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EVALUATION OF FINES CONTENT IN PULPS AND FILTRATES IN PULP PRODUCTION LINES

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Abstract

The raw material for all pulping processes in the pulp and paper industry is wood. The two main fibrous products resulting from pulping processes are fibres and fines. Especially the influence of fines on the final product should not be underestimated. They control the properties of the paper sheet in either a positive or a negative manner. On this account it is of utmost importance to characterize the size and morphology of fines as well as their impact on the final product.

There are different laboratory fractionation methods for the separation of fines and the evaluation of fines content. They differ in construction as well as in their principle. Such devices are the Bauer McNett fractionator, the Tube Flow Fractionator and the Britt Dynamic Drainage Jar Tester (BDDJ). In the present study the BDDJ is used to determine the fines content of different types of pulps and to produce small quantities of fines for further analysis. Furthermore, trials with modified versions of the BDDJ are used in order to produce larger amounts of fines. In the course of these measurements and trials it was possible to improve the sampling procedure for the determination of the fines content. Moreover, mass balances of selected pulp and filtrate flows of four pulp production lines are made to get an estimate about the amount of fines in specific mass flows. Another topic of this thesis is concerned with the analysis of fines using light microscopy and a particle size analyser (Mastersizer 2000). These measurements are carried out in order to validate the fines fractionation measurements obtained by the BDDJ. Light microscopy is used to study the morphology of fines and to verify the reproducibility of instruments, such as the Mastersizer 2000.

The results show that there are considerable amounts of fines in the circulation system of the fibreline of a pulp mill. Filtrates are often used to dilute the pulp at various stages along the fibreline. One has to consider that due to the recirculation of the filtrates a steady state of equilibrium regarding fines content in the process is reached. This means that potential impacts of fines removal are unpredictable. Filtrates of the pulping process, which are transferred to the wastewater treatment plant might be a source of fines that could be used for further applications.

Keywords: fines, fines content, pulp, filtrate, fractionation, mass balance, Britt Dynamic Drainage Jar Tester, Mastersizer 2000, light microscopy.

Kurzfassung

Holz ist das Ausgangsmaterial für die Zellstoff- und Papierindustrie. Die daraus resultierenden Produkte sind Fasern und Feinstoffe. Speziell der Einfluss von Feinstoffen ist nicht zu unterschätzen, da diese die Eigenschaften des Papiers beeinflussen. Deshalb ist die Charakterisierung von deren Größe und Morphologie sowie deren Einfluss auf das Endprodukt von größter Bedeutung.

Es gibt verschiedene Laborfraktioniermethoden zur Abtrennung von Feinstoffen sowie zur Bestimmung des Feinstoffgehalts, wobei diese sich in Aufbau und Prinzip unterscheiden. Beispiele hierfür sind der Bauer McNett sowie der Tube Flow Fraktionierer und der Britt Dynamic Drainage Jar Tester (BDDJ). In der vorliegenden Arbeit wird ein BDDJ zur Bestimmung des Feinstoffgehalts verwendet, um den Feinstoffgehalt von unterschiedlichen Faserstoffen zu bestimmen und zur Produktion von kleinen Mengen an Feinstoffen für weitere Untersuchungen. Des weiteren werden Versuche mit Modifikationen des BDDJ durchgeführt, mit dem Ziel größere Mengen an Feinstoffen zu produzieren. Im Zuge dieser Versuche ist es gelungen, die Probenahme zur Bestimmung des Feinstoffgehalts zu verbessern. Massenbilanzen von Faserstoff- und Filtratströmen werden erstellt, um einen Überblick über die in den Massenströmen enthaltenen Feinstoffmengen zu bekommen. Ein weiteres Thema dieser Arbeit ist die Analyse von Feinstoffen. Hierfür werden ein Lichtmikroskop und ein Partikelanalysator (Mastersizer 2000) verwendet. Diese Messungen sind durchgeführt worden, um die Ergebnisse der Fraktionierung mit dem BDDJ nachzuprüfen. Mittels Mikroskopie wird die Morphologie von Feinstoffen studiert und die Reporduzierbarkeit der Messergebnisse des Mastersizer 2000 verifiziert.

Die Ergebnisse zeigen, dass sich beträchtliche Mengen an Feinstoffen im Kreislaufsystem einer Faserlinie befinden. Filtrate werden oft dazu verwendet, um Faserstoff an verschiedenen Punkten entlang der Faserlinie zu verdünnen. Zu bedenken ist, dass sich der Feinstoffgehalt aufrund der Filtratführung im Gleichgewicht befindet, wodurch die Auswirkungen einer potentiellen Ausschleusung von Feinstoffen nicht vorhersehbar sind. Andererseits könnten Filtrate, die zur Kläranlange geleitet werden, eine Quelle für Feinstoffe und deren weitere Verwendung darstellen.

Schlagwörter: Feinstoffe, Feinstoffgehalt, Faserstoff, Filtrat, Fraktionierung, Massenbilanz, Britt Dynamic Drainage Jar, Mastersizer 2000, Lichtmikroskopie.

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Hannes M. Gruber
Graz, 15 May 2014

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List of abbreviations

Flippr°	Future Lignin and Pulp Processing Reserach
TUG	Graz University of Technology
IPZ	Institute for Paper-, Pulp- and Fibre Technology
KFU	University of Graz
BDDJ	Britt Dynamic Drainage Jar
BMN	Bauer McNett
DIP	Deinked pulp
LWC	Light weight coated
TMP	Thermomechanical pulp
GW	Groundwood
PGW	Pressure groundwood
BOD	Biological oxygen demand
BK	Bleached kraft
SBK	Semi bleached kraft
ECF	Elemental chlorine free
DDÅA	Dynamic Drainage Analyzer of Åbo Academi
PSD	Particle size distribution
TEA	Tensile Energy Absorption
cs	Consistency
fc	Fines content
RI	Refractive index
r.p.m.	Revolutions per minute
SR	Shopper Riegler
OD	Oven dry

Chapter 1

Introduction

1.1 Future Lignin and Pulp Processing Research (Flippr°)

This master thesis was carried out within the frame of the research project Flippr°, which is the abbreviation for "Future Lignin and Pulp Processing Research". Flippr° is a cooperative research project between four companies (see figure 1.1) of the Austrian pulp and paper industry and three universities. The aim of Flippr° is to focus on higher resource efficiency and more extensive utilization of wood based products. To achieve the goal of a higher yield from usable components of wood, Flippr° created two interconnected research areas dealing with lignin on the one hand and pulp fibres on the other (Timmel (2014)). Generally Flippr° is divided into three areas, with this master thesis being part of area 2. The three areas of Flippr° are:

- *Lignin*
  The aim is to get the existing lignin sources utilizable, away from only using them to recover energy for electricity.
- *Pulp fibres*
  Includes the investigation from fibre fractionation, characterization and modification methods as well as their possible applications. One aim of area 2 is the utilisation of fines as product from fibre fractionation.
- *Life cycle sustainability assessment*
  The aim of this technique is to assess environmental impacts associated with all stages of a product regarding lifetime.

In this project two institutes from Graz University of Technology (Institute for Paper, Pulp and Fiber Technology (IPZ), Institute for Process and Particle Engineering (IPPT)) are involved. The major part of the laboratory work was done at the Institute

for Paper, Pulp and Fiber Technology (IPZ) and the analysis of the particle size distribution was carried out at the Institute for Pharmaceutical Science at the University of Graz (KFU).

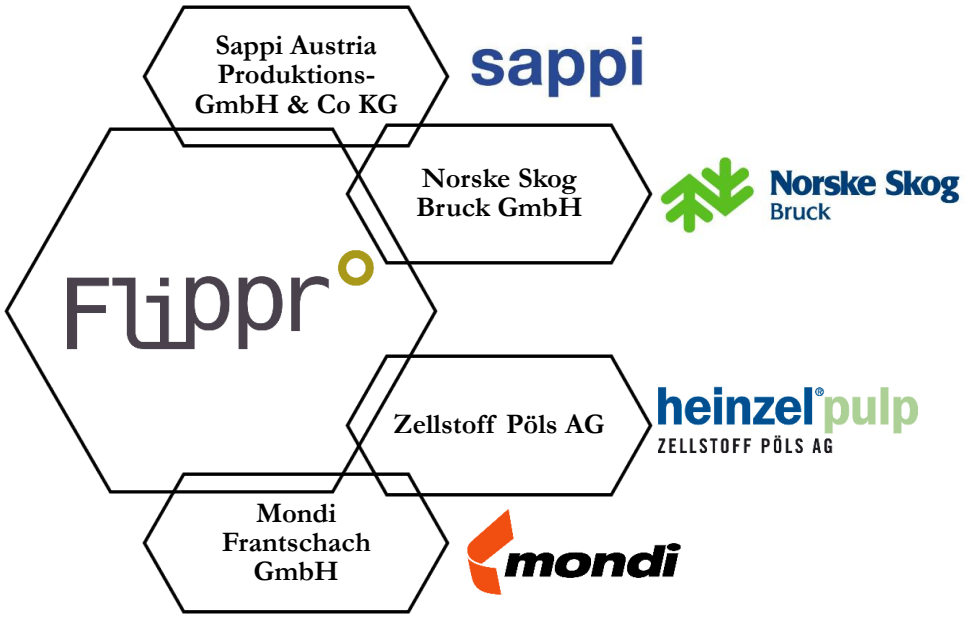

Figure 1.1 Participating companies in Flippr° project

1.2 Scope of the thesis

The scope of this master thesis is the determination of the fines content of pulps and filtrates at different sampling points during the production step of pulp. Mechanical and chemical pulps show considerable differences in both ways quantitatively (yield) and qualitatively (light-scattering ability, tear strength), which was investigated already in work done by e.g. Retulainen et al. (2002, 1993) or Lönnberg (2009). Fines influence the final paper properties tremendously and can be used to tailor them in a meaningful way.

Measurements of the fines content as well as the consistency of samples from all four industrial partners were carried out. These samples cover both pulps and filtrates. Due to the low consistency of the filtrates, fines are suspended in huge amounts of water. Therefore, the determination of the fines content was not possible. This thesis concentrates on measurements as follows:

- the determination of the fines content of various pulps by using the Britt Dynamic Drainage Jar Tester (BDDJ) according to *"SCAN-CM 66:05"*

- the measurement of the particle size distribution (PSD) of fines using the Malvern Mastersizer 2000
- microscopy of fines and filtrates using a Leica DM LM light microscope

Another subject of interest is the evaluation of mass flows of fines in pulps and filtrates. These fines could be used for other possible applications or being fractionated before special production steps in order to save energy and chemicals (Haugan et al. (2006); Hinck and Wallendahl (1999)). Therefore a static mass balance using the measured consistencies and the fines contents of the pulps was carried out in order to get an estimate for the amounts of fines in pulps and filtrates.

1.3 Outline of this thesis

After this introduction chapter, the present thesis is divided into the following five chapters:

Chapter 2 *"Production of primary fibres"*
This chapter briefly describes wood and its composition, fibres and fines. Furthermore, the two pulping types chemical (sulfite and kraft/sulfate) and mechanical pulping are described.

Chapter 3 *"Material and Methods"*
All measurement instruments used in this study are described with the main focus on the BDDJ Tester. Furthermore, a particle size analyzer (Malvern Mastersizer 2000) and a light microscope (Leica DM LM) were used to validate the results obtained by the BDDJ Tester due to their particle size distribution and morphology.

Chapter 4 *"Results and Discussion"*
All results are presented in this chapter. On the one hand results of different measurement devices used in this thesis were examined regarding their accordance with each other. On the other hand influences of the different pulping processes were examined and illustrated.

Chapter 5.1 *"Conclusion and Outlook"*
Conclusions are drawn as well as suggestions for future applications are given.

Chapter 2

Production of primary fibres

The raw material in common for all pulping processes in the pulp and paper industry is wood (see chapter 2.1). Pulping processes are classified into chemical pulping and mechanical pulping (see chapter 2.2). Pulps investigated in the course of this thesis were sulfite pulp, kraft/sulfate pulp and pressurized groundwood pulp. The products resulting from pulping processes are fibres and fines (see chapters 2.3 and 2.4). These products control the properties of the final paper product in either a positive or a negative manner. Especially the influence of fines is not to be underestimated. On this account it is of utmost importance to characterize the size and morphology of fines (see chapter 2.5) as well as their impact on the final product. There are various applications of fines fractionation possible (see chapter 2.6), although most of them are not yet applied in large-scale production processes.

2.1 Wood

Wood is a natural composite with a complex physical macrostructure and also a complex physical as well as chemical microstructure. Wood appears as a compact material, although a dry piece of wood may actually contain more than two-thirds air. On a macrostructure level, wood consists of cellulosic, tube-like fibres that are lengthwise attached to each other by a lignin-rich middle lamella, which is considered as an extra-cellular layer (Lönnberg (2009)).

Wood is divided into two species, softwood and hardwood, which are different regarding their structure and properties. There are about 1000 species of softwoods and 30000 - 350000 species of hardwoods known (Ilvessalo-Pfäffli (1995)). A detailed description of wood components like e.g. fibres is given in chapter 2.3.

The three principle elements woods consists of are: carbon (C), oxygen (O) and hydrogen (H). About 50% of the dry mass of wood is carbon, more than 40% is oxy-

gen and roughly 6% is hydrogen. Besides, small amounts of nitrogen (N) and mineral elements or ash are present too. Combinations of carbon, oxygen and hydrogen form carbohydrates and lignin, which are the principal cell wall constituents. Cellulose, hemicelluloses and lignin intermix in the cell wall in a complex way (Kellomäki (2009)). It is important to mention that the proportions and composition of the different chemical constituents vary among species. Therefore Fengel and Grosser (1975) compiled data on the chemical composition of wood in 153 species of the temperate zones. Table 2.1 shows the average amount of the principal cell wall constituents summarized from the 153 species investigated. Furthermore trees also contain smaller amounts of non-structural carbohydrates such as starch and sucrose. Instead of being cell wall material, these carbohydrates function primarily as energy reserves and are classified as extractives. Among softwoods the highest cellulose contents occur in Siberian larch, Scots pine and Norway spruce, with the first-mentioned being of less commercial importance. Among hardwoods the highest cellulose contents occur in eucalypts, balsa and European silver birch (Sjöström (1993)).

Cell wall constituent *dry matter*	Softwoods [%]	Hardwoods [%]
Cellulose	40 - 45	40 - 45
Hemicelluloses	25 - 30	25 - 35
Lignin	25 - 35	20 - 25

Table 2.1 Average amount of the principal cell wall constituents in temperate softwoods and hardwoods (Fengel and Grosser (1975))

Cellulose is the most abundant organic material on earth and the main constituent of wood. It occurs predominantly in the secondary wall, usually in association with hemicelluloses and lignin. Cellulose consists of glucose molecules ($(C_6H_{10}O_5)^n$) produced by the tree through photosynthesis (Kellomäki (2009)).

Hemicelluloses are heterogenous, low-molecular-weight polysaccharides formed from glucose and other sugar molecules. The proportion is somewhat less in softwoods than in hardwoods. Hemicellulose molecules include a variety of monosaccharides. The main classes of them are xylans and galactoglucomannans (Kellomäki (2009)).

Lignin characteristically differentiates wood from other cellulosic materials. In its natural state as it occurs in wood cells, it is an amorphous, indefinitely large polymer. The most important properties of lignin are the rigidity and stiffness it imparts to the cell wall, which is comparable with reinforced concrete. Lignin is also present in the fine voids within the cell wall where it acts as bulking agent (Kellomäki (2009)). About four-fifths of the lignin, however, is situated in the secondary wall of fibres (Lönnberg (2009)).

2.2 Pulping

The principal reason for pulping is to liberate the fibres from the wood matrix. This can be accomplished either chemically, mechanically or in a combined way (see table 2.2). In the chemical pulping process wood is disintegrated chemically into fibres by cooking wood chips. Thus, fibres can be separated from each other without being destroyed. This is only possible if lignin and hemicellulose (to some extent) are dissolved or more or less softened. Chemical pulp is either produced by the sulphite or kraft/sulphate pulping processes (Sjöström (1993)). The techniques used to produce mechanical pulp are grinding (pressing wood logs against a revolving grindstone) and refining (disintegrating wood chips in a disc refiner). The fibres obtained from chemical and mechanical pulping processes have to be mechanically treated (refined) for most paper grades before they become suitable for papermaking (Lönnberg (2009)).

Mechanical pulps typically have a high yield, ranging from 93 - 97% (PGW even 98.5%) when using Norway spruce, whereas the yield for chemical pulps ranges from 35 - 65% (Lönnberg (2009)). Some wood species can be used for both pulping processes, e.g. Norway spruce, aspen or radiata pine. Others are more suitable for just one pulping type, such as Scots pine, eucalyptus and birch preferably for chemical pulping (Niskanen (2008)). Table 2.2 shows the commercial pulp types available on the global market.

	Pulp type	Yield [% of wood]
(A)	**Chemical**	35 - 65
	Acid sulphite, Bisulphite, Multistage sulphite,	
	Anthraquinone alkali sulphite, Kraft, Polysulphide-kraft	
	Prehydrolysis-kraft, Soda	
(B)	**Semichemical**	70 - 85
	NSSC, Green liquor, Soda	
(C)	**Chemimechanical**	85 - 95
	Chemithermomechanical (CTMP), Chemigroundwood (CGW)	
(D)	**Mechanical**	93 - 97
	Stone groundwood (SGW), Pressure groundwood (PGW)	
	Refiner mechanical (RMP), Thermomechanical (TMP)	

Table 2.2 Commercial pulp types (Sjöström (1993))

Research work carried out on fines in previous studies mainly focused on thermomechanical pulp (TMP). The pulp types relevant in this master thesis are magnesium bisulphite pulp, kraft/sulphate pulp and pressure groundwood (PGW).

Mechanical pulping offers many advantages compared to chemical pulping such as low costs and simple technology. Moreover, mechanical pulps have a higher light-scattering ability, a fairly high brightness, good formation characteristics that result in paper with smooth surface and high bulk. There is only one major drawback using mechanical pulps, which is the higher consumption of electrical energy. In addition to that the bonding ability of the fibres is lower compared to chemical pulp fibres and this in turn leads to lower strength properties. Mechanical pulps quite often also contain impurities and they cannot be bleached to the same extent as chemical pulps, which means that they have a poor brightness stability (Lönnberg (2009)).

2.2.1 Characteristics of chemical and mechanical pulps

The difference between the two pulping methods results in different pulp, fibre and sheet properties. Therefore pulping, bleaching and refining have a crucial effect on paper properties. Paper made from e.g. bleached kraft pulp has 2 to 3 times higher tensile strength than paper made from mechanical pulp. On the other hand the opacity and light-scattering of mechanical pulps is superior to those of chemical pulps, primarily because of the special properties of mechanical pulp fines. In mechanical pulp fines the fraction of long, intact fibres can be less than 20% (by weight) for groundwood (GW) and up to 40% for thermomechanical pulp (TMP). In chemical pulp the long fibre fraction can be as high as 90% (Niskanen (2008)).

Figure 2.1 shows the light-scattering ability of various pulp types. Mechanical pulps show a superior light-scattering coefficient compared to chemical pulps. Figure 2.2 shows the tear strength versus the freeness of different pulp types. Chemical pulps yield higher values compared with mechanical pulps, except chemithermomechanical pulp (CTMP).

Generally chemical pulps have a higher tear index but a lower light-scattering coefficient than mechanical pulps. Although there are differences between softwood and hardwood, the former has a higher tear index but a lower light-scattering index. Among mechanical pulps groundwood pulps like stone groundwood (SGW) and pressure groundwood (PGW) have the highest light-scattering ability but the lowest tear index. With refiner pulps like CTMP and TMP the opposite is true.

