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# Implementation of a Tube Flow Fractionator

MASTER THESIS

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# Abstract

In the present study a tube flow fractionator was implemented as described in the literature. The tube flow fractionator enables the fractionation of pulp fibers and fines over the whole size range based on the behavior of the particles in the flow field of the tube. The velocity of the suspension in the center of the tube is higher than near the tube wall. Flowing particles are randomly distributed in the tube flow due to a slight turbulence. Longer fibers which come close to the tube wall have a higher probability of being recaptured by the faster moving middle flow than smaller fibers or fines. Therefore longer fibers move faster through the tube than shorter ones and finally exit the tube flow fractionator faster (Johansson et al. (1970), Krogerus and Fagerholm (2003)). The main part of the tube flow fractionator is a 100-meter-long teflon tube which is wound onto a wheel. Pulp samples are injected into the tube flow via a three way valve. In order to avoid pulsations and to control the process parameters, a buffer tank and PI-controller were installed. After the fractionation process, the samples were analyzed based on length distributions using an optical fiber analyzer (Kajaani FS-200). To validate the implemented device repeatability tests and a comparison between kraft pulp (more long fibers) and pressure ground wood pulp (PGW, more fines) were carried out and the flow behavior of these two different pulp types in the tube flow device was analyzed. Furthermore a comparison between the tube flow fractionator and the Bauer McNett (BMN) fractionator – representing the standard device for laboratory fractionation – was also carried out. These tests showed that the device works as is described in the literature and allows fractionation of pulp samples over the whole particle size distribution. Further investigations should focus on fractions with narrow length distributions and on further separation of fractions prepared with standard devices. Thereby a deeper insight into the rather wide "fines fraction" (fraction passing 200 mesh) will be possible.

Keywords: tube flow fractionation, Bauer McNett, fiber analysis, particle length, fines

# Kurzfassung

Im Rahmen der vorliegenden Arbeit wurde ein Tube Flow Fraktionierer entsprechend der Beschreibung in der Literatur implementiert. Der Tube Flow Fraktionierer ermöglicht es, Zellstoffproben auf Grund deren Verhaltens in der Rohrströmung über den gesamten Größenbereich aufzutrennen. Die Geschwindigkeit der Suspension ist in der Mitte des Schlauchs höher als an der Schlauchwand. Durch leichte Turbulenz werden die Partikel in der Suspension zufällig über den Schlauchquerschnitt verteilt. Lange Fasern, die in den Bereich der Schlauchwand driften, werden auf Grund ihrer Länge mit höherer Wahrscheinlichkeit wieder in den Bereich höherer Geschwindigkeit gezogen als kurze Fasern und Feinstoff. Lange Fasern, die sich somit tendenziell eher in der Mitte der Strömung befinden verlassen den Fraktionierer schneller als Kurze, die eher durch die Randströmung gebremst werden. Das Herzstück des Fraktionierers ist ein 100 Meter langer Schlauch der auf einer Trommel aufgerollt ist. Zur Probenaufgabe werden zwei 3-Wege-Kugelhähne verwendet. Ein Buffertank und ein PI-Regler gewährleisten einen pulsationsfreien und konstanten Fluss der Strömung. Nach der Fraktionierung werden die Längenverteilungen der Proben mit einem optischen Faseranalysegerät (Kajaani FS-200) ausgewertet. Zur Validierung der Methode wurden Reproduzierbarkeitstests sowie ein Vergleich zwischen einem Langfasersulfatzellstoff (hohe Faserlänge, geringerer Feinstoffanteil) und einem Holzschliff (hoher Feinstoffanteil) durchgeführt. Weiters wurde das unterschiedliche Verhalten dieser beiden Stoffproben im Fraktionierapparat untersucht. Darüber hinaus wurde ein Vergleich mit dem Bauer McNett Fraktionierer - dem Standard Laboraggregat zur Fraktioinierung von Faserstoffen - durchgeführt. Diese Untersuchungen zeigen, dass die Anlage so funktioniert wie es in der Literatur beschrieben wird und dass eine Auftrennung von Stoffproben über alle Längenklassen hinweg möglich ist. Künftige Untersuchungen sollten sich auf Fraktionen mit enger Längenverteilung bzw. auf weitere Auftrennung von bereits mit Standardmethoden fraktionierten Proben konzentrieren. Auf diese Weise könnte beispielsweise der breite Bereich der Feinstofffraktion (Durchgang durch ein Sieb mit Maschenweite 200) für nachfolgende Analytik detaillierter aufgetrennt werden.

Schlagwörter: Tube Flow Fraktionierung, Bauer McNett, Faseranalyse, Partikellänge, Feinstoffe

# Contents

# 1 Introduction

The tube flow fractionator is a device which can be used to fractionate and classify particles over their whole range of sizes. Due to the wall fraction of the fluid, there are differences of flow velocity in the tube. The flow velocity is higher in the center of the tube than close to the tube wall. As a result of their dimension, larger particles have a higher probability to move into the middle flow than the smaller ones. The particles which are captured in the middle flow move faster through the 100 meter long tube than the smaller ones and finally exit the tube flow fractionator earlier (Johansson et al. (1970), Krogerus and Fagerholm (2003)). With this method it is possible to fractionate particles according to their size, particulary to their length, when the pulp is classified. The aim of this work was to implement such a tube flow fractionator which can be used for laboratory fractionation of pulp and fines. For validation of the implemented method, the samples will be analyzed with a microscope or an optical fiber analyzer (Kajaani FS-200).

The traditional fractionator to produce pulp fractions is the Bauer McNett (BMN) apparatus using screens of different mesh openings. Therefore, the range of fractions are limited because of the number of screens. It is impossible to produce different fine fractions for particles less than 76 µm hole diameter. These limitations of the Bauer McNett device shall be overcome by using the tube flow fractionation method. In the present work, a comparison between the tube flow fractionator and the BMN fractionator was carried out. The tube flow fractionator shall offers a possibility to investigate different types of pulps and fines fractions in a more accurate way than other fractionation devices. Therefore, it shall be possible for example

to determine "Scheimtoff" and "Mehlstoff" fractions which are fines fractions of the mechanical pulping process.

# 2 Fractionation and characterization of fibers and fines

## 2.1 Fiber chemistry

### 2.1.1 Cellulose

Cellulose is the structural basis of all plant cells and hence the most important natural substance produced by living organisms. Therefore, cellulose can be described as a linear-polymer glucan with a uniform chain structure. These units are bound by β-(1,4)-glycosidic linkages. Due to the β- position, the following glucose unit needs a turning of 180° around the C1-C4 axis of the pyranose ring in order to be defined as a cellulose molecule.

As presented in figure 2-1, glucose is linked together to long chain molecules. The chemical formula for cellulose is $(C_6H_{10}O_5)^n$, where n represents the value of repeating glucose units or the degree of polymerization (DP). Cellulose in plants and woods has a DP of 7.000–15.000. During pulping or bleaching, where cellulose is exposed to an intensive treatment, the DP-values lead to a strong decrease (Fengel and Wegener (2003)).

The cellobiose unit consits of two consecutive glucose anhydride units. It can easily hydrolyze to single glucose ($C_6H_{12}O_6$) under controlled chemical process parameters. The fiber properties are related to the DP of the molecules. If the

molecular weight decreases under a certain level, it will cause a decrease in single fiber strength (Smook (1989).).

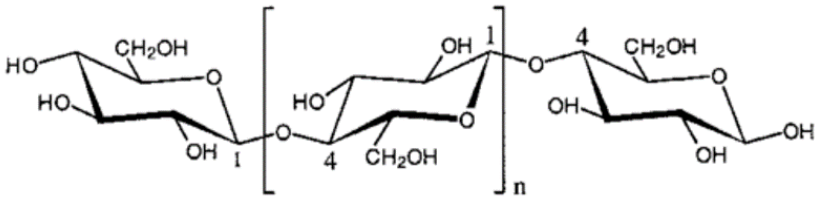


**Figure 2-1** *Stereochemical structure of cellulose (Fardim (2011)).*

### 2.1.2 Hemicelluloses

Besides cellulose, a number of various polysaccharides called polyoses or hemicelluloses are present in wood as well as in other plant tissues. The polyoses differ from cellulose by having various sugar units and by much shorter molecular chains. These polyoses can be divided into four groups (Fengel and Wegener (2003)):

- pentoses,
- hexoses,
- hexuronic acids,
- deoxy-hexoses

These sugars along with uronic acids form various polymeric structures. Some sugars are associated with the cellulosic portion and others are more closely associated with lignin. After the chemical treatment of wood to produce pulp, structures of the hemicelluloses usually change dramatically. They are easily degraded and dissolved, therefore their percentage is always less in the pulp compared to the original wood (Smook (1989)).

### 2.1.3 Lignin

Next to cellulose, lignin is the most important polymeric organic substance in the plant world. Lignin increases the mechanical strength to such an extent, that plants like big trees are able to remain upright. The amounts of lignin is quite variable in

different plants. The percentage of lignin in wood ranges from 20% to 40%. Aquatic and herbaceous angiosperms are less lignified. The lignin content of softwoods is higher than of hardwoods.

The chemistry of lignin is complex, the molecules are very large, having a molecular weight of more than several thousands. Lignin is characterized as a substance, which contains hydroxyl (OH) or methoxyl ($OCH_3$) groups attached to an aromatic ring. Methoxyl groups are located in the lignin in softwoods with a percentage of about 15% and 20% in hardwoods. Hydroxyl groups in the lignin appear in both species with 10% (Clark (1985)). In case of pulping, lignin is more or less dissolved from wood in degraded and altered forms. It represents a large potential carbon source of more than 35 million tons of carbon per year world-wide for lots of chemical and energy processes (Fengel and Wegener (2003)).

## 2.2 Different wood characteristics

Plants can be divided into four different groups. The Spermatophyta (seed plants) contain the trees and other common sources of papermaking fiber. The Spermatophyta can be divided into softwoods (gymnosperms, conifers or evergreens) and hardwoods (angiosperms or deciduous). Figure 2-2 shows the average distribution of the chemical components of softwoods and hardwoods.

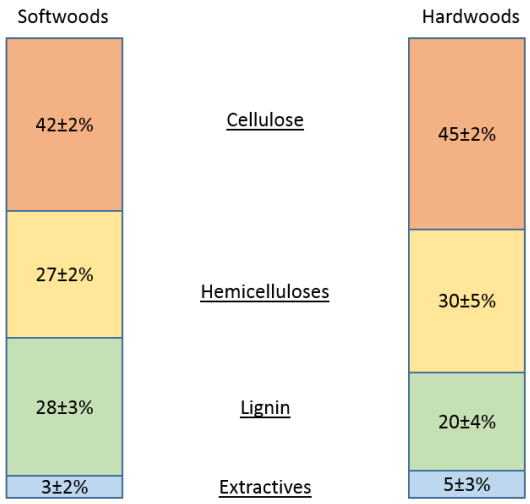


**Figure 2-2** *Chemical composition of hardwoods and softwoods (Smook (1989)).*

### 2.2.1 Softwoods

The vertical structure of softwoods consists of tapering cells called tracheids (long and tapering cells). In some cases, there are also vertical resin canals present. Another structure is the horizontal system. These narrow rays are only one cell wide but often several cells high. There are two different types of ray cells, the ray parenchyma (present in all species) and the ray tracheids (present in only certain species). Softwoods consist of 90 to 95 % of tracheids with a fibrous form. These fibers have an average length between 2 and 6 mm. Parenchyma cells are fixed in horizontal or vertical strands . Annual rings are indicated by a band with higher density, called late wood tracheids. The density of the earlywood zone is in the range one half or one third of latewood (Fardim (2011)). Figure 2-3 shows latewood and earlywood tracheids of softwood. In spruce, minimal radial diameters of 7 µm for latewood tracheids and maximal radial diameter of 32 µm were determined. Softwoods, such as larch, spruce, pine and Douglas fir also contain radially oriented tracheids and ray parenchyma cells (Fengel and Wegener (2003)).

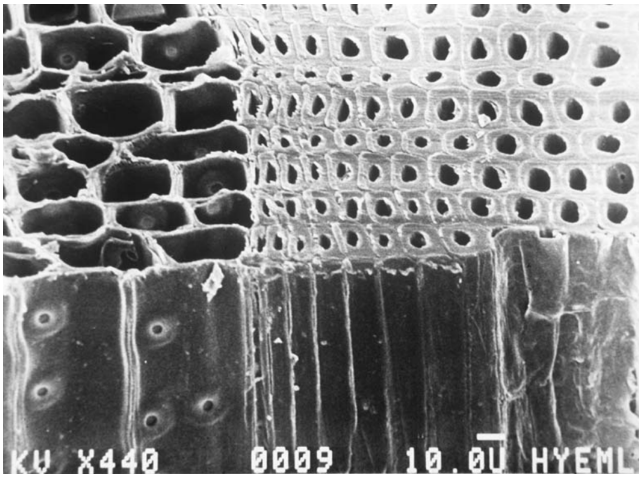


**Figure 2-3** *Tracheids of a pinus sylvestris softwood species (Kellomäki (2009)).*

### 2.2.2 Hardwoods

The vertical structure of hardwoods consists of relatively long fibers of narrow diameter, called libriform fibers. The wider cells are called vessels. In cross sections some of these vessels can be observed by the naked eye as pores, or as a series of long vertical surfaces grooves. Hardwoods also consists of a vertical parenchyma system and a horizontal or ray parenchyma system. Hardwood species show annual rings, made of vessel cells which vary from earlywood to latewood (Smook

(1989)). The dimensions of hardwood fibers are smaller than those of the softwood tracheids. Hardwood fibers have thicker cell walls and smaller lumina, and the differences between wall thickness and lumen diameter is not that big as between earlywood and latewood fibers of softwoods (Fengel and Wegener (2003)). The shape of vessel elements (are dead) is hollow and perforated at their ends to facilitate the upward conduction of water and nutrients from the root system. One vessel can be up to some meters in length (Fardim (2011)). Figure 2-4 shows the formation of vessel elements.

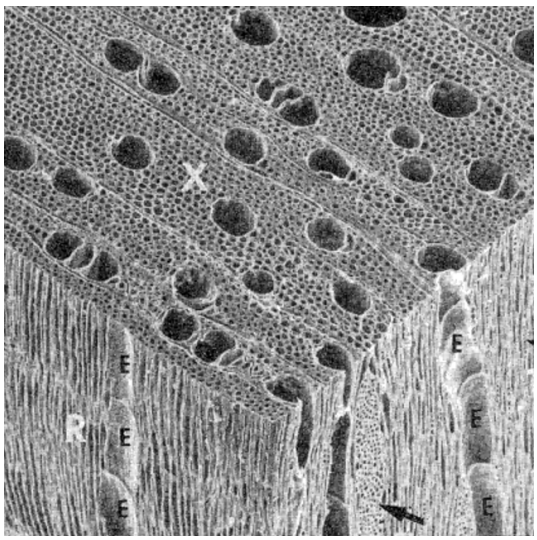


**Figure 2-4** *Three-plane view of a diffuse hardwood xylem (Fardim (2011)).*

### 2.2.3 Comparison between hardwood and softwood

In general, wood contains of many types of cells that have all specific functions. Each of them determines the size, form and perforation of a woody cell. There are three major cell categories: The contacting cells, supporting cells and storage cells. In common, the first two groups are longitudinal or axial with an elongated form and the storage cells are primarily radial but may also be longitudinal.

The structure of softwood cells is simple and they posses only a few types of cells. Figure 2-5 shows, that hardwoods have a more complex structure with a larger number of cells than softwoods (Kellomäki (2009)). Furthermore, the differences between softwood and hardwood cells is shown.

