray cells during the manual selection procedure, reported in section 3.3.2. Results of the amount of ray cells per sample analyzed are available below in table 1



Figure 16 - Examples of ray cells, identified during the manual selection procedure.

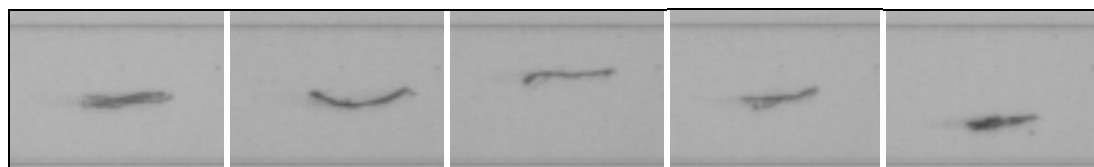


Figure 17 - Examples of particles similar to ray cells, identified as non-ray cells during the manual selection procedure.

Table 1 - Results from manual determination of the amount of ray cells.

Sample	Number of fibers	Number of pictures	Number of Ray Cells	Percentage of Ray Cells, %
MC-PM5_1	4405	1849	56	3.03
MC-PM5_2	4584	1929	50	2.59
TMPchest_1	3435	1500	31	2.07
TMPchest_2	3088	1376	42	3.05
TMP84_1	3682	1537	62	4.03
TMP84_2	3339	1395	39	2.80

From the statistic evaluation of ray cells' morphological properties and according to particle size distributions and cumulative probabilities available in appendix 7, the fiber length, width, curl and fibrillation were selected as the most relevant parameters for ray cells classification.

Table 2 shows the inferior and superior limits, from each selected parameter, favourable for ray cells selection.

Table 2 - Numerical conditions/limits favourable for ray cells selection.

Parameter	Inferior limit	Superior limit
Length	0.2 mm	0.5 mm
Width	25 μm	50 μm
Curl	0 %	0 %
Firbillation index	0	0

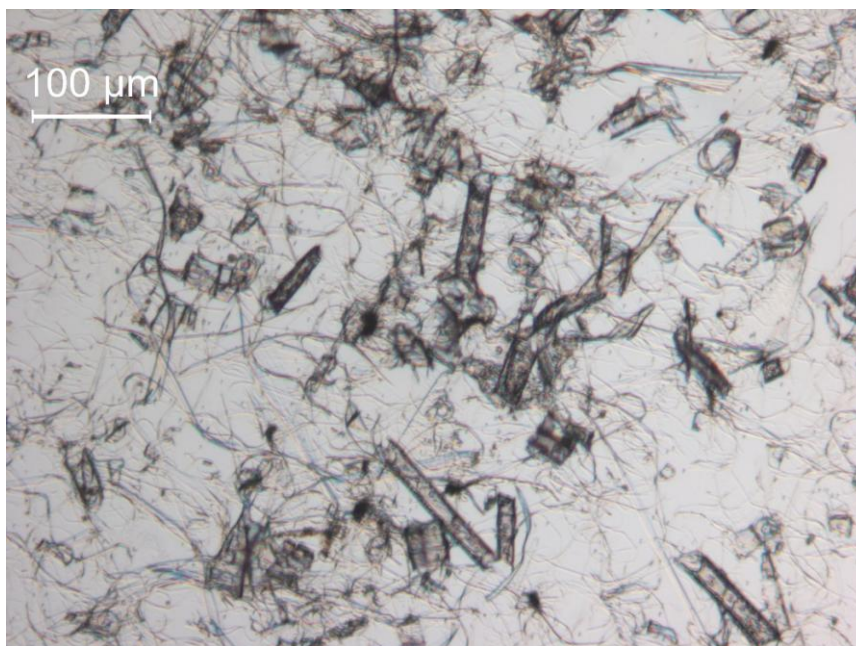
4.3 Ray cells isolation

During the fines extraction process with the Bauer McNett apparatus sample loss occurred in samples 1.1, 1.2 and 2.1.

Table 3 shows ray cells' fractions determined with this method. A microscopic photography of a ray cells fraction is available in figure 18.

Table 3 - Ray cells fractions determined with isolation of ray cells method.

Sample	Isolated Ray Cells fraction, %
1.1	0.34
1.2	0.45
1.3	3.19
2.1	0.91
2.2	2.59
2.3	5.03
3.1	6.07
3.2	3.56

*Figure 18 - Optical microscope photography of isolated ray cells sample.*

4.4 Automatic analysis of ray cells content

The results obtained from the automatic analysis of ray cells content method, are mainly divided in two parts. A first part related to the algorithm evolution and parameters optimization and a second part related to the main results of the final algorithm.

After the first optimization performed with version 1 of the algorithm it was verified that the parameters selected and its respective numerical limits were suitable for ray cells'

identification. It was also verified that fibrillation and curl are essential parameters, showing good results when both are combine together with the other parameters.

The performance of the three different pulp samples have shown similar behaviour however for standpipe TMP 84 sample there is a small discrepancy of the numerical limits when compared with other samples. These can be seen in the figures available in appendix 8.

The results obtained from the optimization of versions 2 and 3 of the algorithm are available below in tables 4 and 5.

Table 4 - Numerical conditions/limits obtained from optimization of version 2.

Parameter	Inferior limit	Superior limit
Length	0.2 mm	0.5 mm
Width	24.5 μm	50 μm
Curl	0 %	0 %
Fibrillation index	0	0

Table 5 - Numerical limits of ray cells 3 (RC3) calculation.

Parameter	Inferior limit	Superior limit
Length	0 mm	0.2 mm
Curl	0 %	0 %
Fibrillation index	0	0

Average contribution of *RC1*, *RC2* and *RC3* for the total amount of ray cells are shown in table 6.

Table 6 - Average contribution of each summand for the total amount of ray cells.

Summand	Average contribution for the total amount of ray cells
Ray cells 1 (<i>RC1</i>)	20 %
Ray cells 2 (<i>RC2</i>)	7 %
Ray cells 3 (<i>RC3</i>)	70 %

The results for the amount of ray cells obtained with the algorithm are presented below in table 7. The deviation between samples from the same pulp sample is also presented.

Table 7 - Automatic Amount of ray cells determined with the last version of the algorithm.

Sample	Collected point	Number of fibers	Percentage of Ray Cells, %	Deviation, %
MC-PM5_1	PM5 Machine Chest	4405	4.72	0.12
MC-PM5_2l		4584	4.60	
1.1Pulp		12246	4.16	0.28
1.2Pulp		12603	4.44	
3.1Pulp		10415	5.24	0.68
3.2Pulp		10473	4.56	
TMPChest_1	PM5 TMP Chest	3435	3.96	0.28
TMPChest_2		3088	4.24	
2.1Pulp		12722	3.99	0.01
2.2Pulp		12755	3.99	

The final results obtained with the automatic analysis of ray cells content and isolation of ray cells methods, were compared and the relative error between both methods was determined. Results are presented below in table 8.

Table 8 - Comparison between automatic analysis of ray cells algorithm results and isolation of ray cells method results.

Sample	Collected point	Automatic Ray Cells, %	Isolated Ray Cells, %	Deviation, %
1Pulp	PM5 Machine	4.30	3.19	1.11
3Pulp	Chest	3.99	3.81	0.18
2Pulp	PM5 TMP Chest	4.90	4.82	0.08

5 Discussion

Concerning the results obtained with the microscopic analysis method, it was verified that this method is not suitable for its purpose. Therefore the hypothesis of doing manual verifications in the microscope and compare this results with FiberLab results for exactly the same sample was rejected. Despite this, the method was important to get familiar with ray cells and other particles' morphology and appearance, making it easier for ray cells' identification in the FiberLab analysis method.

During the image analysis procedure of the kajaaniFiberLab analysis method, several particles were identified as ray cells; however it was possible to find several particles that were similar to ray cells. In figure 16 it is possible to see that, as it was reported before, ray cells usually have a brick-like shape, a small length, a larger cell width. From this picture it is also possible to identify broken ray cells. It was verified that while ray cells tend to be broken or to have open ends, they can also be broken or teared open in the middle of the longer sides. Analyzing the cell surface, not only general shape but also detailed internal structure revealed different color contrasts, where small dark spots can be connected with Piceoid pits, and lighter areas, that can be internal pores, can be linked to resin content. Figure 17 shows some examples of particles that can be taken for ray cells. It is possible so see that while some particles seem to have a brick-like shape, they can simply be small pieces of broken fibres. Fiber positioning when the picture is taken also has some effects on this since they can be twisted and positioned in several angles. It was also verified that even if these particles can have similar length, width and shape; they tend to be fibrillated and curled. Therefore, to identify a ray cell these parameters must be considered altogether. Nevertheless, it sometimes occurs that pieces of broken fibers are identified as ray cells, according to all the parameters. The opposite may also occur, especially with broken ray cells. It is therefore important to obtain define a balance between what should and should not be considered as a ray cell.

It is important to notice that kajaaniFiberLab pictures from the length camera tend to have poor quality, which may affect the accuracy of the manual selection, as well as the precision of FiberLab measurements.

From the results listed in table 1 it is possible to verify that Machine chest and TMP Chest pulps tend to have approximately the same amount of ray cells, as it was expected before. According to theoretical information it was also expected that Standpipe pulps should have a lower content of ray cells, however this was not verified. This difference can be directly related to Standpipe pulp characteristics, since it is a pulp from the beginning of the process,

it has a higher percentage of unbroken fibers and consequently a higher percentage of unbroken ray cells when compared to Machine Chest and TMP Chest pulps. Therefore KajaaniFiberLab will easily detect ray cells and save the respective pictures. Another cause for that difference is related to the method used, as it is a manual method an error is related to the operator criterion. Fines samples results are not reported in table 1, because the purpose was to obtain a higher amount of ray cells morphological information, important for the data analysis procedure. Withal, it was confirmed that fines samples have a higher amount of ray cells, as it was expected before.

Results of the analysis of the particle size distributions for ray cells and non-ray cells particles from all the samples (MC-PM5, TMPchest, TMP84 and MC-PM5_Fines), available in appendix 7 verified that fiber length, width, curl and fibrillation were the most relevant parameters. Results from the present investigation show that ray cells have smaller length than regular fibers and that fiber length is one of the most important parameters. This has also been reported by several authors. According to the results obtained (Appendix 7 - Figure 32) this fact was also confirmed. Fiber width was also shown to be a suitable parameter detection of ray cells. According to fiber width distribution (Appendix 7 - Figure 33) in general ray cells have larger width when compared to other particles, this statement is in agreement with the literature. Wall thickness was not considered because its distribution curves are overlapped (Appendix 7 - Figure 34), meaning that there are no significant differences between ray cells and non-ray cells particles' wall thickness. According to the bibliography it was expected that ray cells would have thicker walls, however this difference can be related to the equipment accuracy for measuring particles with shorter length. Fibrillation and curl indexes have both shown to be good parameters for selecting and characterizing ray cells. As it has been reported in the literature ray cells tend to have no fibrillation and no curl indexes, this was also confirmed from the particle size distributions (Appendix 7 - Figures 35 and 36). Fiber volume and cross section area (CSA) are calculated from other KajaaniFiberLab parameters and are therefore not independent parameters and not considered to be suitable parameters.