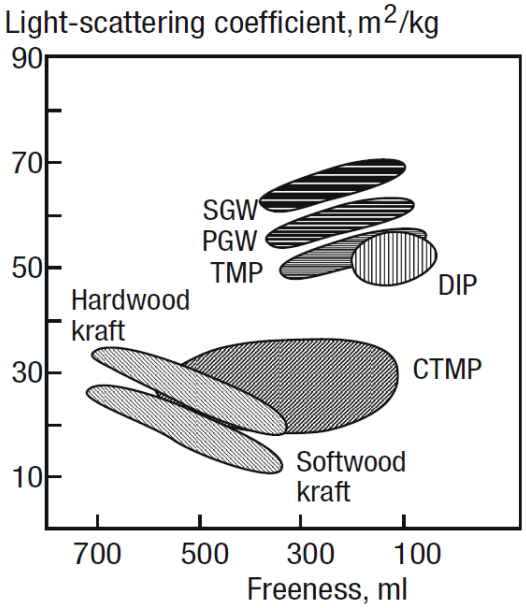

Figure 2.1 Light-scattering ability of various chemical pulps and mechanical pulps (wood: Norway Spruce) Lönnberg (2009)

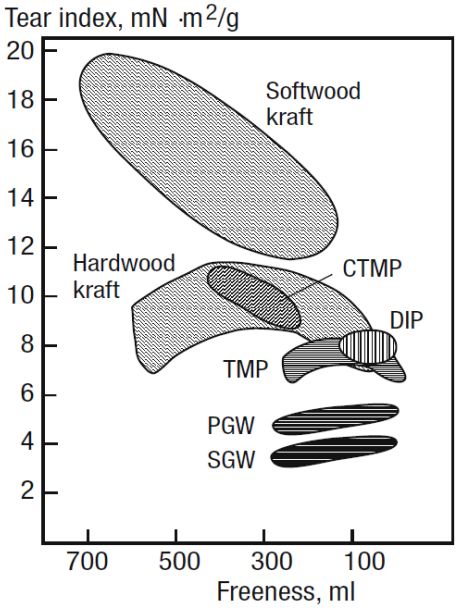

Figure 2.2 Tear strength of various chemical pulps and mechanical pulps (wood: Norway Spruce) Lönnberg (2009)

Differences between chemical and mechanical pulps are illustrated in table 2.3 and 2.4. Almost all properties differ between the two pulp types. For example the lignin

Property	**Mechanical pulps**	**Chemical pulps**
Yield on wood	High	Low
Amount of lignin	High	Low
Amount of hemicelluloses	High	Low
Degree of polymerization	High in cellulose	High
Charge in water suspension	More anionic	Less anionic
Water affinity	More hydrophobic	More hydrophobic
Long fibres per unit mass	Few	Many
Specific surface area	Large	Small
Fines content	High	Low

Table 2.3 General differences between the properties of mechanical and chemical pulps from softwoods (Niskanen (2008))

content of mechanical pulps is in the range of about 30% whereas in bleached kraft pulps it is almost zero. Since the content of lignin and hemicelluloses is reduced, chemical pulps also have a lower yield. Thus, compared with mechanical pulps the ratio of pulp mass to wood mass is smaller for chemical pulps (Niskanen (2008)).

Property	Mechanical pulps	Chemical pulps
Fines:		
Structure	Lamellar	Fibrillar
Bonding ability	Good	Excellent
Fibres:		
Structure	Stiff, coarse, straight	Slender, curly, kinky
Shape	Short and wide	Long and narrow
Bending stiffness	High	Low
Degree of collapse	Less collapsed	More collapsed

Table 2.4 Differences between the properties of mechanical and chemical pulp fibres and fines from softwoods (Niskanen (2008))

2.2.2 Advantages and drawbacks of chemical pulping methods

A comparison of kraft pulping and sulphite pulping is shown in table 2.5 and 2.6, in which advantages and drawbacks of the pulping processes are listed.

Kraft pulping	
(+)	**(−)**
increased delignification rate	darker pulp
stronger pulp	pulp harder to beat
higher yield	pulp harder to bleach
wide range of wood species applicable	causes malodorous gases
insensitive to bark and wood quality	
relatively short cooking time	
little pitch problems	
efficient chemicals regeneration	
efficient energy regeneration	
valuable by-products obtained	

Table 2.5 Advantages and drawbacks of kraft pulping pulping (Fardim (2011))

Sulphite pulping	
(+)	**(−)**
wide pH range	high BOD of mill effluents
pulp has higher initial brightness	few wood species applicable
pulp is easier to bleach	
smaller odor problems	
smaller investment costs	
flexibility of producing speciality pulps	

Table 2.6 Advantages and drawbacks of sulphite pulping (Fardim (2011))

2.3 Fibres

Pulps consist of fibres and fines. The geometric structure of paper is primarily controlled by the fibres and they are the principal structural element of paper. In addition to fibres, fines and fillers are the most typical particles in papermaking (Niskanen (2008)). More precisely, only fibres and fines are produced during the pulping process. Fibres are plant cells that have a high length-to-width ratio, approximately 50 - 100. The average fibre length (from both hard- and softwoods) scatters in a range from 0.8 - 7.0 mm and the fibre width from 14 - 65 µm. Hardwood fibres are generally thinner and shorter than softwood fibres. The volume fraction of fibres in wood vary from 89 - 95% for softwoods and 37 - 65% for hardwoods (Ilvessalo-Pfäffli (1995)).

A description of the cell wall structure and the constituents of a fibre is given in figure 2.3 and in figure 2.4. The fibre wall consists of a thin primary wall (P) as

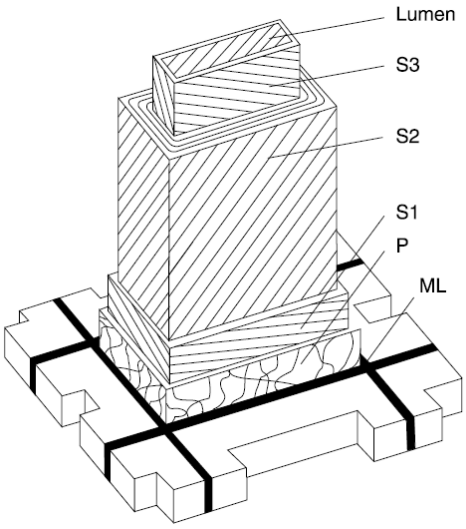

Figure 2.3 Cell wall structure of a wood fibre (conifer tracheid)
middle lamella (ML), primary wall (P), secondary wall 1 (S1), secondary wall 2 (S2), secondary wall 3 (S3)
(Niskanen (2008))

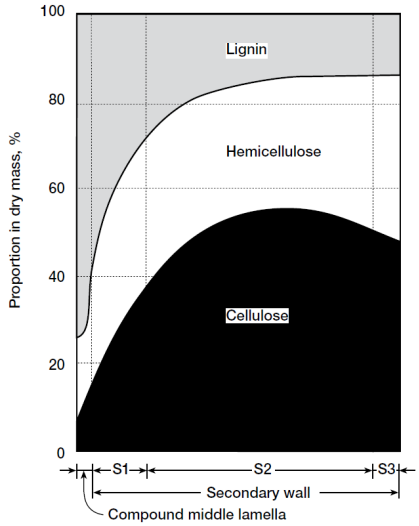

Figure 2.4 Distribution of cellulose, hemicelluloses and lignin within the cell wall of conifers
(Kellomäki (2009))

an outermost, amorphous layer. A bulky secondary wall (S) underneath the primary wall can be divided into three different layers (S1, S2 and S3). As outermost layer of the secondary wall, the S1 layer is thin and mostly amorphous (0.1 - 0.3 µm thick). The S2 layer (1 - 5 µm thick) is bulky and mostly crystalline, because it contains crystalline cellulosic chains on a microstructural level. Cellulosic chains form cellulosic microfibrils, which are surrounded by amorphous lignin. Bunches of these cellulosic

fibrils form spiral lamellae or macrofibrils around the fibre axis at a constant angle. Macrofibrils in turn are surrounded by amorphous substances, mostly lignin. The S3 layer (thinner than approx. 0.1 µm) is amorphous and protects the fibre from bacterial attack through the lumen, which is the hollow part inside the fibre. The wall thickness varies depending on tree species, cell type, season of formation and other factors (Lönnberg (2009)). Fibre properties are important because:

- they affect paper structure in terms of formation and consolidation
- they are responsible for the properties of both wet and dry paper

The raw material controls the fibre structures and the fibre dimensions, which are modified by the pulping process later on. Moreover, the different types of pulps have different effects on the properties of paper (see chapter 2.2).

2.4 Fines

In the papermaking process fine cellulosic particles are called "fines". Fines can play an important role in the papermaking process. Due to their small size and large total specific surface they have a significant impact on many surface chemistry phenomena (Krogerus et al. (2002)). On the one hand, fines can help to fill up spaces between fibres in a sheet of paper. This effect typically results in a denser, stronger and more uniform product. On the other hand, high levels of fines have a negative effect on the dewatering behavior of paper during the sheet forming process. This has also a negative impact on the manufacturing process (reduced production speed) and leads to a higher energy demand in the drying section of the paper machine (Chen et al. (2009)).

The fraction of pulp which passes through a screen with a certain mesh size or through the holes of a wire with an equivalent hole diameter of a given fractionator is defined to be the fine fraction. Common devices are the Bauer McNett Fractionator (BMN) or the Britt Dynamic Drainage Jar Tester (BDDJ). There are also other types of fractionators which are used for the production of fines, i.e. a combination of bow screen and Attisholtz filter in a row (Haugan et al. (2006)).

There is no exact line of separation between fibres and fines, basically the size of particles is an arbitrary decision. The most common size limit is a 200-mesh screen, which corresponds to a 76 µm hole diameter. According to literature it is therefore often called P200 (pass 200-mesh). Some tests were also carried out with P100 (fraction passing through 100 mesh screen) fractions (Chen et al. (2009)) or a wire plate with the equivalent hole diameter (Lindqvist et al. (2011)). However, even relatively long particle (100 - 250 µm) bands may pass through the screen, because they are very thin (width < 1 µm). That is why fines are a heterogenous fraction in terms of particle

size, shape and composition (Krogerus et al. (2002)).

Fines differ in their morphology as well as in their composition. The classification of fines is shown in table 2.7. The difference between fines produced during chemical pulping and mechanical pulping is clarified in the following two subchapters 2.4.1 and 2.4.2.

Type of fines	Origin	Morphology	Content [%]
Mechanical fines	TMP, GW	Fibrils, flakes, ray cells, etc	10 - 40
Primary fines	Unbeaten chemical pulp	Ray cells, lignin, flakes from middle lamella, etc	2 - 10
Secondary fines	Beaten chemical pulp	Fibrils peeled of from fibre wall	2 - 10
Tertiary fines	DIP, broke from mill	Fibre fines, fillers, coating pigments, latexes, additives, stickies, etc	variable

Table 2.7 Classification of fines (Krogerus et al. (2002))
TMP...thermomechanical pulp, GW...groundwood, DIP...deinked pulp

The effects of fines on the physical properties of pulp and sheet are shown in table 2.8.

Property	Effect	Property	Effect
Drainage resistance	++	Air permeability	−−
Wet web strength	+	Specific bond strength	
Sheet density	+	- *primary fines*	−
Shrinkage potential	+	- *secondary fines*	+
Tensile strength	+	Light-scattering	
Elongation	+	- *chemical pulp fines*	−
Tensile stiffness	+	- *mechanical pulp fines*	++
Tear strength	−	Fibre rising	−
Compression strength	+	Linting	−
Folding endurance	+		

Table 2.8 Effect of fines on the pulp and sheet properties (Retulainen et al. (1993))
(+) the property value increases, (-) the property value decreases

Fines lead to an increase in the density of the sheet, which is the result of a closer packing within the sheet due to the smaller fines particles. This yields in greater bonding, which has a corresponding effect on many physical sheet properties. Increased bonding within the sheet improves the tensile properties of paper. Furthermore, the wet web strength, the folding endurance as well as the compression strength are also improved. A drawback of the void filling effect of fines is that they decrease the air permeability of a paper web (Pruden (2005)).

2.4.1 Chemical pulp fines

Table 2.7 before shows that the fines content in chemical pulp (2 - 10%) is decisively lower compared with mechanical pulp (10 - 40%). Chemical pulp fines can be divided into two types, primary fines and secondary fines.

Primary fines result mainly from pulping as a result from the cooking and chipping processes. Secondary fines are generated during refining of the pulp by rubbing and crushing actions. Figures 2.5 and 2.6 show micrographs of various types of chemical pulp fines.

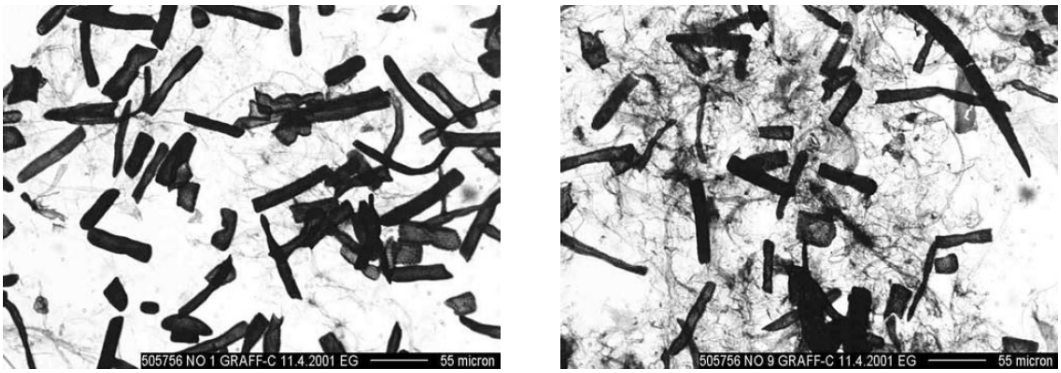

Figure 2.5 Primary fines (left) and secondary fines (right) from hardwood chemical pulp (Krogerus and Tiikkaja (2002))

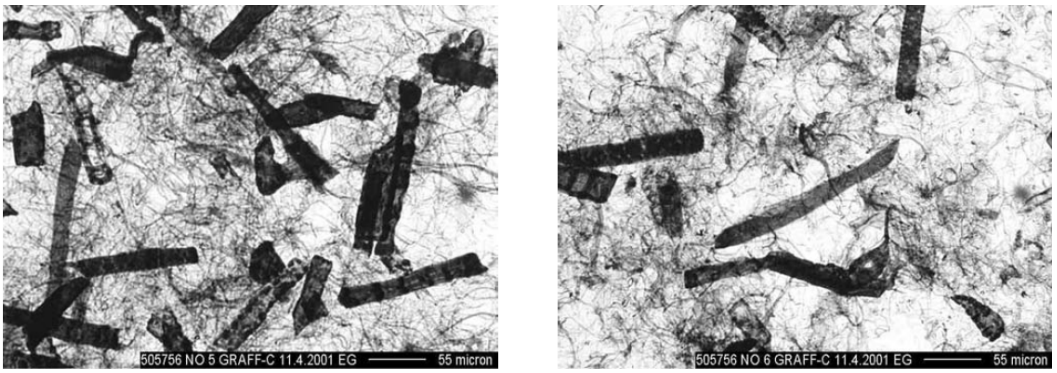

Figure 2.6 Primary fines (left) and secondary fines (right) from softwood chemical pulp (Krogerus and Tiikkaja (2002))

Tertiary fines are brought in with already processed paper which is returned as raw material, like broke or DIP.

Primary fines
They comprise short or broken fibres or parts of it and consist mainly of ray cells (a) and parenchyma cells (b). Also, their constituents differ between softwood (a, b) and hardwood (a, b, and vessel elements). Primary fines consist of a coarser fraction (rich in ray cells) and a finer fraction (fibrils and lamellae). Both fibrillar and ray cell content influence paper properties such as the tensile index and light-scattering. Thereby, fines with higher fibrillar and lower ray cell content affect paper properties more strongly (Yin et al. (2013)). According to micrographs in work done by Cole et al. (2008) primary fines are rectangular objects with a length of 30 - 60 µm and a width of 10 - 30 µm. This is consistent with other work, in which primary fines are described using a length-to-thickness ratio of less than five (Chen et al. (2009)).

Chemical pulps produced from hardwood contain larger proportions of primary fines than those of softwood. The difference in bonding ability of primary fines in different pulp types is small: Regardless of whether softwood is compared with hardwood or bleached softwood is compared with unbleached softwood (Yin et al. (2013), Chen et al. (2009)).

Secondary fines
Secondary fines are produced during beating of the pulp. The general effects of beating are fibrillation of fibres and fibre shortening, which are quite similar effects that occur in mechanical pulping. The main effects are as follows: Ferreira et al. (2000)

- *External fibrillation* - Result of the removal of parts of the primary wall and secondary wall layers. Furthermore, fibrils are spread out from the fibre surface.
- *Internal fibrillation* - Result of the breakage of intermolecular bonds, which corresponds to a partial delamination of the fibre wall.
- *Fibre shortening* - Result of cutting action during beating.

Figure 2.7 shows a beaten kraft fibre. The fibres were freeze-dried in order to visualize the external fibrillation. Conventional drying causes a collapse of the fibrils on the fibre wall and due to this they are not visible. Figure 2.8 shows internal fibrillation of a beaten kraft fibre. In this image the delamination of the fibre walls is clearly visible. Fibrillation generally increases fibre-to-fibre bonding, due to an increase in the area available for bonding and in fibre swelling. Fibrillation also increases fibre

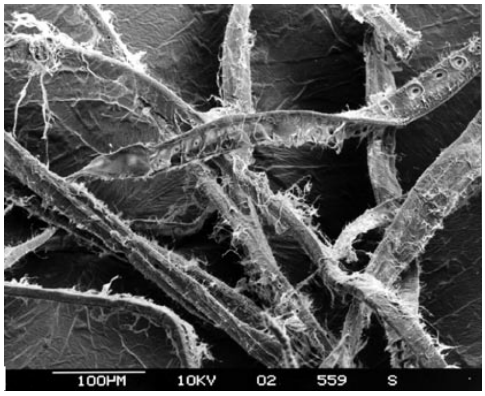

Figure 2.7 External fibrillation of a kraft fibre (freeze-dried) (Niskanen (2008))

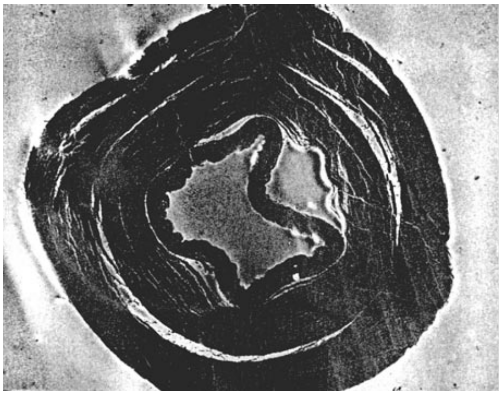

Figure 2.8 Internal fibrillation of a kraft fibre (cross section shown) (Niskanen (2008))

flexibility and collapsibility, thus improving fibre conformability in the fibre network. Both external fibrillation and fibre shortening produce fines, while internal fibrillation does not (Ferreira et al. (2000)).

The constituents of secondary fines are lamellar and fibrillar parts of the fibre wall and colloidal material, which originate mainly from the S1 and S2 layers. Further, secondary fines consist of broken fibre fragments, fibrils and thin lamellae from the fibre surface. Moreover, they show an increased specific surface area, which in turn increases the relatively bonded area between the fibres. Thus, secondary fines increase bonding strength, whereas primary fines decrease it. Higher refining amounts of fines contribute to higher bonding potential, higher surface charge, higher hydrodynamic specific volume and higher fibrillar content, but lower ray cell content (Yin et al. (2013)).