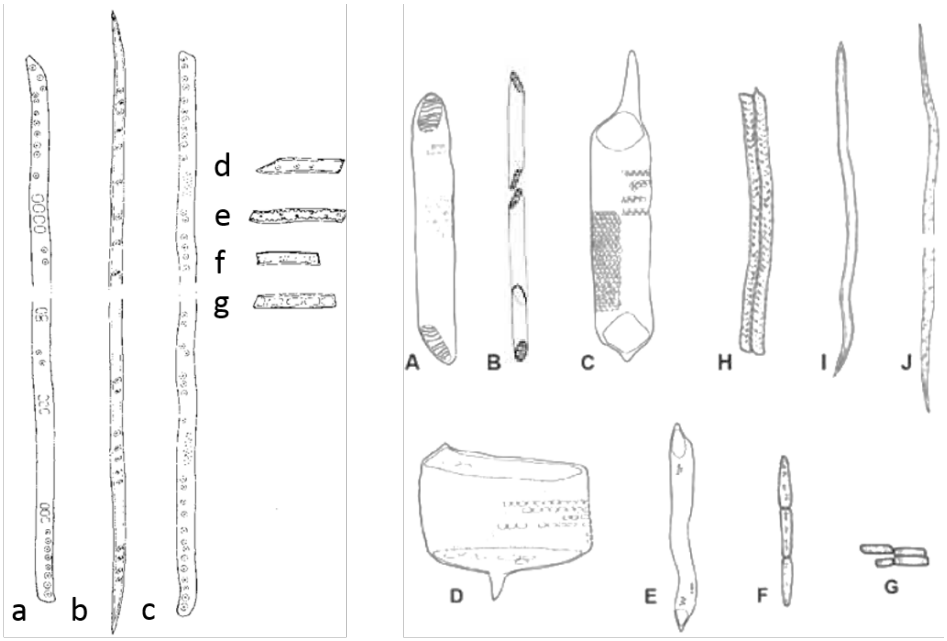


**Figure 2-5** *Cell types of softwoods: (a): Earlywood pine tracheid; (b): Latewood pine tracheid; (c): Earlywood spruce tracheid; (d): Ray tracheid of spruce; (e): Ray tracheid of pine; (f),(g): Ray parenchyma cell of spruce (f) and pine (g). Cell types of hardwoods: (A): Vessel element of birch; B: Birch vessel; (C): Vessel element of aspen; (D), (E): Vessel element of oak in earlywood (D) and oak in latewood (E); (F): Longitudinal parenchyma cell of oak; (G): Ray parenchyma cell of birch; (H), (I): Tracheids of oak (H) and birch (I) and libriform fiber of birch (J) (Fardim (2011)).*

Another considerable difference between this two types is the fiber length. Table 2-1 shows the dimensions of different types of softwoods and hardwoods.

| Species | Latin name | Length, mm | Width, µm |
|---|---|---|---|
| German spruce | *Abies alba* | 3.4–4.6 | 25–65 |
| Spruce | *Picea abies* | 1.7–3.7 | 20–40 |
| Pine | *Pinus sylvestris* | 1.4–4.4 | 10–50 |
| Beech | *Fagius sylvatica* | 0.6–1.3 | 15–20 |
| British oak | *Quercus robur* | 0.3–1.6 | 10–30 |
| Poplar | *Populus spec.* | 0.7–1.6 | 20–40 |

**Table 2-1** *Fiber dimensions of different wood species (adopted from Sixta).*

By far, fiber length and cell wall thickness are the most important structural characteristics. To achieve enough interfiber bonding, a minimum length is required and the length is proportional to tear strength (Smook (1989)).

## 2.3 Types of pulp

The production of pulp is the most important process for the chemical and mechanical conversion of wood. The term pulp is used generically for stone groundwood, refiner mechanical pulps, semichemical and chemical pulps. Novel pulping processes are alkaline pulping with additives (e.g. soda-AQ, CTMP) or sulfur-free pulping (e.g.soda-oxygen pulping, organosolv pulping) (Fengel and Wegener (2003)). In the pulping process, wood is reduced to a fibrous mass. This can be accomplished chemically, mechanically or by a combination of these processes (Smook (1989)).

### 2.3.1 Mechanical Pulping

The oldest pulping process is the stone groundwood process which was invented by Keller 1843. Since that time, wood has ben the dominant raw material for the paper production. In this process, a block of logs is pressed against a roughened grinding stone. Fibers are torn out and washed away with water. The principle is very simple but not easy to control. Another method to produce mechanical pulp is shredding and grinding chips of wood between rotating discs of a so called refiner. The product is called refiner mechanical pulp (RMP). RMP contains more long fibers than stone groundwood. Modern RMP installations includes thermal or chemical process steps in order to improve pulp quality and to reduce mechanical energy consumption. Therefore, higher yields are reachable. Table 2-2 presents the survey of mechanical pulping.

| Process/Pulp type | | Chemical treatment | Mechanical treatment | Yield, % |
|---|---|---|---|---|
| Mechanical pulping | | | | 80–99 |
| Stone grinding | Groundwood (SGW) | None | Grindstone | 93–99 |
| | Pressure Groundwood (PGW) | None | Grindstone | |
| Refiner pulping | Refiner Mechanical pulp (RMP) | None | Disk refiner | 93-98 |
| | Thermomechanical pulp (TMP) | Steam | Disk refiner | 91-98 |

**Table 2-2** *Different mechanical pulping processes (adopted from Fengel and Wegener (2003)).*

### 2.3.2 Chemical pulping

The goal of chemical pulping is to remove the lignin and to retain most of the cellulose and hemicelluloses in the original form. The yield of this process is much lower compared to mechanical pulping, in general between 40 and 50% of the original wood substance. The principle of chemical pulping is to cook wood chips in a solution at elevated temperature and pressure. There are two types of chemical pulping, the kraft process (alkaline environment) and the sulfite process (acid environment). The kraft process has become the dominant process because of its good chemical recovery and pulp strength (Smook (1989)).

#### *2.3.2.1 Kraft process*

The main chemicals of kraft cooking are sodium hydroxide (NaOH) and sodium sulfide ($Na_2S$). These chemicals initiates breaking of the lignin molecule into smaller segments. The main advantages of sulfate pulping are listed below (Fengel and Wegener (2003)):

- low demands on wood quality, including all types of softwoods and hardwoods
- short cooking times
- good recovery of the pulping chemicals generation of process heat and the possibility to get products such as tall oil and turpentine
- good strength properties

#### 2.3.2.2 Sulfite process

The cooking chemicals are a mixture of sulfurous acid ($H_2SO_3$) and bisulfite ions ($HSO_3^-$). The bisulfite ion is used to attack and solubilize the lignin. Sulfite pulp treatment can be deployed over a wide range of pH (pH 1-5). This processes can be characterized by the composition of the cooking liquor, which influences the cooking pH. The groups are (Fengel and Wegener (2003)):

- acidic sulfite pulping
- bisulfite pulping
- multi-stage sulfite pulping
- neutral sulfite pulping
- alkaline sulfite pulping

Sulfite pulps are easier to bleach because they are brighter in color than kraft pulps. In contrast to equivalent kraft pulps sheets, a sheet made of sulfite pulp is weaker.

Resinous softwoods and tannin-containing hardwoods are difficult to handle. Table 2-3 gives an survey of the comparison of kraft and alkaline sulfite pulp data.

| | Yield % | Kappa No. | Brightness GE | C.S.F. ml |
|---|---|---|---|---|
| Kraft, 166°C | 47,2 | 26,8 | 28,9 | 300 |
| Alkaline sulfite, 175°C, pH 8.0 | 43,9 | 43,6 | 37,9 | 300 |
| Alkaline sulfite, 175°C, pH 9.5 | 43,9 | 43,6 | 37,9 | 300 |

**Table 2-3** *Comparison of kraft and alkaline sulfite pulp data(adopted from Fengel and Wegener (2003)).*

## 2.4 Fibers

The term pulp indicates collectively chemical, semichemical, chemimechanical and mechanical pulps. Pulp is mostly utilized for making paper and board. Some pulps are processed into various cellulose derivatives and regenerated celluloses (e.g., viscose).

In general, there are two different pulp types which are of special interest. These are the mechanical and the chemical pulp. Mechanical pulp consists of a variety of fiber or particulate matter in different sizes, from intact whole fibers to small cellwall fragments (fines). For chemical pulp, the wood gets disintegrated chemically to single whole fibers. If pulp is refined, fines are produced. The cell wall of

fibers may be delaminated to a greater or less extent and the surfaces of fibers are fibrillated. Fines interact with chemicals used in papermaking. Furthermore, they improve interfiber bonding (Alen (2007)).

## 2.5 Fines

Pulps consist of fibers and smaller fragments called fines. The latter comprise very short or broken fibers, cut ends of fibers, wall fragments, microfibrils and small amounts of other particles such as ray and parenchyma cells (Ferreira et al. (1998)). These differences are presented in figure (2-6). The content of fines is defined as the weight fraction that passes through a 76 $\mu$m diameter round hole screen of a fiber length classifier. Fines may act as small fibers which fill voids between fibers and can assist in forming fiber-fiber bonds (Sirvio and Nurminen (2003)). Furthermore, fines of mechanical pulp are quite different compared to chemical pulp fines. The following section describes the fundamental differences between these fines. Mechanical pulps contain a much greater amount of fines (20-35% by weigth). These fines lead to higher opacity and strength of printing paper, as they improve bonding between fibers. In general, mechanical pulp contains a larger portion of fines than chemical pulp. The fines of both pulps have similar effects on pulp and sheet properties and on the bonding degree of the sheet. These higher bonding ability results in higher apparent density, tensile strength and elastic modulus (Retulainen et al. (1993)).

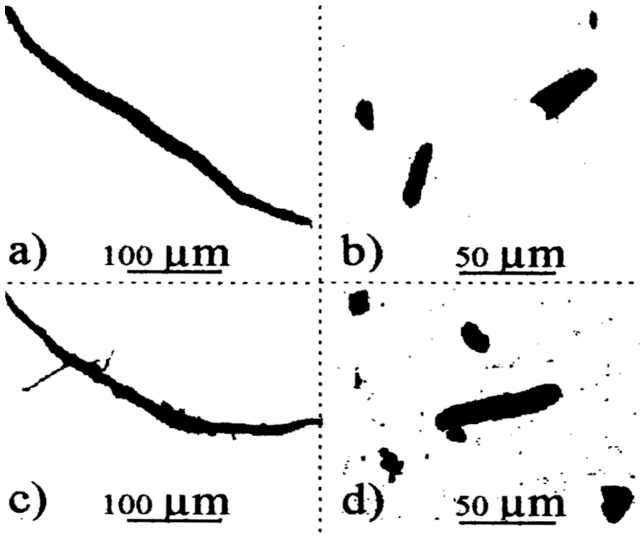


**Figure 2-6** *Microscopic binary image: a.) unbeaten fiber; b.) unbeaten pulp primary fines; c.) beaten fiber and d.) beaten pulp primary and secondary fines (Sirvio and Nurminen (2003)).*

### 2.5.1 Classification of fines

Fines can be classified based on the pulping method, i.e. mechanical or chemical. Mechanical pulp fines can be classified according to their morphology into so-called "Mehlstoff" and "Schleimstoff". Mehlstoff consists of chunky lignin-rich particles with low bondability. In contrast, Schleimstoff fines consist of cellulose-rich finer and more fibrilar particles with a high bonding potential. Pulp fines can be devided into primary and secondary fines. Primary fines are present in the pulp before refining and secondary fines are produced due to mechanical loading during beating (Retulainen et al. (1993)). Small particles of wood, for instance vessel elements, shortened fibers, ray and parenchyma cells are defined as primary fines. Their mass amount is usually very small (Seth (2003)). In comparison to primary fines, secondary fines offer more fibrillated and lamellar material, which comes from the $S_1$- and $S_2$- layers . They are also more beneficial to paper properties, as they have better bonding properties (Retulainen et al. (1993)). The amount of secondary fines can be large and depends on fiber morphology, pulping processes and mechanical treatment (Seth (2003)). Secondary fines increase and primary fines decrease the bonding strength (Retulainen et al. (1993)).

### 2.5.2 Properties of fines in a paper sheet

The fines fraction of both mechanical and chemical pulps is capable of pulling fibers closer together. A fiber network containing fines, shows a decrease in thickness and porosity, as well as increased density. Another effect caused by fibril-like particles is the sheet condensing effect. Flake-like fines, in most cases broken pieces of fibers or cell walls may contribute as well, but their influence is not comparable to fibrillar fines. Usually an increase in kraft fines increases the tensile index of paper. The light-scattering coefficient remains unchanged until a certain amount of fines is reached (about 15% of fines). On the other hand, mechanical pulp fines significantly increase the light- scattering coefficient. The tensile index shows a clear dependence on fines quality. The higher the fibrilar-fines content, the higher the tensile index. Porosity and density are the most relevant structural parameters which influence the mechanical properties of paper. Therefore, the quality of fine particles seems to be of utmost importance for pulping quality. The appropriate quality of fines and fibers can be achieved by ideal wood raw material and processing conditions (Sirvio and Nurminen (2003)). Table 2-5 gives an overview of paper sheet properties of unbeaten and beaten pulp, with and without fines.

| Pulp properties | With fines unbeaten | With fines beaten | Without fines unbeaten | Without fines beaten |
|---|---|---|---|---|
| Paper properties | | | | |
| tensile index ($Nm/g$) | 70,9 | 99,4 | 48 | 81,7 |
| tear index ($mNm^2/g$) | 8.8 | 9.5 | 6.7 | 10.2 |
| burst index ($Kpam^2/g$) | 3.8 | 6.9 | 2.1 | 5.0 |
| light scattering ($m^2/kg$) | 35.1 | 25.6 | 35.3 | 28.0 |
| density ($kg/m^3$) | 668 | 767 | 600 | 708 |

**Table 2-4** *Paper sheet properties of unbeaten and beaten pulp, with and without fines (Sirvio and Nurminen (2003)).*

## 2.6 Fractionation of pulp

Fractionation implies the separation of particles according to one or more characteristics, such as particle size or particle density. Pulp fibers obtain different characteristics, such as fiber length. If fibers are bleached or refined, they are not treated at the same intensity because pulp treatment can be observed as a statistical process. This leads to a wide range of particle size distribution. For the use in paper products, in depth knowledge on the fiber and fines characteristics is of considerable interest. Important characteristics for papermaking are mechanical and optical properties such as fiber length, fiber wall thickness, fiber strength, etc. As mentioned above, it is important to have fractionation processes available which can separate fibers according to relevant papermaking parameters. Some fractionation processes are well established in the industry. For example, in the packaging grade stock preparation, the fractionation according to fiber length plays an important role. Fractionation for laboratory investigation are also well established and will be described in the following. Laboratory separation devices which produce two fractions (fibers and shives) are the Heindl separator, the Brecht-Holl separator and the Somerville separator. They all use meshes to separate the different fractions. The disadvantage of those fractionator devices is that they are only able to produce two fractions (Höke (2009)). Therefore the Bauer McNett fractionator uses 4 screens for fractionation and produces as well fines fractions. Figure 2-7 presents the fiber length distribution of a TMP pulp with a typical particle size distribution, as produced using the Bauer McNett that has multiple stages for separation.

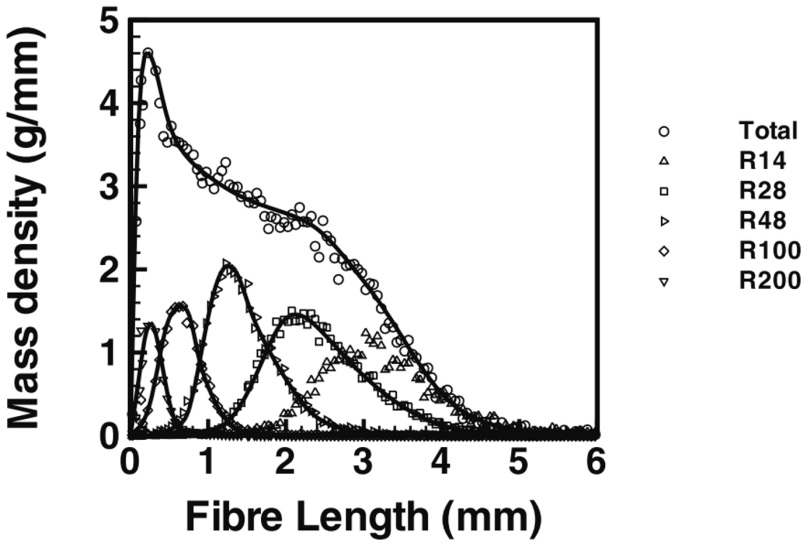


**Figure 2-7** *Mass density of pulp from individual BMN classes (Gooding and Olson (2001)).*

### 2.6.1 Mathematical description of fractionation

In this section an overview on the mathematical description of fractionation is given. A fractionation apparatus should be considered as a black box. The feedstock (mass $m_f$) gets classified into a coarse material (mass $m_c$) and a smooth material (mass $m_s$). The indices $m_F$ is defined as the mass feedstock with the particle size distribution $q_F(x)$ and the indices $m_c$ is defined as the mass of coarse material with the particle size distribution $q_c(x)$. The indices of the mass of the smooth material ($m_s$) is defined as the mass with the particle size distribution $q_s(x)$ (Stieß (1995)). An example for a fractionator is presented in Figure 2-8.