Considering ray cells' size distributions for the selected parameters, it was verified that the majority of the ray cells analyzed have a length between 0.2 and 0.5 mm, width between 25 and 50 μm and curl and fibrillation indexes equal to zero. Curl and fibrillation are in agreement with theoretical values. However, length and width have larger values. This can be due to several reasons that can be related to equipment accuracy. KajaaniFiberLab measurements are done with image analysis based on reflectance differences and resolution clearly limits all investigations. Higher effective resolution leads to that smaller particles with a fine structure are detected, which clearly affects the measured distributions. Another reason for this can be related to the fact that in this case some ray cells are broken, and

consequently they tend to have larger dimensions. Apart from this, it is important to notice that the method used to obtain the theoretical values is unknown and therefore its accuracy can also be questionable.

Observing the results obtained from ray cells' isolation method, it is verified that with this method it is possible to isolate ray cells. As it can be seen in figure 11 there is a higher content of ray cells in the isolated fraction. From the results present in table 3 it was verified that samples 1.1, 1.2 and 2.1 have very low ray cells fractions. This is related to losses during Bauer McNett's fines extraction. This happens because fines tend to accumulate near the 635-mesh sieve blocking the water output, and consequently the amount of water in the tank increases and overflows. The problem was controlled with a permanently cleaning of the sieve surface. Regarding other samples it was verified that the values obtained do not differ from what was expected. Even so, there are some differences between same pulp samples, what can be related to the accuracy of the separation. Since the method was developed with the purpose of obtaining an approach of the amount of ray cells in the sample, its accuracy can be questionable, and therefore a higher precision of the results should not be expected. Thereby, in order to minimize the errors, only average results were considered.

Analyzing the results obtained with the automatic analysis of ray cells content method, after the first parameter optimization, it was seen that parameters and numerical limits selected before were suitable for the method purpose. Fibrillation and curl have shown to be essential parameters, as they give a higher contribution to method efficiency. All the different pulp samples analysed have shown similar performances. However for standpipe TMP84 it was found that the optimal numerical limits for length and width differ from the ones obtained for the other samples. These can be related to the difference that exists between standpipe pulp and the other samples.

For the remaining steps no Standpipe pulp was used. This exclusion is not directly related to the differences between pulp samples, but mainly to the aim of this project. Since the project is related to paper printing performance it is desirable to analyse pulp samples collected nearby the paper machine as is the case for TMP chest and machine chest samples, but not for standpipe samples.

A second parameter optimization was performed using version 2 of the algorithm. For this optimization only the width was considered. From the analysis of prior results it was verified that width would be the only parameter with capacity to be improved, as curl and fibrillation have shown to be favourable with numerical limits equal to zero and the length parameter can not have an inferior numerical limit below 0.2 mm due to camera resolution. For the optimization study of the parameter with, only the inferior numerical limit was considered, and it is justified by width distributions of ray cells and non-ray cells (Appendix 7 - Figure 33).

Results give an optimized value for width of, 23.2 μm for TMP chest and 24.5 μm for Machine chest (Appendix 9), a small difference of only 1.3 μm between both pulp types.

This difference can be related to several factors, namely to small differences between both pulp types, because there is a certain amount of fines from the wet end of the paper machine, going back with the water to the machine chest, but it can also be related to sample preparation, due to the manual disintegration process. The optimal numerical limits differ with approximately 1 μm , a level considered to be not very significant and therefore a single value was chosen for both pulp types. Machine chest optimal value, 24.5 μm , was selected, mainly due to the higher importance of studying this pulp type for linting problems, thus it is located immediately before the paper machine. Another factor that contributed to this decision is that this value is higher and is in agreement with the results obtained with the data analysis method.

The new numerical limits are now 0.2 and 0.5 mm for length, 24.5 and 50 μm for width and zero for curl and fibrillation (Table 4). Using exactly the same classification criteria (parameters and limits/conditions) a new calculation is added to the algorithm to determine the amount of ray cells for fibers without an associated picture. The same classification criteria is used because these particles are assumed to be equal of particles with an associated picture for the same length range, the only difference between them is that the picture is not saved because it has poor image quality. Besides that, size distributions of particles with picture and without picture and with length between 0.2 mm and 0.5 mm shown similar behaviour.

A third calculation was included in the algorithm to determine the amount of ray cells for particles smaller than 0.2 mm in length. This is a very important step of the algorithm thus according to theoretical mean values of ray cells' length (≈ 0.09 mm), the fraction of cells smaller than 0.2 mm can be approximately 80 % of the total amount. From the results available in table 5 it is seen that the parameter width is not considered in this calculation, this is due to an equipment limitation as kajaaniFiberLab is unable to calculate fibre width for particles smaller than 0.2 mm in length. Fibrillation and curl kept the same numerical conditions mainly because they represent particular ray cells' characteristics and also because it did not lead to any improvements for other fibrillation and curl values.

The algorithm consists now of three different ray cells' calculations where the amount of ray cells in a pulp sample is determined by the sum of each fraction. Analysing the results obtained for the contribution of each fraction to the total amount of ray cells, it is possible to observe that ray cells 3 (RC3) is the summand that gives the highest contribution, nearly 70 % of the total amount of ray cells, which is in agreement to the estimation of 80 %, obtained from the combination of ray cells length distributions with its theoretical mean

length. Since ray cells are mainly smaller than 0.2 mm in length, the contribution of ray cells 1 and 2 is, low, nearly 30 %, as it was expected. Ray cells 1 (*RC1*) gives a contribution of approximately 23 % while ray cells 2 (*RC2*) contributes with only 7 %. This lower contribution of ray cells 2 is also in agreement with what was expected before, since this fraction of ray cells is calculated from the smallest fraction of the entire data set. These differences between *RC2* and *RC3* also contribute to the minimization of the deviations between pulp types verified during the parameter optimization.

Analyzing the results obtained, for the amount of ray cells, with the final version of the algorithm (Table 7) it is verified that these results are in general in agreement with the theoretical values of ray cells fraction for Norway spruce (4.7 %), since they differ less than 1 %. However this difference is not considered significant as the precision of the theoretical value is questionable, because there is no information available regarding the experimental procedure used for its determination. The results obtained for the same pulp sample shows a good accuracy of the method. These small differences can be related to FiberLab accuracy for fibers/fines detection and to sample preparation. Therefore to determine the amount of ray cells in a pulp sample it is recommended to do at least a double analysis and calculate its average. It was also verified that in general Machine chest samples tend to have a larger amount of ray cells when compared to TMP chest samples, this can be explained with what happens in the process, thus there is an addition of fines, coming from the wet end of the paper machine, in the machine chest.

The average results obtained with both methods, isolation of ray cells and automatic analysis of ray cells content, have shown to be similar for the same pulp sample. As it is possible to see in table 8 for samples 2pulp and 3pulp the deviation between both methods is almost zero. Although sample 1pulp has a larger deviation, where both methods results differ nearly 1 %, this is probability related to the precision of isolation of ray cells method's result. Since, it is a single sample value, because as it was mentioned before the other samples were rejected due to problems during the fines extraction process. Therefore the comparison between both methods has shown that the automatic analysis of ray cells content method gives a good estimative of the amount of ray cells.

Analyzing the method developed for ray cells' content automatic analysis, several advantages were verified, since it is a novel method able to determine the amount of ray cells directly from pulp samples. It is necessarily less than 30 min since the sample is collected until the results are obtained. The method only needs equipment that is already available at the company and the sample preparation is very simple. Another advantage is that the method was specifically designed for detecting ray cells in Thermomechanical pulps of Norway spruce.

6 Conclusions

The goal of this project was to develop a method suitable for analysis of the ray cells content in TMP of Norway spruce. Ray cells are known for being the main reason for linting during the printing process. Therefore its prediction is an important part for quality control in the pulp and paper industry.

The first approach of the entire project was the microscopic analysis method. This method gives low and not-well known reliability, since it is not possible to verify the results with the kajaaniFiberLab. This method is also time consuming and requires an experienced operator, nevertheless it was important to get familiar with ray cells.

The kajaaniFiberLab method can give detailed information about ray cells morphological properties. It was observed that kajaaniFiberLab pictures from the length camera tend to be of poor quality, which may affect the accuracy of the measurements. Withal, FiberLab is still one of the best options for fiber analysis, since it is able to analyze, in a short time, individual fiber properties directly from a pulp sample. Concerning ray cells information obtained with this method it was observed that the most important morphological parameters for its classification were the fiber length, fiber width, curl and fibrillation.

Regarding the ray cells' isolation method, it was verified that it is possible to isolate ray cells from a pulp sample and determine an estimation of its percentage in the sample. However, in order to minimize the errors, a method optimization is recommended, by the optimization of the materials used and the settling times. It is also recommend a permanent control during the fines extraction process with the Bauer McNett, since lost of is possible.

The automatic analysis of ray cells content by use of the kajaaniFiberLab instrument is the main product of this thesis. A novel method able to determine the amount of ray cells from a pulp sample was developed. The method's input is kajaaniFiberLab's individual fiber results, and the algorithm is based on a decision tree structure to classify the fibers into ray cells and non-ray cells. The results obtained have shown to be accurate and have confirmed the reliability of the method. An advantage of the method is that the measurement accuracy is independent of the circumstances, meaning that it is possible to compare samples analyzed in deferent days. It is a fast method, easy to implement, and it was specially designed for ray cells analysis in TMP of Norway spruce. Another advantage is that it does not have any limitation for the amount of ray cells that can be calculated, so it can be used for analysis of different types of samples. Depending on the sample used the method accuracy can vary, therefore a priori verification is recommended.

7 Project Evaluation

This chapter presents a brief personal evaluation of the project together with some recommendations for future work.

7.1 Goals Accomplished

In the beginning of the project it was purposed by the company to development a method for analysis of ray cells content in thermomechanical pulp of spruce, it was also required that it should be based on equipment available at the company, such as the online instrument PulpEye or the laboratory kajaaniFiberLab. The method developed fulfils these criteria's since it provides a method to determine the amount of ray cells in a pulp sample. Therefore it is considered that the initial goals of this project were fully accomplished.