Secondary fines slow the drainage rate to a much greater extend than primary fines do. Hence, the energy demand for dewatering on the paper machine can be reduced by removing water during forming and pressing more effectively. Higher levels of fines in paper production can result from increased use of mechanical pulp, increased refining and from recycling paper without deinking. Moreover, secondary fines are suggested to be used as effective aids for mechanical filtration (Cole et al. (2008)).

Generally secondary fines yield higher strength properties than primary fines and among them higher strength properties are obtained from highly refined pulps. Compared to primary fines, they have a strong swelling ability and clearly better bonding properties. Therefore, they are more advantageous to paper properties. (Yin et al. (2013), Chen et al. (2009)).

2.4.2 Mechanical pulp fines

Mechanical pulp fines were defined in the pioneering work of Brecht and Klemm (1953) in which they focused on quality aspects of the fines components in groundwood pulp. They described some of the fines to be granular (flour stuff = "Mehlstoff"), whereas other fines in groundwood are mucilaginous, consisting of slender fibrils (slime stuff = "Schleimstoff") (Brecht and Klemm (1953), Chen et al. (2009)). Other terms for slime and flour stuff are fibrillar and flake-like material (Luukko (1999)). Mechanical pulps contain a large number of primary fines, which tend to be enriched in lignin. These amounts of lignin are a result of the mechanical forces applied to the wood to separate the fibres. Many fibre fragments are produced and they mostly originate from the S1 and S2 layers of the secondary wall (Pruden (2005)). The main particle types in mechanical pulps are: (Krogerus et al. (2002))

- Flakes and lamellae
- Band- and thread-like fibrils
- Pores (tori from bordered pits, from the fibre cell wall and from ray cells)
- Ray cells

Fibrillar material contains fibrils and thin lamellae as well as fibrils that are attached to, e.g. a fibre wall fragment. Fibrillar material consists of cellulose-rich material with a high specific surface area (volume) and degree of swelling (originating largely from the S2 layer of the fibre).

Flake-like material contains fibre wall fragments, thick lamellae and ray cells. Furthermore, it consists of lignin-rich cell wall and middle lamella fragments as well as ray cells with a low surface area and swelling ability. Figure 2.9 shows images of fibrillar (left) and flake-like material (right). The samples were obtained from a Bauer

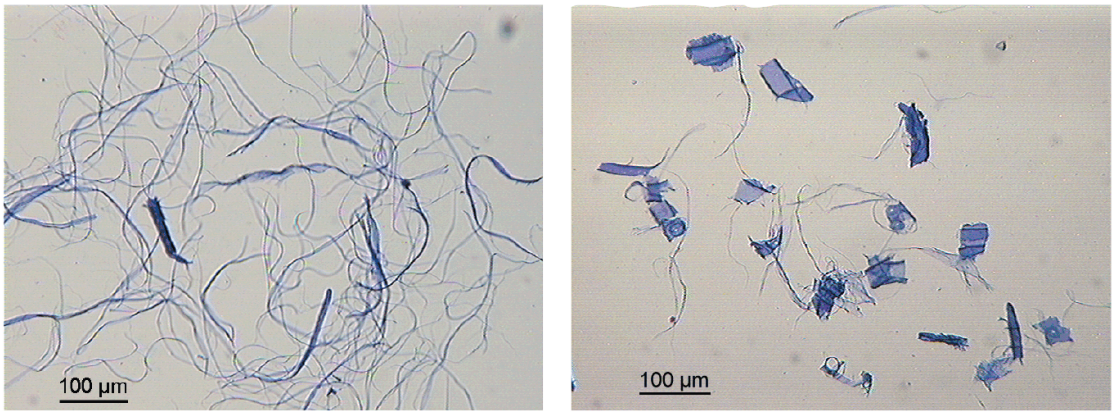

Figure 2.9 Microscopy of fibrillar material (left - P100/R200 fraction) and flake-like material (right - P300/R400 fraction) of TMP fines (Luukko and Paulapuro (1999))

McNett Fractionation process and the images were captured by a light microscope (Luukko and Paulapuro (1999)).

The chemical composition of fines is as important as their morphology. In the work done by Sundberg et al. (2003) isolated fractions of fines from a TMP pulp were produced by using a combination of filtration (equipped with a BDDJ Tester) and subsequent sedimentation techniques. The composition of the various fractions is illustrated in figure 2.10. The fractions separated and investigated were fibres, fibrils

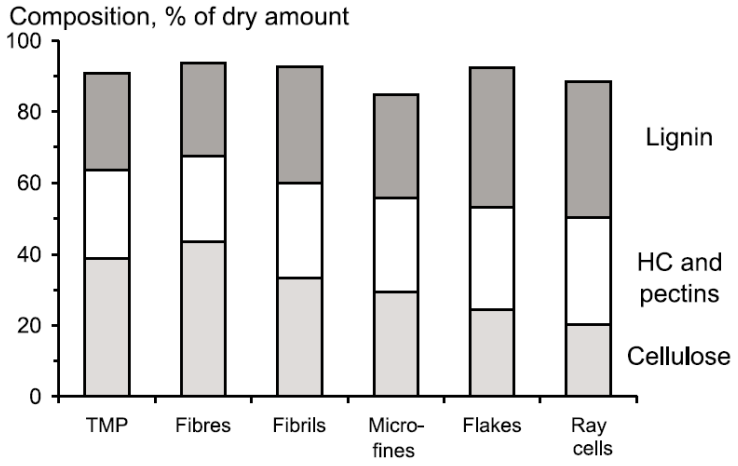

Figure 2.10 Composition of cellulose, hemicelluloses (and pectins) and lignin in fibres and fines from unbleached TMP (Sundberg et al. (2003))
HC...hemicelluloses, TMP...thermomechanical pulp

(i.e. long, thin threads), microfines (i.e. colloidal fines), flakes (i.e. thin lamellae from tori or bordered pits) and ray cells (i.e. ray parenchyma cells and ray tracheids). All types of TMP fines contained more lignin and less cellulose than fibres. The fibrils contained slightly more cellulose and less lignin than the flakes and ray cells. Most of the lignin of the separated fractions contained ray cells and flakes (Sundberg et al. (2003)).

Mechanical pulp fines generally increase paper properties like opacity and strength (Retulainen et al. (1993)). Fibrillar material contributes to densification and consolidation of the sheet, while flake-like particles improve light-scattering effects (Luukko and Paulapuro (1999)). The particle size of mechanical pulp fines also has an influence on sheet properties: The smaller the particles, the higher the sheet density and sheet strength.

With most groundwood properties it is a matter of the quantitative relation of fibres to fines. This means that mechanical pulp fines are a powerful instrument to adjust mechanical pulp properties (Chen et al. (2009)).

2.4.3 Tertiary fines

Tertiary fines originate from already processed paper which is returned to the paper production as raw material. They are either fines from coated and uncoated broke inside the mill or from deinked pulp (DIP). Tertiary fines have many constituents such as different types of fibres, fillers, coating pigments, latexes as well as agglomerates of paper additives (see table 2.7). Figure 2.11 shows a scanning electron microscopy (SEM) image of a ground calcium carbonate (GCC) coating pigment. The amount of

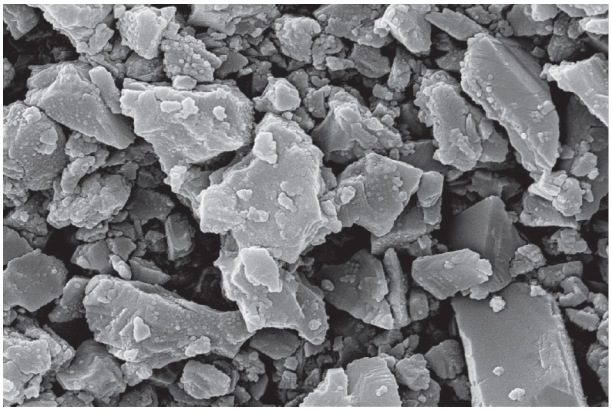

Figure 2.11 SEM image of a ground calcium carbonate (GCC) coating pigment (Paltakari (2009))

fines depends strongly on the paper grade produced. Uncoated LWC (light weight coated) base paper may contain as much as 60% fines, coated LWC broke even 70%. The mineral content in the fines fraction is about 40% (Krogerus et al. (2002)).

2.5 Characterization of fines

The characterization of fines by means of measuring their particle size or their particle size distribution (PSD) can be carried in different ways. For this different types of instruments were used. The following subsection gives an overview about such devices used in previous studies.

- **Malvern Mastersizer 2000**
  The Mastersizer 2000 from Malvern Instruments GmbH uses laser diffraction in order to analyse the PSD of either dry or wet samples (fines suspension). With this device it is possible to analyse particles with a size ranging from 0.02 to 2000 µm *(Mastersizer 2000 manual, 2005)*. A detailed description of the Malvern Mastersizer 2000 is given in chapter 3.3.
  Mosbye (1999) investigated fines from TMP using a Malvern Mastersizer µ+. The different fractions were analysed in order to estimate the fractionating efficiency and reproducibility. Data obtained by the Mastersizer should be treated

with care since the instrument assumes, that the particles are spherical. This assumption is not valid to measure fines. Nevertheless, in this work as well as in others the size distribution curves were considered useful to measure a change in the PSD and to assess the reproducibility of the fractionation process.
Chen et al. (2009) tested fines of unrefined and refined bleached hardwood kraft pulp (BK) and chemithermomechanical pulp (CTMP). The particle size distribution (PSD) of cellulosic fines matter were determined using a "Horiba LA-300" laser diffraction device. Default conditions for ultrasonication (before the measurement) and recirculation of the suspension were used.
Xu and Pelton (2005) tested fines of a semi bleached kraft pulp (SBK) using a Malvern Mastersizer 2000. Fines dispersed in deionized water were sonicated before the measurements.

- **Coulter Multisizer II^{TM}**
  Fines from eucalyptus kraft pulp were tested in work of Ferreira et al. (2000). Those fines were analysed using a Coulter Multisizer II unit. Due to the measurement principle particles flow through an aperture filled with a conductive electrolyte. As the particle occupies the cross section of the channel, the resistance across the channel increases. The change of the resistance is measured by applying a defined voltage and the amplitude of this change is proportional to the particle size.

- **Light microscopy**
  Light microscopy was performed in various works in order to study fractions visually and to verify the reproducibility of the fines measured by particle size analyzers. Examples would be: Zeiss "Axioplan" (Xu and Pelton (2005)), Olympus "BH-2" (Ferreira et al. (2000)) and Zeiss "Axioskop 2" (Kangas and Kleen (2004)). A detailed description of microscopy carried out is given in chapter 3.4.

2.6 Possible applications of fines fractionation

Various applications of fines fractionation were found due to literature research carried out. The possible applications were structured into the categories "property improvement", "bleaching and energy" and "biorefinery".

Property improvement

- Increased porosity (Olson et al. (2001))
  The removal of fines and short fibres resulted in a long fibre pulp fraction. This long fibre fraction was refined to a certain tensile energy absorption (TEA), which led to a pulp with higher porosity.

- Strength improvement due to addition of fines (Bäckström et al. (2008))
  An increased consolidation of the fibre network due to creation of capillary forces between surfaces can be achieved. These capillary forces are higher with secondary fines than with primary ones.

- Positioning of fractionation step (investigated for a sulphite pulp mill) with fines removal between washing and bleaching plant (Hinck and Wallendahl (1999))
  An improved pulp quality with better drainage, absorption and paper making properties was reached.

- Fibre fractionation followed by triple layered paper formation (outer layers long fibre fraction, middle layer fines rich fraction) (Sood et al. (2007))
  Paper produced in the work cited was made from agricultural residues pulp. It showed a higher tensile strength, tear strength, bulk stiffness and surface strength.

- Fibre fractionation (Yu et al. (1994) and Asikainen et al. (2010))
  The process was carried out by means of a fractionator with a washing device set afterwards. The fractionator removed excess ash, inactive fillers or fines. This results in an increased freeness and tear index of the pulp. The fines fraction obtained can be used as bonding material in various fibre furnishes.

Bleaching and energy

- Quality improvement and cost reduction by bleaching of fractions (Haugan and Gregerson (2007))
  A fractionation step before bleaching can reduce the chemical consumption, because fines consume more bleaching chemicals like NaOH and H_2O_2 as fibres do.

- Positioning of fractionation step (investigated for a sulphite pulp mill) with fines removal between washing and bleaching plant (Hinck and Wallendahl (1999))
  An Improved operation of downstream bleaching processes, of the pulp drying and on the paper machine is possible. Apart from that, the reduced demand on bleaching chemicals allows milder bleaching conditions.

- Fibre fractionation (Yu et al. (1994) and Asikainen et al. (2010))
  A reduction of operation costs due to less energy consumption is possible. Thereby only the fractionated long fibre fraction gets refined. Moreover, less chemicals are used and brightness is improved too.

Biorefinery

- Positioning of fractionation step (investigated for a sulphite pulp mill) with fines removal between washing and bleaching plant (Hinck and Wallendahl (1999))
  Increased recovery of fines for use as e.g. on-site generated fuels.
- Fibre fractionation (Asikainen et al. (2010))
  The biorefinery concept in this work uses the fines fraction as source for xylan, fatty acids and sterols.

Production, chemical oxygen demand (COD) and toxicity

- Fibre fractionation (Yu et al. (1994))
  The refining equipment is oftentimes the bottleneck of the production process. A fractionation step where only the long fibre fraction is treated can help to maintain the quality of production.
- Positioning of fractionation step (investigated for a sulphite pulp mill) with fines removal between washing and bleaching plant (Hinck and Wallendahl (1999))
  Reduced effluent discharges of extractives, reduced COD, reduced toxicity and reduced loading on the effluent treatment system can be reached.

Chapter 3

Material and Methods

The Britt Dynamic Drainage Jar Tester (BDDJ) was used to investigate the pulps of all industrial partners participating in the Flippr° project. The BDDJ Tester was used to:

- determine the fines content of the pulps
- produce small quantities of fines for further analysis (Mastersizer, microscope, etc.)

Chapter 3.1 describes why the BDDJ Tester was chosen and which other devices are available. Chapter 3.2 gives an overview of the BDDJ Tester itself. Chapter 3.3 describes the Malvern Mastersizer 2000, which was used to verify the results obtained from the BDDJ Tester concerning the particle size distribution (PSD) of the fines suspension. Finally in Chapter 3.4 microscopic images of both fines suspensions and filtrates were captured to check visually whether the PSD obtained from the Mastersizer 2000 correlated with them.

3.1 Fractionation methods

There are different fractionation methods for the production of fines available. Essentially they differ slightly in their construction, even though all of them have the same principle. The water of a pulp suspension passes through a wire and the filtrate, which contains the fines is collected. Devices available at the Institute of Paper-, Pulp- and Fibre Technology (IPZ) automatically excluded other fractionators, which were used in previous studies (Haugan et al. (2006), Lindqvist et al. (2011), Paavilainen (1992)). The two devices at the IPZ for the determination of the fines content are the Britt Dynamic Drainage Jar Tester (BDDJ) and The Bauer McNett Fractionator (BMN). In comparison with the latter, in which fines are suspended in huge amounts of water (more than 200 l per test) the BDDJ Tester only needs small

amounts of water (5 l per test). Besides that the fines content can be determined easily.

Methods for fines fractionation used in previous studies are:

- **Britt Dynamic Drainage Jar Tester (BDDJ)**
  There are several studies in which different types of pulps were fractionated using the BDDJ Tester (Ferreira et al. (2000), Sundberg et al. (2003), Sirviö and Nurminen (2004), Mosbye (1999), Kangas and Kleen (2004), Rundlof et al. (2000), Lindqvist et al. (2011), etc). All fractionation trials in this thesis were carried out with this device. Therefore a detailed description is given in chapter 3.2.

- **Bauer McNett Fractionator (BMN)**
  The BMN Fractionator classifies fibres mainly according to their length and it is used to quantify the size distribution of fibres. This laboratory device simulates the fractionation progress using a pressure screen at industrial scale. The Bauer McNett Fractionator (BMN) is the most common laboratory fractionation device among this equipment (Gooding and Olson (2001)). Table 3.1 shows the specifications used for this fractionation method.

Specifications: Bauer McNett Fractionator	
Sample size of pulp	10 g (o.d.)
Active volume of one chamber	9.8 l
Standard flow-through rate	12 l/min
Standard set of BMN screens used	14, 28, 48, 100 and 200-mesh

Table 3.1 Bauer McNett Fractionator specifications (Gooding and Olson (2001))

Figure 3.1 shows a fractionation chamber of the BMN Fractionator. The fractionator usually consists four or five chambers (see a 4-chamber model in figure 3.4). These chambers are equipped with screens of decreasing mesh size. The pulp suspension is transferred into the chamber through the inlet (see top of figure 3.1). A motor driven rotor inside the chamber is used to introduce circular motion (clockwise rotation) to the suspension. Furthermore, this leads to a homogeneous distribution of the fibers within the suspension. A part of the pulp suspension leaves the chamber through the outlet. The outlet of each chamber is equipped with a screen having a certain mesh size (see table 3.1).

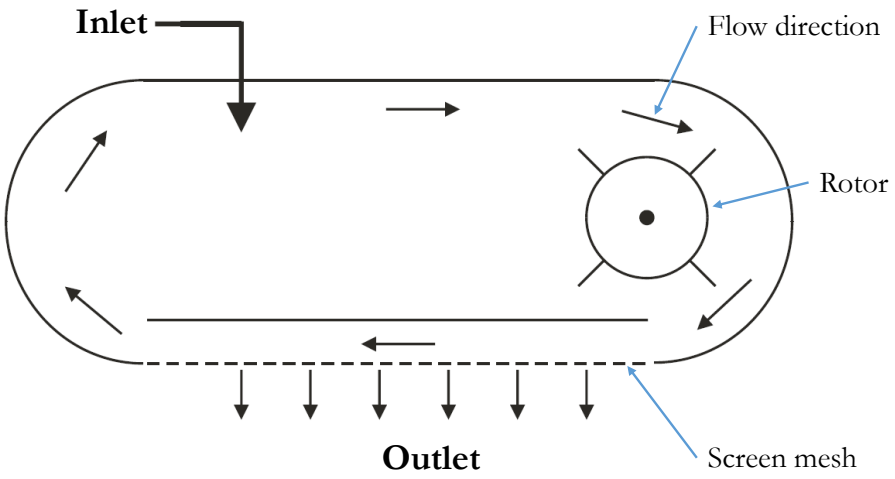

Figure 3.1 Chamber of a Bauer McNett Fractionator (BMN) in plan view (Gooding and Olson (2001))

A comparison of the BDDJ Tester with the BMN Fractionator is given in table 3.3.

- **Tube flow fractionation**
  In the tube flow fractionator particles having a size range from 1 to 5000 µm can be fractionated. This corresponds to the entire size range of fibres and fines. The tube flow fractionation process, also called field flow separation process, uses the fractionation phenomena in flowing pulp suspensions. The particles in the pulp suspension are separated axially while flowing through the tube of the fractionator. This is due to differences in flow rate over the entire cross section of the tube, i.e. higher velocity at the center of the tube than near the wall of the tube. Smaller particles drift close to the tube walls, whereby bigger particles stay in the middle of the tube. The largest particles accumulate at the front end of the flow and the smallest ones at the rear of the flow (Laitinen (2011)).
  The fractionation principle of the tube flow fractionation is shown in figure 3.2. There are three steps during the fractionation process. At the beginning the sample is injected (see a) and all particles are distributed randomly in the tube. Over the entire length of the tube the separation of large and small particles develops more and more (see b). At the end of the fractionation process (see c) in figure 3.2, the larger particles come out first.