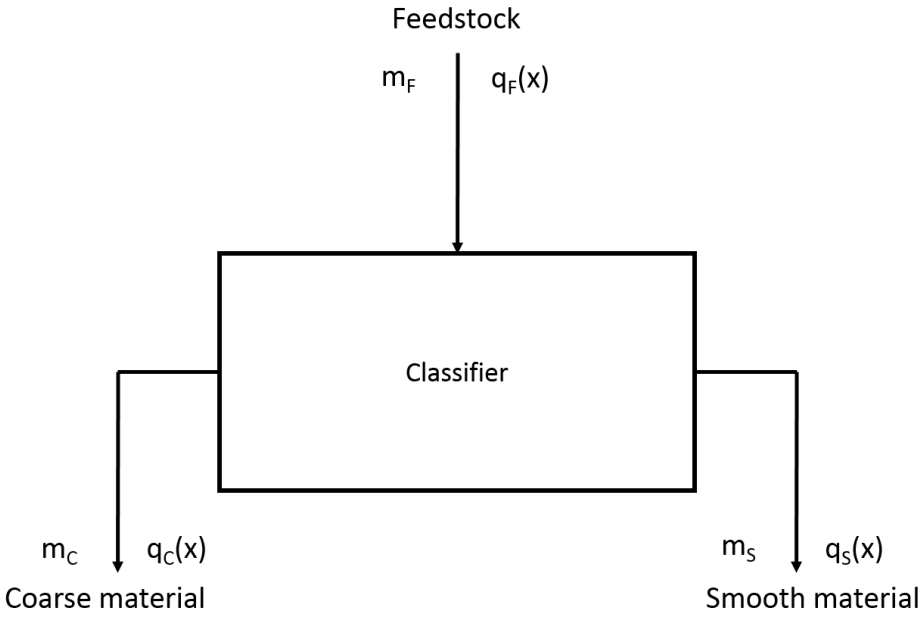


**Figure 2-8** *Schematic illustration of a fractionation device (Stieß (1995)).*

According to (Stieß (1995)), the fractionation process can be calculated as follows: Point balance: $F$ stands for particle feedstock, $C$ for coarse particles and $S$ for smooth or fine particles

$$m_F = m_C + m_S \tag{2.1}$$

For mass flow balances $[kg/s]$:

$$\frac{dm}{dt} = \dot{m}_F - \dot{m}_C - \dot{m}_S \tag{2.2}$$

For steady state stream:

$$\dot{m}_F = \dot{m}_C + \dot{m}_S \tag{2.3}$$

Steady-state mass fraction for coarse material:

$$c = \frac{m_C}{m_F} = \frac{\dot{m}_C}{\dot{m}_F} \tag{2.4}$$

Steady-state mass fraction for smooth material:

$$s = \frac{m_S}{m_F} = \frac{\dot{m}_S}{\dot{m}_F} \tag{2.5}$$

Equation 2.4 and equation 2.5 applied in equation 2.1 results in

$$1 = c + s \tag{2.6}$$

Balance of fraction (particle size x): Particle size x stands for a measure of length (for instance defined as mesh or equivalent diameter).

$$\dot{m}_F(x) = \dot{m}_C(x) + \dot{m}_S(x) \tag{2.7}$$

$q_{r,i}$ is defined as the ratio of the total amount between a certain size range and a certain width of particle classes $\Delta x_i$. $\Delta x_i$ can be a mass amount between two wires with a mesh of $x_i$ and $x_{i-1}$. Therefore, $q_{r,i}$ is also called distribution of frequency. i.g.:

$$q_{r,i} = \frac{Subset\ x_{i-1}...x_i}{Total\ quantity\ \cdot Interval\ width} \tag{2.8}$$

Another description which is called distribution sum is defined as (Stieß (1995)):

$$Q_{r,i} = Q_r(x_i) = \frac{Subset\ x_{min}...x_i}{Total\ amount\ x_{min}...x_{max}} \tag{2.9}$$

Balance of fraction (q=$q_3$):

$$\dot{m}_F(x) = \dot{m}_F \cdot \dot{m}_F(x) = \dot{m}_F \cdot \frac{dQ_F(x)}{dx} \tag{2.10}$$

$$\dot{m}_C(x) = \dot{m}_C \cdot \dot{m}_C(x) = \dot{m}_C \cdot \frac{dQ_C(x)}{dx} \tag{2.11}$$

$$\dot{m}_S(x) = \dot{m}_S \cdot \dot{m}_S(x) = \dot{m}_S \cdot \frac{dQ_S(x)}{dx} \tag{2.12}$$

$$q_F(x) = c \cdot q_C(x) + s \cdot q_S(x) \tag{2.13}$$

$$Q_F(x) = c \cdot Q_C(x) + s \cdot Q_S(x) \tag{2.14}$$

The distribution number $D(x)$ is defined as:

$$D(x) = \frac{\dot{m}_C(x)}{\dot{m}_F(x)} \tag{2.15}$$

In the section that follows, the different types of amounts are described (Stieß (1995)).

- Amount: Particles are counted inside the class. The results of the particle size analyze are amount distributions
- Area: The surface of the particles is the type of amount
- Volume, Mass: The classes are determined by weighing. If the density of the particles is independent to their size, the mass- and volume distribution are will be equal.

Table 2-5 presents the different types of amounts. P(x) stands for pass of particles.

| Index | Type of amounts | Distribution |
|---|---|---|
| r=0 | amount ($x^0$) | $Q_0(x)$, $q_0(x)$ |
| r=1 | length ($x^1$) | $Q_1(x)$, $q_1(x)$ |
| r=2 | area ($x^2$) | $Q_2(x)$, $q_2(x)$ |
| r=3 | volume ($x^3$) | $Q_3(x)$, $q_3(x)$ |
| r=3* | mass ($\rho \cdot x^3$) | $Q_3(x)$=P(x), $q_3(x)$ |

**Table 2-5** *Different types of amounts (Stieß (1995)).*

## 2.7 Laboratory methods of fractionation

### 2.7.1 Fractionation in a Bauer McNett classifier

The Bauer McNett classifier is composed of a series of chambers, typically four or five, each with a progressively smaller screen mesh. At the beginning of the test, a defined amount of pulp is added to the first chamber. Thereafter, a continuous flow of water passes through the chambers. Smaller fibers pass through the mesh and flow into the basin. The collection of fines fraction with the Bauer McNett fractionator is associated with a high effort. After 20 minutes fractionation time the test is stopped and then the chambers are drained and the mass of solid material in each chamber is measured. The upstream velocity of this device is about 2 $m/s$ and the aperture velocity is 0.02 $m/s$. The consistency in a BMN is 0.1 %. In Figure 2-9, a schematic figure of an BMN classifier for laboratory investigations is presented. Therefore, the flow of the suspension through the chambers are visible. Chamber 2 to chamber 4 collect the fibers and chamber 5 the fines. An impeller which moves the flow past the mesh surface creates turbulence in the chamber without producing pressure pulsations. The standard set of an BMN screen is: 14, 28, 48, 100 and 200 mesh, which coincides with screen openings of 1410 µm, 600 µm, 300 µm, 150 µm and 76 µm, respectively (Gooding and Olson (2001)).

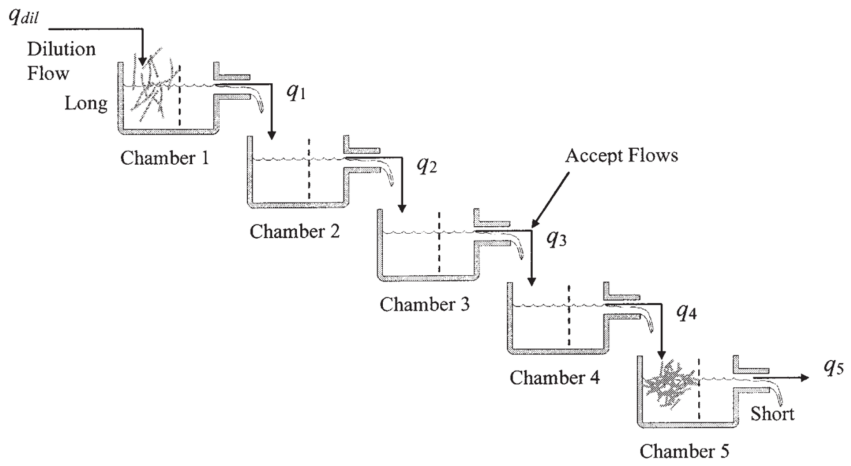


**Figure 2-9** *Schematic figure of a BMN fractionator (Young and Olson (2004)).*

The BMN fractionator separates fibers mainly on the basis of length. As presented in figure 2-10, the BMN fractionator has an impeller which moves the flow past the mesh surface and causes mixing in the chamber (Gooding and Olson (2001)). Detailed investigations about the Bauer McNett fractionator can be found in literature.

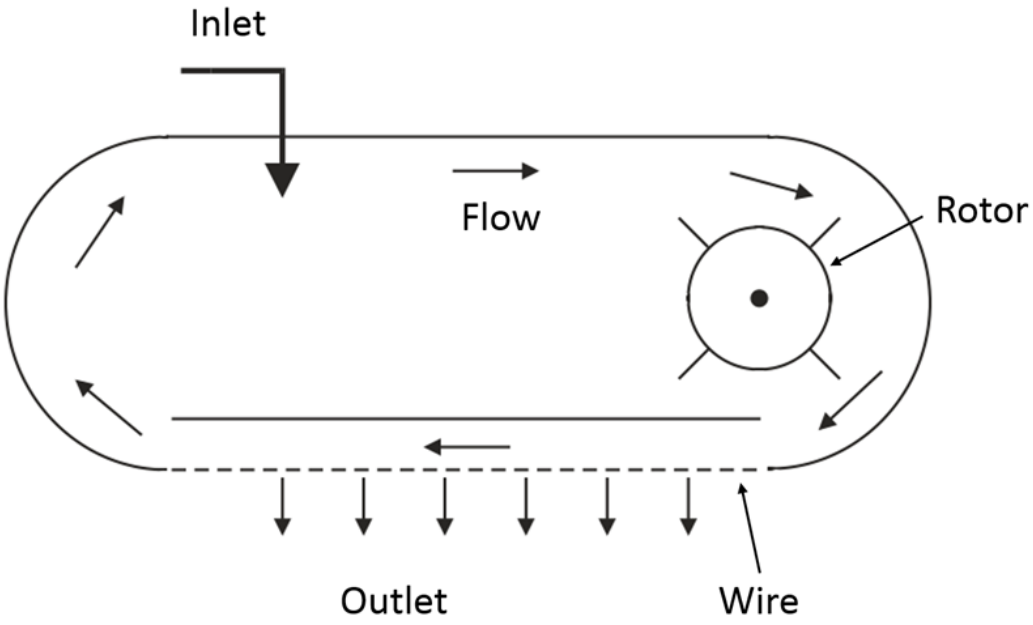


**Figure 2-10** *Bauer McNett classifier in plane view. The inlet flow comes from the top. The circulation is caused by the rotor. A part of the flow splits of and passes through the screen mesh at the bottom (Gooding and Olson (2001)).*

### 2.7.2 Fractionation with a tube flow fractionator

In chapter 2.7 it was shown that pulp can be distributed into different length classes as fibers and different types of fines and also measured with different types of optical fiber analyzers. It is clear, that if the fractions are produced with the Bauer McNett classifier, there will be always sharp delineation between the different fractions. Another way to produce fractions without these constraints is the fractionation using the tube flow classifier. With this device different size classes are easy to produce. The method of fractionating particles and fibers over the entire range of size may be carried out with the tube flow fractionator. Figure 2-11 illustrates the principle of the tube flow fractionation method (Laitinen (2011)).

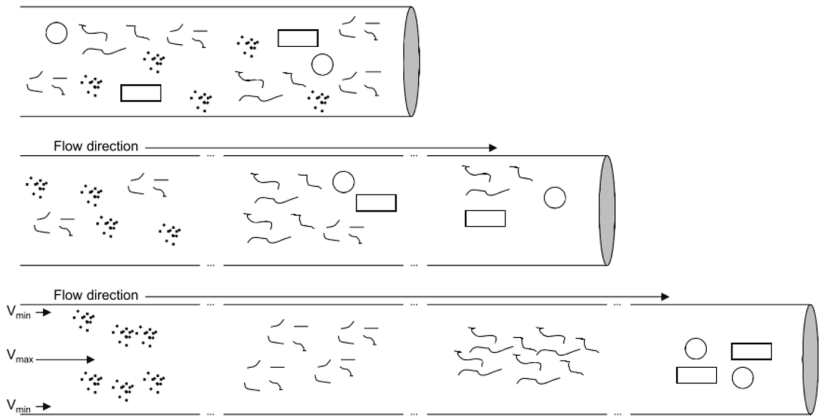


**Figure 2-11** *Schematic design of the principle of fractionation with tube flow fractionator (Laitinen (2011)).*

### 2.7.3 Analysis of Fibers and Fines

During the past few years, the technology of optical fiber analyzers has changed significantly. Therefore, many of new instruments have been brought to market. These new instruments are able to produce fast, reliable on-line and off-line measurements. Furthermore the new instruments also include information such as width, kink, curl and other fiber properties like fiber morphology. Different types of modern fiber analyzers are the Kajaani FS 200, STFI Fibermaster and IPS MorFi. With these analyzers, it is also possible to measure the properties of fines. In case of optical fiber length analyzers, fines are defined as objects, which are less than 0.2 mm in length. The percentage on a length weighted basis is defined as the sum of the fines length divided by the total number of fibers multiplied by 100. For example, the Kajaani FS-200 calculates the percentage of fines based on an arithmetic basis as well as a length weighted basis (Guay et al. (2005)).

The most popular particle size measurements are the numerical, the length-weighted and the weight-weighted(mass weighted) fiber length, defined as (Carvalho et al. (1997)):

$$L_n = \frac{\sum n_i \cdot l_i}{\sum n_i} \tag{2.16}$$

$$L_l = \frac{\sum n_i \cdot l_i^2}{\sum n_i \cdot l_i} \tag{2.17}$$

$$L_w = \frac{\sum w_i \cdot l_i}{\sum w_i} \tag{2.18}$$

$L_n =$ *numerical average length*
$L_l =$ *length-weighted average length*
$L_w =$ *weight-weighted average length*
$n_i =$ *number of fibers in the ith class*
$l_i =$ *mean length of the ith class*
$w_i =$ *weight or mass of fibers in the ith class*

# 3 Materials and methods

The tube flow fractionation device is presented in Chapter 3.1 and the construction in Chapter 3.2. Chapter 3.3 presents the experimental part and chapter 3.4 describes the materials used in the fractionation process. In the end, chapter 3.5 presents the the fiber analysis using the Kajaani FS-200.

## 3.1 Principle of the tube flow fractionation

The fractionation phenomena in flowing pulp suspensions was investigated in the 1950's by Masonat at Paprican as well as Johansson and Kubat at the Swedish Pulp and Paper Research Institute (STFI) in 1956. In the 1970's investigations with the first tube flow fractionator were carried out at the Ecole Française de Papeterie et des Industries Graphiques (EFGP) in Grenoble (Laitinen (2011)). The separation of particles and fines with the tube flow fractionation method is a field flow separation process. With this device, it is possible to fractionate particles over the whole size range of pulp and fines (1 - 5000 $\mu$m) with high selectivity. In this experiment the particles and fibers are separated axially. The largest ones approach at the front end of the flow plug and the smallest ones at the end. At the beginning of the experiment, the sample is injected into the tube which is full of water and all particles are distributed randomly. After the beginning of the flow, the particles begin to move with the flow and start to move randomly inside the flow because of the slight turbulence in the transition flow regime. The small particles will remain in the fluid regime that is close to the tube wall. For efficient particle separation, the flow condition should differ from a laminar flow profile. In this study, a Reynolds

number of 6240 was used, which is defined as a turbulent flow condition. A laminar or turbulent flow depends on fluid friction (viscosity) and flow inertia. The ratio of inertia to viscous forces is defined as the Reynolds number $Re = \frac{U \cdot d}{\nu}$, where U stands for the flow velocity, d for the tube diameter and ν for the kinemtatic viscosity of the fluid. As a general rule, the tube flow is laminar if the Reynolds number is less than 2300 and turbulent if the Reynolds number is higher than 2300 (Spurk and Aksel (2006)) .

Large particles also drift close to the tube wall, but due to their larger dimensions, they may be recaptured by the middle flow due to their higher velocity. The probability of being captured by the middle flow is higher for single long dimensions (e.g. fibers) than for short particles (e.g. fines). It has been observed, that separation according to the length of the pulp particles is the dominant effect of fractionation for tube flow fractionation. The consistency of the sample must be below 0.5 %, otherwise the fibers are going to form a coherent fiber network (Krogerus and Fagerholm (2003)). In fact of suspensions, fibers will interact with each other, so the diameter of the tube as well the concentration of particles have an effect on the separation (Hemström et al. (1976)). Hydrodynamic forces play an important role in particle migration. The particles generally tend to avoid the tube wall. Some particles that settle to the bottom of the tube do not pass through the tube as easily as the particles carried out by the water flow. Furthermore, the Brownian motion effect plays a minor role, because pulp particles components are too large (1 $\mu$m) (Schimpf et al. (2000)).

Finally, the density of the suspension must be close to that value of water to ensure that lift forces are the most dominant forces to keep the particles from the so called lubrication layer away, which is a thin layer of pure water, which is developed close to the tube wall (Jäsberg et al. (2008)).

## 3.2 Design of the tube flow fractionator

The tube flow fractionator produces fractions from different types of pulp. The main component of the tube flow fractionator is a 100-meter-long teflon tube that is wound around a wheel, which is presented in figure 3-1. The sample is injected into a three way valve which is connected with a second one (see figure 3-3). These valves are used to allow any air that is inside the pipe to be removed from the fractionation system. After the sample is injected and free of air bubbles, the three-way valve (figure 3-3) is turned so that the continuous water flow can transport the sample into the tube system.