7.2 Parallel work

It was concluded that the method developed can be applied online. However, with the equipment presently available at the mill it is impossible to utilize this method for online determination of the amount of ray cells. This limitation is not directly related to the algorithm developed, it is related to the equipment used in the mill to access fiber information of the pulp. Since the equipment used for online determination at the mill, designated as PulpEye, only gives the average information of the sample analyzed and the developed algorithm has as input individual fiber information. Nevertheless if it will be possible to obtain individual fiber information online, regarding fiber length, width, curl and fibrillation it will be possible to do an online implementation of this method. A possible solution could be the combination of PulpEye ability to perform online pulp analysis with kajaaniFiberLab capacity to determine individual fibers' morphological properties.

Some samples were sent to be analyzed in a new equipment that is being developed by Metso Automation, however no useful results were obtained from it.

A VBA (Visual Basic for Applications) application of the algorithm and a user manual were also written, although these will not be included in the report, since it is to be exclusively used within the company.

7.3 Recommendations for future work

Since this is a novel method several recommendations for further work can be given.

First of all, it will be interesting to measure several pulp samples and relate the results obtained, with costumers' feedback concerning linting problems during the printing process. With a long term analysis it may be possible to obtain a correlation between pulp ray cells' fraction and linting, which may contribute to an easier prediction of pulp linting propensity at the mill.

It is also suggested to analyze lint particles collected at the print machine and do a deeper analysis of its morphological properties. It is recommended doing the analysis with kajaaniFiberLab, since the measurement principle is the same and therefore the results can be combined with the results obtained during this project. From the results obtained with this analysis it would be interesting to verify ray cells' differences between both samples. Since, the contribution of ray cells with length larger than 0.2 mm to the total fraction is very low it is recommended to investigate if its fraction in the lint sample is significant, or if it is sufficient to consider only ray cells 3 results, in order to determine the total amount of ray cells in the sample.

This method has proven to be suitable to obtain an approximation of the ray cells fraction of a pulp sample. However, if depth studies with higher precision are required; it is recommended to replace or modify kajaaniFiberLab with an equipment designed for fines analysis. Increasing kajaaniFiberLab length camera resolution and modifying its software in order to save pictures from the second camera in a proper way so that they can be directly linked with the fiber measurement data, is also possible option.

Regarding ray cells' isolation method recommendations for future work, two options must be considered. In order to use the actual method it is necessary to provide material and settling times optimization, even so this method is only suitable for laboratory usage, and has the drawback that the separation takes several hours up to days. Therefore it is recommended considering other separation methods, such as flotation or the usage of hydrocyclones.

7.4 Global Appreciation

As well as many other projects, this project started almost from zero, where the first question that I asked myself was "What is ray cell?". This question was followed by many other questions, in a process of understanding new concepts and getting familiar with new equipment. After these five months, looking to the results obtained and knowing that they will be useful for the company, it can only result in a good appreciation of the work done.

Apart from the results obtained, I can affirm that the circumstances where this project was developed together with its own development contribute to the development of my soft and technical skills.

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Appendix 1 Samples

1.1 Pulp samples

Table 9- Pulp samples details.

Sample Identification	Collect point	Collect date
MC-PM5	PM5 Machine Chest	30-9-2010
TMPchest	PM5 TMP Chest	30-9-2010
TMP84	Standpipe TMP84	30-9-2010
1Pulp	PM5 Machine Chest	17-10-2010
2Pulp	PM5 TMP Chest	15-11-2010
3Pulp	PM5 Machine Chest	15-11-2010

Samples identification according to the following nomenclature: MC-PM5_*i*, TMPchest_*i* and 1.*i* Pulp referees to samples collected from the respective pulp sample, where *i* can vary from 1 to the number of samples collected. Fines samples are identified by the word Fines after the pulp identification (MC-PM5-Fines_*i*)

1.2 Isolation of Ray cells samples

Table 10 - Samples used for ray cells' isolation.

Samples Identification	Pulp Sample	Consistency / %	Mass of wet pulp added to Bauer McNett / g
1.1	1Pulp	3.3	336
1.2			311
1.3			310
2.1	2Pulp	3.8	285
2.2			268
2.3			267
3.1	3Pulp	3.4	310
3.2			304

Appendix 2 Pulp Consistency Determination

Consistency is one of the most common parameter of pulp analysis, thus it is needed for almost all pulp analysis methods. Pulp consistency or, more properly, concentration is defined as the percent in weight of oven-dry fiber in a pulp-water mixture. This procedure is based on Tappi T240 om-93 test method.

Procedure used for pulp consistency determination for thermomechanical pulp samples:

1. In a bucket, manual disintegrate the pulp and mix it, in order to have a homogenous water distribution in the sample;
2. Weight 100-400 g of pulp, m_{wet0} , in a 500 mL tared bucket and dilute it with water;
3. Place a preciously dried, tared filter paper in the Büchner funnel, moisten with water, then apply suction to the funnel and filter the sample. Clean the bucket with water in order to avoid losing fibers;
4. Remove the resulting pad and filter paper, cover the pad with a pre-weighted dried filter paper and place it in a press dryer until it ceases to steam;
5. Wight the pad and the filter papers, m_{total} and calculate the amount of dry pulp, m_{dry0} with equation A2.1;

$$m_{dry0} = m_{total} - (m_{paper1} + m_{paper2}) \quad (A2.1)$$

6. The percent consistency, c of the sample is then calculated with equation A2.2.

$$c = \frac{m_{dry0}}{m_{wet0}} \times 100 \quad (A2.2)$$

Appendix 3 Bauer McNett apparatus

The Bauer McNett is a laboratory device used to assess fiber size distribution. It consists of a series of tanks, typically four or five mounted in cascade and each tank has a progressively smaller screen mesh. At the beginning of the process, a prescribed amount of pulp is poured into the first tank, then a continuous water flow passes through the tank and the followed serie of tanks and an agitated pulp suspension is fractionated according to fiber/particle size. At the end of the process the desired fractions are collected at the bottom of the respective tank. Pictures from the equipment are shown in figure 21.



Figure 21 - Bauer McNett classifier used.

3.1 Bauer McNett procedure for fines isolation

This procedure follows SCAN-CM 6:05 test method for fibre fractionation in the Bauer McNett classifier.

1. Manual disintegration of Pulp;
2. Calculate pulp consistency following procedures in appendix 2;
3. Weight a sample of pulp in order to have 10 g of dry pulp;
4. In a bucket dilute the sample with water until a concentration of 3-4 g dry pulp/L;
5. Check that the water flow is (10.0 ± 0.2) L/min;
6. Check that the agitators rotate at (580 ± 25) rpm;
7. Add 30, 50, 100, 200 and 635-mesh to tanks 1, 2, 3, 4 and 5 respectively;
8. Close all the tanks and fill them with water;
9. Add a cup with filter paper at the bottom of tank number 5;
10. When the tanks are full with water, turn the engine on and add the sample to tank number 1;
11. After 15 min turn the engine off and open all the tanks;
12. When tank number 5 is empty, open the cup and collect the sample with water to a bucket.

The collected sample comprises all the fibers that passes through 200-mesh and are retained at 635-mesh (P200/R635), this fibers are designated as fines.

Appendix 4 kajaaniFiberLab apparatus

KajaaniFierLab is a fast and easy to use fiber analyzer developed by Metso Automation Corporation, Finland.

The equipment measures individual fibers with two cameras at 10 μm and 1.5 μm resolutions, with sensitivity of 10 μm and 0.1 μm respectively. Measurement results are available as averages, distributions, fractions and individual fiber results. Fiber length, fiber width, curl, coarseness, fines content, cell wall thickness, cross sectional area, fiber volume and fiber fibrillation are some of the measurements reported by kajaaniFiberLab.



Figure 22 -kajaaniFiberLab equal to the used one (Metso Automation, 2010)).

4.1 Measurement Principle

A beaker containing the sample is placed in the sample unit. After the start command the analyzer mixes and dilutes the sample to the correct consistency for measurement. The sample is then sucked in, so that it flows through the capillary. Length measurement optics (with Laser and Xenon light sources) controls the fiber measurements that are carried out by means of image processing technology. Length measurement optics (laser) detects the presence of a fiber and monitors its passage through the capillary. When the fiber reaches the center of the optics section, the Xenon lamp flashes. The fiber length and cross-sectional cameras simultaneously capture an image of the same fiber (Figure 23)(Oikarinen and Rakkolainen, 2002).

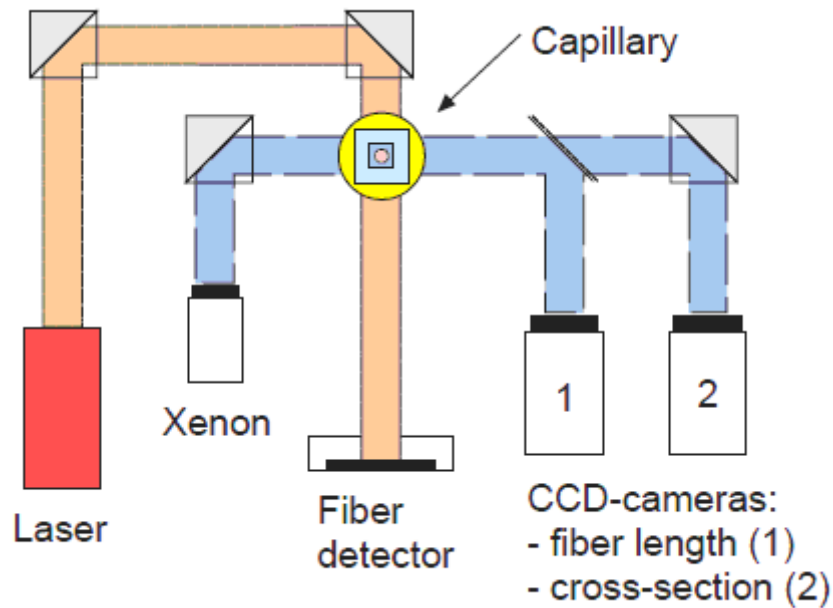


Figure 23 - Measurement principle of kajaaniFiberLab (Oikarinen and Rakkolainen, 2002).

Table 11 shows the measurement performance information of both length and cross-section cameras.

Table 11 - KajaaniFiberLab performance (Oikarinen and Rakkolainen, 2002).

Camera	Measurement range	Resolution	Sensitivity
Length	0.01-7.60 mm (length)	10 μm	< 0.1 μm
Cross-section	1-500 μm (width)	1.5 μm	

The fiber image from the length measurement camera can be captured in different formats such as: normal fiber image (online image), difference image (=reference image-normal fiber image), binary fiber image and reference image. Examples of normal fiber images from length and cross-section cameras are shown in figures 24 and 25 respectively.