  Due to the small sample size (50 ml per run), this device was not suitable for further investigations in the course of this master thesis.

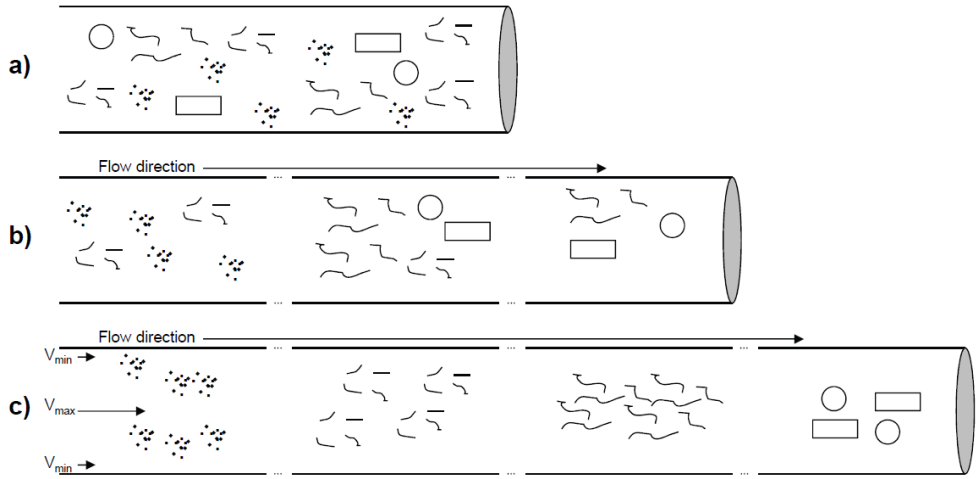

Figure 3.2 Schematic of a tube flow fractionation (Laitinen (2011))
a...at sample injection, b...at the start of fractionation, c...at end of fractionation

- **Bow screen and Attisholtz filter**
  In work done by Haugan et al. (2006) they produced TMP fines from Norway spruce for experiments with a bow screen (100-mesh) and an Attisholtz filter (200-mesh).

- **Dynamic Drainage Analyzer of Åbo Academi (DDÅA)**
  Lindqvist et al. (2011) investigated elemental chlorine free bleached (ECF) pine pulp. They removed fines for analytical measurements with a conventional Dynamic Drainage Jar (200-mesh) and determined the fines content with a BMN Fractionator (200-mesh). Furthermore, they used a custom built combined drainage analyzer and a sheet former, the so called Dynamic Drainage Analyzer of Åbo Academi (DDÅA). Differences compared to a conventional BDDJ Tester are the construction, such as a built-in computer control, adjustable conductivity and pH during measurements, etc.

3.2 Britt Dynamic Drainage Jar Tester (BDDJ)

The BDDJ Tester is a single-screen fibre classifier and can be used for all kinds of pulps. The screening procedure is the same for both chemical pulps and mechanical pulps, although the mass of the test portion vary. Furthermore, modifications concerning the sampling procedure were carried out (see chapter 3.2.6). The BDDJ Tester is described in the standard *SCAN-CM 66:05*. Figure 3.3 and figure 3.4 show the devices for the determination of fines content, which are available at the Institute for

Paper-, Pulp and Fibre Technology (IPZ). The BDDJ Tester and the BMN Fractionator (according to standard *SCAN-M 6:69*).

Figure 3.3 Britt Dynamic Drainage Drainage Jar Tester (BDDJ) at IPZ

Figure 3.4 Bauer McNett Fractionator (BMN) at IPZ

3.2.1 Measuring principle

In the first step the pulp sample is disintegrated in a laboratory disintegrator using deionized water. Afterwards the suspension is screened through the wire, which corresponds to a 200-mesh screen of the BMN Fractionator. This wire is a perforated plate with round holes with a diameter of 76 µm. Both, the material retained on the screen and that which passes through the screen are collected in beakers. In the next step the water is removed by using a Nutsche Filter which is equipped with a filter paper. After this the filter papers are dried and weighed separately. In the final step the fines content is calculated (see chapter 3.2.4).

It is important to mention that the mass of the test portion and the volume of water are different for mechanical pulps and chemical pulps (see table 3.2). It is important to mention that according to the *SCAN-CM 66:05* there is no significant difference in the results obtained using a perforated plate or a wire in the BDDJ Tester. The screening procedure can be used for all pulp samples. Provided that the sample can be fully disintegrated.

3.2.2 Sample preparation

In the first step the pulp sample is disintegrated according to the following ISO standards:

- *ISO 5263-1:2004* for chemical pulps

- *ISO 5263-2:2004* for mechanical pulps

The required pulp mass and the volume of water needed differ considerably between chemical pulp and mechanical pulp (see table 3.2). The reason for this is the fines content, which can be 10 times higher for mechanical pulp. The oven dry mass (OD) used for chemical pulps was chosen 5 g to guarantee a good precision even for pulps with very low fines content. Moreover, the total volume of water needed per test also differs between 0.5 and 5 liters.

Pulp grade	Oven-dry mass of test portion [g]	Total volume of water [ml]
Chemical pulp - fines content < 10 %	5.0 ± 0.5	5000
Mechanical pulp - fines content > 10 %	0.5 ± 0.1	2500

Table 3.2 Mass of test portion and volume of screening water for different pulps with the Britt Dynamic Drainage Jar Tester (BDDJ) (*SCAN-CM 66:05*)

3.2.3 Components and fractionation principle (*SCAN-CM 66:05*)

The screening effect results from the turbulence and the pressure developed by a stirrer which is equipped with a three-bladed propeller. The BDDJ Tester consists of a cylindrical sample holder which has a diameter of 100 (± 10) mm and an electrically driven propeller (see figure 3.5). The stirrer must be centered and the distance

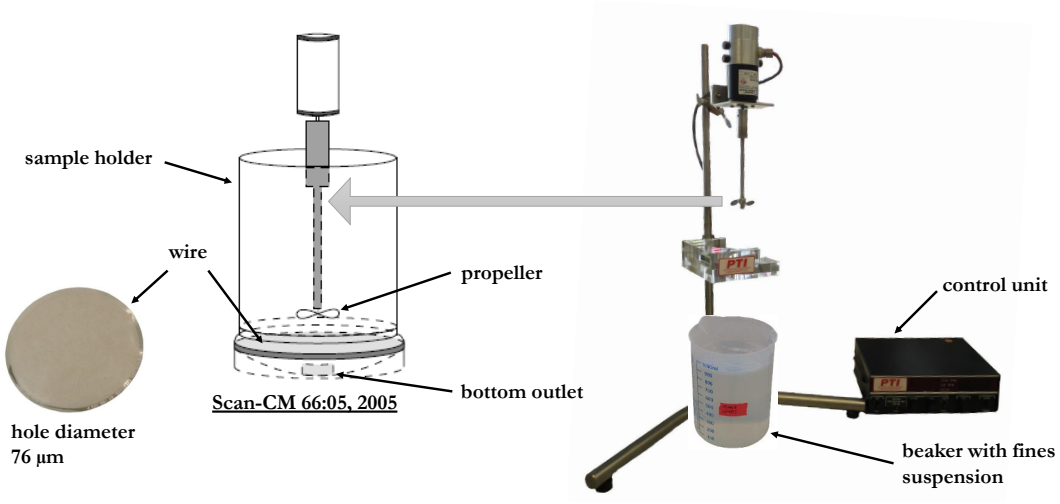

Figure 3.5 Detailed view of the BDDJ from the stirrer and the wire

between the propeller and the screen below it shall be 3.5 (± 1) mm. The rotation direction of the propeller has to be adjusted that the pressure of the propeller blades

is directed towards the screen. The speed of the propeller needs to be in a range of 750 ($\pm$ 50) rpm. A detailed view of the BDDJ Tester with sample holder and propeller is given in figure 3.5. The individual components of the BDDJ Tester are shown in figure 3.6. The screening procedure of each sample has to be carried out in duplicate.

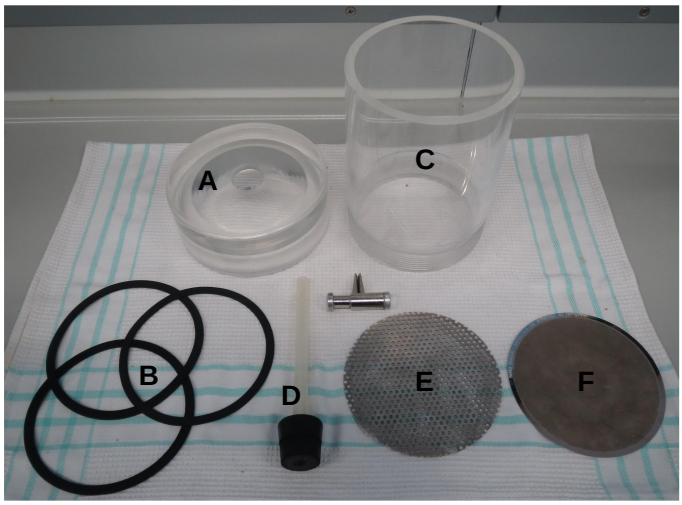

Figure 3.6 Components of the BDDJ Tester
(A)...Bottom of the sample holder, (B)...Gaskets, (C)...Sample holder, (D)...Outlet plug, (E)...Supporting plate, (F)...Wire (200-mesh)

The test portions have to be taken from a well stirred, disintegrated pulp suspension (for modifications carried out see chapter 3.2.6). Dilute the weighed test portion in a beaker up to 1000 ml and transfer it to the sample holder of the BDDJ Tester (bottom outlet closed). Set the stirrer in position above the screen and start it. Then open the outlet. Let the suspension drain through the wire into a beaker. As soon as the level of suspension in the jar is as low as 5 mm above the screen, add an additional portion (1 liter) of deionized water. Continue this procedure until the total volume of deionized water required (see table 3.2) has been used.

Finally, collect and filter both fractions obtained (fibres and fines) separately. Therefore use pre-weighed filter papers. Afterwards, dry and weigh the filter papers from both fibres and fines and calculate their oven-dry mass.

3.2.4 Calculation of fines content

The fines content can be calculated using equation 3.1 (according to *SCAN-CM 66:05*):

$$X = \frac{100 \cdot a}{a + b} \tag{3.1}$$

X ..... fines content of the investigated suspension [%]
a ..... oven-dry mass of the fines fraction [g]
b ..... oven-dry mass of fibre fraction [g]

The fines content of a given pulp sample is obtained as the mean value of the two or three measurements taken.

3.2.5 Pros and cons of BDDJ towards BMN

	Pros	Cons
BMN	bigger amounts of fines producible multiple fractions of fines per run	strong washing effect on fines uses large amounts of water large energy demand
BDDJ	determination of fines content small amounts of water needed small washing effect on fines	small amounts of fines producible

Table 3.3 Pros and cons of the Britt Dynamic Drainage Jar Tester (BDDJ) towards the Bauer McNett Fractionator (BMN)

3.2.6 Modified sampling procedure with the BDDJ Tester

Due to problems that came up during the determination of the fines content, the sampling procedure was modified. These modifications are as follows:

- **Difficulties to keep the variations of the sample mass within the range according to *SCAN-CM 66:05* standard:**
  At the beginning of this study, the required amount of pulp (see table 3.2) was given into a beaker by using a ladle. Due to this an accurate dosage of the test portion was quite difficult. In order to overcome this problem it has been decided to transfer the pulp after the disintegration step into a laboratory pulp distributor. This device is normally used for pulp suspensions which are used to produce laboratory handsheets. The distributor consists of a 10 liter beaker which is a equipped with a stirrer and an outlet valve at the bottom.
  Compared to the former procedure, the new method ensures a much easier and more accurate sampling. Another advantage of it is that the distribution of fibres and fines within the sample are much more uniform.

- **Dewatering problems when using mechanical pulps:**
  When using mechanical pulps the dewatering time for 1 liter of pulp suspension

was almost one hour. It is assumed that a filter cake is built on the surface of the wire and this in turn extended the dewatering process considerably. In comparison with the chemical pulp samples (dewatering time for most of them < 10 minutes) the dewatering time for mechanical pulps bore no relation to them.
To solve this problem the filter cake was removed from the wire after each liter. This was done by means of a spade shaped test tube cleaner. Due to this the dewatering time decreased from almost 60 minutes to less than 10 minutes per liter.

3.2.7 Modified trial versions of the BDDJ Tester

In order to produce bigger amounts of fines, tests with modified versions of the BDDJ Tester were performed. The reason for this was that the colleagues at BOKU in Tulln needed bigger amounts of fines for their tests. All modified versions of the tester were equipped with pumps at the bottom outlet with the aim to support the dewatering speed. The following modifications were tested:

- A BDDJ Tester supported by the pump of a BMN Fractionator and equipped with a 200-mesh wire from a BMN Fractionator (see figure 3.7)

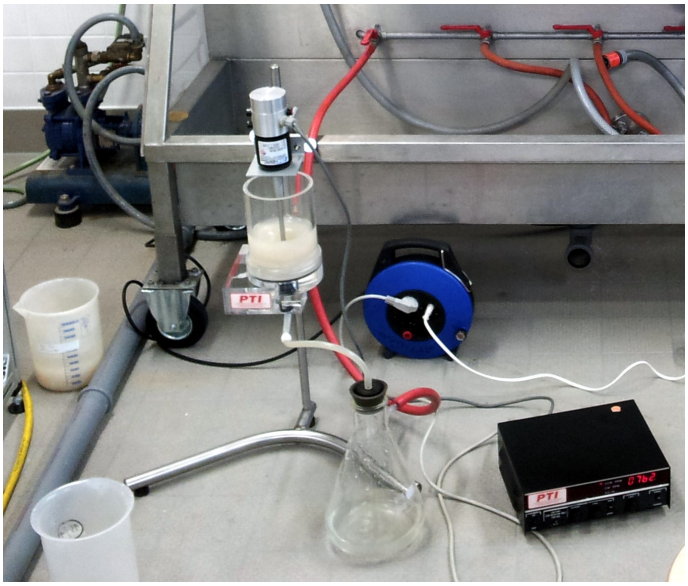

Figure 3.7 BDDJ Tester modified with the pump from a BMN Fractionator

- A BDDJ Tester supported by a laboratory pump and equipped with a 200-mesh wire from a BMN Fractionator
- Reconstruction of a Shopper Riegler (SR) device which is supported by a lab-

oratory pump and equipped with a 200-mesh wire from a Bauer McNett Fractionator (BMN).

There were no positive results obtained from these tests. The vacuum applied with the pumps could not speed up the dewatering process, the trials even showed the opposite. The dewatering time was extended instead of shortened. It is assumed that the application of vacuum promotes the formations of a filter cake on the surface of the wire which in turn has a negative effect on the dewatering process (already discussed in chapter 3.2.6).

3.3 Particle size distribution measurements

The Mastersizer 2000 particle size analyzer from Malvern Instruments GmbH was used to determine the particle size distribution (PSD) of fines suspensions. This device uses laser diffraction in order to measure the size of particles of either dry or wet samples (fines suspension). Figure 3.8 shows the Malvern Mastersizer 2000 at University of Graz (KFU), with which the PSD measurements were performed. The

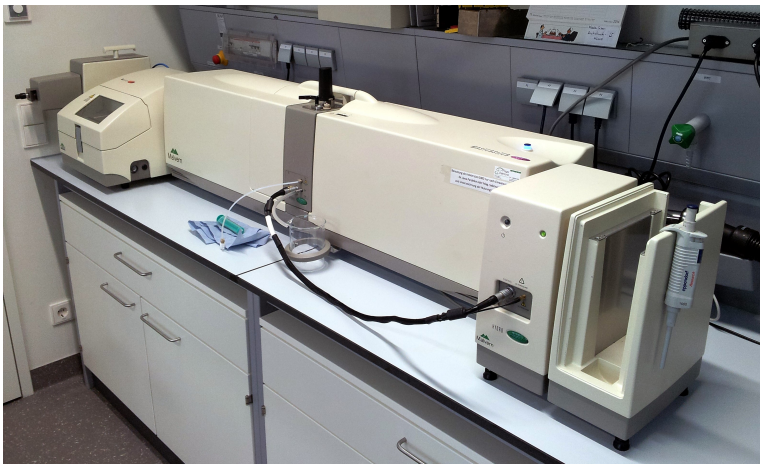

Figure 3.8 Malvern Mastersizer 2000 particle size analyzer at KFU Graz

intensity of the scattered light from laser a beam that passes through the particles is measured by various detectors. The sample dispersion unit is equipped with both a stirrer with adjustable rotation speed and an ultrasound unit with adjustable intensity. Ultrasound and stirring can be used in order to detach agglomerated particles (e.g. fines). The influence of both units on fines was tested and documented (see chapter 4.6).

The PSD of various fines suspensions of all industrial partners were investigated in order to validate the fines fractionation measurements obtained from the BDDJ Tester.

General information regarding the specification of the Malvern Mastersizer 2000 was taken from the user manual by Malvern Instruments (*(Mastersizer 2000 manual, 2005)*). The specification is shown in table 3.4.

Specification Mastersizer 2000	
Size range	0.02 - 2000 µm
Measurement principle	Mie Scattering
Detection systems	"blue light": wide angle forward and back scattering "red light": forward scattering, side scattering and back scattering
Light sources	"blue light": solid state light source "red light": helium neon laser
Optical alignment system	Automatic rapid align system with dark field optical reticle and multi- element alignment detector
Sample dispersion unit	Sample dispersion units automatically recogni- zed, configured and enabled on insertion of measurement cell cassettes into optical bench interchange system

Table 3.4 Specification of the optical bank in the Mastersizer 2000 *(Mastersizer 2000 manual, 2005)*

For investigations with the Mastersizer 2000 the following settings were used:

- only fines dispersions in deionized water were added (to the sample injection)
- fines dispersions were not sonicated before measurements (except for comparative measurements)
- non-spherical mode regarding particle shape
- the default light source used was "blue light"
- refractive indexes (RI) for cellulose 1.46 (and 1.53 for comparative measurements)

3.4 Microscopy of fines and filtrates

In several studies light microscopy was used in order to characterize both the size and shape of fines (Sundberg et al. (2003), Kangas and Kleen (2004), Chen et al. (2009), Retulainen et al. (2002)). Besides the investigation of fines morphology, light microscopy was used to study different fractions and to verify the reproducibility of measuring instruments, such as the Mastersizer 2000. Another possibility to investigate the morphology of fines apart from light microscopy is scanning electron microscopy(Sundberg et al. (2003), Kangas and Kleen (2004)).

In this thesis a Leica DM LM light microscope connected with a camera was used (see figure 3.9). Images were taken and stored on a computer. The results

Figure 3.9 Leica DM LM microscope at IPZ

of these investigations are shown in chapter 4.5. The basic functions of the light microscope include the reflected light and transmitted light mode. In case of the reflected light mode, the sample is illuminated from above whereas in transmitted light mode it is illuminated from the bottom side of the microscope. In both cases the sample is observed from above. Transmitted light microscopy provides information about the internal structure of transparent samples. It is used to investigate fibres as well as the morphology of particles. Reflected light microscopy is used to study the morphology of opaque surfaces (Bart (2006), Evans and Evans (2001)).

Furthermore, polarized light can also be used. In the work of Kappel et al. (2010), polarized light microscopy was used to determine the optical bonded area of fibre-

fibre bonds.

All images in this thesis were captured using transmitted light mode, because the image quality obtained was better compared to those obtained with incident light. The difference between oriented and crystalline materials is enhanced by using polarized light (Bart (2006)). Due to the fact that in the present study only the morphology of fibres and fines was of interest, the polarized light microscopy was not used.