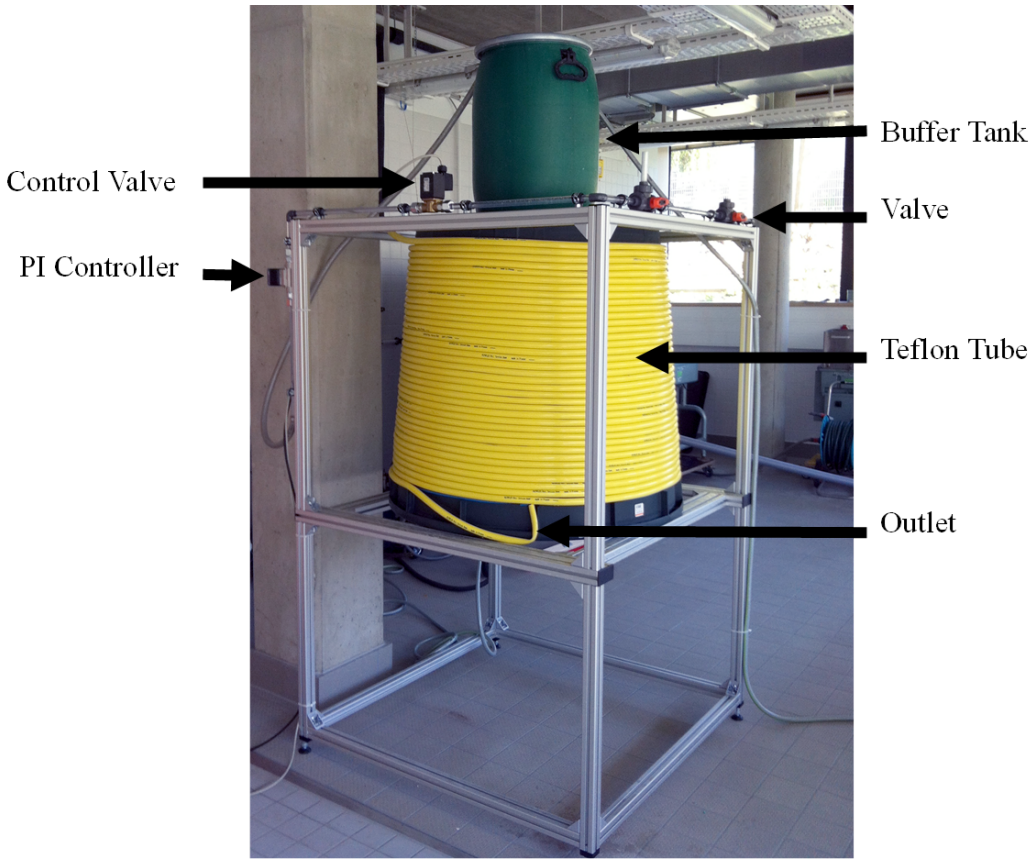


**Figure 3-1** *The tube flow fractionator device.*

The tube is mount onto a wheel that is mount on a frame construction and is positioned 700 mm above the ground to ensure easier sampling. A buffer tank is mounted on top of the drum. The tank is needed, to dampen pulsations originating from the groundwater supply. Pulsations adversely affect the distinctive turbulent flow that enables particles to separate cleanly. To ensure a continuous and controlled flow, a PI (Type Bürkert e-Control 8611) flow controller and flow sensor (Type Bürkert S030 inline fitting) are installed and connected to a proportional valve (Type Bürkert 2835) (figure 3-2).


**Figure 3-2** *PI controller for flow measurement and flow control.*

The pulp sample is fractionated into different size categories when the suspension flows through the tube. When the mixture of water and pulp exits the long tube, it is divided into size fractions according to the setup of the device.In the next step different fractions are analyzed using devices such as a fiber length analyzer or a microscope.

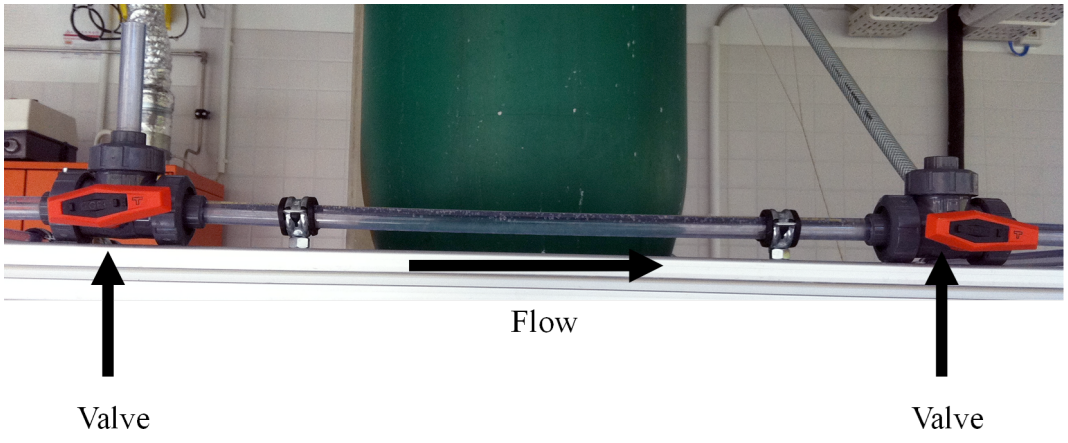


**Figure 3-3** *Three-way valves for sample filling.*

Parameters of importance during fractionation, including flow velocity, temperature, sample volume and consistency, were all maintained precisely at fixed levels to determine the effect that each parameter has on fractionation. The essential variables are shown in table 3-1.

| Parameter or variable | Value |
|---|---|
| Flow rate | 2.5 l/min |
| Water temperature | 19°C |
| Length of fractionator | 100 m |
| Inner diameter of fractionation tube | 16 mm |

**Table 3-1** *Different parameters and values of the fractionation process.*

Due to close control of parameters such as temperature and flow velocity, good reproducibility of fraction separation was observed when pulp samples were tested repeatedly. The most important factor for the fractionation process was to identify the correct Reynolds value since fractionation can only work at a turbulent flow condition ($Re > 2300$).

## 3.3 Measurement procedure

The next section describes the measurement procedure with the tube flow fractionator, sample preparation, sample injection and fraction sampling.

### 3.3.1 Sample preparation

The consistency of the original pulp is too high for fractionation with the tube flow device. In a first step a sample with a consistency of 0.5% was produced. To avoid damaging of damaging the capillary of the Kajaani FS-200, it was necessary to remove nibs and shives from the pulp. A laboratory mixer was used to prepare the samples for fractionation. After blending, the feed pulp was put into a 500 ml beaker for storage.

### 3.3.2 Sample injection

The sample injection unit consists of two 3 way valves which keeps air from the injected sample. The two valves are connected via a transparent PVC pipe that enables an observation of the sample injection process (3-4).

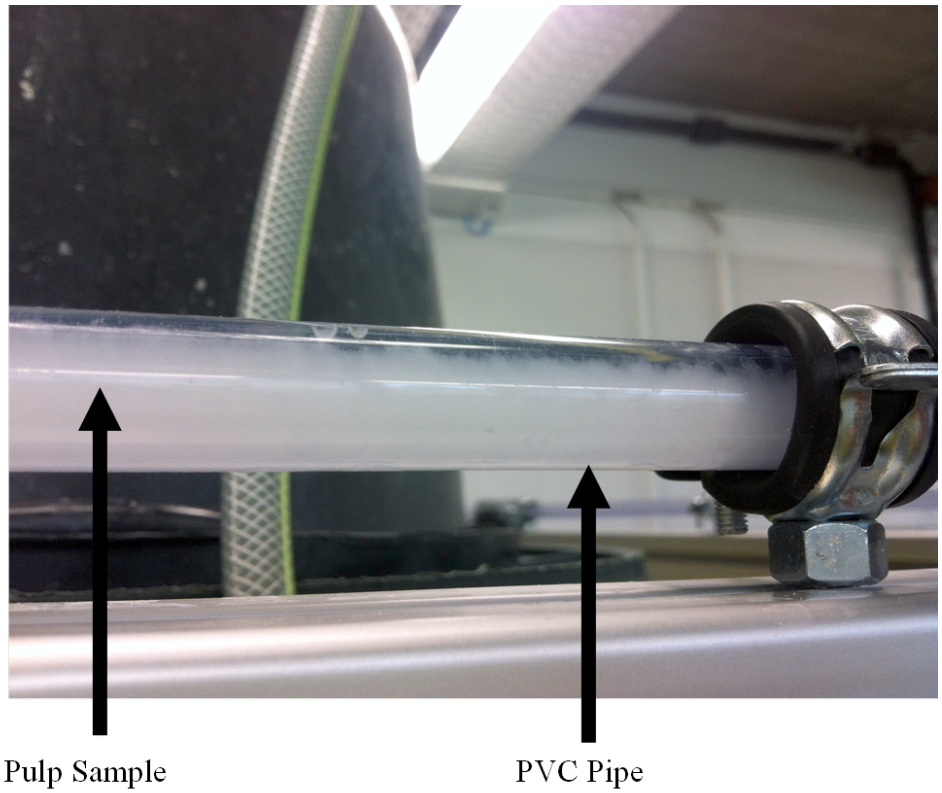


**Figure 3-4** *Visual control of sample filling.*

The sample injection is performed using a common 50 ml laboratory pipette. After addition of the sample into the measurement chamber and returning the valves into flow position, the fractionation process can then proceed. Figure 3-5 shows sample injection procedure.

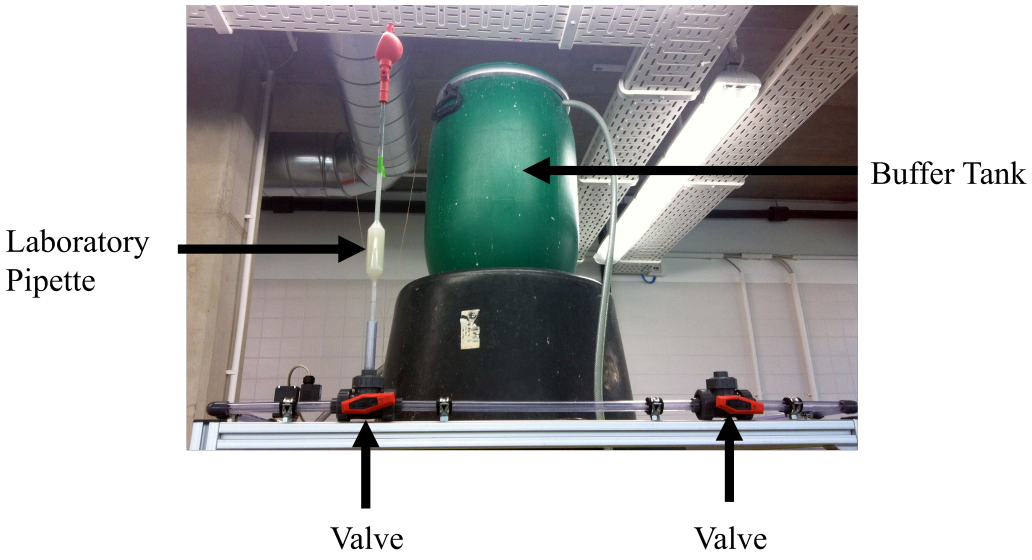


**Figure 3-5** *Sample injection with inserted 50 ml pipette.*

### 3.3.3 Sampling

The fractionated samples are collected in different beakers where the first contains the largest particles and the last contains the smallest ones. Figure 3-6 illustrates the method used for sampling. In general, 7 to 10 beakers are prepared. These beakers are put under the end of the tube. At predefined time intervals, the fractionated suspension was directed into the predefined beakers. In this study, time intervals ranging from 15 to 30 seconds were used. Shorter time periods are not possible because of the low consistency of the suspension, which makes it impossible to measure it with the Kajaani FS-200 device. The measurement time depends on both, the flow rate and the type of pulp (fines content, length of fibers etc.). Sulfate pulp (unbleached kraft pulp) takes less time (5 min) to go through the fractionator compared to PGW pulp (5.5 min) which contains more fines and short fibers.

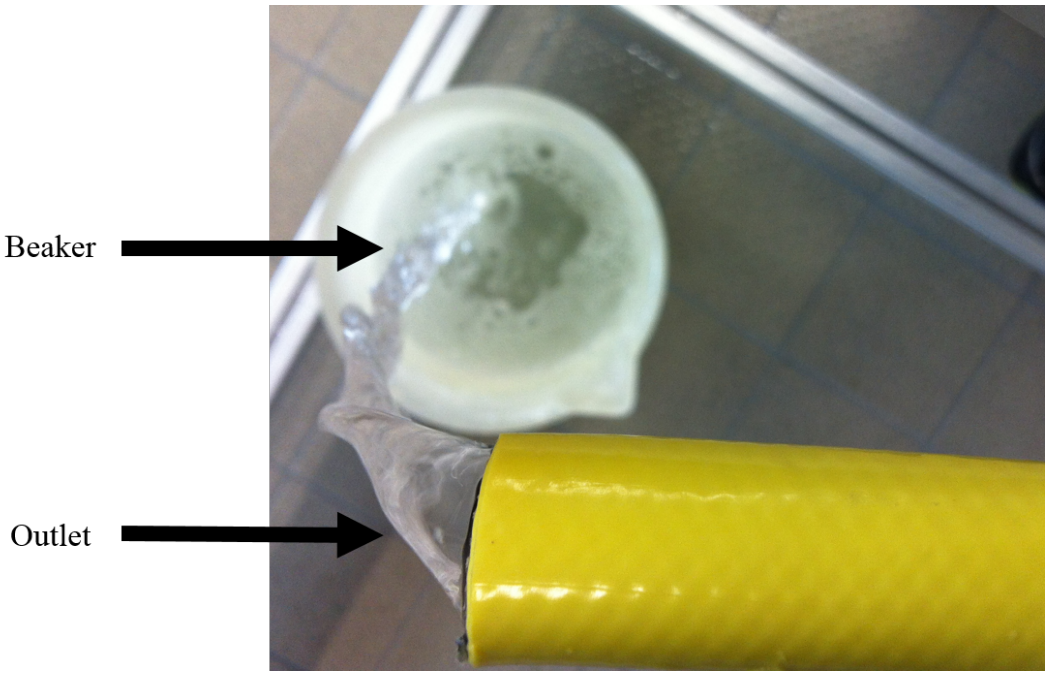


**Figure 3-6** *Sample taking by a flow rate of 2.5 l/min and sample filling time of 30 seconds.*

### 3.3.4 Pre tests for time strategy

Between sample injection and sample collection, the dwell time of the sample which flows through the tube has to be determined. It is necessary to develop a sampling strategy. First a model system was conducted by using plastic granulates. Therefore, the elution time was measured for a given flow rate. After this initial step, tests on pulp samples were carried out. The dwell time of fibers and fines in the tube flow fractionator depends on the shape and size.

Another way to determine the dwell time is to estimate the flow velocity. The flow can be calculated, if the flow rate and the diameter of the tube are well-known. For an exact determination of the dwell time, a wire of a paper machine was placed over a beaker. The elution time is defined as the time when the first fibers are retained at the wire. After the determination of the dwell time it is possible to establish a time strategy for sampling.

### 3.3.5 Characterization of fractions

In this study, fiber fractions from the first to the last beaker can be characterized to contain long fibers, short fibers and fines. Table 3-2 presents the fractions obtained with the tube flow fractionator and the average particle size as measured by the

Kajaani FS-200 of a kraft pulp with a flow rate of 2.5 l/min (0.39 m/s). In the case of a chemical pulp, the fraction 20–40 sec to fraction 60–80 sec observed to contain long and short fibers, while fraction 80–100 sec contained mostly fines. When the first sample is taken, a certain volume presents a certain size of particles. Therefore it is possible to control the collected size ranges for each fraction which may differ depending on the type of pulp.

| fractions sec | tube flow liter ranges l | average fiber length mm |
|---|---|---|
| 0–20 | no fibers/fines | unable to analyze |
| 20–40 | 0–0.825 | 2.70 |
| 40–60 | 0.825–1.665 | 1.04 |
| 60–80 | 1.665–2.500 | 0.22 |
| 80–100 | 2.500–3.325 | 0.13 |

**Table 3-2** *Liter ranges of different fractions.*

If the flow velocity is known, the volume or mass of each fraction can be determined. Therefore, the tube length of a fraction (sample which is distributed as a fraction in the tube after 100 m)which comes out of the tube flow fractionator can be also calculated. Table 3-3 shows the tube length distribution in the tube of fractions taken at time intervals of 15, 20 and 30 seconds at the end of the 100 m tube. In contrast to the tube fraction length at the end of the tube flow fractionator, the injected sample offers a tube length of about 200 mm. In order to determine the mean arithmetic length of fibers and fines with an optical analyzer device, it was necessary to further condition the various sample fractions. The consistency of these samples had to be increased, which was achieved by letting the fibers settle and then decanting the supernatant liquid.

| fraction time range sec | tube fraction length after 100 m |
|---|---|
| 15 | 3,15 |
| 20 | 4,2 |
| 30 | 6,3 |

**Table 3-3** *Tube fraction length after 100 m of fractionation.*

## 3.4 Samples

In the present study two different types of pulps were used. The first one was a pressurized groundwood (PGW) which was fully bleached, unrefined and never-dried. The pulp was produced at Norske Skog Bruck/Mur (3-7). The second sam-

ple was an unbleached softwood kraft pulp (mixture of spruce and pine) that was unrefined and never-dried from Mondi Frantschach (3-8). For each pulp type two batches were delivered, both used for the trials. The pulp samples were delivered in a never-dried state, with a consistency of 0.4 % and stored at a temperature of 6°C. The chemical pulp was used to investigate the behavior of typical long fiber pulps in the tube flow apparatus. Fractions of the PGW pulp were used to investigate the behavior of the fines fractions (PGW pulps containing more fines than kraft pulps).

