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Investigation of pulp dewatering – relating freeness to filtration properties

**Master of Science Thesis in the Master Degree Program, Innovative
and Sustainable Chemical Engineering**

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Abstract

For Sweden the forest sector is of great importance and contributes significantly to the Swedish export. One of the most important products is pulp, which can be used for production of packing material, printing paper and tissue paper. Pulp is also produced in several other countries (e.g. Finland, Canada and Brazil) and the world production is around 180 million tons during 2010. There are several different methods used for pulp production, both mechanical and chemical methods can be used. Among the chemical methods the kraft process is the most common.

One important product based on pulp is paper. Paper is formed by dewatering a mix of pulp and additives, to achieve a proper formation the starting pulp needs to be severely diluted, consistency as low as 0.1 % is required to form a paper of a good and even quality. The formation of the paper takes place on a wire section, where large amounts of water are removed by filtration. Further dewatering is then performed in the pressing section and finally the paper is dried. In the industry the dewatering properties of the pulp are measured using simple equipment measuring the freeness of the pulp, normally no filtration properties are measured.

The goal for this study has been to investigate pulp dewatering behaviour and relate freeness measurements to measured filtration properties. The fibres used in this study are produced in a kraft process based on softwood material

To investigate how the filtration properties change with the dewatering rate, the pulp was treated in a PFI-mill to change the structure on the pulp and thus the dewatering rate. The dewaterability was measured using two industrial methods Canadian standard freeness (CSF) and Schopper-Riegler (SR). The filtration resistance of the pulp was also measured at 1 bar filtration pressure. A model for CSF from the literature was used to compare a theoretical relation between freeness values and specific filtration resistance and the relation found from the filtration experiments in this study.

The range of the freeness value of the processed pulp was from 695 ml to 125 ml for CSF, which corresponds to a specific filtration resistance of 9×10^9 m/kg to 7.7×10^{12} m/kg at 1 bar filtration pressure. The model for CSF indicated lower filtration resistances than the ones measured in the experiments. An important explanation for this is the difference in filtration pressure and with the following difference in compression of the filter cake.

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1 Introduction

The world's current pulp production is about 186 million tonnes of pulp 2010 (Skogsindustrier, 2012). The pulp is used as starting material in production of packing material, printing paper or tissue paper. For pulp production two different approaches can be taken either mechanical or chemical. There are also processes which uses both approaches.

The mechanical process involves fibre separation using applied shear forces on to the wood material. A drawback of this method is a shortening of the fibre length. Mechanical pulping method has a high yield but ageing properties are an issue. Mechanical pulp is often used for pulp in production of newspaper, where for example ageing is not that crucial. Also thanks to the low production cost of pulp via mechanical treatment compared to chemical based provide further advantage in for example newspaper production. The high lignin content will not affect the product that much because the lifetime of the product is not that long.

For the chemical process a mixture of chemicals (sodium hydroxide and sodium sulphide) is added and temperature is elevated in a digester (Brännvall, 2009). During these conditions the lignin is broken down into smaller fragments. This type of treatment allows a higher degree of separation of lignin from the wood fibres and liberates the fibres without too high decomposition of the cellulose fibres and is suitable for products with high strength and chemical stability.

The pulp and paper industry handle large heterogeneous flows with desired materials that require separation. To separate the liquid phase from the solid material different techniques are available; e.g. evaporation of liquid and filtration. The evaporation manages to large extent remove the liquid contra filtration which will perform the separation with very little energy input but cannot remove the same amount of liquid from the pulp. During production of paper the paper must be formed and this is only possible through filtration which is why this is method is used in paper mills at the beginning of the paper machine. The filtration section is followed by a number of rolls and presses to increase the dry content enough for an introduction of the drying section which is the last part in the paper production process (Norman, 2000).

Depending on the interaction between the solid materials in the filter cake, the flow through the cake will vary greatly. The water flow can be increased by increasing the filtration area or decreasing the filtration resistance of the pulp. One important factor is to control the charge of the system. Today the dewaterability is investigated with Schopper-Riegler and Canadian standard freeness equipments, while measurements of the filtration resistance are uncommon.

A constructed paper machine can be improved via an increase in dewatering rate of the formed paper matrix on the wire screen. This will allow a higher production as the residence time on the wire screen will be lower to reach a specific dry content. The same understanding can be used during the design of a new paper machine to form a machine that will perform the same work as the already constructed one but using a smaller filtration area.