Chapter 4

Results and Discussion

In this chapter the results of all samples are discussed. Each subchapter (4.1 to 4.4) includes the following results:

- Results from measurements of the fines content using the BDDJ Tester. Furthermore, the consistencies of all pulp samples and filtrates were determined. All in all 22 pulp and 24 filtrate samples were investigated.
- Mass balances of selected pulp and filtrate flows to get an estimate for the amount of fines. Two types of mass flows were investigated:
  - the mass flows in the circulation within the fibreline of the pulp mills
  - the mass flows which are transferred to the wastewater treatment plant

Calculation of mass flows of fines

The mass flows were calculated in order to estimate the amounts of fines. For calculating these mass flows the same procedure for all samples of each project partner was used. The calculation procedure is illustrated by means of a sample (pulp to wash press 1) from Sappi. All data of this sample are shown in table 4.3.

Information regarding the volume flows $\dot{V}_{pulp}$ were provided by the industrial partners. The consistency (cs) and the fines content (fc) content were determined within the frame of this thesis. Equations 4.1 to 4.3 show how the fines mass flow $\dot{m}_{fines}$ is calculated.

$$\dot{V}_{pulp} = \dot{V}_{total} \cdot cs = 936 \cdot \frac{3.18}{100} = 29.765 \ \frac{m^3}{h} \tag{4.1}$$

$$\dot{V}_{fines} = \dot{V}_{pulp} \cdot fc = 29.765 \cdot \frac{2.93}{100} = 0.87 \ \frac{m^3}{h} \tag{4.2}$$

$$\dot{m}_{fines} = \dot{V}_{fines} = 870 \ kg/h = 20859 \ \frac{kg}{d} \tag{4.3}$$

Since the consistencies of the filtrates are within a small range, the amount of fibres and fines are small as well. Due to this fact the determination of the fines content by means of the BDDJ Tester was not possible. Therefore a fines content of 50% was assumed. This value was used as a starting point to detect potentially interesting mass flows. For example if the calculated $\dot{m}_{fines}$ is still very small even though a fines content of 50% is used, it should be neglected because the actual fines content of filtrates is much lower.

- Discussion of the results

In chapter 4.5 light microscope images of fines suspended in filtrates and those, which were removed from the pulp using the BDDJ Tester are presented. Furthermore, in chapter 4.6 the results of the particle size distribution (PSD) measurements, using the Malvern Mastersizer 2000, are discussed. Finally, in chapter 4.7 a comparison of results of the company partners is given.

4.1 Sappi Austria Produktions-GmbH & Co KG

Sappi Austria Produktions-GmbH & Co KG produces sulphite pulp (magnefite pulping process). The following samples from Sappi were investigated:

- 5 pulp samples taken at different positions along the fibre line
- 8 filtrate samples
- 3 pulp samples from the pulp dewatering machine

Fines content & consistencies

Figure 4.1 shows a flow chart of the fibreline from Sappi. Both, the sampling points of the pulp and filtrate samples are shown. Table 4.1 contains the measurement results of consistency as well as the fines content (sampling points are shown in figure 4.1). The results show that the fines content of the pulp samples increases moderately along the fibreline. At the beginning of the fibreline, the fines content is 2.92% (Sappi_1010) and at the end it is in the range of 6.09% (Sappi_1014). An important remark is that the fines content significantly increases between the last two sampling points (Sappi_1013 = 3.76% and Sappi_1014 = 6.09%). From figure 4.2 it is apparent that the white water of the dewatering machine is used to dilute/wash the pulp after the pulp thickener. This might be an explanation for the increase in the fines content. Moreover, images of potentially affected filtrates show (see chapter 4.5.3) that they contain parts of fibres as well as fines.

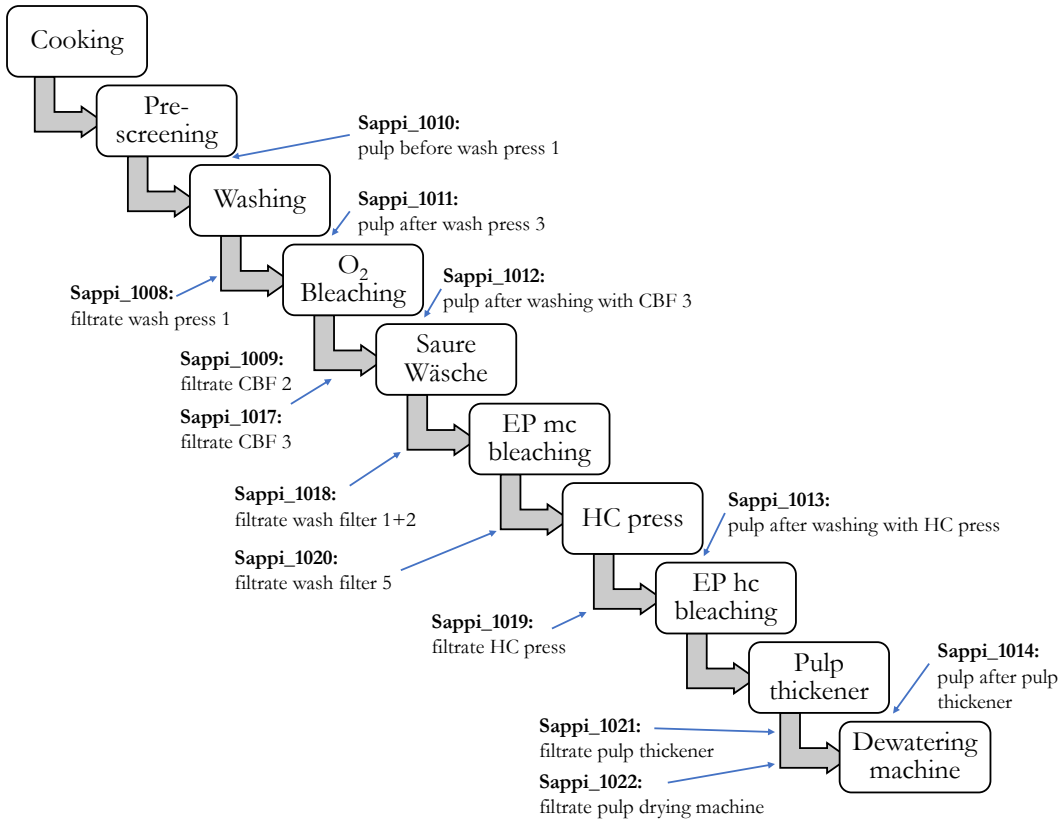

Figure 4.1 Flow chart of the fibreline at "Sappi Austria Produktions-GmbH & Co KG" with all sampling points of pulps and filtrates. Consistencies and fines content were measured

On this account three samples from the dewatering machine were taken and analyzed. The objective was to see if the circulation of white water has an influence on the fines content. Figure 4.2 shows a flow chart of the dewatering unit in which the sampling points of the three additional pulp samples are illustrated. These samples were needed to examine the influence of filtrates on the fines content of the pulp in the dewatering machine. Table 4.2 illustrates the results from consistency and fines content measurements of these three samples. The sample "Sappi_1024" was taken from the same sampling point as "Sappi_1014", but both samples differ in their fines content (4.73% vs 6.09%). An explanation is that they were taken at different dates (13th of November 2013 and 30th of January 2014). Therefore, the proportion of wood types used (spruce and beech) and further the fines content were different.

Apart from that, the assumption from before was confirmed. Table 4.2 shows that the fines content varies considerably between the sampling points. The pulp of the pulp thickener is diluted before the headbox of the dewatering machine. The white water, which is used for dilution to headbox consistency most likely contains

Sampling point	Name	Consistency [%]	Fines content [%]
Pulp			
Sappi_1010	pulp before wash press 1	3.18	2.92
Sappi_1011	pulp after wash press 3	35.42	3.42
Sappi_1012	pulp after CBF 3	17.44	3.45
Sappi_1013	pulp after HC press	34.22	3.76
Sappi_1014	pulp after pulp thickener	2.32	6.09
Filtrates			
Sappi_1008	filtrate wash press 1	0.0189	-
Sappi_1009	filtrate CBF 2	0.0207	-
Sappi_1017	filtrate CBF 3	0.0163	-
Sappi_1018	filtrate wash filter 1+2	0.0043	-
Sappi_1019	filtrate HC press	0.0236	-
Sappi_1020	filtrate wash filter 5	0.0050	-
Sappi_1021	filtrate pulp thickener	0.0059	-
Sappi_1022	filtrate pulp drying machine	0.0190	-

Table 4.1 Consistencies and fines content of pulps and filtrates from "Sappi Austria Produktions-GmbH & Co KG"

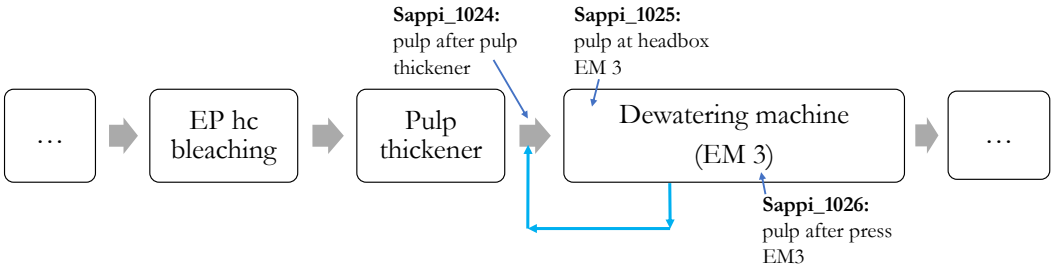

Figure 4.2 Flow chart of the dewatering machine at "Sappi Austria Produktions-GmbH & Co KG" with the sampling points of three additional pulps investigated. Consistencies and fines content were measured.

huge amounts of fines. This means that fines are introduced by filtrates at this stage. Furthermore, it is apparent that the fines content decreases again considerably after the dewatering machine.

Sampling point	Name	Consistency [%]	Fines content [%]
Pulp			
Sappi_1024	after pulp thickener	19.06	4.73
Sappi_1025	at headbox dewatering machine (EM3)	1.94	6.32
Sappi_1026	after press dewatering machine (EM3)	45.99	4.40

Table 4.2 Consistencies and fines content of the dewatering machine "Sappi Austria Produktions-GmbH & Co KG"

Mass balance of pulp and filtrate flows

In table 4.3 the results of the mass balance of flows in the circulation sytem within the fibreline of the pulp mill at Sappi are shown. Data of all pulp and filtrates samples were provided by Sappi.

Considerable amounts of fines in pulps and filtrates can be expected according to the these calculations. The results range from 1934 to 24128 $\frac{kg\ \ fines}{day}$. Especially the amount in the filtrates of wash press 1+2+3 is remarkable. The mass flow of fines calculated for this filtrate is 1934 $\frac{kg\ \ fines}{day}$.

Sampling point	$\dot{V}_{total}$ $[m^3/h]$	cs [%]	$\dot{V}_{pulp}$ $[m^3/h]$	fc [%]	$\dot{V}_{fines}$ $[m^3/h]$	$\dot{m}_{fines}$ $[kg/h]$	$\dot{m}_{fines}$ $[kg/d]$
Pulp							
to wash press 1	936.0	3.18	29.765	2.92	0.87	870.00	20859
from wash press 3	82.8	35.4	28.311	3.43	1.01	1005.37	24129
total pulp from bleaching	27.36	34.22	9.363	3.762	0.35	352.22	8453
pulp production dewatering machine	20.8	20.18	4.204	6.098	0.26	256.37	6152
Filtrates							
filtrate wash press 1+2+3	852.7	0.0189	0.161	*50*	0.08	80.58	1934

Table 4.3 Mass balance of fines in pulps and filtrates (in the circulation) from "Sappi Austria Produktions-GmbH & Co KG"
cs...consistency, fc...fines content

Table 4.4 contains the results of the calculated amounts of fines, which were transferred in direction to the wastewater treatment plant (ARA). Even though a fines

Sampling point	$\dot{V}_{total}$ $[m^3/h]$	cs [%]	$\dot{V}_{pulp}$ $[m^3/h]$	fc [%]	$\dot{V}_{fines}$ $[m^3/h]$	$\dot{m}_{fines}$ $[kg/h]$	$\dot{m}_{fines}$ $[kg/d]$
filtrate from bleaching	0.52	0.0161	0.0001	*50*	0.00004	0.04	1

Table 4.4 Mass balance of fines in filtrates (to ARA) from "Sappi Austria Produktions-GmbH & Co KG"
cs...consistency, fc...fines content, ARA...wasterwater treatment plant

content of 50% is assumed, only 1 $\frac{kg\ \ fines}{day}$ is transferred to the wastewater treatment plant. Due to the fact that the true fines content will be much lower than 50%, this mass flow do not provide a suitable amount, which can be used for other applications.

4.2 Norske Skog Bruck GmbH

Norske Skog Bruck GmbH produces pressure groundwood (PGW) on site. The following samples from Norske Skog were investigated:

- 3 pulp samples taken at different positions along the fibre line
- 7 filtrate samples

Fines content & consistencies

Figure 4.3 illustrates a flow chart of the fibreline at Norske Skog Bruck. Both, the sampling points of the pulp and filtrate samples are shown.

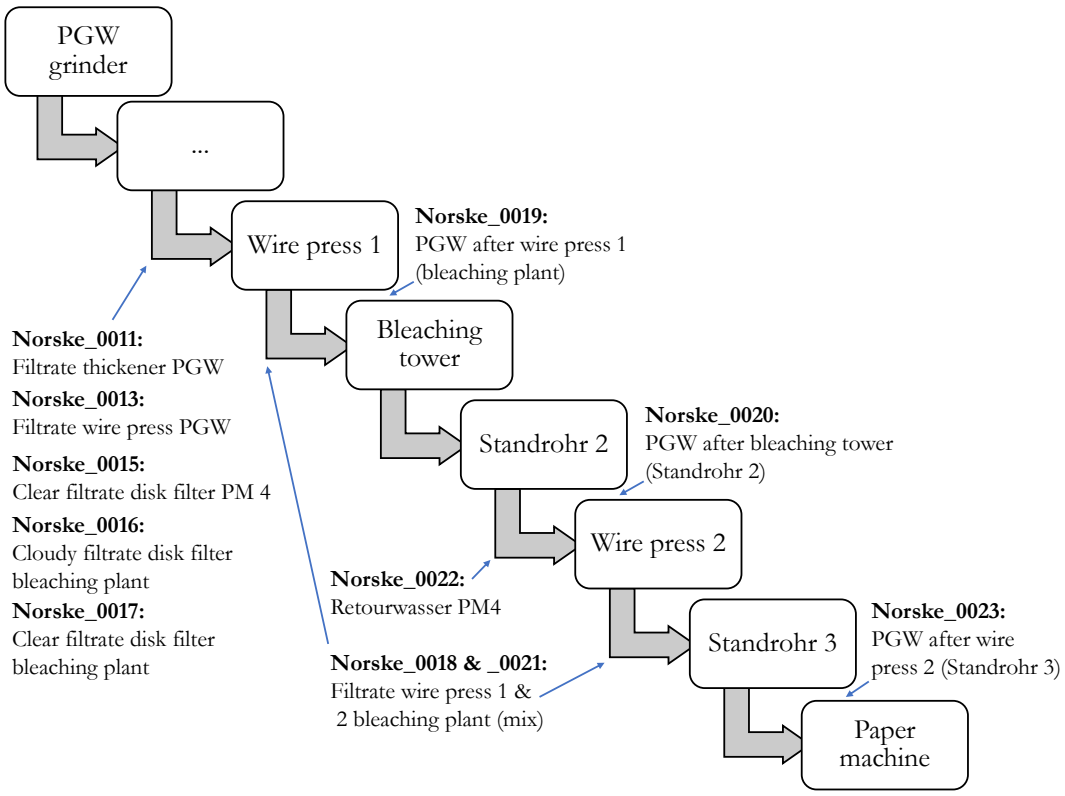

Figure 4.3 Flow chart of "Norske Skog Bruck GmbH" with all sampling points of pulps and filtrates. Consistencies and fines content were measured

Table 4.5 shows the results of consistency and fines content measurements (sampling points are shown in figure 4.3). The results show that the fines content of the three pulp samples firstly increases moderately and then decreases again. The first pulp sample was taken right before the bleaching tower after the wire

press (Norske_0019 = 23.48%). Samples two (Norske_0020 = 25.86%) and three (Norske_0023 = 25.02%) were taken after the bleaching tower. During the bleaching process, a slight increase of the fines content was observed (before/after: 23.48%/25.86%). This is in accordance to the results of the work done by Karlsson and Agnemo (2010). They found out that the fines content in pulps increases with increased bleaching intensity (for hydrogen peroxide bleaching). At the end of the fibreline the fines content decreases again (from 25.86% to 25.02%). The reason therefore might be the washing and subsequent thickening of the pulp in "wire press 2" between the two sampling points (see figure 4.3).

Sampling point	Name	Consistency [%]	Fines content [%]
Pulp			
Norske_0019	PGW after wire press 1 (bleaching plant)	27.22	23.48
Norske_0020	PGW after bleaching tower (Standrohr 2)	7.98	25.86
Norske_0023	PGW after wire press 2 (Standrohr 3)	9.92	25.02
Filtrates			
Norske_0011	Filtrate thickener PGW	0.0105	-
Norske_0013	Filtrate wire press PGW	0.0282	-
Norske_0015	Clear filtrate disk filter PM 4	0.0044	-
Norske_0016	Cloudy filtrate disk filter bleaching plant	0.0110	-
Norske_0017	Clear filtrate disk filter bleaching plant	0.0082	-
Norske_0018 & _0021	Filtrate wire press 1 & 2 bleaching plant (mix)	0.1194	-
Norske_0022	Returning water PM 4	0.0980	-

Table 4.5 Consistencies and fines content of pulps and filtrates "Norske Skog Bruck GmbH"

Mass balance of pulp and filtrate flows

In table 4.6 the results of the mass balance of flows in the circulation system within the fibreline of the pulp mill at Norske Skog Bruck are shown. Data of all pulp and filtrates samples were provided by Norske Skog.

According to the calculations, huge amounts of fines in pulps (because of the high

levels of fines in mechanical pulps) and smaller amounts of fines in filtrates can be expected. The values range from 108 to 73208 $\frac{kg\ fines}{day}$. Especially the fines content in the filtrate of the water recirculated from the paper machine 4 (PM 4) is significant. The mass flow of fines calculated for this filtrate is 1148 $\frac{kg\ fines}{day}$.

Sampling point	$\dot{V}_{total}$ $[m^3/h]$	cs [%]	$\dot{V}_{pulp}$ $[m^3/h]$	fc [%]	$\dot{V}_{fines}$ $[m^3/h]$	$\dot{m}_{fines}$ $[kg/h]$	$\dot{m}_{fines}$ $[kg/d]$
Pulp							
PGW to PM 3 (unbleached)	55.4	2.50	1.384	23.48	0.33	325.05	7801
PGW to PM 4 (bleached)	487.7	2.50	12.192	25.02	3.05	3050.36	73208
Filtrates							
Recirculated water from PM 4	97.6	0.0980	0.096	*50*	0.05	47.84	1148
Clear filtrate from PM 4	206.3	0.0044	0.009	*50*	0.005	4.54	108

Table 4.6 Mass balance of fines in pulps and filtrates (in the circulation) from "Norske Skog Bruck GmbH"
cs...consistency, fc...fines content

Table 4.7 contains the results of the balanced amounts of fines, which were transferred in direction to the wastewater treatment plant (ARA).