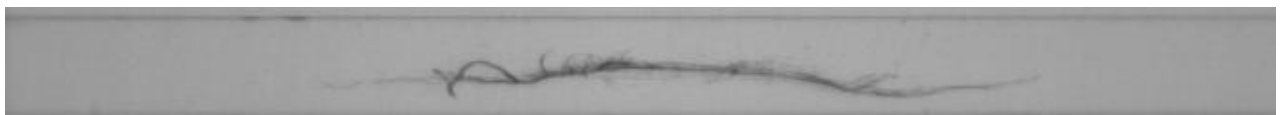


Figure 24 - Normal fiber image from kajaaniFiberLab length camera.



Figure 25 - Normal fiber image from kajaaniFiberLab cross-section camera.

Both camera images are then processed by FiberLab software and the following results are calculated: fiber length, projected fiber length, fiber width, cell wall thickness, curl, fibrillation, cross-sectional area and fiber volume index.

The fiber length, L_c , is the length along the fibre while the projected length, L_p , is the maximum linear distance between fibre ends. During this project all references made to length are related to fiber length, L_c (Figure 26).

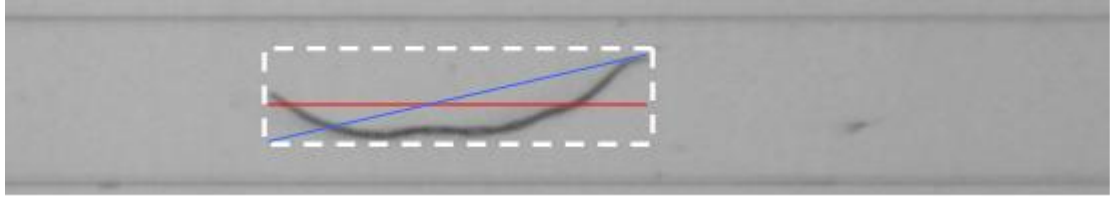


Figure 26 - Scheme of fiber length, L_c (red line) and projected length, L_p (blue line)

4.1.1 Calculation formulas

a) Fiber length measurement

Average length in class i ($i=1... 152$)

$$l_i = (0.05 * i) - 0.025 \quad [\text{mm}] \quad (\text{A4.1})$$

Arithmetic average fiber length (n_i = number of fibers in class i)

$$L(n) = \frac{\sum (n_i * l_i)}{\sum n_i} \quad [\text{mm}] \quad (\text{A4.2})$$

Length-weighted average fiber length

$$L(l) = \frac{\sum (n_i * l_i^2)}{\sum n_i l_i} \quad [\text{mm}] \quad (\text{A4.3})$$

Weight-weighted average fiber length

$$L(w) = \frac{\sum (n_i * l_i^3)}{\sum n_i l_i^2} \quad [\text{mm}] \quad (\text{A4.4})$$

b) Cross-sectional measurements

Average fiber width of width class i ($i=1...100$), where w_i = width, kw = width calibration factor

$$w_i = kw * (i - 0.5) \quad [\mu\text{m}] \quad (\text{A4.5})$$

Average fiber width ($i=1...100$)

$$Aw = \frac{\sum (n_i * w_i)}{\sum n_i} \quad [\mu\text{m}] \quad (\text{A4.6})$$

Average fiber width of cell wall thickness class i ($i=1...100$), where CWT_i = cell wall thickness, and kt = cell wall thickness calibration factor.

$$CWT_i = kt * (i - 0.5) \quad [\mu\text{m}] \quad (\text{A4.7})$$

Average cell wall thickness ($i=1...100$)

$$ACWT = \frac{\sum (n_i * CWT_i)}{\sum n_i} \quad [\mu\text{m}] \quad (\text{A4.8})$$

Average cross-sectional area in area class i ($i=1...100$), where CSA_i = cross-sectional area

$$CSA_i = \pi * (CWT_i * (w_i - CWT_i)) \quad [\mu\text{m}^2] \quad (\text{A4.9})$$

Average cross-sectional area ($i = 1...100$)

$$CSA = \frac{\sum (n_i * CSA_i)}{\sum n_i} \quad [\mu m^2] \quad (A4.10)$$

Average fiber volume in volume class i ($i = 1...100$), where V_i = fiber volume and L_c =true length of fiber (along the centre line)

$$V_i = CSA_i * L_c(n)_i \quad [10^6 \mu m^3] \quad (A4.11)$$

Average fiber volume ($i = 1...100$)

$$V = \frac{\sum (n_i * V_i)}{\sum n_i} \quad [\mu m^3] \quad (A4.12)$$

c) Curl measurement

Fiber curl ($i = 0...100$), where $CURL_i$ = curl of fiber, L_c =true length of fiber (along the centre line), L_p =projected length of fiber (linear measurement)

$$CURL_i = \left(\frac{L_c}{L_p} - 1 \right) * 100 \quad [\%] \quad (A4.13)$$

Average curl ($i = 1...100$), where $CURL$ = average fiber curl

$$CURL = \frac{\sum (n_i * CURL_i)}{\sum n_i} \quad [\%] \quad (A4.14)$$

An example of individual fiber measurement result is shown in table 12.

Table 12 - kajaaniFiberLab individual fiber results example.

Photo	Lp	Lc	Width	Wall	Curl	CSA	Volume	Vessel	Kink k1	Kink k2	Kink k3	Kink k4	Max SS	Ave SS	Fibrillation	File
176	0.74	1.17	58.432	25.56	58	2639.56	3.09	0	0	0	0	0	0	0	7.14	sieve1_00194.tif
179	1.23	1.34	25.199	0	8	0	0	0	0	0	0	0	0	0	6.03	
180	2.24	3.61	26.192	0	61	0	0	0	0	0	0	0	0	0	11.38	
182	1.07	1.12	24.818	0	4	0	0	0	0	0	0	0	0	0	4.51	
194	1.22	1.51	15.694	4.26	23	153.02	0.23	0	0	0	0	0	0	0	2.96	
197	0.64	0.81	36.432	14.2	26	991.8	0.8	0	0	0	0	0	0	0	10.66	sieve1_00218.tif
201	0.41	0.5	16.62	0	21	0	0	0	0	0	0	0	0	0	20.05	
203	0.41	0.45	9.14	0	9	0	0	0	0	0	0	0	0	0	0	
208	0.48	0.51	19.592	0	6	0	0	0	0	0	0	0	0	0	3.97	
214	0.44	0.5	11.297	0	13	0	0	0	0	0	0	0	0	0	3.54	
218	1.82	2.59	36.31	7.1	42	651.54	1.69	0	0	0	0	0	0	0	19.35	sieve1_00232.tif
220	0.2	0.36	7.876	0	80	0	0	0	0	0	0	0	0	0	0	
221	0.26	0.27	24.401	0	3	0	0	0	0	0	0	0	0	0	0	
232	1.39	1.41	35.206	6.39	1	578.48	0.82	0	0	0	0	0	0	0	2.83	
233	2.47	2.55	45.08	3.55	3	463.17	1.18	0	0	0	0	0	0	0	3.19	sieve1_00233.tif
234	0.48	0.9	23.012	0.71	87	49.74	0.04	0	0	0	0	0	0	0	16.44	sieve1_00234.tif
238	0.3	0.39	8.634	0	29	0	0	0	0	0	0	0	0	0	0	sieve1_00241.tif
241	0.69	0.78	21.56	2.84	13	167.02	0.13	0	0	0	0	0	0	0	7.01	
243	1.35	1.66	29.467	14.2	22	681.08	1.13	0	0	0	0	0	0	0	2.57	
245	0.22	0.23	38.192	1.42	4	164.04	0.04	0	0	0	0	0	0	0	0	sieve1_00245.tif
247	0.83	0.94	16.743	2.84	13	124.05	0.12	0	0	0	0	0	0	0	1.96	sieve1_00247.tif
248	1.45	1.56	63.653	2.13	7	411.69	0.64	0	0	0	0	0	0	0	7.58	sieve1_00258.tif
255	0.03	0.03	0	0	7	0	0	0	0	0	0	0	0	0	0	
258	1.2	1.24	34.277	2.84	3	280.48	0.35	0	0	0	0	0	0	0	2.95	
259	0.69	0.72	26.796	12.78	4	562.76	0.41	0	0	0	0	0	0	0	2.99	
261	0.12	0.12	0	0	4	0	0	0	0	0	0	0	0	0	0	
262	1.54	1.63	27.55	2.84	5	220.47	0.36	0	0	0	0	0	0	0	2.88	sieve1_00262.tif
263	0.19	0.24	11.364	0	26	0	0	0	0	0	0	0	0	0	0	sieve1_00265.tif
265	0.62	0.68	22.925	7.1	9	352.99	0.24	0	0	0	0	0	0	0	5	
266	0.24	0.46	21.438	0	91	0	0	0	0	0	0	0	0	0	0	

4.2 Measurement Procedure

4.2.1 Sample preparation

According to kajaaniFiberLab user manual wet samples must be diluted in order to obtain a fiber count of 90 fibers per second. For thermomechanical pulp of spruce this corresponds to a solution with a concentration of 0.1 g of dry pulp/L.

The amount of wet pulp need to prepare a solution with a concentration of 0.1 g of dry pulp/L was determined according to appendix 2. The weighted pulp was then disintegrated using a manual disintegrator delivered (Figure 27).

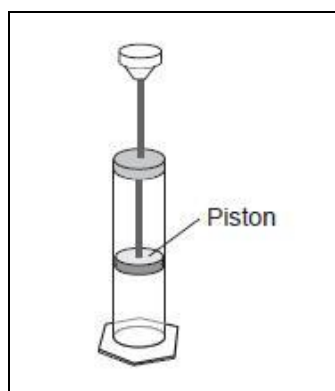


Figure 27 - Manual disintegrator (Oikarinen and Rakkolainen, 2002)

After the disintegration process, the samples were diluted with water until the desired concentration and samples of 50 mL were collected into the analyzer cups, delivered with the equipment.

4.2.2 Equipment verification

The equipment condition is verified by analyzing control fibers (Rayon fibers) and it is recommended to do it regularly.

In this case the verifications were done with kajaani Rayon fibers number 2 and 3, and for all the analysis taken the results were acceptable.

4.2.3 Measurement instructions

Place the cups in kajaaniFiberLab rotate disc and specify the number of samples and sample identification in FiberLab 4 software by doing File -> Open Sample Set -> Default. Double click on the sample name and remove options “Short measurement” and “Blockage remove”.

In the main window go to Setting->Sieve Analysis and set 1 for save (saving all the pictures), for length set 0 and 7, for width set 0 and 50 and for wall set 0 and 10. Finally go to File and choose Save Sample Set, after that click start to initiate the measurement.