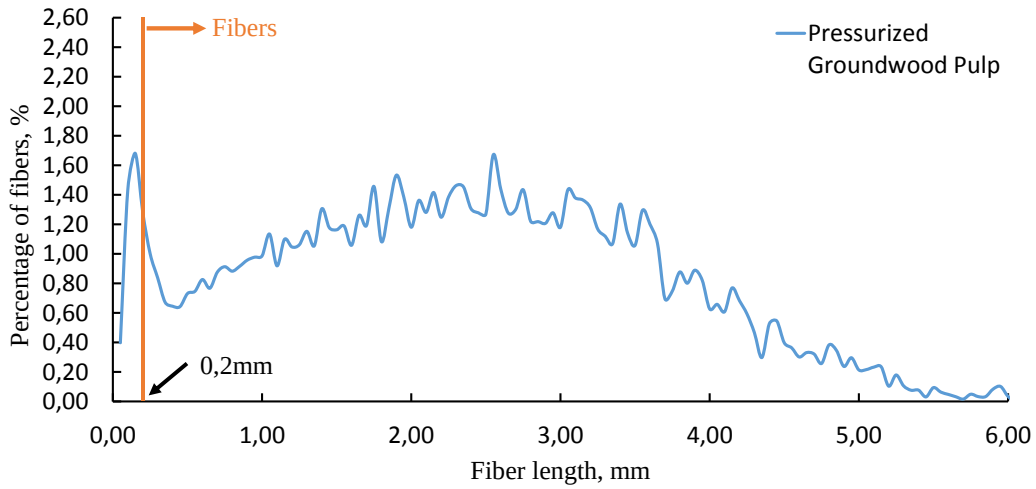


**Figure 3-7** *Bleached PGW pulp sample investigated with Kajaani FS-200.*

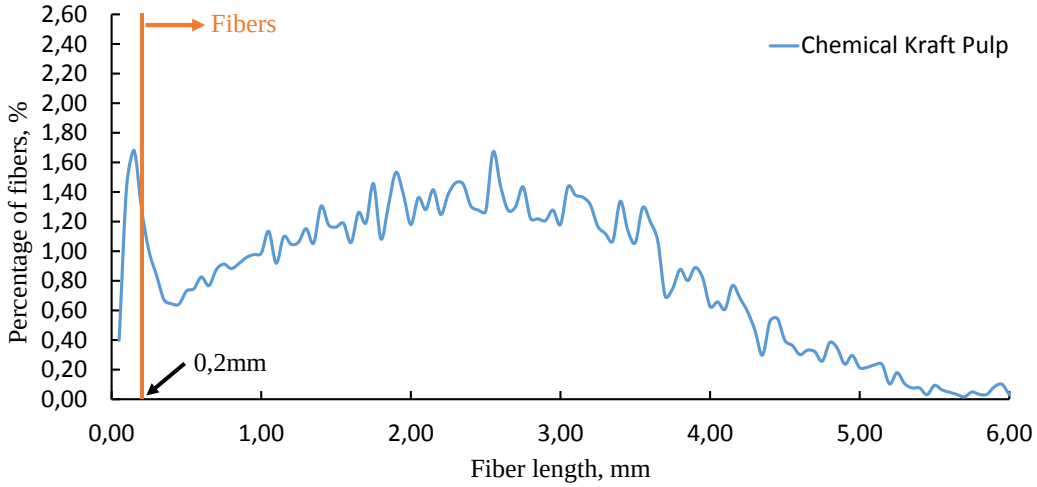


**Figure 3-8** *Kraft pulp sample investigated with Kajaani FS-200.*

## 3.5 Fiber Analysis using the Kajaani FS-200

The Kajaani FS-200 unit is designed to measure the fiber-length distributions of fibers and fines. The FS-200 is used extensively in the pulp and paper industry because the method is simple and fast. The Kajaani FS-200 analyzer is designed to be a process control instrument. It is fully automated and includes a capillary tube which changes the tube diameter from 0.2 to 0.4 mm automatically. There is always enough vacuum to ensure that fibers are straight in the capillary tube. A laser light source for illuminating fibers and fines is used and on the opposite side, a highly sensitive detector is set up to determine these particles (Cöpür and Makkonen (2007)).

This analyzer uses an indirect optical technique for fiber analyzing, so it is subject to artifacts that might cause errors in the fiber length analysis. For example, if two fibers stick together and enter the capillary tube together, they will be counted as a single fiber. Also, if fibers do not enter the capillary tube perfectly aligned, then the measured projected length will be smaller than the aligned actual. There are also problems when using samples with a low consistency due to a little amount of fibers and fines. All samples were prepared as stock suspensions of approximately 0.010 % consistency before dilution in the Kajaani instrument. The number of evaluated objects per sample was about 30000 fibers or fines. To achieve the desired consistency, it was necessary to allow the fibers and fines to settle and

than decant a defined amount of the supernatant water. Duplicate measurements of the suspension were performed.

# 4 Results

## 4.1 Fractionation of different pulp samples

In this chapter the fractionation of different pulp samples using the tube flow fractionator is demonstrated. The distribution of fibers and fines of the fractions of the tube flow device on the one hand and the Bauer McNett fractionator on the other is investigated.

### 4.1.1 Experiments on PGW pulp

If the wood is ground in a pressurized and sealed grinder the pulp is defined as pressure groundwood pulp (PGW). PGW offers a high amount of short fibers and fines. Based on preliminary testing of PGW pulp fractions, the parameters in table 4-1 were used for subsequent experiments.

| Flow rate | Time range | Dwell time in tube | Temperature |
|---|---|---|---|
| 2,5 l/min | 15 sec | 5 min | 19°C |

**Table 4-1** *Basic parameters for PGW pulp investigations.*

Figure 4-1 shows the distribution of fibers of the first fraction after 15 seconds (TF 15) and the distribution of fibers of the whole sample. Fraction 15 shows a high amount of fibers with an average arithmetic fiber length of about 1.65 mm. The fraction clearly consists only of long fibers ranging from 0.5 mm to 4 mm.

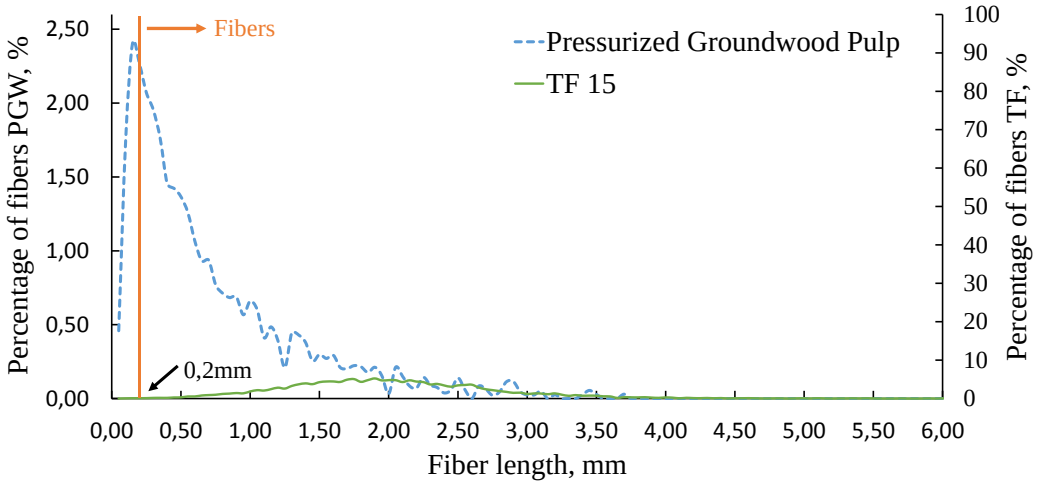


**Figure 4-1** *Length distribution of the PGW pulp fraction after 15 seconds compared with the whole PGW pulp.*

Figure 4-2 shows the second fraction collected after 30 seconds (TF 30). The value of the mean arithmetic fiber length is 0.56 mm. For fraction 45 (TF 45), shown in figure 4-3, the mean arithmetic fiber length reaches values of 0.2 mm. These two fractions are considered to be composed mostly of pulp fibers with only a small amount of fines. The boundary where particulate matter is considered as fibers greater than 0.2 mm. Particles finer than 0.2 mm are considered to be fines in this study.

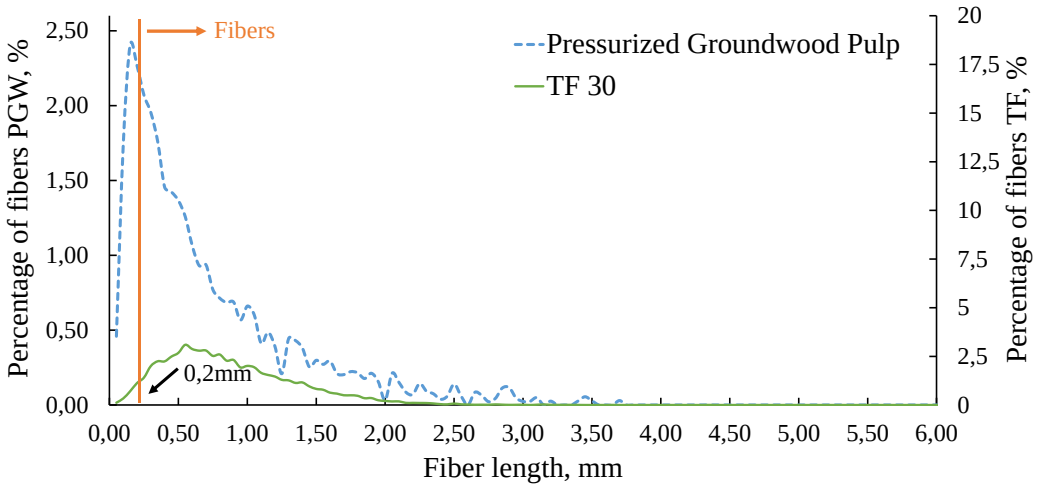


**Figure 4-2** *Length distribution of the PGW pulp fraction after 30 seconds compared with the whole PGW pulp.*

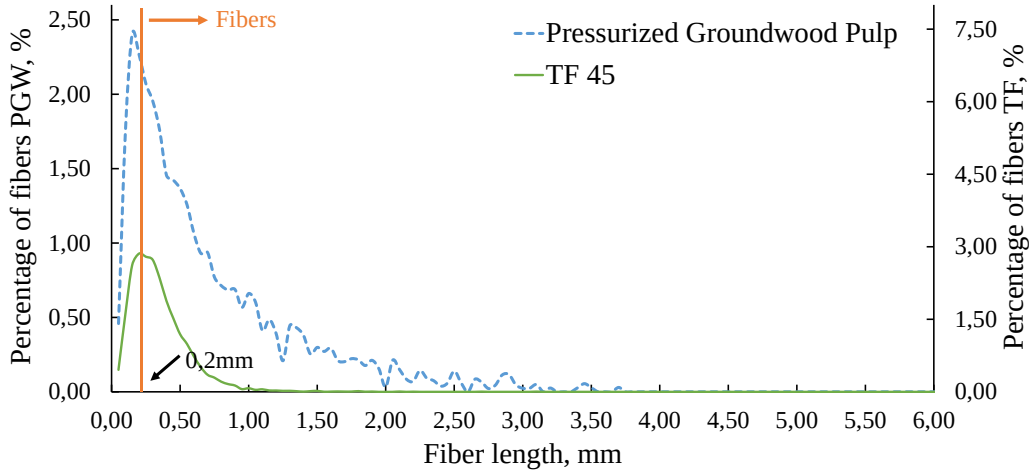


**Figure 4-3** *Length distribution of the PGW pulp fraction after 45 seconds compared with the whole PGW pulp.*

The average arithmetic particle length of fraction 60 (Figure 4-4) is 0.12 mm and is identified as the first fraction composed almost entirely of fines. Fraction 75 offers an arithmetic fines length of 0.10 mm, which is also composed of fine particulates. The tube flow fraction 75 is presented in figure 4-5. It was not possible to measure the size distribution of fraction 90 because of its low consistency (small amount of particle in fraction).

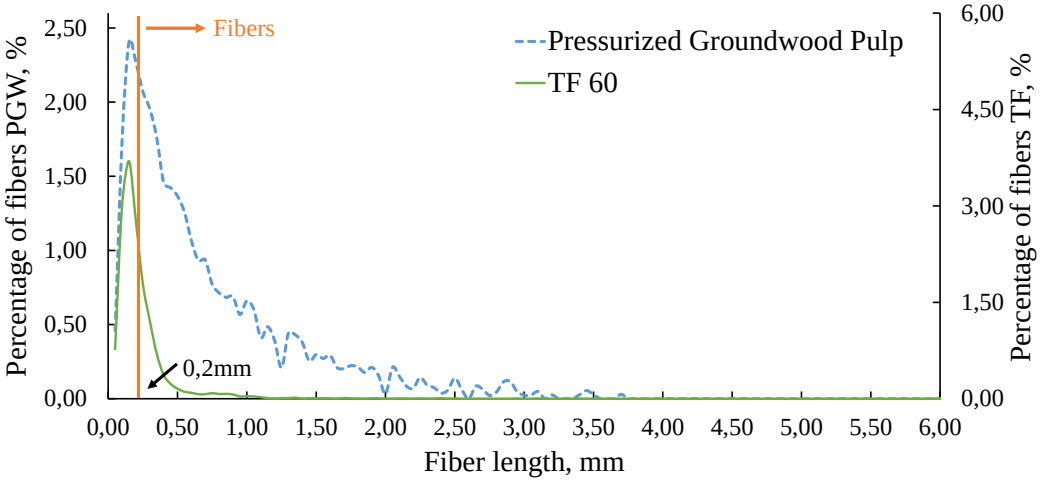


**Figure 4-4** *Length distribution of the PGW pulp fraction after 60 seconds compared with the whole PGW pulp.*

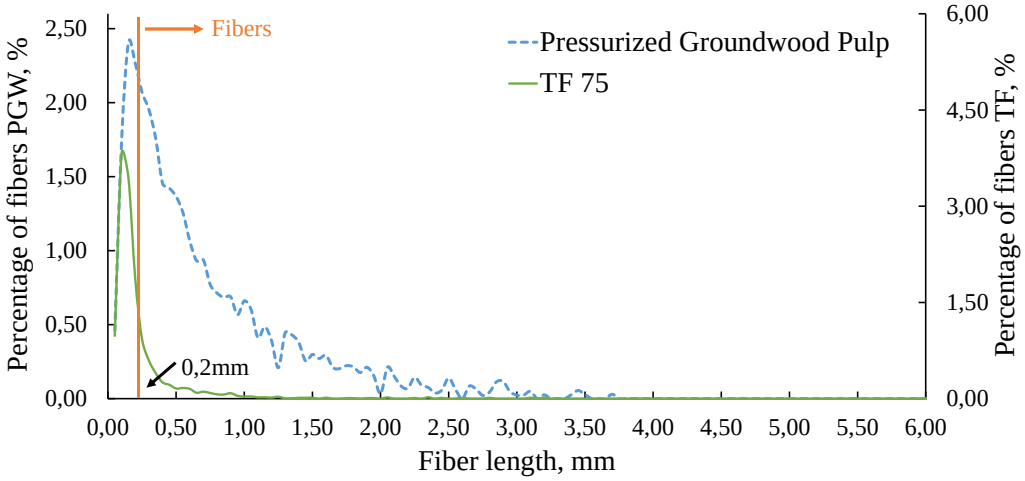


**Figure 4-5** *Length distribution of the PGW pulp fraction after 75 seconds compared with the whole PGW pulp.*

#### 4.1.1.1 Comparison of PGW fractions

Figure 4-6 depicts the whole fractionating process containing fraction 15 to fraction 75. Fraction 15 to fraction 45 consist mostly of fibers while fraction 60 to fraction 75 are considered fine particles. In this figure, the distribution of the fractions are adjusted to fit under the distribution of the whole pulp sample. With increasing time, the fines content increases and the fiber content decreases. Fraction 0 could not be analyzed due to the low amount of mass in the fraction.