Sampling point	$\dot{V}_{total}$ $[m^3/h]$	cs [%]	$\dot{V}_{pulp}$ $[m^3/h]$	fc [%]	$\dot{V}_{fines}$ $[m^3/h]$	$\dot{m}_{fines}$ $[kg/h]$	$\dot{m}_{fines}$ $[kg/d]$
Clear filtrate bleaching	108.5	0.0082	0.009	*50*	0.004	4.45	107

Table 4.7 Mass balance of fines in filtrates (to ARA) from "Norske Skog Bruck GmbH"
cs...consistency, fc...fines content, ARA...wasterwater treatment plant

The fines content for all filtrates was assumed to be 50%. According to the calculations 107 $\frac{kg\ fines}{day}$ are transferred to the wastewater treatment plant. This clear filtrate is introduced from the disk filter of the bleaching plant. Even though its consistency is very low (0.0082%), this mass flow of fines could provide a suitable amount to be used for other applications. It is however questionable whether the fractionation of fines from filtrates is worthwhile from an investment and energy cost point of view.

4.3 Zellstoff Pöls AG

At Zellstoff Pöls AG kraft/sulphate pulp is produced. The following samples from Zellstoff Pöls AG were investigated:

- 7 pulp samples taken at different positions along the fibre line
- 5 filtrate samples

Fines content & consistencies
Figure 4.4 gives an overview of the fibreline at Zellstoff Pöls. Both, the sampling points of the pulp and filtrate samples are shown.

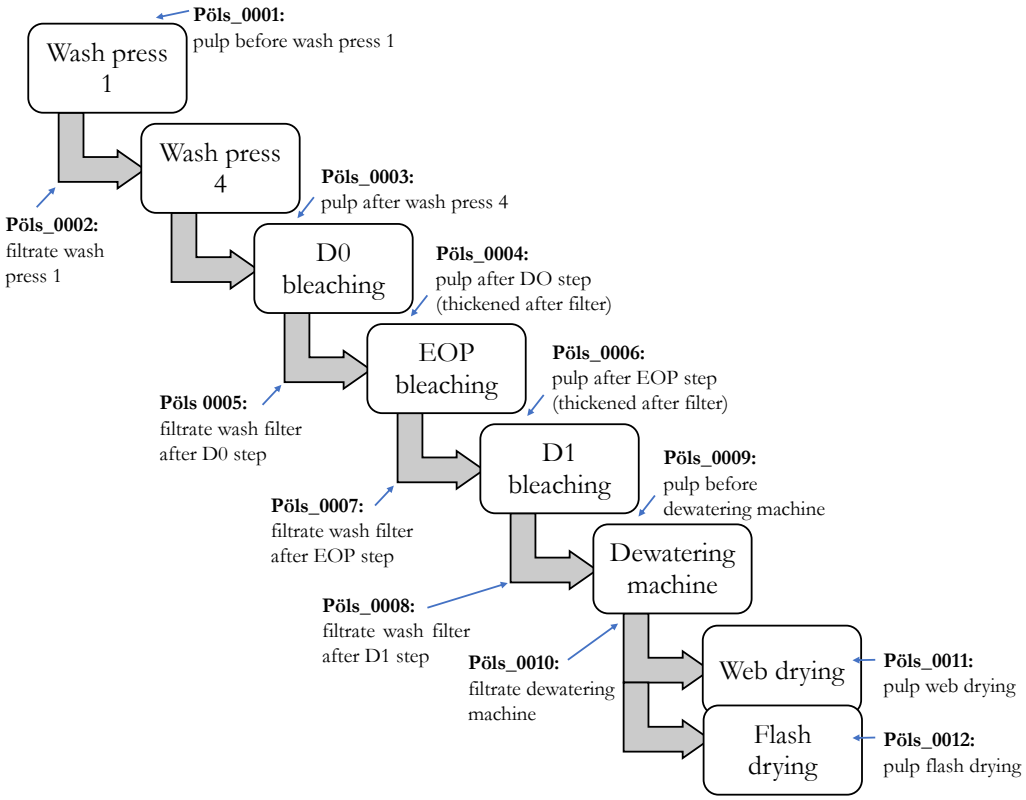

Figure 4.4 Flow chart of "Zellstoff Pöls AG" with all sampling points of pulps and filtrates. Consistencies and fines content were measured

Table 4.8 shows the results of consistency and fines content measurements (sampling points are shown in figure 4.4). From the results it is apparent that the pulp before wash press 1 has the highest fines content (Pöls_0001 = 6.02%). It is assumed that the high fines content originates from extractives and colloidal substances of the

cooking process before. These substances mostly get washed out in the subsequent wash presses.

After wash press 4 the fines content decreases considerably (Pöls_0003 = 3.22%) and in the subsequent bleaching steps there is again a slight decrease. The fines content after the D0 step (Pöls_0004 = 2.91%) and after the EOP step (Pöls_0006 = 2.85%) are similar. The next sampling point is located before the dewatering machine (Pöls_0009 = 4.56%). Here the fines content increases again. Finally, the pulp is split up into two lines: The web drying (Pöls_0011 = 4.22%) and the flash drying unit (Pöls_0012 = 4.68%). The fines content is in the same range for both drying lines.

Sampling point	Name	Consistency [%]	Fines content [%]
Pulp			
Pöls_0001	before wash press 1	2.88	6.02
Pöls_0003	after wash press 4	34.07	3.22
Pöls_0004	after D0 step	14.01	2.91
Pöls_0006	after EOP step	14.02	2.85
Pöls_0009	before dewatering machine	1.99	4.56
Pöls_0011	web drying	94.46	4.22
Pöls_0012	flash drying	88.39	4.68
Filtrates			
Pöls_0002	wash press 1	0.0213	-
Pöls_0005	after D0 step	0.0036	-
Pöls_0007	after EOP step	0.0004	-
Pöls_0008	after D1 step	0.0023	-
Pöls_0010	dewatering machine	0.0133	-

Table 4.8 Consistencies and fines content of pulps and filtrates "Zellstoff Pöls AG"

Mass balance of pulp and filtrate flows

In table 4.9 the results of the mass balance flows in the circulation system within the fibreline of the pulp mill at Zellstoff Pöls are shown. Data of all pulp and filtrates samples were provided by Zellstoff Pöls.

From the results it is apparent that the mass flows of fines decrease considerably between the first (Pöls_0001 = 84298 $\frac{kg\ fines}{day}$) and the last two samples (Pöls_0011 + Pöls_0012 = 24396 + 7049 = 31445 $\frac{kg\ fines}{day}$). It is assumed that huge amounts of fines are washed out in various process steps along the fibreline. The mass flows in some

filtrates (Pöls_0002, Pöls_0005, Pöls_0010) are significant as well, but those are part of the circulation, which means that they are not removed from the process.

Sampling point	$\dot{V}_{total}$ $[m^3/h]$	cs [%]	$\dot{V}_{pulp}$ $[m^3/h]$	fc [%]	$\dot{V}_{fines}$ $[m^3/h]$	$\dot{m}_{fines}$ $[kg/h]$	$\dot{m}_{fines}$ $[kg/d]$
Pulp							
Pöls_0001	2025.9	2.88	58.346	6.02	3.51	3512.42	84298
Pöls_0003	168.7	34.07	57.476	3.22	1.85	1850.73	44418
Pöls_0004	292.8	14.01	41.021	2.91	1.19	1193.72	28649
Pöls_0006	336.6	14.02	47.191	2.85	1.34	1344.95	32279
Pöls_0009	1844.2	1.99	36.700	4.56	1.67	1673.50	40164
Pöls_0011	25.5	94.46	24.087	4.22	1.02	1016.48	24396
Pöls_0012	7.1	88.39	6.276	4.68	0.29	293.70	7049
Filtrates							
Pöls_0002	1877.3	0.0213	0.400	*50*	0.20	199.93	4798
Pöls_0005	2837.8	0.0036	0.102	*50*	0.05	51.08	1226
Pöls_0007	2680.8	0.0004	0.011	*50*	0.01	5.36	129
Pöls_0008	2672.1	0.0023	0.061	*50*	0.03	30.73	737
Pöls_0010	1720.0	0.0133	0.229	*50*	0.11	114.38	2745

Table 4.9 Mass balance of fines in pulps and filtrates (in the circulation) from "Zellstoff Pöls AG"
cs...consistency, fc...fines content

Table 4.10 shows the results of the balanced amounts of fines, which were transferred in direction to the wastewater treatment plant (ARA).

Sampling point	$\dot{V}_{total}$ $[m^3/h]$	cs [%]	$\dot{V}_{pulp}$ $[m^3/h]$	fc [%]	$\dot{V}_{fines}$ $[m^3/h]$	$\dot{m}_{fines}$ $[kg/h]$	$\dot{m}_{fines}$ $[kg/d]$
from wash press 4	90.0	0.0036	0.0032	*50*	0.0016	1.62	39
from Pöls_0005	179.6	0.0036	0.0065	*50*	0.0032	3.23	78
from Pöls_0007	287.6	0.0004	0.0012	*50*	0.0005	0.58	14
from Pöls_0008	120.4	0.0023	0.0028	*50*	0.0013	1.38	33
from dewatering machine	180.4	0.0133	0.0240	*50*	0.0120	12.00	288

Table 4.10 Mass balance of fines in filtrates (to ARA) from "Zellstoff Pöls AG"
cs...consistency, fc...fines content, ARA...wasterwater treatment plant

According to the calculations only the mass from the dewatering machine (288 $\frac{kg\ fines}{day}$) could provide a suitable amount to be used for other applications. It is however questionable whether the fractionation of fines from filtrates is worthwhile from an investment and energy cost point of view.

4.4 Mondi Frantschach GmbH

The Mondi Frantschach GmbH pulp mill produces kraft/sulphate pulp. The following samples from Mondi Frantschach GmbH investigated:

- 4 pulp samples taken at different positions along the fibre line
- 4 filtrate samples

Fines content & consistencies
Figure 4.5 presents a flow chart of the fibreline at Frantschach. Both, the sampling points of the pulp and filtrate samples are shown.

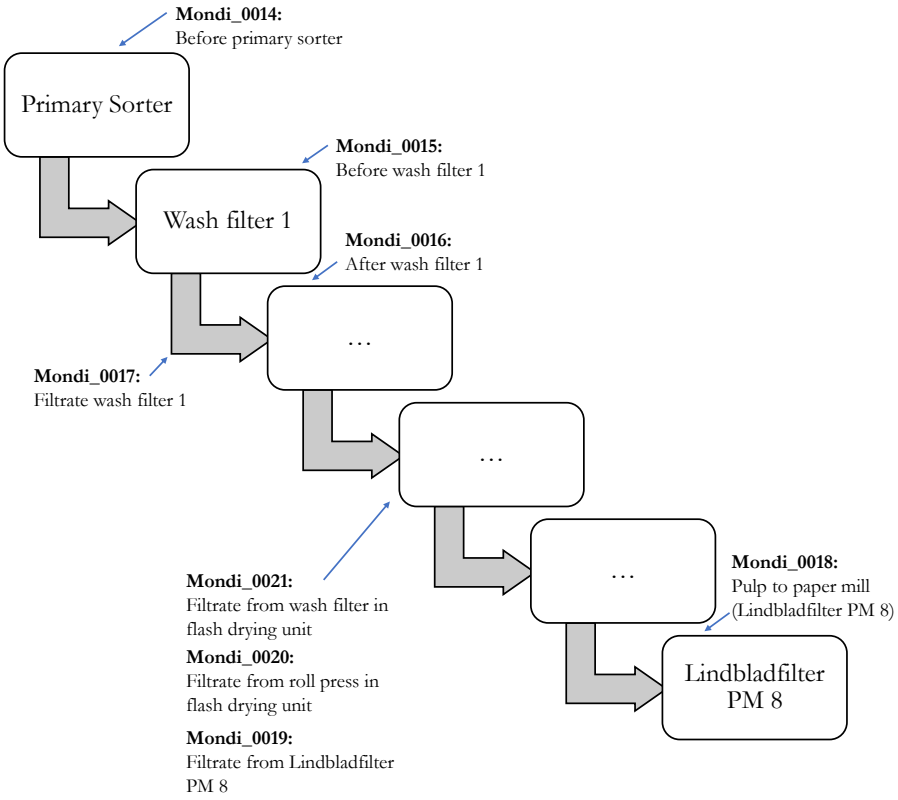

Figure 4.5 Flow chart of "Mondi Frantschach GmbH" with all sampling points of pulps and filtrates. Consistencies and fines content were measured

Table 4.11 shows the results of consistency and fines content measurements (sampling points are shown in figure 4.5). The results show that the fines content of the pulp samples is very similar at the first three sampling points (Mondi_0014 = 3.50, Mondi_0015 = 3.88 and Mondi_0016 = 3.73%). The subsequent sampling point shows an increase in fines content (Mondi_0018 = 5.06%). No conclusions could be drawn

why the fines content increased at this point on the basis of flow charts provided by Mondi. The pulp at this point is transferred to the paper mill.

Sampling point	Name	Consistency [%]	Fines content [%]
Pulp			
Mondi_0014	before primary sorter	2.53	3.50
Mondi_0015	before wash filter 1	1.45	3.88
Mondi_0016	after wash filter 1	12.75	3.73
Mondi_0018	to paper mill (Lindbladfilter PM 8)	4.71	5.06
Filtrates			
Mondi_0017	wash filter 1	0.00008	-
Mondi_0019	from Lindbladfilter PM 8	0.00347	-
Mondi_0020	roll press in flash drying unit	0.03167	-
Mondi_0021	wash filter in flash drying unit	0.00044	-

Table 4.11 Consistencies and fines content of pulps and filtrates "Mondi Frantschach GmbH"

Mass balance of pulp and filtrate flows

In table 4.12 the results of the mass balance of flows in the circulation system within

Sampling point	$\dot{V}_{total}$ $[m^3/h]$	cs [%]	$\dot{V}_{pulp}$ $[m^3/h]$	fc [%]	$\dot{V}_{fines}$ $[m^3/h]$	$\dot{m}_{fines}$ $[kg/h]$	$\dot{m}_{fines}$ $[kg/d]$
Pulp							
to paper mill	13.70	4.71	0.645	5.06	0.0327	32.65	784
before wash filter 1	267.31	1.45	3.876	3.88	0.1504	150.39	3609
after wash filter 1	30.40	12.75	3.876	3.73	0.1446	144.57	3470
Filtrates							
wash filter 1	236.91	0.00008	0.0002	*50*	0.0001	0.095	2
Filtratbehälter 1	720.00	0.00008	0.001	*50*	0.0003	0.288	7

Table 4.12 Mass balance of fines in pulps and filtrates (in the circulation) from "Mondi Frantschach GmbH"
cs...consistency, fc...fines content

the fibreline of the pulp mill at Mondi are shown. Data of all pulp and filtrates

samples were provided by Mondi.

According to the these calculations very small amounts of fines can be expected for filtrates. The results are in the range of 2 and 7 $\frac{kg\ \ fines}{day}$. The amount of fines in the pulp samples is significantly higher (784 and 3609 $\frac{kg\ \ fines}{day}$).

Finally, no information was obtained from Mondi regarding volume flows, which are transferred in direction to the wastewater treatment plant (ARA).

4.5 Microscopy of fibres and fines

In this section images of fibres, fines and filtrates are illustrated. The initial trials using a light microscope are a first step towards image analysis, which is one of the topics within the Flippr° project. In this thesis the microscopy was used to compare different fines from different types of pulp. Furthermore, fines fractions obtained were compared with results of previous studies.

All images were captured using transmitted light mode. Polarizing filters were not used. A magnification of 10x was used (detailed information about light microscopy is given in chapter 3.4).

4.5.1 Fibres

Images of fibres were taken in order to illustrate how pulp fibres differ in their appearance from one pulping process to another. The figures show unbeaten fibres after fractionation with the BDDJ Tester, i.e. the primary fines were removed from the samples.

Figure 4.6 shows unbeaten kraft pulp fibres from Mondi (Mondi_0018). Figure 4.7 presents pressure groundwood (PGW) pulp fibres from Norske Skog (Norske_0023). These two samples were investigated in order to illustrate the difference between chemical and mechanical pulp fibres. Mechanical pulp fibres are much stronger fib-

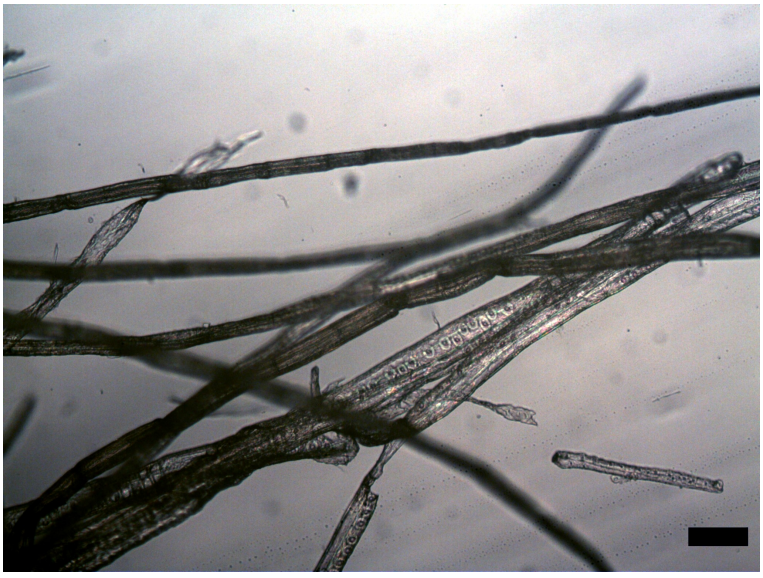

Figure 4.6 Unbeaten kraft pulp fibres without primary fines (Mondi_0018)
Magnification: 10x, bar at bottom right = 100 µm

rillated and shortened than chemical pulp fibres, which is a result of the groundwood pulping process.

Since all industrial partners mainly use spruce as raw material for their pulping

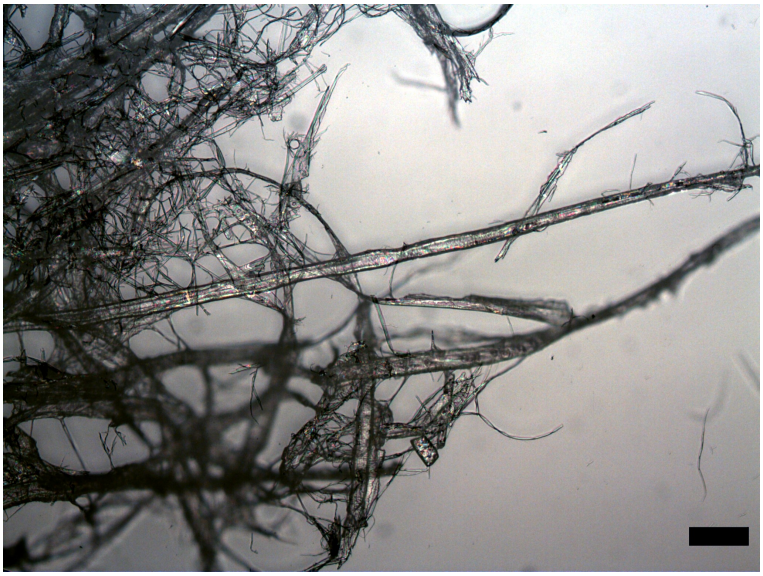

Figure 4.7 Unbeaten PGW pulp fibres without primary fines (Norske_0023) Magnification: 10x, Bar at bottom right = 100 µm

processes, images from Sappi and Zellstoff Pöls are similar to those from Mondi and were therefore not included here. Apart from spruce the industrial partners also use smaller quantities of fir (Norske Skog), beech (Sappi) and pine (Zellstoff Pöls and Mondi).