Appendix 5 Linear correlation between DDJ and Bauer McNett

Linear correlation between fines content of DDJ and Bauer McNett apparatus.

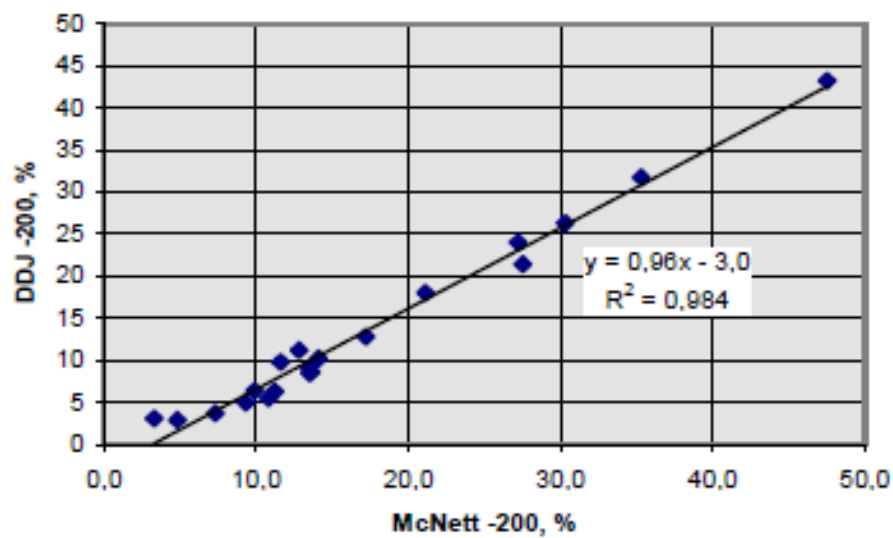


Figure 28 - Linear correlation between DDJ and Bauer McNett fines content
(Krogerus et al., 2002a)

Appendix 6 Automatic quantification of ray cells algorithm versions diagrams

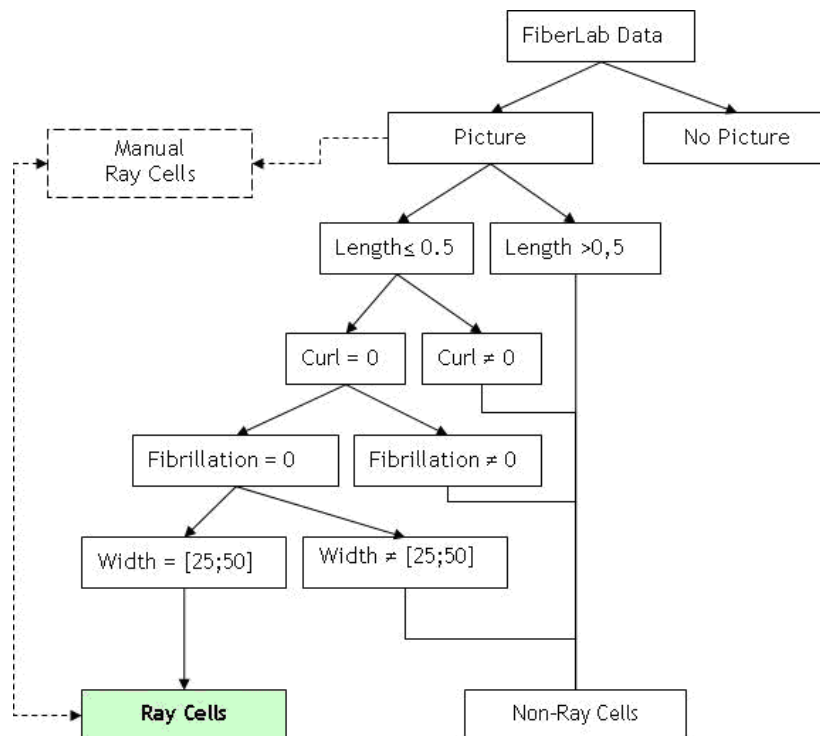


Figure 29 - Version 1 of the automatic ray cells quantification algorithm.

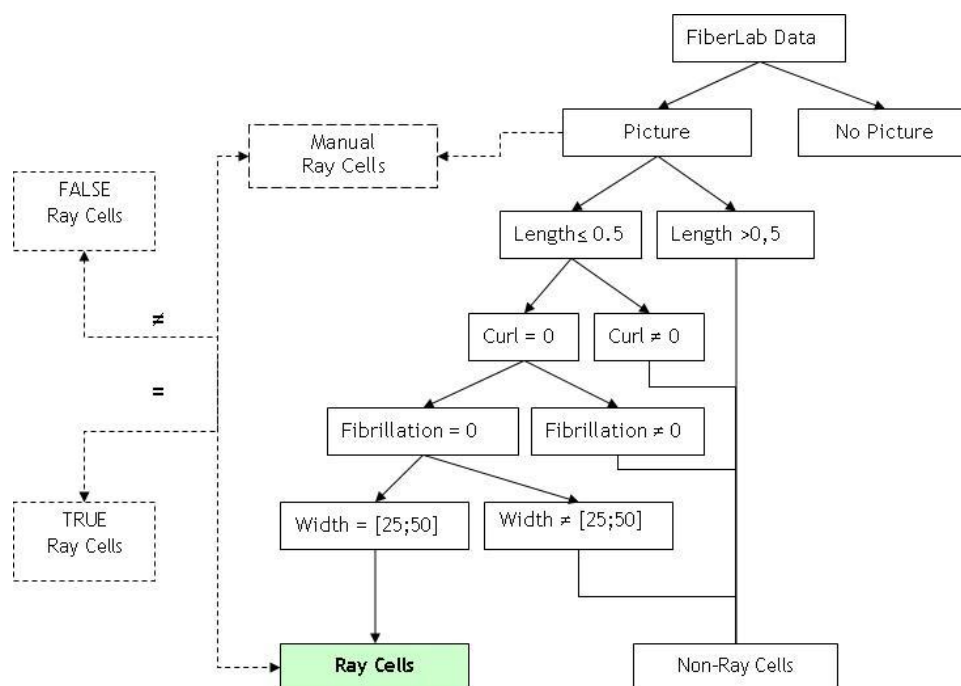


Figure 30 - Version 2 of the automatic ray cells quantification algorithm.

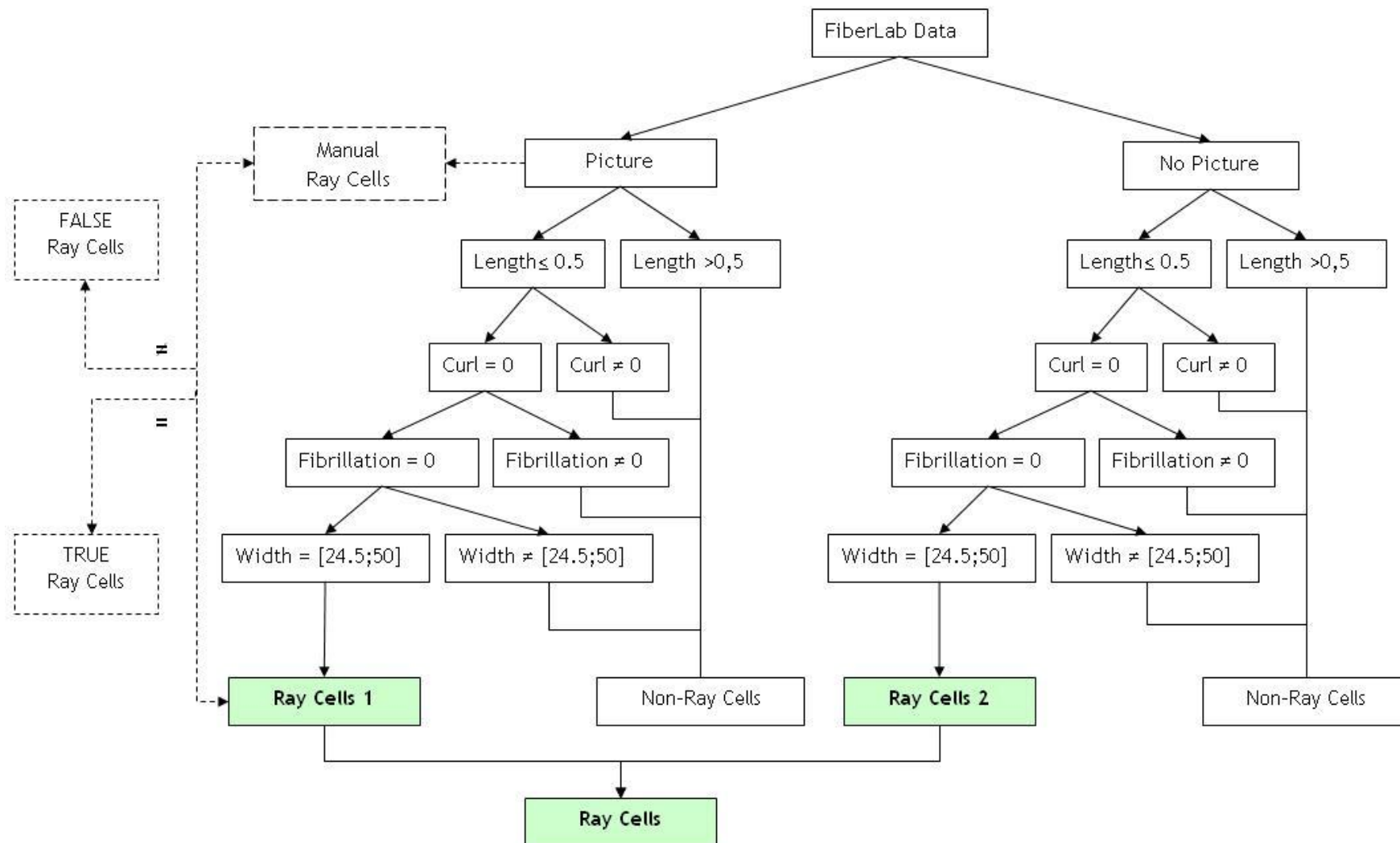


Figure 31 - Version 3 of the automatic ray cells quantification algorithm.

Appendix 7 Ray cells and non-ray cells particle size distributions

Ray cells and non-ray cells normal distributions and cumulative probability for all the morphological parameters.