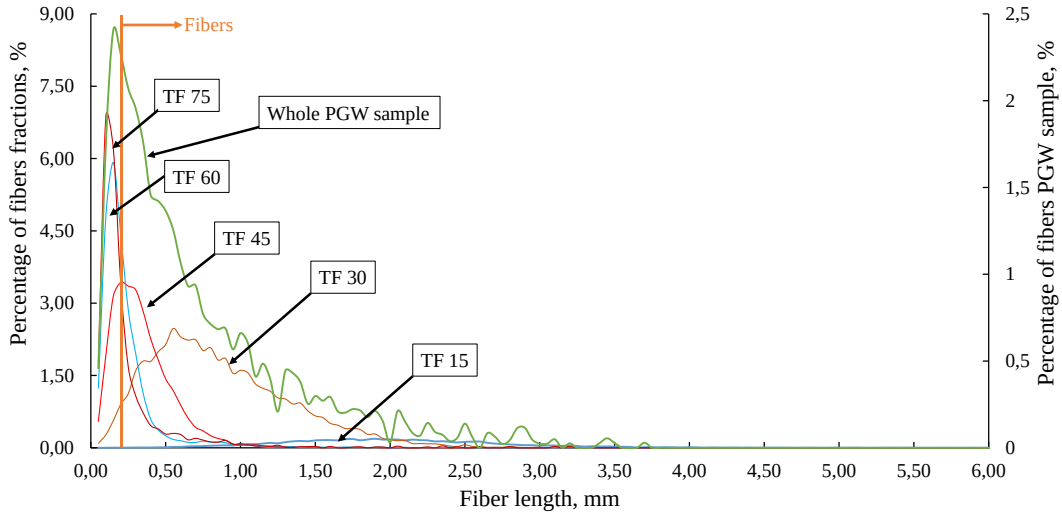


**Figure 4-6** *Distributions of all fractions and the whole pulp sample combined within one diagram for illustration.*

### 4.1.2 Experiments with unbleached kraft pulp

The fiber length distribution of chemical pulps shows more long fibers and less fines compared to PGW pulp. Based on previous investigations, a flow rate of 2.5 l/min, a time range of 20 sec and a temperature of 19°C were chosen. From a comparison of PGW pulp fractions and chemical pulp fractions, it is evident that PGW pulps contain more fines and chemical pulps have longer fibers. In the study of kraft pulp, four fractions with a time range of 20 seconds per fraction were collected and a comparison was done for all fractions, as is shown in the figures that follow. Fraction 0 could not be analyzed due to the small amount of fibers that were collected.

In figure 4-7 the TF 20 fraction and the whole kraft pulp sample are compared. For the TF 20 fraction, the mean arithmetic fiber length reaches a value of 2.70 mm. This fraction is considered to be composed mostly of long pulp fibers with a small amount of short fibers.

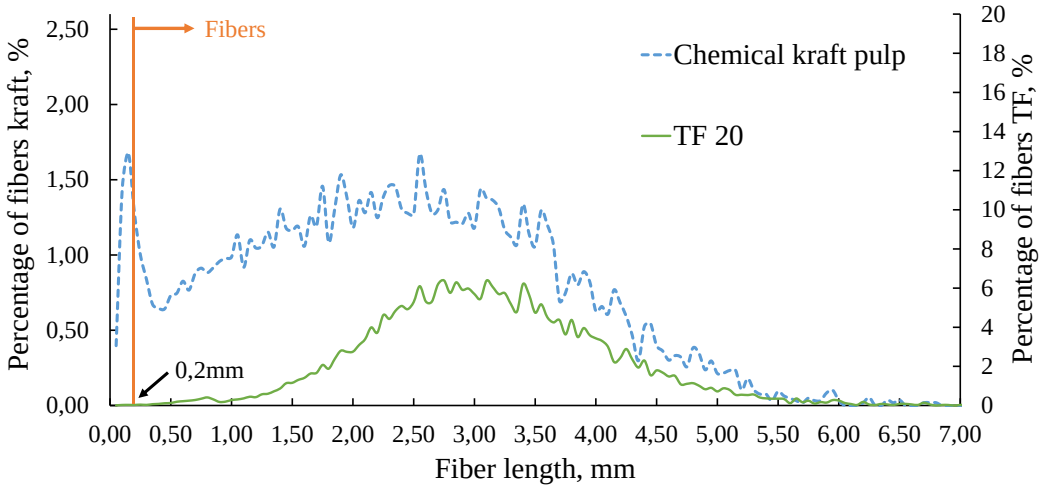


**Figure 4-7** *Length distribution of the kraft pulp TF 20 fraction and the whole kraft pulp.*

Figure 4-8 shows the TF 40 fraction and the whole kraft pulp sample. This fraction shows an increase in short fibers and is also considered to be composed of a small amount of fines. In this fraction, the mean arithmetic fiber length reaches a value of 1.04 mm.

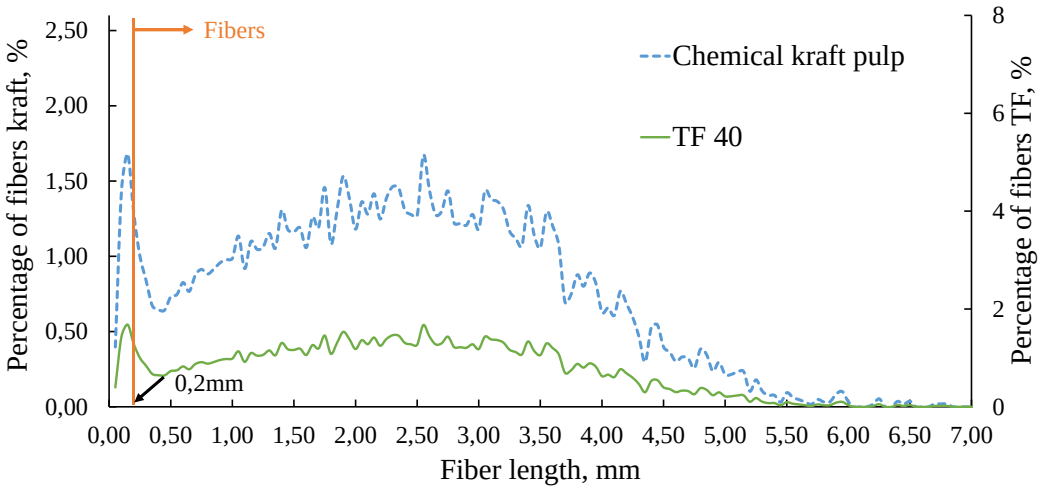


**Figure 4-8** *Length distribution of the kraft pulp TF 40 fraction and the whole kraft pulp.*

The TF 60 fraction is shown in figure 4-9. It reaches a mean arithmetic fiber length of 0.22 mm. Results of the Kajaani FS-200 fiber classes calculation show, that there are more than 23 percent of all particles under a fiber length of 0.20 mm. It is evident, that there is an increase in fines and a decrease in long fibers.

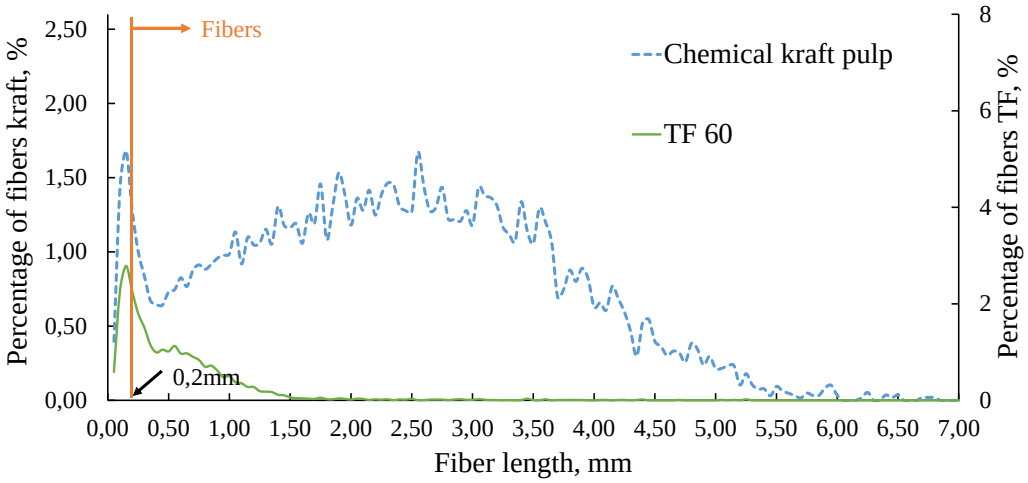


**Figure 4-9** *Length distribution of the kraft pulp TF 60 fraction and the whole kraft pulp.*

In figure 4-10, the length distribution of the TF 80 fraction is presented. The mean arithmetic fiber length is about 0.12 mm. It is evident that this fraction shows a sharp peak for the percentage of fines. In this length distribution, the percentage of particles which are smaller than 0.2 mm is about 31%. This fraction is defined as the fine fraction of the kraft pulp sample.

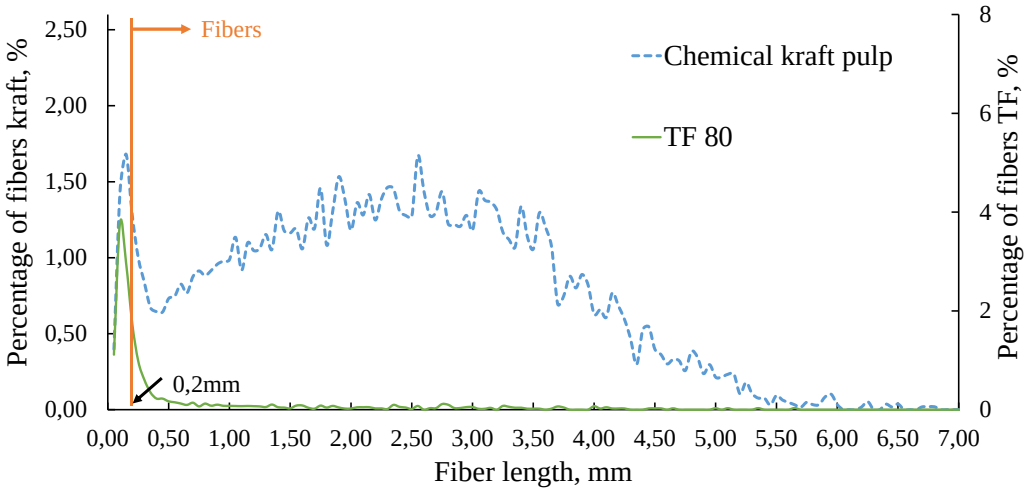


**Figure 4-10** *Length distribution of the kraft pulp TF 80 fraction and the whole kraft pulp.*

#### 4.1.2.1 Comparison of the tube flow kraft pulp fractions and the whole kraft pulp sample

In this section, a comparison between different tube flow fractions of kraft pulp and the whole kraft pulp is shown. Figure 4-11 shows all tube flow fractions of the chemical pulp. The TF 20 and TF 40 fractions consist mostly of fibers while fraction 60 to fraction 80 contain an increased amount of fines, TF 80 fraction is beeing composed of fines to a high extent. In this figure, the length distribution are adjusted to fit under the distribution of the whole pulp sample for better illustration of the composition of the fractions. The resolution of the fractions of the kraft pulp is not that good compared to that of PGW pulps mostly because it's fines content is lower. It is also clear that the kraft pulp shows much less fines.

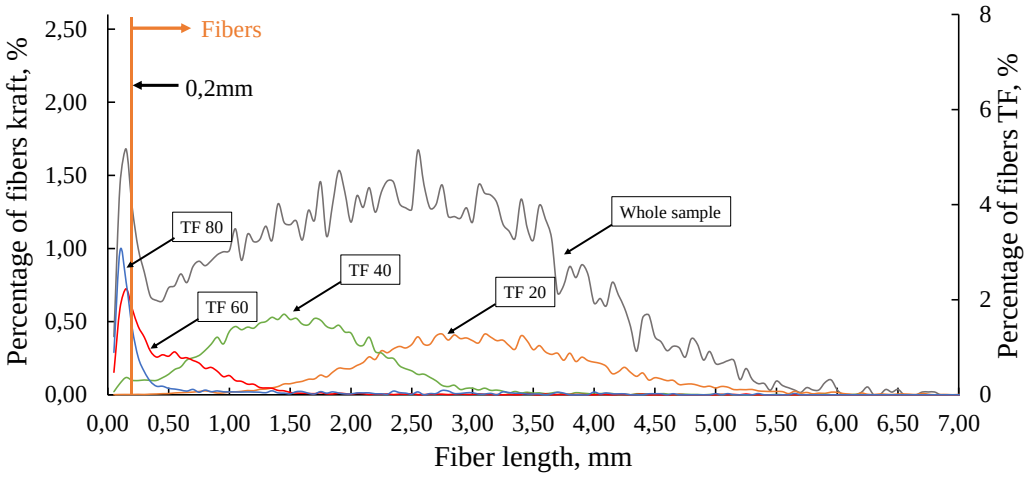


**Figure 4-11** *Distributions of all kraft pulp fractions and the whole kraft pulp sample combined within one diagram for illustration.*

## 4.2 Repeatability of the method

It is widely accepted, that the repeatability of different Bauer McNett devices is limited (Bos (1966), Gooding and Olson (2001)). In this chapter, the repeatability of the tube flow fractionator is examined. Four independent samples were prepared and each was tested in the tube flow device. The results of the repeatability tests are shown in table 4-2, where the arithmetic mean of each run is given. It was not possible to measure the size distribution of run 4 for the 0 seconds fraction due data transfer problems with the Kajaani FS-200.

There are possible effects and process steps which affect the repeatability: Unprecise sample preparation might cause inaccuracies which can be observed in

the following experiments. It is possible that inaccuracies in sample injection such as air bubbles between the pulp sample in the tube, or inaccurate sample collection from the tube flow device, or problems during fiber length determination with the Kajaani FS-200 analyzer might have contributed to the observed differences.

| | TF after 0 sec | TF after 30 sec | TF after 60 sec |
|---|---|---|---|
| arithmetic mean run 1 | 1,081 | 0,288 | 0,112 |
| arithmetic mean run 2 | 1,135 | 0,296 | 0,116 |
| arithmetic mean run 3 | 1,128 | 0,317 | 0,118 |
| arithmetic mean run 4 | - | 0,284 | 0,112 |

**Table 4-2** *Arithmetic mean of fiber length of 4 independent pulp samples.*

The tube flow device shows a high repeatability based on the arithmetic mean. It should be noted that the variations of the values are getting smaller with increasing fractionation time (table 4-2). Figure 4-12 presents the 0 seconds tube flow fraction of the PGW pulp for three independent samples. In figure 4-13, the mean length distribution of three independent 0 seconds tube flow fractions including confidence intervals for each class is shown. In this length distribution, all trails produce similar results with minor changes. Also the deviation of the arithmetic mean of the fiber length represents only small differences between testing repetitions.

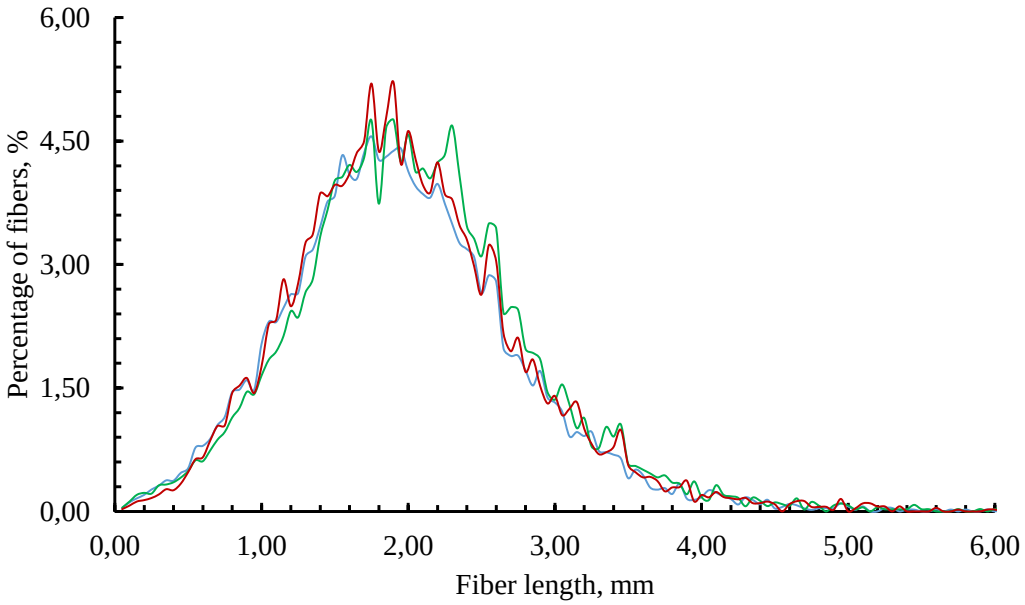


**Figure 4-12** *Repeatability length distribution of the 0 seconds tube flow fraction of the same PGW sample.*

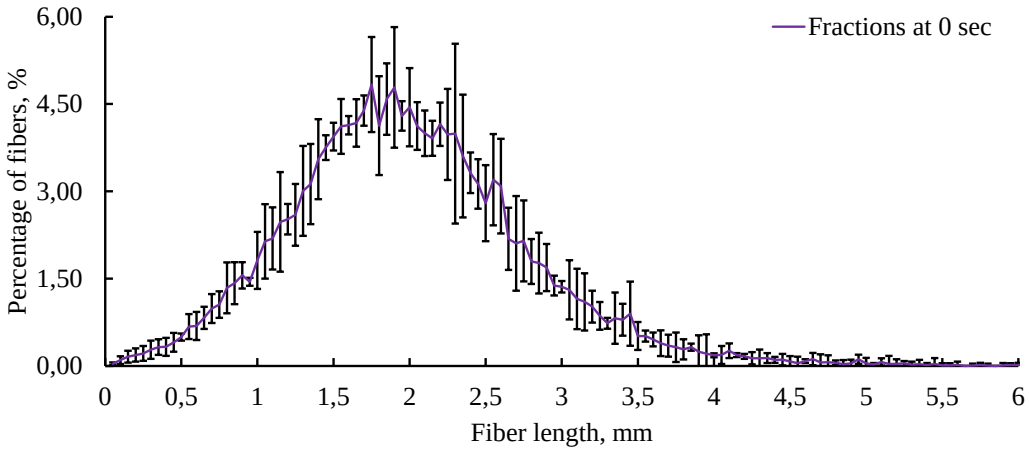


**Figure 4-13** *Repeatability mean of the length distribution including 95% confidence intervals of arithmetic fiber length of the 0 seconds PGW tube flow fraction.*

Figure 4-14 and figure 4-15 depict the examination of a PGW pulp sample within the fraction range between 30 and 60 seconds. The length distribution of the 30 seconds tube flow fraction, shown in figure 4-14 offers a similar shape. The 95% confidence intervals along the length classes in the distribution are rather small ( see figure 4-15).