This study aims to investigate the correlation between freeness values and specific filtration resistance.

1.1 Overview of the thesis

This thesis deals with filtration resistance during constant pressure with a selected range in refining of the pulp. The pulp used in the thesis is Black R from Södra's mill in Värö. In chapter 2 introductions to flow through porous beds and filtration theory is presented, followed by an overview of the pulping process and corresponding paper production. The last is a review of the Schopper-Riegler and Canadian standard freeness apparatuses and for each one of them a derivation of an equation that explains how the liquid level changes with time. In chapter 3 a presentation of materials used in filtration experiments with equipment set-up and analysing methods used during the thesis.

2 Theory

This section will introduce a simple model for flow through porous beds and continue with filtration in general with a presentation of models with their limitations. Further in this section, short concepts around pulping process (types of processes and theory around paper formation) will be presented. The last part will deal with empirical measurements of freeness values and present a derived model for each freeness equipment.

2.1 Flow through porous beds

In 1865 Darcy proposes a correlation for flow through porous bed where flow rate are related to the pressure drop over the bed Eq. 1:

$$u = \frac{1}{A} \frac{dV}{dt} = \frac{K\Delta P}{\mu\Delta z} \quad [\text{Eq. 1}]$$

where u is the superficial flow velocity, ΔP is the total pressure drop over the bed, μ the viscosity of the fluid, Δz the height of the bed, K the permeability of the bed, A the area of the bed, V the filtrate volume and t the time. Permeability is a property of the bed and therefore can be used to formulate an expression for the specific filtration resistance for the bed:

$$\alpha \equiv \frac{1}{K\rho_s\phi} \quad [\text{Eq. 2}]$$

where α is the specific filtration resistance, ρ_s the density of the solid material and ϕ the solidosity.

The solidosity is then a description of the amount of the mass in filter cake.

$$\phi = \frac{V_{solid}}{V_{total}} \quad [\text{Eq. 3}]$$

where V_{solid} is the volume of the solid material in the porous bed and V_{total} the total volume of the bed.

2.2 Filtration

During filtration a build-up of filter cake will occur and this makes the direct use of Darcy's equation not viable. Therefore Ruth (Ruth, 1935) formulated a basic filtration equation (Eq. 4) based on Darcy's law, which describes the flow through the formed filter cake.

$$\frac{dt}{dV} = \frac{\mu(\alpha_{av}cV + R_m A)}{A^2\Delta P} \quad [\text{Eq. 4}]$$

where α_{av} is the average specific filtration resistance, c the mass of solid per unit filtrate volume and R_m resistance of the filter. In this equation ΔP represent the sum of pressure drop over the filter cake and filter medium. The specific filtration resistance in Eq. 4 present by Ruth is a mean value of the local specific filtration resistance and are formulated through integration of the pressure drop over the filter cake.

$$\frac{1}{\alpha_{av}} \equiv \frac{1}{P_c} \int_0^{P_c} \frac{1}{\alpha} dP_s \quad [\text{Eq. 5}]$$

where P_c is pressure drop over the filter cake and P_s is the solid compressive pressure. It is problematic to isolate and measure the pressure drop over the filter cake, and therefore it is more convenient to use the total pressure drop over both the cake and the medium as Ruth does in equation Eq. 4.

One limitation of the basic filtration equation proposed by Ruth is the assumption of an incompressible filter cake and therefore is only suitable for materials that form

incompressible filter cakes. Tiller and Shirato (Tiller & Shirato, 1964) presented a modification of classical filtration equation that compensate for the compressibility of the filter cake. The equation introduced a factor that varies with concentration and approaches one at low slurry concentration.

$$\frac{dt}{dV} = \frac{\mu(\alpha_{av}J_R cV + R_m A)}{A^2 \Delta P} \quad [\text{Eq. 6}]$$

where

$$J_R = \frac{A dt}{dV} \int_0^1 \left(v - \frac{1-\phi}{\phi} v_s \right) d \left(\frac{w}{w_c} \right)$$

v is the superficial velocity along the drainage chamber, v_s is the superficial velocity of the solid, w is the surface weight and w_c the surface weight of the filter cake.