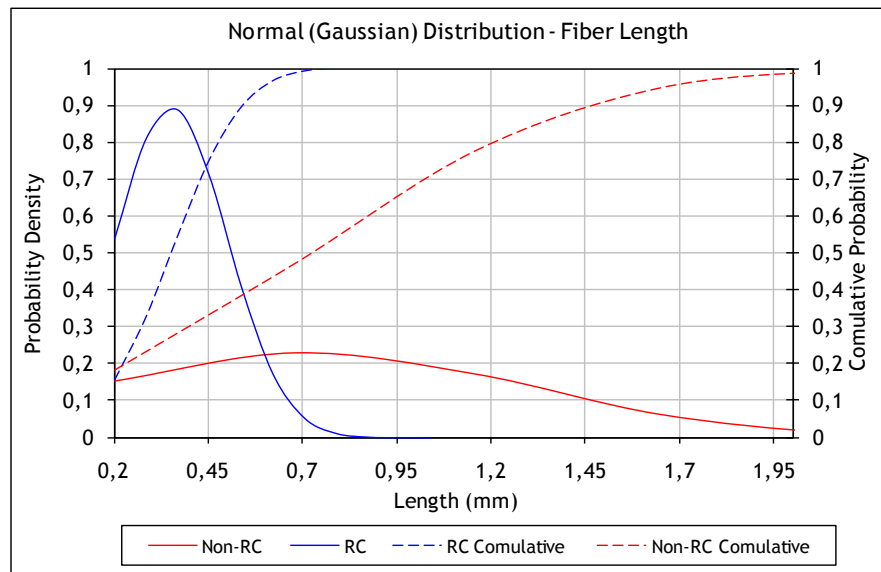


Figure 32 - Ray cells and non-ray cells normal distributions and cumulative probability for fiber length.

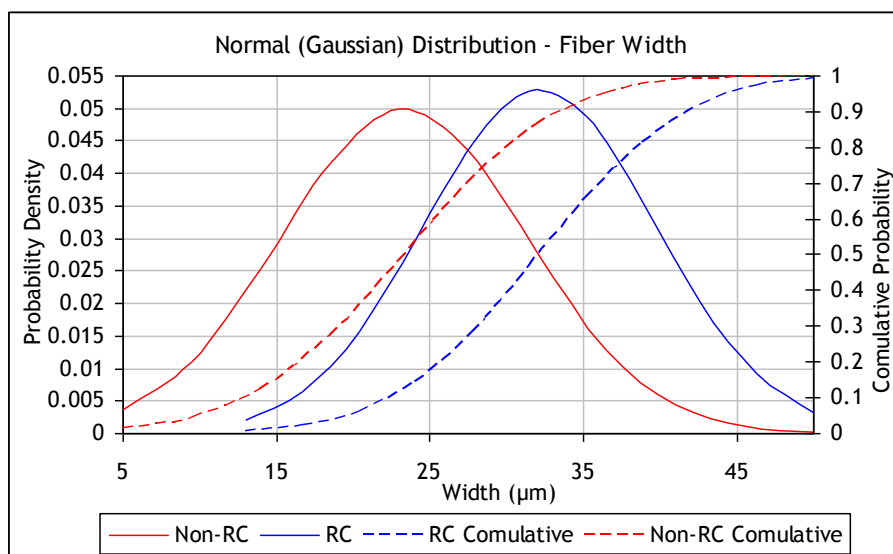


Figure 33 - Ray cells and non-ray cells normal distributions and cumulative probability for fiber width.

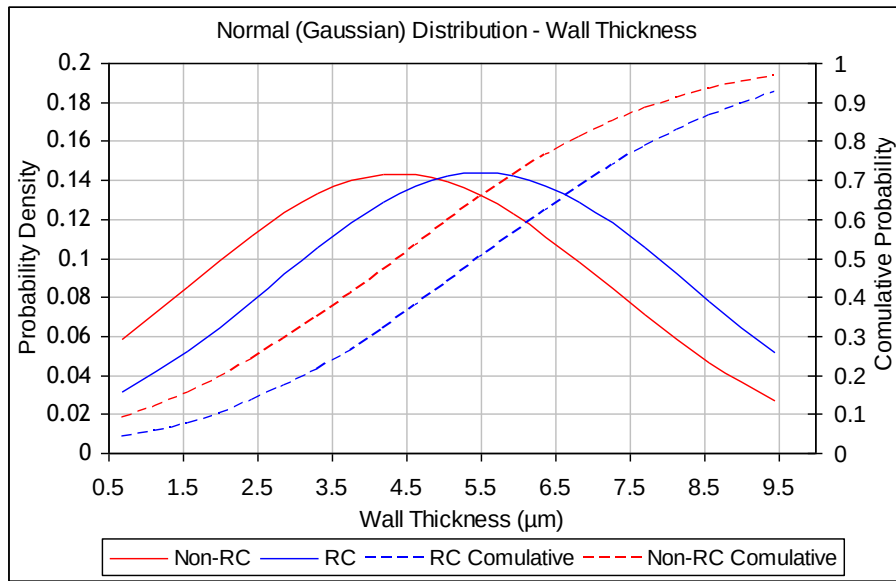


Figure 34 - Ray cells and non-ray cells normal distributions and cumulative probability for fiber wall thickness.

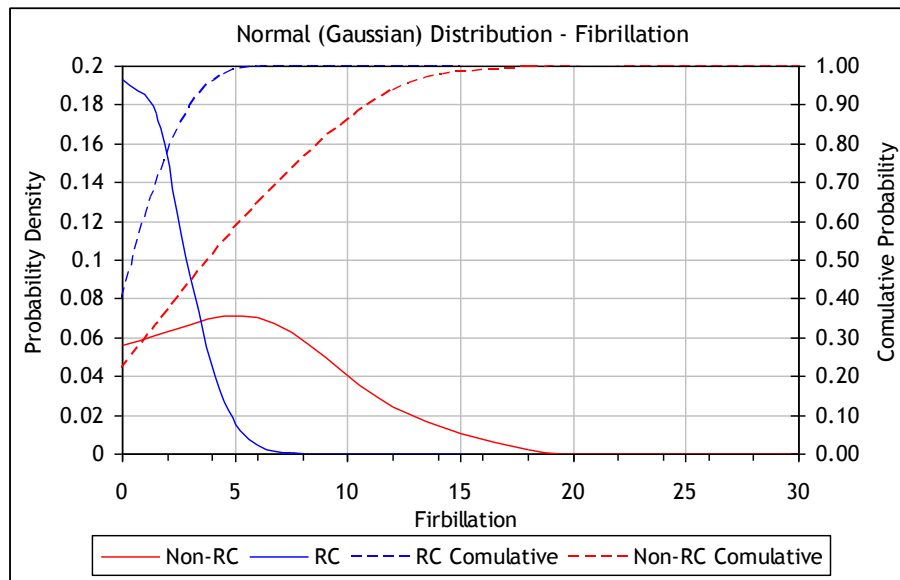


Figure 35 - Ray cells and non-ray cells normal distributions and cumulative probability for fiber fibrillation index.

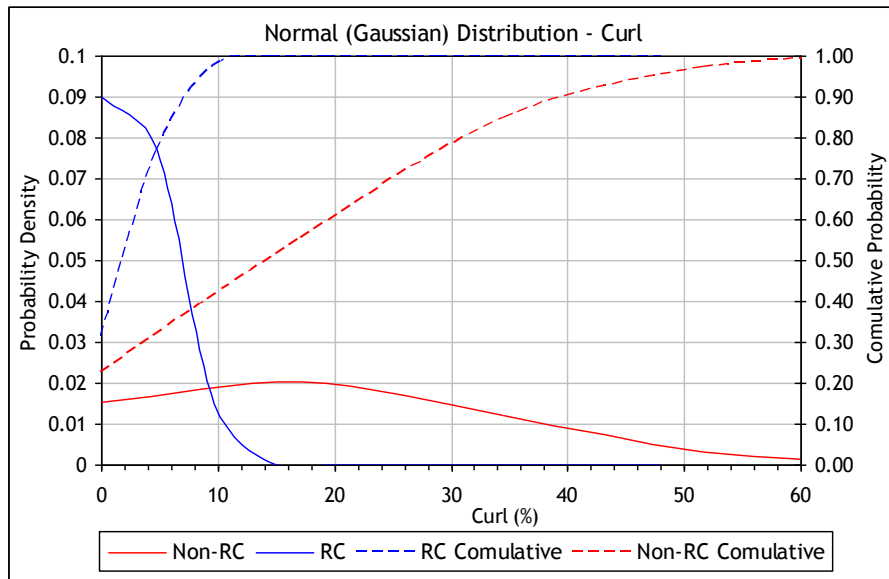


Figure 36 - Ray cells and non-ray cells normal distributions and cumulative probability for fiber curl.

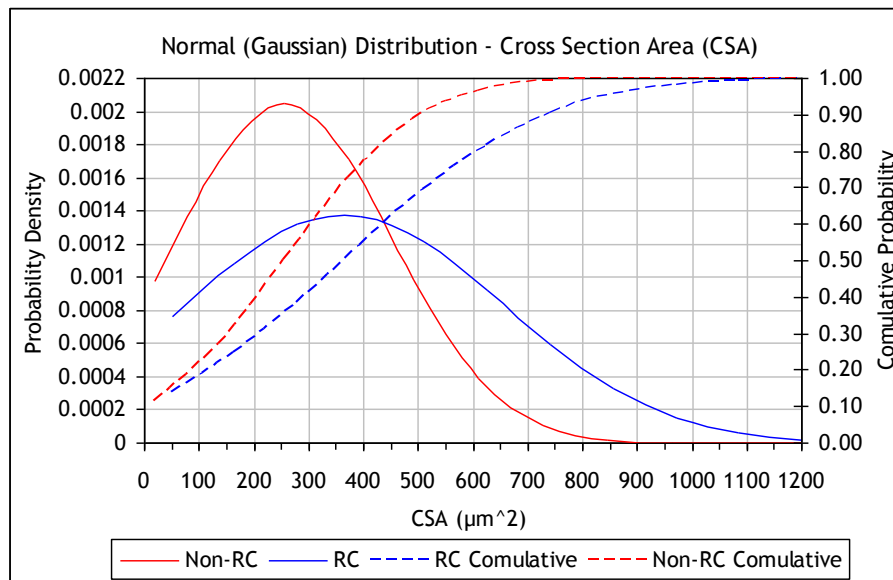


Figure 37 - Ray cells and non-ray cells normal distributions and cumulative probability for fiber cross section area.