4.5.2 Fines

In this subsection, representative images of fines of each investigated pulp are presented (see figures 4.8 to 4.11). Images of primary fines suspended in filtrate after fractionation with the BDDJ Tester were taken. The results regarding size and morphology are in accordance with those obtained in previous studies (see chapter 2).

Primary fines from chemical pulps comprise short or broken fibres or parts of it. Furthermore, they consist mainly of ray cells, parenchyma cells and vessel elements and generally have a coarser and a finer fraction. Moreover, they are rectangular objects with a length of 30 - 60 µm and a width of 10 - 30 µm, which results in a length-to-thickness ratio of less than five. (Yin et al. (2013), Chen et al. (2009), Cole et al. (2008)).

In contrast, mechanical pulps contain a large number of primary fines. Many fibre fragments are produced, such as flakes, fibrils, lamellae, pores and ray cells. The two main fragments of mechanical pulp fines are fibrillar and flake-like material. Regarding morphology, mechanical pulp fines are more heterogenous than those of chemical pulps. Bands of fibrillar material can even be as long as 100 - 250 µm. (Krogerus et al. (2002), Pruden (2005), Luukko (1999)).

As can be seen from the figures 4.8 to 4.10 all chemical pulp fines look similar. Their two main fractions can be easily identified: The first one is the coarse fraction rich in ray cells and the other one is the finer fraction, which comprises fibrils and lamellae (see chapter 2.4.1).

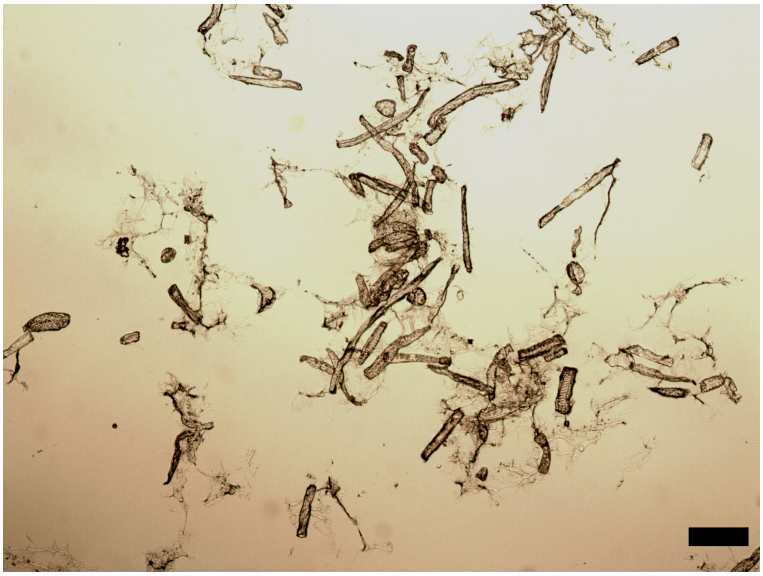

Figure 4.8 Fines from Sappi Austria Produktions-GmbH & Co KG (Sappi_1011)
Magnification: 10x, Bar at bottom right = 100 µm

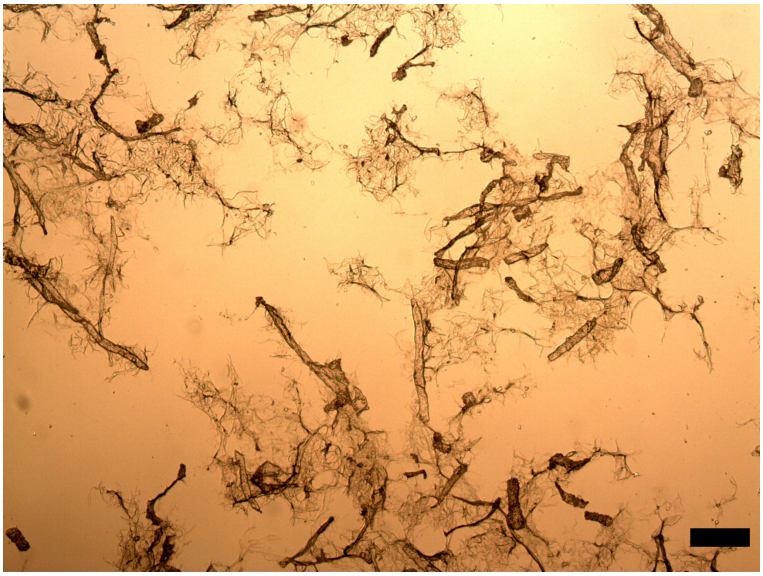

Figure 4.9 Fines from Zellstoff Pöls AG (Pöls_1012)
Magnification: 10x, Bar at bottom right = 100 µm

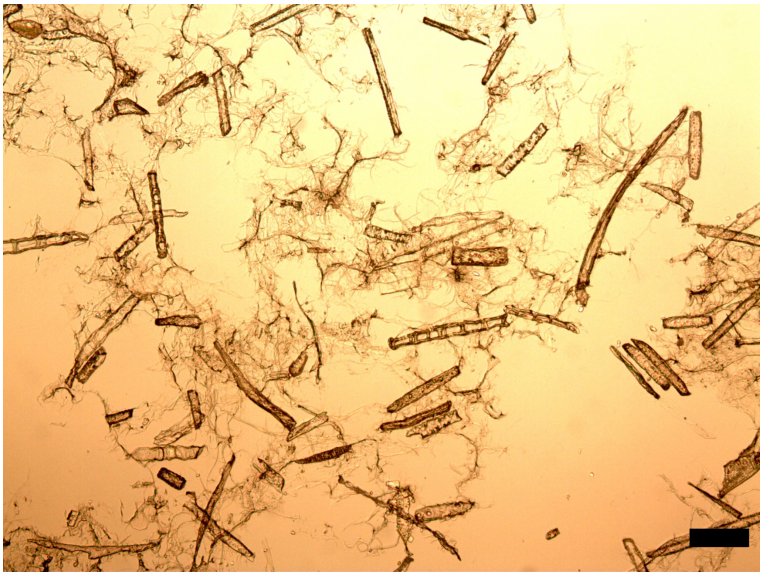

Figure 4.10 Fines from Mondi Frantschach GmbH (Mondi_0018)
Magnification: 10x, Bar at bottom right = 100 µm

From figure 4.11 it is apparent that mechanical pulp fines differ considerably in comparison to chemical pulp fines. Besides the two main types of fragments, which are fibrillar and flake-like material. They comprise more different types of particles, these are produced during mechanical pulping (see chapter 2.4.2).

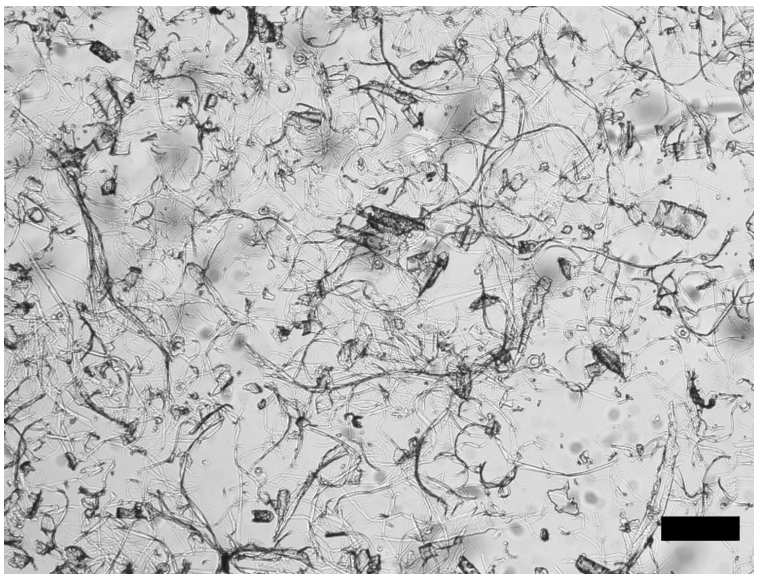

Figure 4.11 Fines from Norske Skog Bruck GmbH
Magnification: 10x, Bar at bottom right = 100 µm

4.5.3 Fibres and fines in filtrates

Due to the reason described in chapter 4.1 (recirculation of white water from dewatering unit), only filtrates from Sappi were analyzed. It was assumed that the filtrates contain bigger amounts of fine material and fines, which are introduced by white water from the dewatering machine downstream.

Figure 4.12 shows the filtrate of the high consistency press (Sappi_1019), which contains a large amounts of fines. Even pieces of fibres can be identified. Figure 4.13

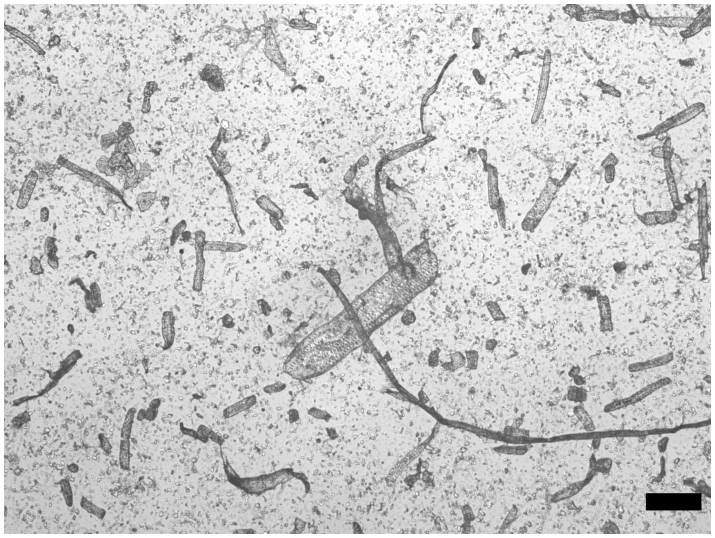

Figure 4.12 Image of filtrate "Sappi_1019"
Magnification: 10x, Bar at bottom right = 100 µm

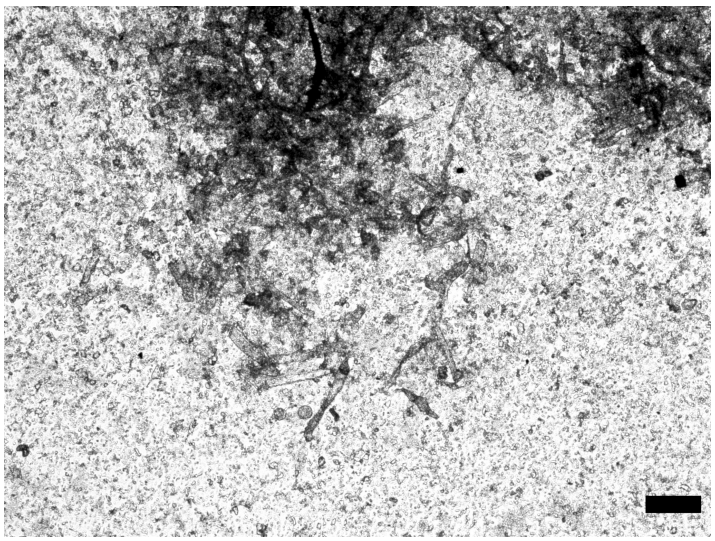

Figure 4.13 Image of filtrate "Sappi_1022"
Magnification: 10x, Bar at bottom right = 100 µm

shows the filtrate of the pulp drying machine (Sappi_1022). This filtrate also contains large amounts of fine material too. In this case the composition of the filtrate is not known and due to this it is difficult to distinguish between fibre fines and other materials. To clarify this question further investigations are needed. Figure 4.14 shows the filtrate of the pulp thickener (Sappi_1021). In comparison to the other filtrate samples

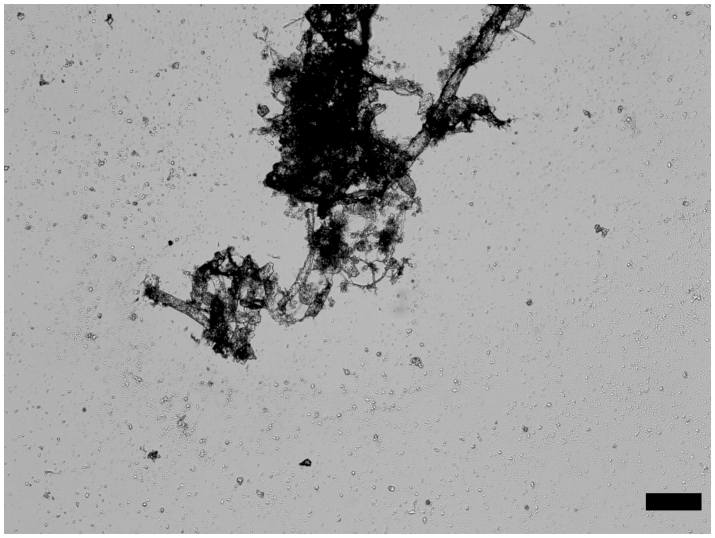

Figure 4.14 Image of filtrate "Sappi_1021"
Magnification: 10x, Bar at bottom right = 100 µm

it contains much less material. An important remark is that the fines and fibre fragments shown in the image tend to built agglomerates. These agglomerates have an influence on particle size measurements (see chapter 4.6).

4.6 Particle size distributions

The particle size distributions of fines were analysed with a Malvern Mastersizer 2000 (detailed description is given in chapter 3.3). Fines samples from all industrial partners were investigated. The diagrams show the influence of settings, which were adjusted during the measurements:

- Influence of the revolutions (r.p.m.) of the stirrer in the measuring chamber (see figures 4.15, 4.17 and 4.18)
- Influence of the refractive index (RI). It was either RI = 1.46 or RI = 1.53 (see figures 4.21, 4.20 and 4.19)
- Influence of sonication. Measurements were taken with and without ultrasound unit (see figures 4.22 and 4.23)

The settings of the Mastersizer 2000 were adjusted in order to estimate the influence of the 3 mentioned parameters on the measurement results. A first trial using 1750 r.p.m. obviously led to wrong results. The results using default settings (revolutions) showed a volume fraction up to a particle size of 800 µm (see figure 4.15). The same behavior was noticed for samples of Norske Skog (figure 4.17) and, to a very small extent for samples from Mondi Frantschach (figure 4.18). The sample was fractionated with the BDDJ using a wire which corresponds to a 200 mesh screen. Due to this it is almost impossible to have such large particles inside this fraction. It is assumed that the fines tend to build agglomerates. Therefore it has been decided to increase the revolutions per minute. From figure 4.15 it is apparent that larger particles disappear when using higher revolutions of the stirrer. Thus, the sample preparation for particle size distribution measurements is of utmost importance.

There are similarities for samples from all industrial partners, which are illustrated in the diagram in figure 4.16. Firstly, all samples have their maximum volume fraction (7 to 8%) at the same particle size (25 µm). Furthermore, all chemical pulp samples show a bimodal graph with 2 peaks: The first at 25 µm and the second at 70 to 90 µm. It is assumed that the bimodal distribution is associated with the two main fractions of chemical pulps, the finer and the coarser fraction. The mechanical pulp samples from Norske Skog did not show a bimodal distribution.

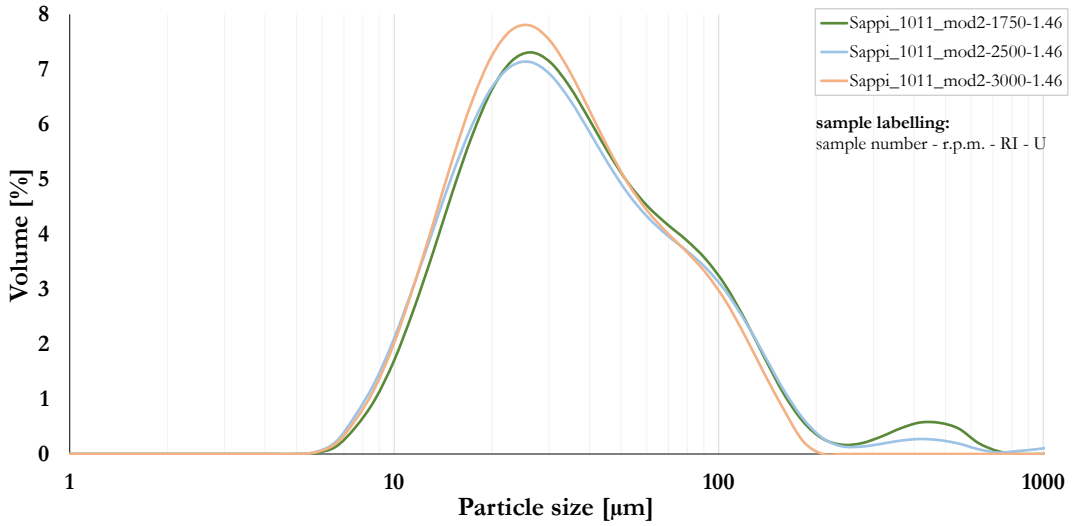

Figure 4.15 Fines from Sappi Austria Produktions-GmbH & Co KG at different r.p.m.
r.p.m....revolutions per minute, RI...refractive index, U...with ultrasound

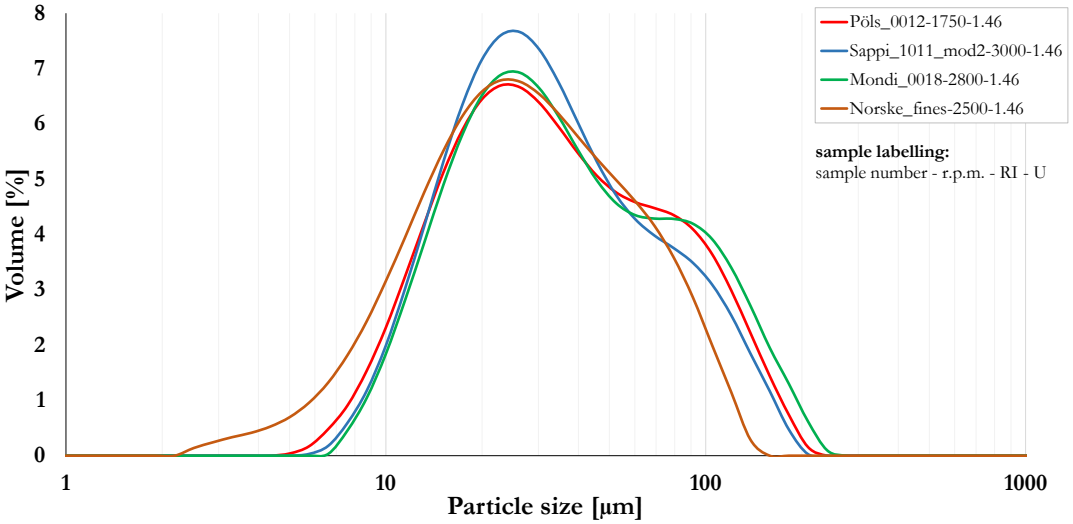

Figure 4.16 Fines of all four industrial partners
r.p.m....revolutions per minute, RI...refractive index, U...with ultrasound