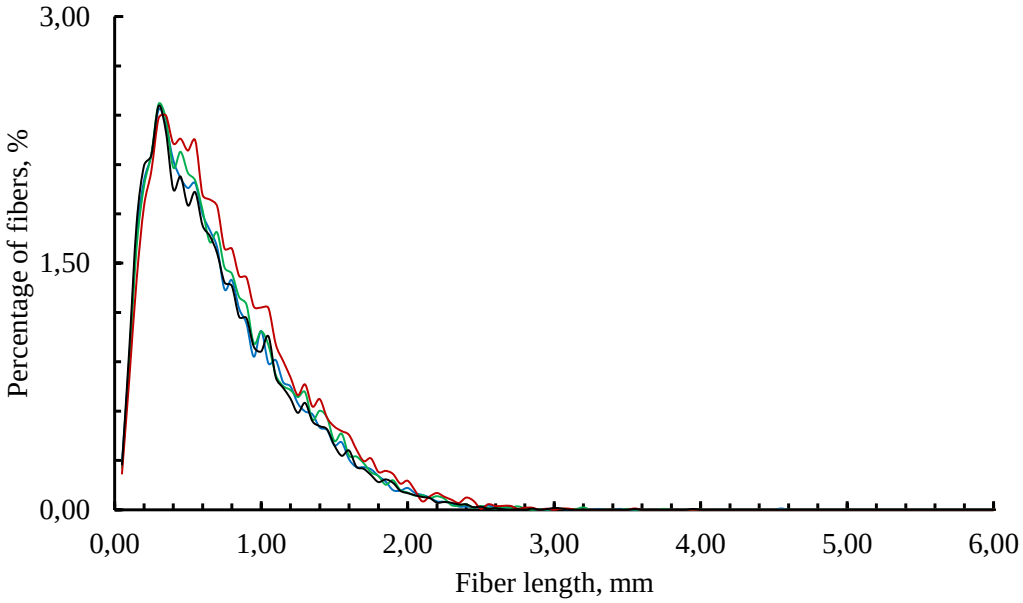


**Figure 4-14** *Repeatability length distribution of the 30 seconds tube flow fraction of the same PGW sample.*

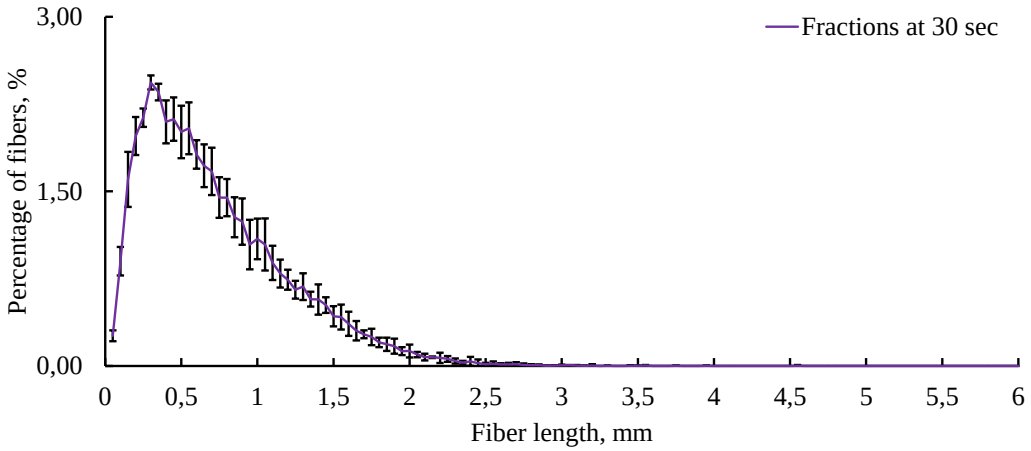


**Figure 4-15** *Repeatability mean of the length distribution including 95% confidence intervals of arithmetic fiber length of the 30 seconds PGW tube flow fraction.*

Figure 4-16 depicts the 60 seconds tube flow fraction of a PGW pulp of four independent samples. This length distribution shows a similar shape, only one distribution shows a small difference at the peak, which might result from the artifacts mentioned above. Figure 4-17 shows the mean length distribution of four independent 60 seconds tube flow fractions including 95% confidence intervals for each class. The variations of the arithmetic mean of the fiber length also show minor changes.

In summary, it can be concluded that the tube flow fractionator works very accurately if all important parameters such as water temperature, flow rate and consistence are kept constant.

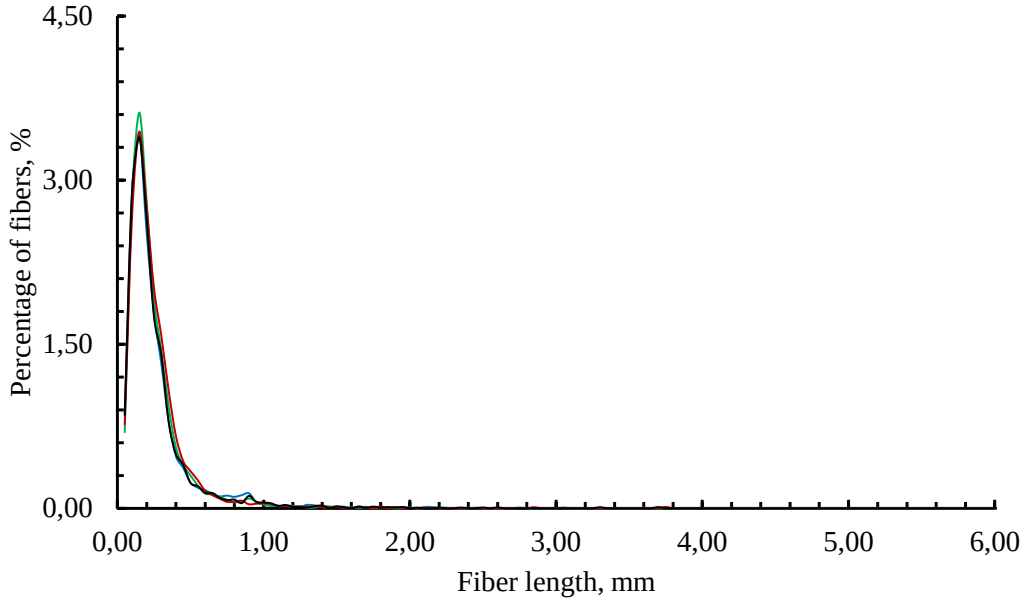


**Figure 4-16** *Repeatability length distribution of the 60 seconds tube flow fraction of the same PGW sample.*

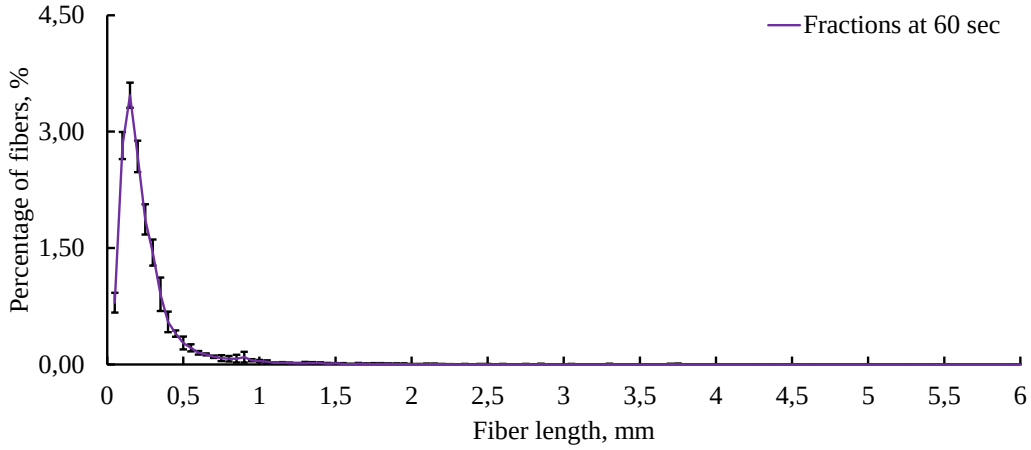


**Figure 4-17** *Repeatability mean of the length distribution including 95% confidence intervals of arithmetic fiber length of the 60 seconds PGW tube flow fraction.*

## 4.3 Experiments with the Bauer McNett fractionator

To compare the tube flow frctionator with the standard device used for fractionation, testing of the same samples with the Bauer McNett fractionation device was performed.

For the fractionation with the BMN-device, the same PGW and kraft pulp samples with similar sample sizes were used. The fractionation experiments with both types of pulp were carried out under equal conditions (flow rate, temperature etc.). For the fractional determination of fines, the 76 µm pass of particles of the BMN device was collected. Further on the fractionation with the BMN-device, all samples were analyzed with the Kajaani FS-200 analyzer.

The Bauer McNett fractionator also separates the pulp based on fiber length although fiber thickness and slenderness as well as flexibility has some impact (Ullman et al. (1968)). This is an important fact for the study of different pulp types, such as hardwood vs. softwood or chemical vs. mechanical pulps. Care should be taken when comparing the results obtained from different types of pulps. An in depth discussion of the BMN device can be found in literature (Ullman et al. (1968)).

### 4.3.1 Results of PGW pulp fractions

In this chapter, a comparison of different PGW pulp fractions were carried out with the Bauer McNett fractionator. In figure 4-18, different PGW pulp fractions are shown. For a better illustration of the composition of the fractions, the length distribution of the fraction sample were fit under the whole PGW pulp sample. About 87 per cent of the 30-mesh screen (FR30) fraction offers fibers which are bigger than 0.2 mm. The arithmetic mean of fiber length is 1.01 mm. For fraction 50 (FR50), which is defined as the 50-mesh screen fraction, the mean arithmetic fiber length reaches a value of 0.49 mm and for the 100-mesh screen fraction (FR 100) 0.24 mm. In fraction 200-mesh (FR200) an increase in finer particles is evident, also the amount of particles which are smaller than 0.2 mm shows an increases. In this case, the fines fraction is defined as particles which pass through the 76 µm BMN screen. When using finer screens the length distributions diverge more and more and is running quickly towards a higher fines content. For the fines fraction (FR fines), the mean arithmetic fiber length is 0.13 mm and about 97 per cent of particles are smaller than 0.2 mm.

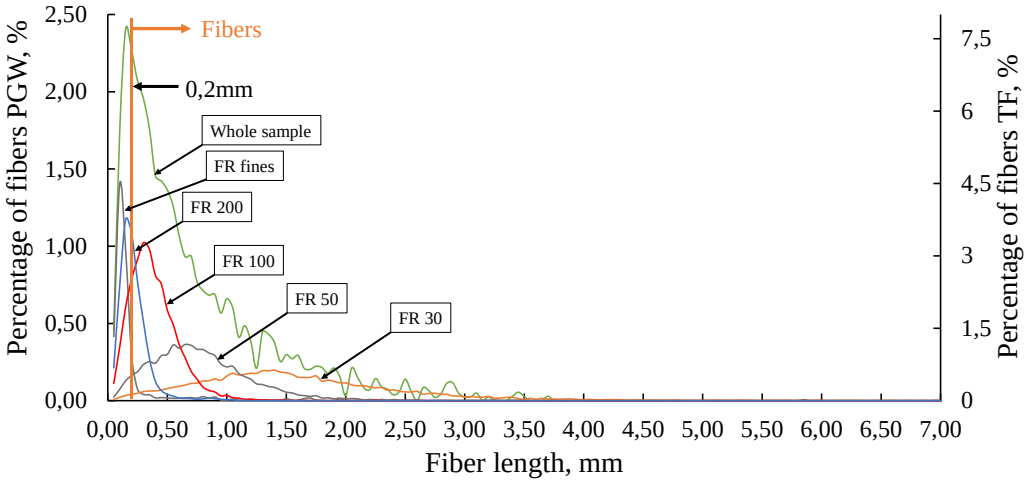


**Figure 4-18** *Length distributions of all PGW pulp fractions and the whole PGW pulp sample combined within one diagram for illustration.*

### 4.3.2 Results of kraft pulp fractions

In this section, a comparison of BMN-fractions of a kraft pulp was carried out. As mentioned above, all length distribution samples are adjusted to fit under the length distribution of the whole kraft pulp sample for a better illustration. Figure 4-19 shows the kraft pulp BMN fraction samples and the whole kraft pulp sample. Fraction 30-mesh (FR30) is defined as particles which retain on the first screen of the BMN apparatus. There are about 97 percent of fibers which are bigger than 0.2 mm. The mean arithmetic fiber length is 2.38 mm. For fraction 50-mesh (FR50), the value of the mean arithmetic fiber length is 1.12 mm. For fraction 100 about 0.60 mm. Fraction 200-mesh (FR200) indicates a higher amount of fines which reaches a value of about 97 percent for particles smaller than 0.2 mm. When particles pass through the 76 µm screen of the BMN-device, they are defined to be the fines fraction. The arithmetic fiber length of the fines fraction is 0.07 mm. In comparison with the distributions mentioned above, it is evident that there is a high amount of particles which is smaller than 0.2 mm. Furthermore, the length distribution of the fines fraction shows a narrow distribution. There are about 99 percent of particles which are smaller than 0.20 mm.

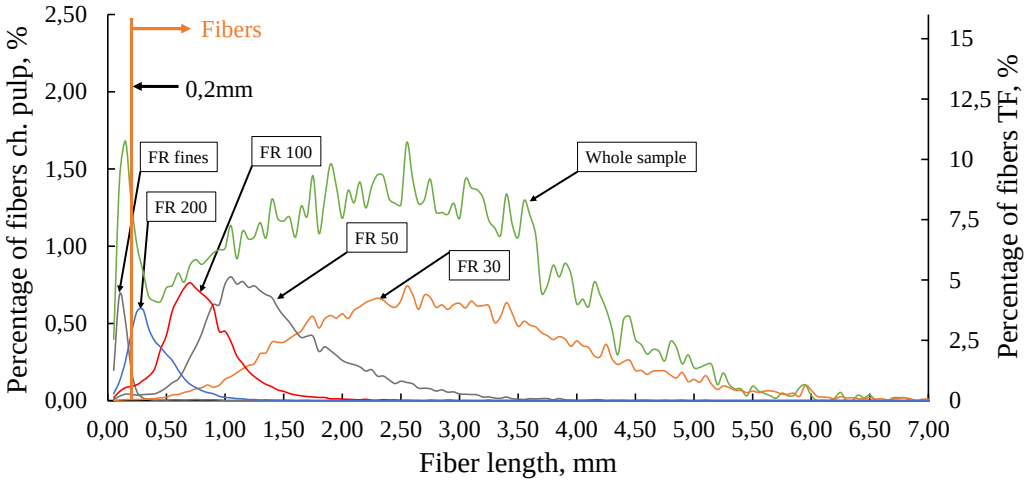


**Figure 4-19** *Length distributions of all kraft pulp fractions and the whole kraft pulp sample combined within one diagram for illustration.*

# 5 Discussion

Chapter 5.1 describes the method of fractionation with the tube flow fractionator, its validation based on experiments using two different pulp samples and the repeatability of the method. Chapter 5.2 shows a comparison between the tube flow fractionator and the Bauer McNett fractionator as the standard laboratory fractionator. To conclude, Chapter 5.3. gives an outlook about future applications with the tube flow device.