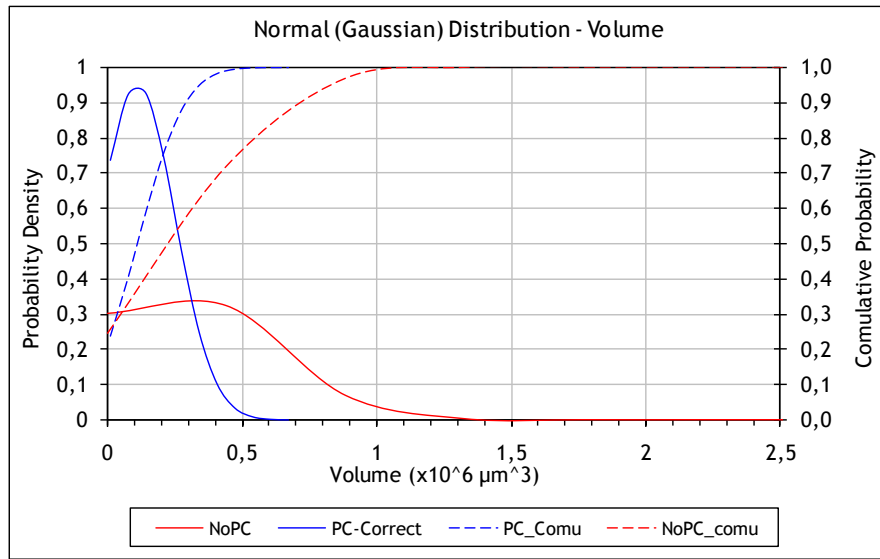


Figure 38 - Ray cells and non-ray cells normal distributions and cumulative probability for fiber volume.

Appendix 8 Parameter optimization of version 1 of the algorithm.

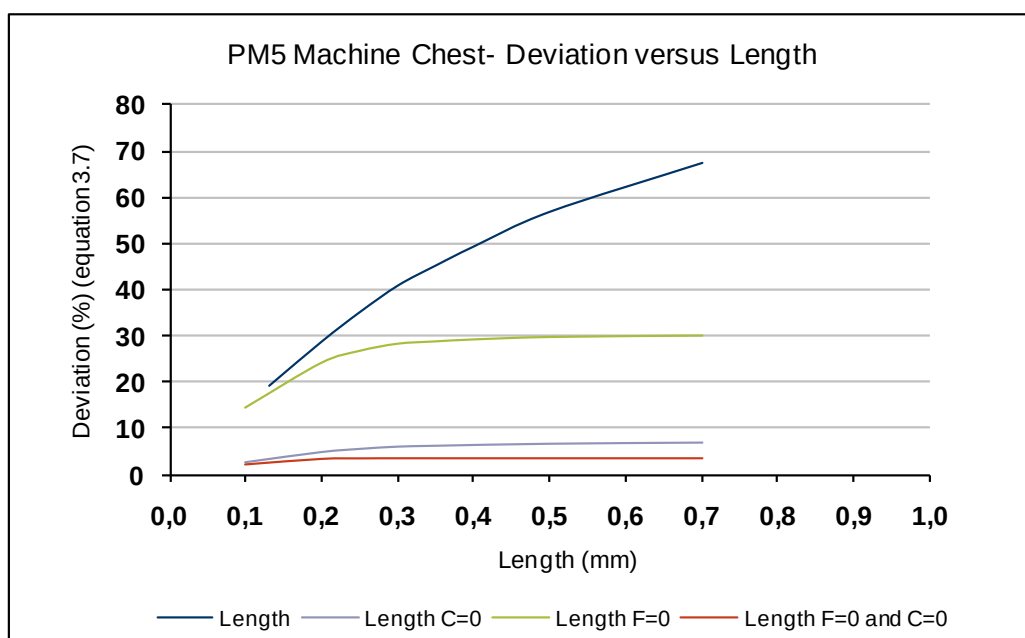


Figure 39 - Length optimization of version 1 of the algorithm for Machine Chest samples.

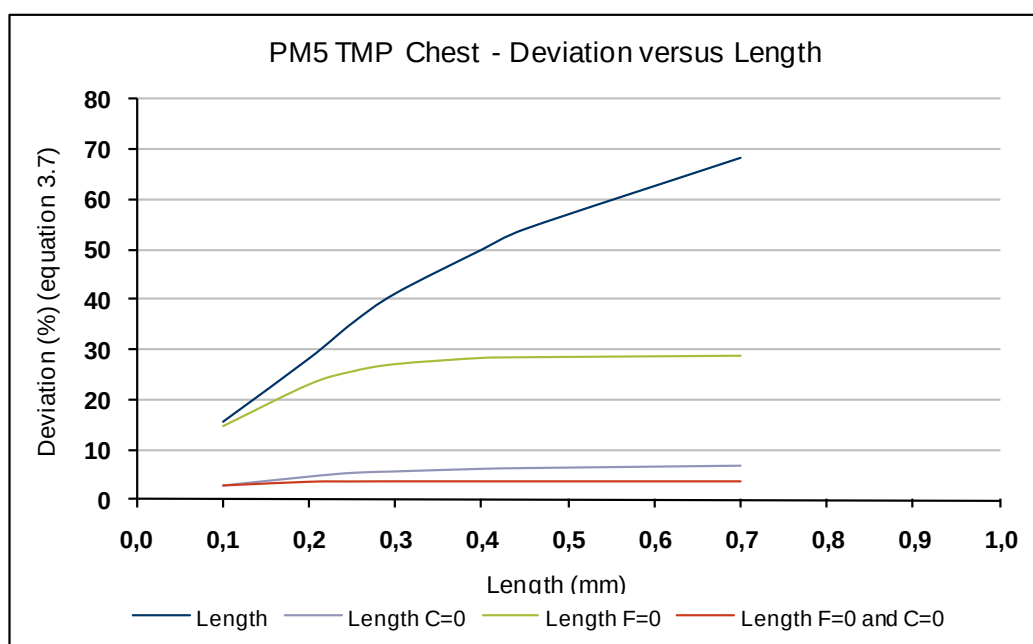


Figure 40 - Length optimization of version 1 of the algorithm for TMP Chest samples.

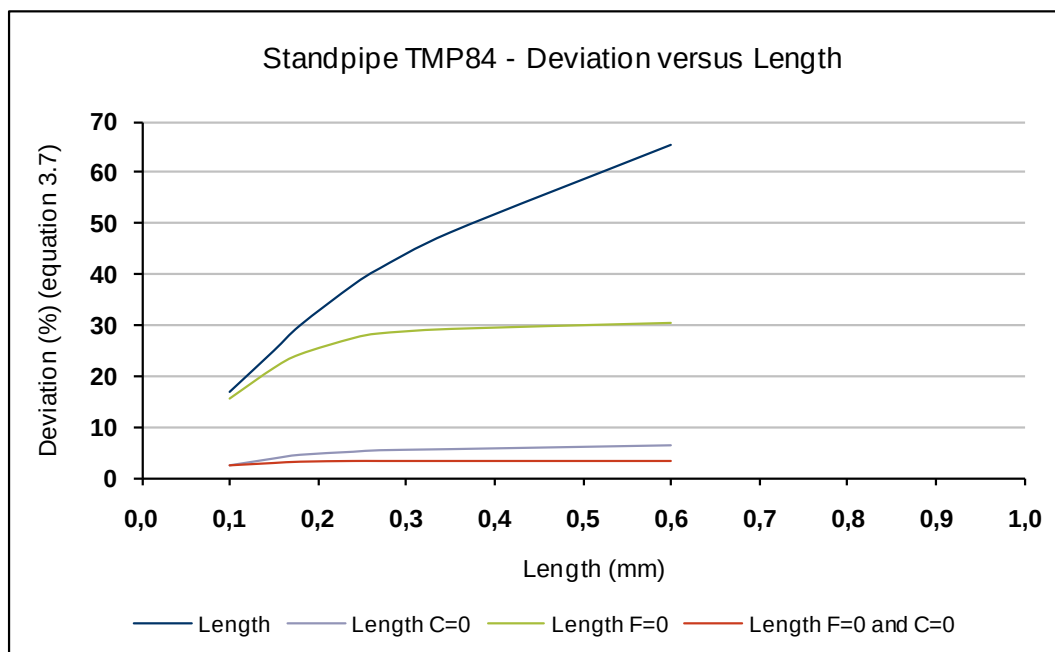


Figure 41 - Length optimization of version 1 of the algorithm for Standpipe TMP84 samples.

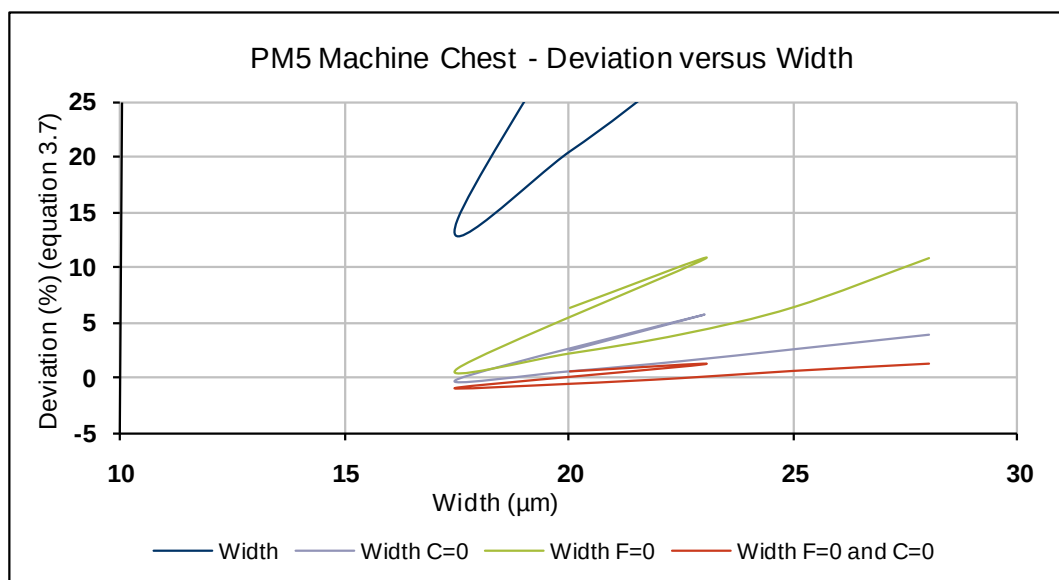


Figure 42 - Width optimization of version 1 of the algorithm for Machine Chest samples.