2.3 Pulping processes

The pulping can be divided into two main groups for treatment of wood either mechanical based or chemical based processes. Where mechanical process use applied mechanical force to separate the wood fibres and chemicals processes use as the name indicate chemicals to separate the wood fibres. These two approaches can be further combined in different ways to utilize the advantages associated with each type when certain products are demanded.

2.3.1 The kraft process

The kraft process is the most common chemical pulping process. An overview of the kraft process can be found in several sources in the literature (e.g. Brännvall, 2009) which involve the introduction of the wood chips to the suspending of the bleaching stage. As chipped wood material enters the part of the chemical process that represent the cook, the chips are mixed with cooking chemicals, which are sodium hydroxide and sodium sulphide. The active components of the chemicals are hydroxide (OH^-) and hydrogen sulphide (HS^-) ions. To perform an efficient cook it is important that the chemicals are evenly distributed over the entire chip. A good impregnation of cooking chemicals into the chips will lead to a more even delignification of the chips. The delignification can be divided into three steps where the first step is a fast and take places during the heating of the chips. The second part is the bulk delignification where the major part of the lignin is removed and is also the most selective phase. As the lignin content decrease the selectivity of cooking chemicals decrease and with an increase of degraded cellulose. The cook is then preferable ended when the lignin content reach the critical concentration level where the selectivity reach the acceptable limit. The remaining lignin is called the residual lignin and is treated in the upcoming bleach steps (Germgård, 2009). Before the treatment in the bleaching process the pulp must be separated from the cooking chemicals or the present chemicals in the pulp will consume the bleaching chemicals without any delignification performed. The separation is performed through wash sequences. With the appropriate conditions of the pulp, the bleaching sequence can be designed in various ways to reach the desired end result. Both in how each bleaching steps is designed (the flow direction of the pulp through the bleaching stage, the consistency of the pulp and the reaction time for the pulp) and the order of how the bleaching chemicals is introduced will vary from process to process. As the desired brightness is reached the bleaching process is terminated and pulp is further transported to the paper machine.

2.3.2 The mechanical process

An overview of the mechanical pulp process can be found in several sources of the literature e.g. Sundholm (Sundholm, 1999), where he provides the concept around

mechanical pulping. The first version of the process used grindstones to liberate the wood fibres and create stone ground wood. The idea is by applying shear forces on to the wood pieces the fibres are liberated to desired extent without dissolving the lignin. Due to the high presents of lignin, the paper made from mechanical pulp will be sensitive to exposure of sun light and might change colour after exposure. The advantages with mechanical pulp include the fairly easy process with a cheap production procedure and a high yield.

2.4 Paper production

Norman present the paper production steps in the chapter Web forming (Norman, 2000). After the processing of the wood material either through chemical or mechanical treatment where the fibres have been liberated in different extent, the beginning of paper formation starts with dilution of the pulp to around 0.1 %. The dilution is implemented so possible flocculation breaks up by the applied shear forces from the flow but during the dilution great attention towards the mixing is needed to avoid concentration variation further downstream. The diluted mixture is then delivered to the headbox whose main purpose is to evenly distribute the mixture over the wire section via nozzles. As the mixture is applied on to the wire the separation of the water starts with filtration where the criteria for the wire section is a dry content on the formed paper web around 20 %.

Filtration resistance is the major factor of influence on the design of the wire section. A good dewaterability (low filtration resistance) allow a more compact design of the wire section of the paper machine or a higher production rate.

The wire section is then followed by a combination of rolls and presses that will increase the dry content from 20 % to around 50 %. The rolls use much less energy per unit water removed compared to the energy requirement of drying to perform the same work. To reach the desired dry content the design of the press section will depend on the limitation formed after properties on the paper matrix. If the press section would be compression limited the dry content will only depend on the applied surface pressure. Compare this to a flow limited process where the dry content is not limited by applied pressure but by residence time due to the flow resistance. With a dry content of 50 % the paper web enters the drying section where the last removal of water to reach the specification of the product.

2.5 Impact of fibre properties on to the filtration behaviour

With the mechanical process a high yield is reached at the expense on the fibre length, shorter and stiffer fibres are produced compared with the chemical process. In the chemical process the yield will be lower but instead the fibre length is kept fairly close to the origin and is more flexible. The difference in physical properties will have major impact on the filtration process and the ending result on produced paper.