## 5.1 Fractionation with the tube flow device

The tube flow device was implemented as is described in the literature and a series of experiments was conducted to produce different fractions of different pulp types. It was shown, that the tube flow fractionator separates different pulp samples in classes over their entire fiber length. The results of these experiments can be compared with results from the literature (Krogerus and Fagerholm (2003)). An exemplary result based on a mechanical pulp sample (PGW) is shown in figure 5-1. After the 100 m long tube the injected pulp sample is distributed over about 16 to 20 m of tube length representing about 80 sec of flowing suspension at the exit of the tube. Sampling is done by collecting the suspension over a certain time interval. By changing this time interval one can change the composition of the collected sample without clear restrictions as they are evident in the standard laboratory fractionators due to the mesh of the applied screens. In this work the time intervals had to be large enough to collect enough objects for a Kajaani FS-200 measurement. Based on the pulp type the time interval for sampling has to be adjusted. As the

flow time of fibers, fines or generally objects depends on their length. Longer fibers or samples containing longer fibers will reach the tube exit after a shorter time period than a sample composed mostly of shorter fibers and fines. In case of the experiments discussed earlier in this work the softwood kraft pulp sample shows a shorter flow time than the PGW which is composed of shorter fibers and which has a higher amount of fines (as is evident in figure 5-2).

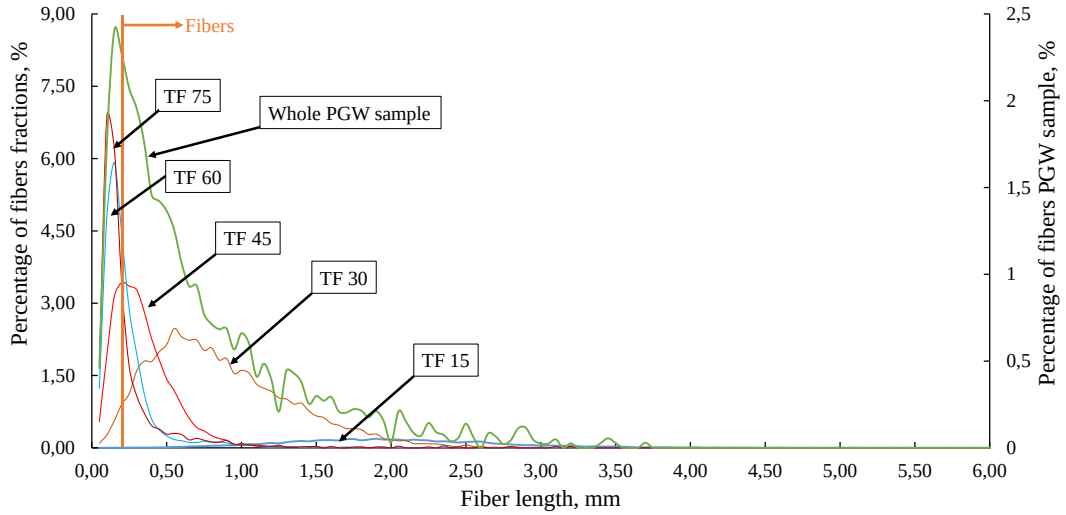


**Figure 5-1** *Fractionation results of the tube flow fractionator.*

When different pulp types are compared, a clear difference between both fractions can be observed in the distribution of length (Figure 5-2). Furthermore it can be observed that the shape of each length distribution is a result of the pulp type.

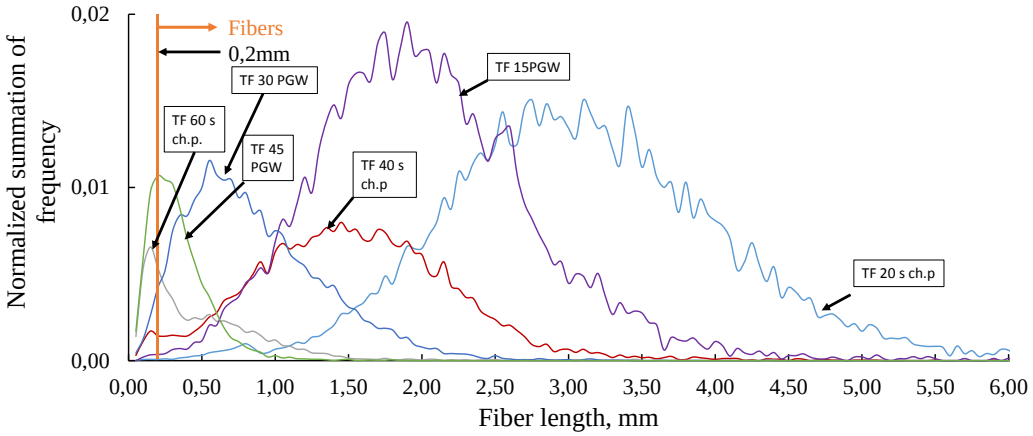


**Figure 5-2** *The comparison between a PGW pulp fraction and a kraft pulp fraction established with the tube flow fractionator.*

## 5.2 Repeatability

To investigate the repeatability of the whole fractionation procedure using the implemented tube flow device four runs based on a PGW sample were conducted. Sampling, dilution to the necessary consistency, injection of the sample, sampling with defined time intervals at the exit of the tube and analysis of the fractions using the Kajaani FS 200 was done four times independently. Possible variations my arise during sampling, the fractionation process in the tube flow and in the Kajaani measurements.

| | TF after 0 sec | TF after 30 sec | TF after 60 sec |
|---|---|---|---|
| arithmetic mean run 1 | 1,081 | 0,288 | 0,112 |
| arithmetic mean run 2 | 1,135 | 0,296 | 0,116 |
| arithmetic mean run 3 | 1,128 | 0,317 | 0,118 |
| arithmetic mean run 4 | - | 0,284 | 0,112 |
| standard deviation | 0,024 | 0,012 | 0,002 |

**Table 5-1** *Arithmetic mean of fiber length and the standard deviation of 4 independent pulp samples.*

Table 4-2 shows the mean fiber length of the three fractions (corresponding to the time intervals 0-30, 30-60 and 60-90 seconds) for each of the four independent runs and the corresponding standard deviation between the mean values. As is evident in this table the standard deviation is smaller for the finer fractions which can be attributed to the length distribution of the PGW sample. The number of

objects in the finer fractions is much higher than in the longer fractions, therefore the variation of the mean length in these fractions is smaller for statistical reasons and the length distributions are smoother for the same reason. This is also evident in figure 5-3 and in particular in figure 5-4 which shows the 95% confidence intervals for each length class based on the four independent measurements. Based on these tests it can be concluded that the whole measurement procedure shows good reproducibility.

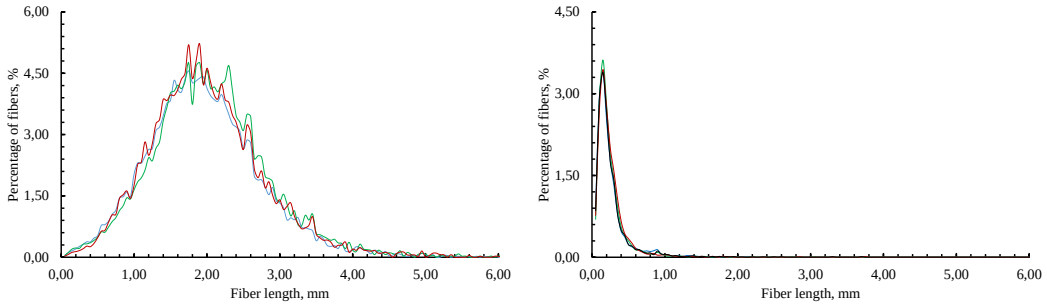


**Figure 5-3** *The comparison between the 0 seconds and the 60 seconds repeatability test length distribution.*

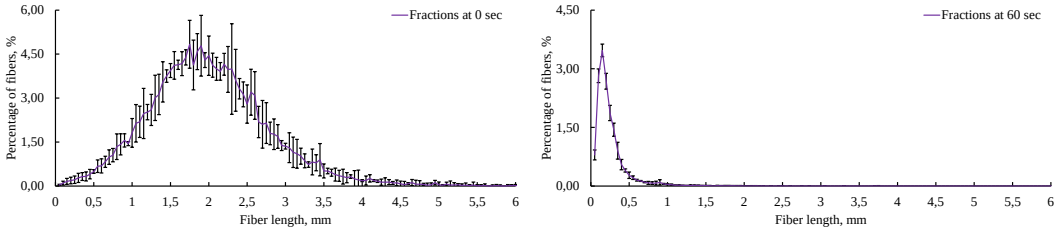


**Figure 5-4** *The comparison between the 0 seconds and the 60 seconds repeatability test mean length distribution including 95% intervals.*

## 5.3 Comparison of fractions using the Bauer McNett and tube flow fractionator

The Bauer McNett fractionator is the standard device used for laboratory fractionation. This method uses different screens of different meshes (see also Chapter 2.7.1). A comparison between the Bauer McNett fractionator and the tube flow fractionator was carried out. The Bauer McNett fractionator was chosen because of its recognition as the standard laboratory fractionation device for pulp samples. For that reason, the idea was to develop similar tube flow fractions which can then be compared with fractions of the Bauer McNett classifier. After the first pretests with

a PGW pulp it showed up that tube flow fractions within a time range of 15 seconds offers fiber length distributions similar to those of the Bauer McNett fractionator. It now becomes particulary clear that both length distributions show a similar degree of separation across all fractionation samples.

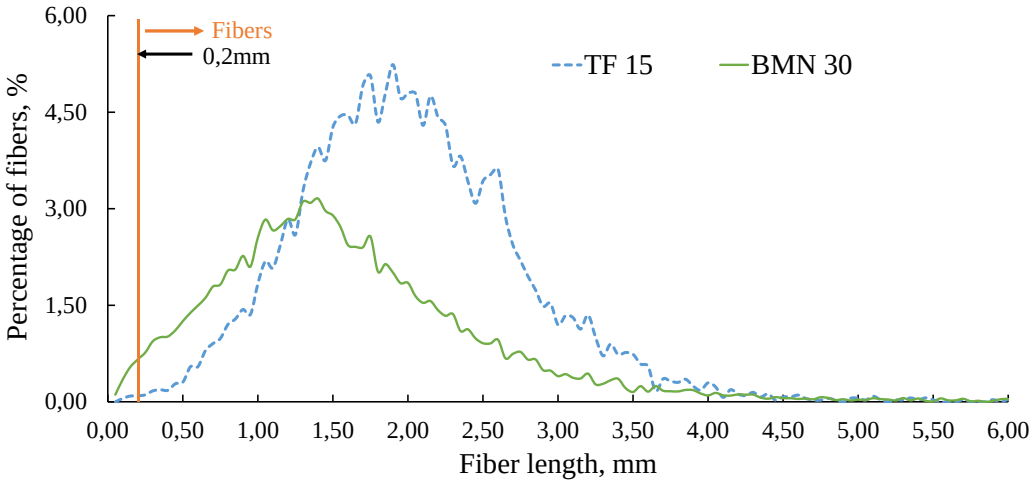


**Figure 5-5** *Length distribution of the TF 15 fraction and the BMN 30 fraction.*

Figure 5-5 shows a comparison between a 15 seconds tube flow fraction and a Bauer McNett 30 fraction for a PGW pulp. Taking a closer look at figure 5-6, it should be noted that fractions containing more short fibers and fines show a very good agreement of the fiber length distribution. Similar results were also achieved for the comparison of the tube flow fraction after 75 sec and the Bauer McNett fines fraction which can be seen in figure 5-7.
It can be concluded that in consideration of the right choice of time intervals a comparison of tube flow fractions and fractions from the BMN device can be done. It has been found out that in those circumstances, both length distributions offer a similar degree of selectivity.

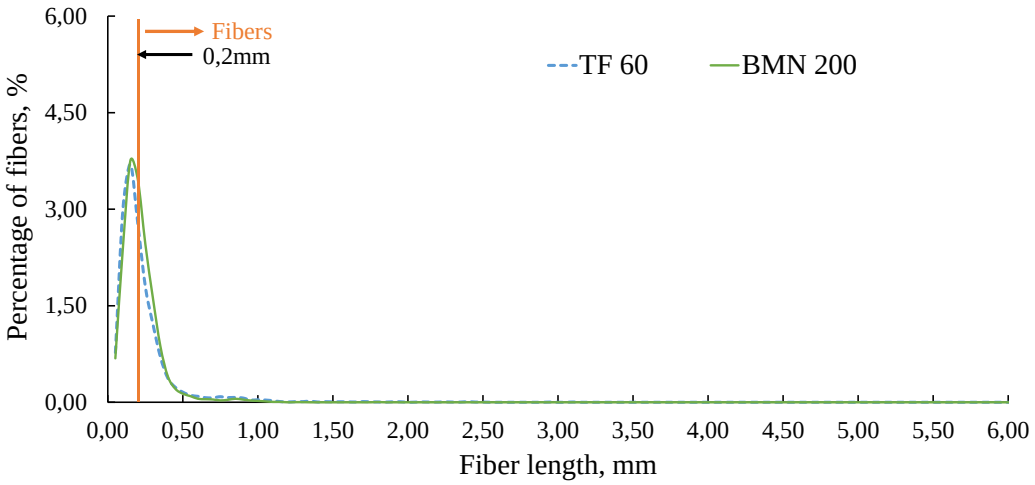


**Figure 5-6** *Length distributions of TF 60 fraction and the BMN 200 fraction.*

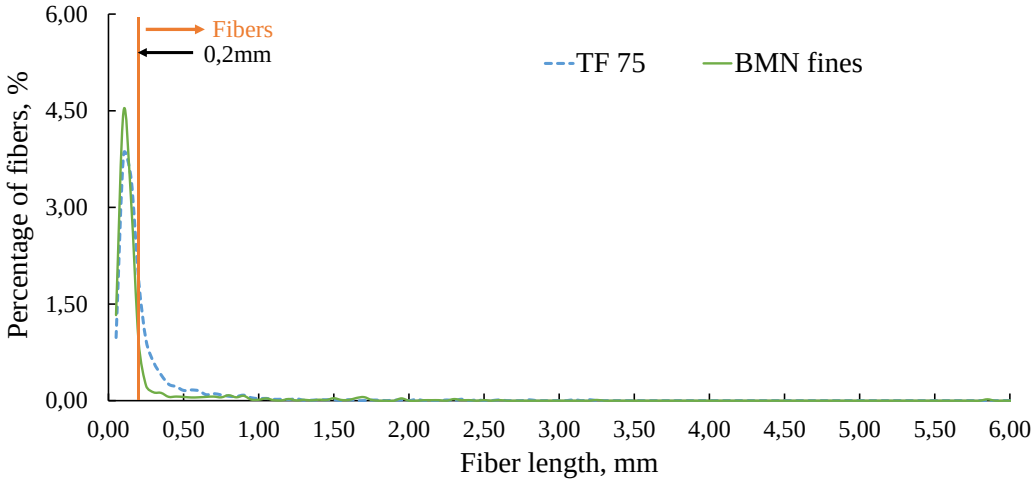


**Figure 5-7** *Length distributions of TF 75 fraction and the BMN fines fraction.*

## 5.4 Possible applications for tube flow fractionation

Due to the fact that the tube flow fractionator offers no limitations regarding the size of fraction length distributions, there will be some new possibilities of investigating pulp fractions. This insight inspires the possibility to the analytical fractionation of different pulp fractions. In the knowledge that the tube flow fractionator produces fractions with independent time intervals, many new opportunities are given in the laboratory fractionation method. Possible future applications of the analytical fractionation with the tube flow device can probably the fractionation of previously produced samples using other devices. This makes it possible to subdivide the previously produced fractions in more finer fractions for analytical purposes.

# 6 Conclusions

The aim of this work was to implement a tube flow fractionator for pulp fractionation based on similar devices described in the literature. Experiments with different pulp types and a comparison with the Bauer McNett fractionator were carried out for validation of the method based on Kajaani FS-200 length distribution. Testing on different pulp types shows that the tube flow system fractionates pulp samples according to their length distribution. Fractions of longer and shorter fibers as well as fractions of fines can be produced. Due to the fact that long fibers exit the tube first and fines in the end, samples with different time intervals allows different fractions with different length distributions. Repeatability tests were carried out based on 4 independent measurements of a PGW sample showing a high repeatability of the method. When the tube flow fractionator is compared to the BMN fractionator, it is shown that based on certain time intervals of the tube flow fractionator both devices offer samples with length distributions of almost identical shape.

Further investigations should focus on experiments with different time ranges and different types of pulps. Another interesting field of application will be the fractionation of pulp fractions-obtained with other devices. Thereby a separation of for example Mehlstoff and Schleimstoff for analytical purposes might be possible.

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