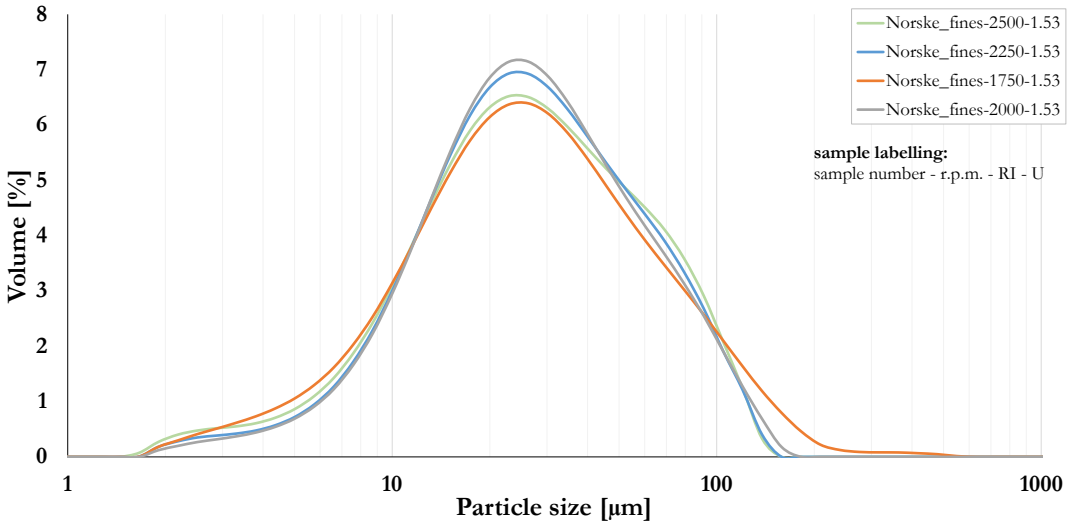

Figure 4.17 Fines from Norske Skog Bruck GmbH at different r.p.m.
r.p.m....revolutions per minute, RI...refractive index, U...with ultrasound

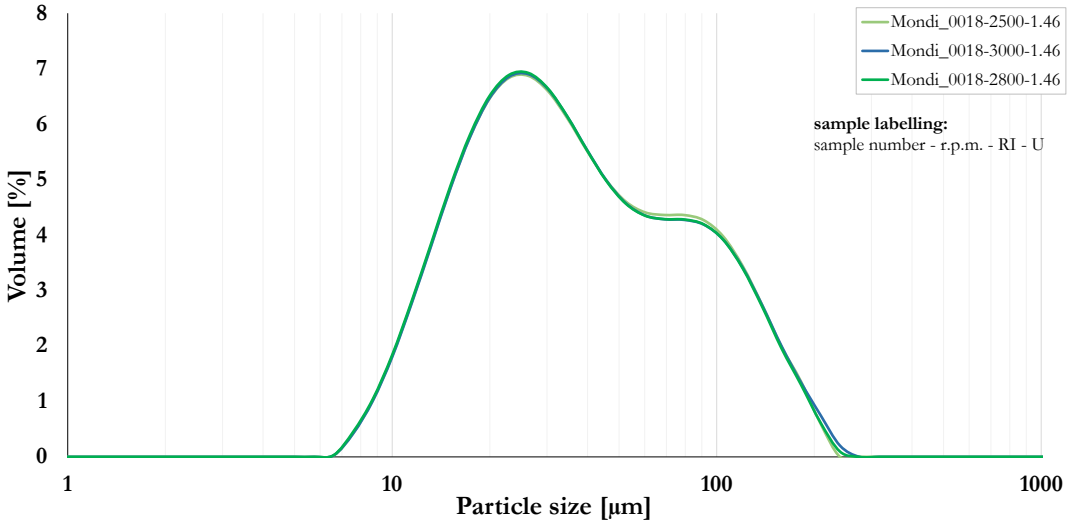

Figure 4.18 Fines from Mondi Frantschach GmbH at different r.p.m.
r.p.m....revolutions per minute, RI...refractive index, U...with ultrasound

The refractive index (RI) of cellulose is in the range of 1.46 (Kasarova et al. (2007)). This RI is also used as a default value of the Mastersizer 2000. In the work of Xu and Pelton (2005) a refractive index of 1.53 was used. In order to see if there is a significant difference in the results, trials with the default and adjusted refractive index values were performed. From the results (see figures 4.19, 4.20 and 4.21) it is apparent that using a refractive index of either 1.46 or 1.53 did not show a significant difference

in the results. An important remark is that samples of Mondi Frantschach using a RI = 1.53 showed a higher volume at first peak than with a refractive index of 1.46. In contrast, the samples from Norske Skog and Zellstoff Pöls showed the opposite behavior.

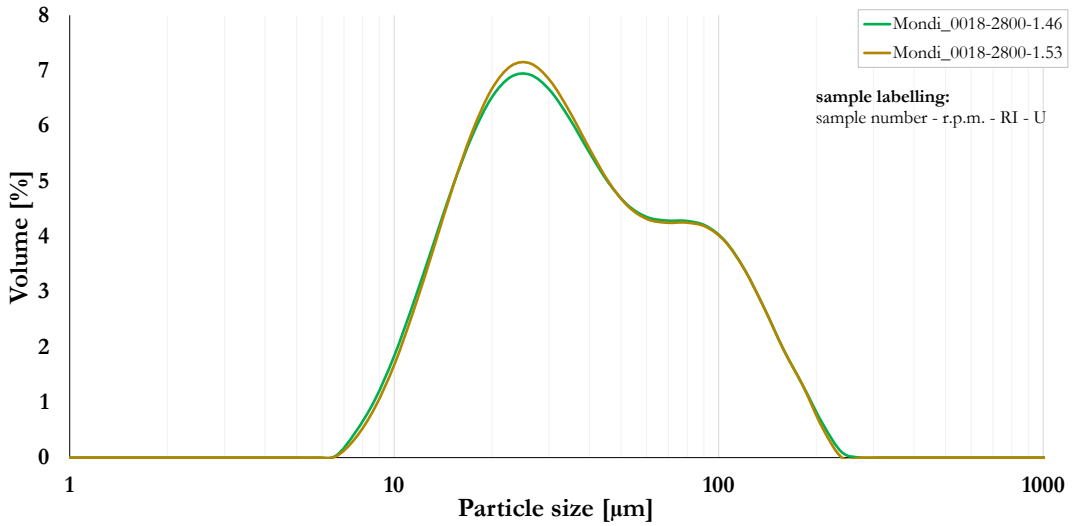

Figure 4.19 Fines from Mondi Frantschach GmbH at different RI
r.p.m....revolutions per minute, RI...refractive index, U...with ultrasound

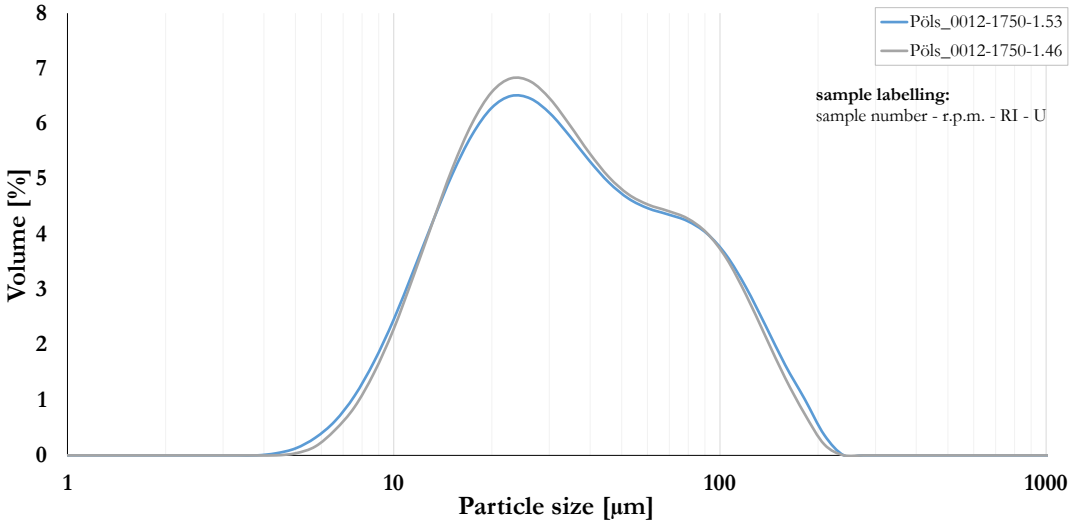

Figure 4.20 Fines from Zellstoff Pöls AG at different RI
r.p.m....revolutions per minute, RI...refractive index, U...with ultrasound

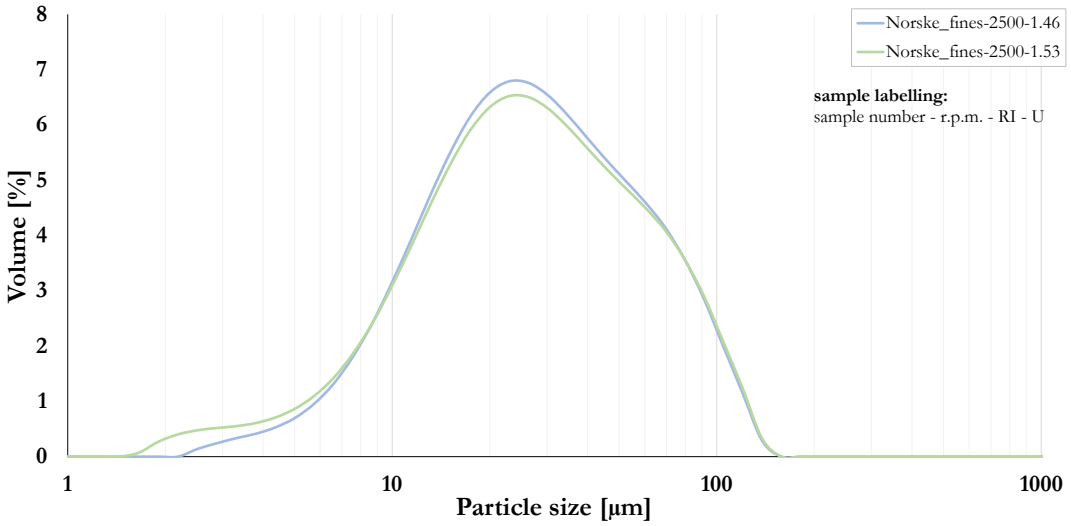

Figure 4.21 Fines from Norske Skog Bruck GmbH at different RI
r.p.m....revolutions per minute, RI...refractive index, U...with ultrasound

As already mentioned, fines tend to build agglomerates, which have an influence on the particle size measurements. The Mastersizer 2000 used in this study is also equipped with an ultrasound unit, which might be another option to destroy agglomerated flocs. Due to this tests with and without ultrasound treatment were performed. Sonication of the fines suspensions did not show a significant change in the results (see figures 4.22 and 4.23).

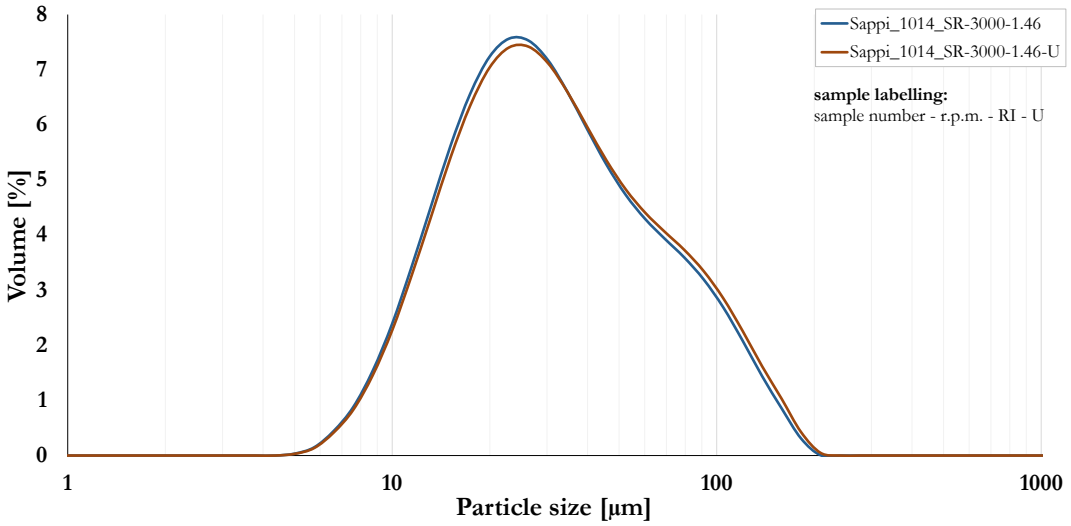

Figure 4.22 Fines from Sappi Austria Produktions-GmbH & Co KG with/without ultrasound
r.p.m....revolutions per minute, RI...refractive index, U...with ultrasound

It is important to mention that the samples from Sappi, which were exposed to ultrasound showed a higher volume at first peak than without sonication. The samples from Zellstoff Pöls showed the opposite behavior.

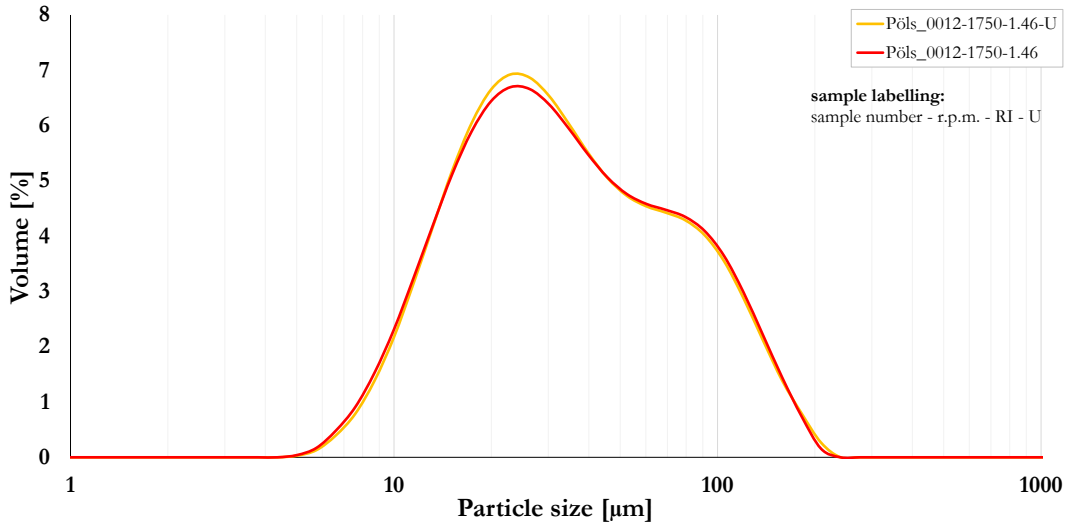

Figure 4.23 Fines from Zellstoff Pöls AG with/without ultrasound
r.p.m....revolutions per minute, RI...refractive index, U...with ultrasound

4.7 Comparison of fines contents from industry partners

Table 4.13 shows a comparison of all fines contents. All results obtained in this thesis are in accordance with literature (compare table 2.7). From the results shown in table 4.13 it is apparent that there is a difference in the development of the fines content along the fibrelines of the industrial partners.

The final fines contents at the end of the fibreline are in the same range for all chemical pulps (4.73%, 4.22%/4.68% and 5.06%). Nevertheless, the fines contents at the first sampling points right after the digester differed considerably (2.92%, 6.02% and 3.05%).

In contrast, however, the final fines content for the mechanical pulp sample was considerably higher (25.02%). Moreover, no considerable changes regarding fines content were observed along the fibreline (23.48%, 25.86% and 25.02%).

Sampling point	cs [%]	fc [%]	Sampling point	cs [%]	fc [%]
Sappi_1010	3.18	2.92	Norske_0019	27.22	23.48
Sappi_1011	35.42	3.42	Norske_0020	7.98	25.86
Sappi_1012	17.44	3.45	Norske_0023	9.92	25.02
Sappi_1013	34.22	3.76			
Sappi_1014	2.32	6.09			
Sappi_1024	19.06	4.73			
Sappi_1025	1.94	6.32			
Sappi_1026	45.99	4.40			
Pöls_0001	2.88	6.02	Mondi_0014	2.53	3.50
Pöls_0003	34.07	3.22	Mondi_0015	1.45	3.88
Pöls_0004	14.01	2.91	Mondi_0016	12.75	3.73
Pöls_0006	14.02	2.85	Mondi_0018	4.71	5.06
Pöls_0009	1.99	4.56			
Pöls_0011	94.46	4.22			
Pöls_0012	88.39	4.68			

Table 4.13 Comparison of all fines contents and consistencies determined cs...consistency, fc...fines content

Chapter 5

Conclusion and Outlook

Fines can influence the properties of paper in either a positive or a negative manner. Until the present day a lot of work has been done in order to investigate their influence on mechanical and optical properties. The present work gives an overview about the development of the fines content between different process stages of the pulping process. Apart from that, differences between chemical and mechanical pulping processes can be quantified by means of the fines content. The Britt Dynamic Drainage Jar Tester (BDDJ) is used to determine the fines contents of pulp samples. This device is easy and simple to handle, the energy and water consumption is low and it provides quick results.

The measured fines contents are in the same range for all chemical pulps (4.73%, 4.22%/4.68% and 5.06%) at the end of the fibreline. Nevertheless, the fines contents at the first sampling points right after the digester differed considerably between the pulping lines of the industrial partners (2.92%, 6.02% and 3.05%). As to be expected, the final fines content for the mechanical pulp sample was much higher (25.02%). Moreover, no considerable changes regarding fines content were observed along the fibreline (23.48%, 25.86% and 25.02%).

The initial trials using a light microscope are the first step towards image analysis, with what morphological differences between different types of fines can be investigated.

Evaluation of pulp and filtrate flows illustrate that there are remarkable amounts of fines in the circulation system of the fibreline of a pulp mill. Filtrates are often used to dilute the pulp at various stages along the fibreline. For example in wash presses and thickeners, filtrates often introduce large amounts of fines into the pulp. There are several positions along the fibreline of pulp mills where fines can be introduced to the process. Due to this the fines content increases punctual along the pulping process.

Particle size analysis with the Mastersizer 2000 shows, that all fines samples have their maximum volume fraction (7 to 8%) at the same particle size (25 µm). In addition all chemical pulp samples show a bimodal graph with 2 peaks: The first at 25 µm and the second at 70 to 90 µm. However, the mechanical pulp samples did not show a bimodal distribution. Due to the fact that fines tend to build agglomerates, some settings were adjusted to investigate their influence on the results. Adjusted settings are the revolutions (r.p.m.) and the influence of ultrasound. The results using default revolutions (1750 r.p.m.) showed a volume fraction up to a particle size of 800 µm, which is almost impossible for a fractionation using the BDDJ equipped with a 200-mesh wire. These peaks disappear in case of higher revolutions, whereas there is no difference if the fines suspensions are sonicated. A refractive index chosen of either 1.46 or 1.53 does not show a significant difference in the results.

In conclusion, it is evident that due to the recirculation of the filtrates a steady state of equilibrium regarding fines content in the process is reached. This means that potential impacts of fines removal are unpredictable.

5.1 Future outlook

Several methods (see chapter 3) used in this thesis might be interesting for further investigations. The BDDJ Tester is used for the determination of the fines content, but there are also similar devices. In order to overcome sampling problems that arise during the trials with the BDDJ Tester, the Dynamic Drainage Analyzer at Åbo Academi (see chapter 3.1) could be rebuilt at IPZ. The fines content of filtrates was assumed to be 50%. Due to this the actual fines content of potentially interesting mass flows ($\dot{m}_{fines}$ calculated with 50% fines content) should be determined. Trials with a Coulter Multisizer II^{TM} (see chapter 2.5) should be carried out in order to evaluate whether if the results obtained by this device are in accordance with those from the Mastersizer 2000. Initial trials using light microscopy showed that it is possible to distinguish between different types of fines. These tests are a first step towards image analysis and should be used as a general basis for further investigation within the Flippr° project.

Filtrates of the pulping process transferred to the wastewater treatment plant might be a source of fines, and could be used for further applications. It is however questionable whether the fractionation of fines from filtrates is worthwhile from an investment and energy cost point of view. Tests using industrial fractionation equipment in the production processes of paper mills might be an option to clarify this question.

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