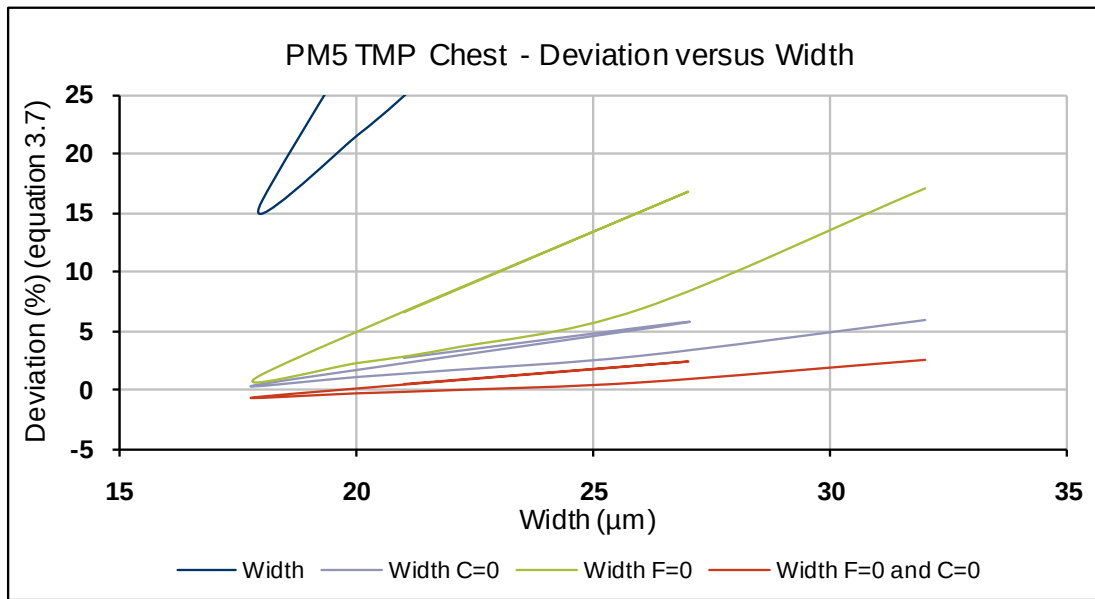


Figure 43 - Width optimization of version 1 of the algorithm for TMP Chest samples.

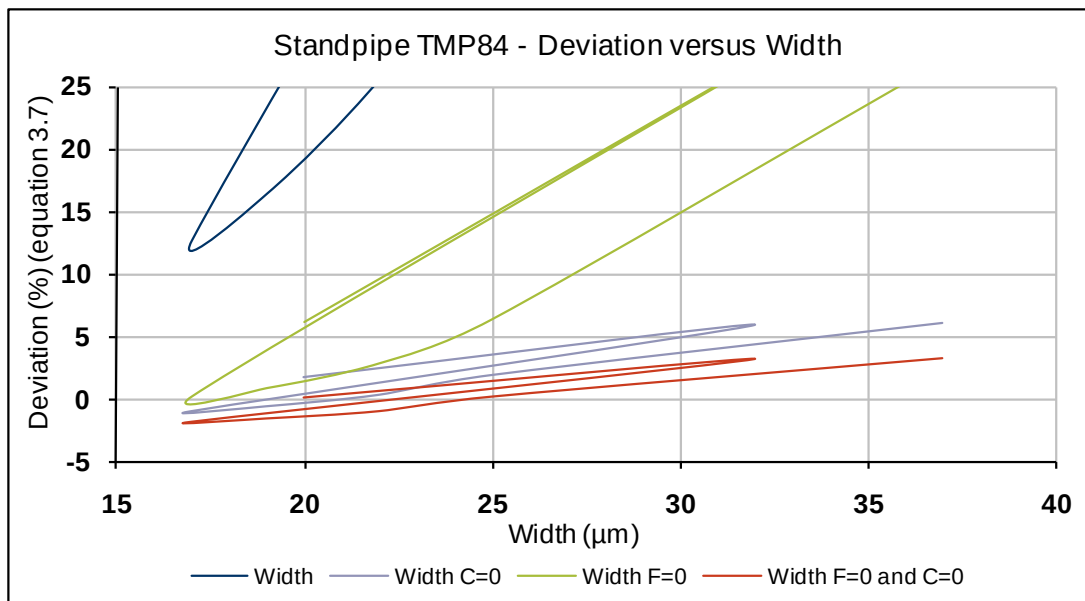


Figure 44 - Width optimization of version 1 of the algorithm for Standpipe TMP84 samples.

Appendix 9 Parameter optimization of version 2 of the algorithm.

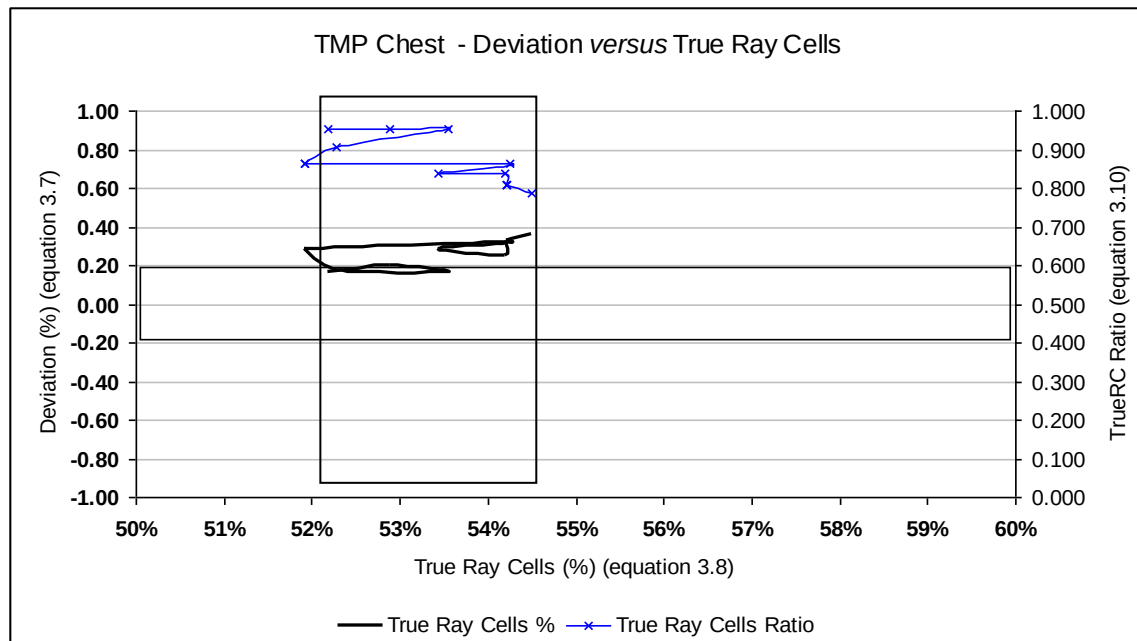


Figure 45 - Deviation and true ray cells ratio versus the percentage of true ray cells of width optimization of TMP chest samples.

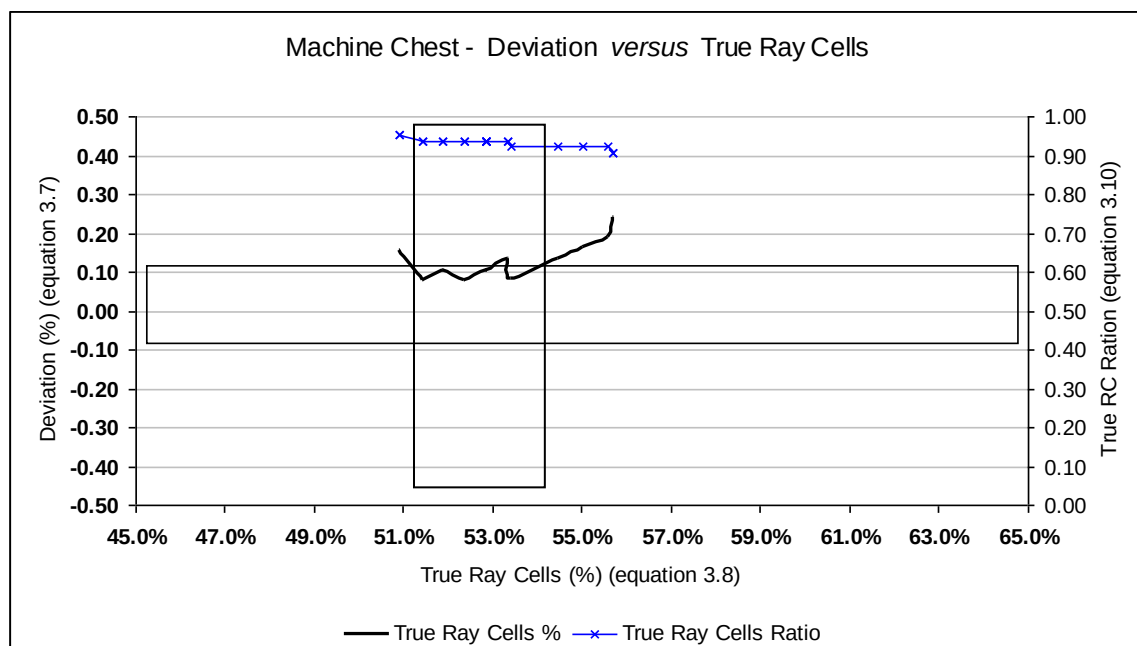


Figure 46 - Deviation and true ray cells ratio versus the percentage of true ray cells of width optimization of machine chest samples.

Table 13 - Width inferior limit optimization performed with version 2 for Machine Chest samples.

Width inferior limit (μm)	Deviation %	True Ray Cells (%)	True Ray Cells Ratio
23	0.16	50.9	0.95
23.1	0.16	50.9	0.95
23.2	0.08	51.4	0.94
23.3	0.08	51.4	0.94
23.4	0.10	51.9	0.94
23.5	0.10	51.9	0.94
23.6	0.08	52.4	0.94
23.7	0.08	52.4	0.94
23.8	0.11	52.8	0.94
23.9	0.11	52.8	0.94
24	0.11	52.8	0.94
24.1	0.11	52.8	0.94
24.2	0.11	52.8	0.94
24.3	0.13	53.3	0.94
24.4	0.13	53.3	0.94
24.5	0.08	53.4	0.92
24.6	0.14	54.5	0.92
24.7	0.16	55.0	0.92
24.8	0.19	55.6	0.92
24.9	0.24	55.7	0.91
25	0.24	55.7	0.91

Table 14 - Width inferior limit optimization performed with version 2 for TMP Chest samples.

Width inferior limit (μm)	Deviation %	True Ray Cells (%)	True Ray Cells Ratio
23	0.17	52 %	0.952
23.1	0.20	53 %	0.952
23.2	0.17	54 %	0.952
23.3	0.17	52 %	0.904
23.4	0.28	52 %	0.864
23.5	0.28	52 %	0.864
23.6	0.32	54 %	0.864
23.7	0.29	53 %	0.837
23.8	0.25	54 %	0.837
23.9	0.32	54 %	0.809
24	0.32	54 %	0.809
24.5	0.36	55 %	0.789