In the mechanical treatment a lot of fines are formed during the beating and to bind these fines to the rest of the fibres at wire section during the paper formation, addition of retention aids is required (Kilpeläinen et al., 2000). These polymeric additives will have a charged surface which is preferable opposite to the pulp stock components. The additives will neutralize the surface of the fibres with the possibility to form flocculation with result of an enhanced retention. Due to the large amount of fines formed during the mechanical treatment the produced fibre web will have a compact matrix with low dewater ability which leads to a lower filtrate rate which further will

give a low freeness value (Sundholm, 1999). The final paper product based on mechanical pulp is a paper with a smooth paper surface due to the presents of fines but the tear strength is not as high as for chemical treated fibres. With the same analyse applied on the chemical process, the result show that the fibres is not exposed to same amount of mechanical treatment and therefore the fine content is lower, instead there is a greater loss of carbohydrate during the cooking section. The advantage is the flexibility which will give good tear strength for the paper and with the low content of fines, a high freeness values is reached which indicate on a good dewaterability, leading to a faster filtration process.

The brightness of the pulp will vary between the two methods where the kraft pulp will have bright appearance compared to mechanical based. This is more suitable for products such as books where a high brightness simplifies the reading experience and low ageing effects is desired.

2.6 Paper formation

As mentioned before the paper formation process starts with a dilution of the pulp before the fibres are delivered to the headbox. After the delivery of suspension to the headbox the following step is taken to create an even paper web. The suspension is supplied over the wire via nozzles where each nozzle must have the same conditions (fluid velocity, concentration and volumetric flow rate) to enable creation of a uniform paper web. To achieve this Norman (Norman, 2010) suggests that the jet out from each nozzles shall be flat and have laminar flow conditions when it leave the nozzle openings. Using nozzles that creates flat jets will allow a perpendicular contact to the machine direction but this phenomenon rarely happened and is more a goal then a criteria. With a contraction over the nozzles an acceleration on to the fluid is applied that will generate a pressure drop with consequence of a reduction in relative turbulence. With the same conditions in all the nozzles the orientation of the fibres can be set by changing the relative velocity between the wire and the jets, with a higher wire velocity the fibres will orientate along the wire direction because the fibres will be dragged along the wire during the impact.

With the mentioned theory around the headbox and corresponding nozzles it is clear that the properties of the resulting paper can vary greatly. With a similar wire versus jet velocity the fibre orientation will be more random and the paper will have good strength properties in all directions. As the wire velocity increase the fibres will change their orientation more along the machine direction which will lead to higher strength properties in the machine direction but perpendicular the strength will be poor. Further the surface of the paper might sometimes appear to be like it should be but the properties do not correspond to the appearance, then a self-healing might have occurred (Norman, 2000). This comes from present flocculation in the suspension which creates a variance in distributed fibres over the wire. As the dewatering process proceeds areas with less disposed fibres will appear in the web. Because the filtration resistance in this region will be lower a higher flow rate of liquid will be the result compared to the regions with greater amounts of disposed fibre material. This phenomenon smooths out possible variation in thickness but the creation of interaction between the fibres will take place in two steps. This leads to lower number of interaction compared to if the fibres would be even spread from the beginning.

2.7 Filtration of pulp fibres

A several of articles have been written regarding filtration of pulp fibres, e.g. Roux (Roux, 2011; Chellappah et al., 2009). Roux comes to conclusion that the filtration

mechanism is the dominating mechanism and that homogeneous filter cake is formed. Roux base the conclusion on absences of difference between formed cake from early disposed fibre and later drained fibres along the wire section. Due to the difficulty with preforming measurements directly on the paper machine, simplifications are needed to collect representative data. To represent the wire section of the paper machine in lab scale, the formation process of the fibre matrix is considered as a dead-end filtration process. This can be assumed to be valid since a cake is being built up over the wire and not removed until the end of the wire. With this simplification dead-end filtration equipment could be used for simulation of the wire section.

2.8 Measurement of Canadian standard freeness and Schopper-Riegler

Measuring freeness is a method commonly used in the industry for characterisation of the dewatering ability of the pulp. In these empirical methods either dewaterability or dewater resistance of a pulp is measured. In 1907 the first freeness tester were introduced by Klemm which consist of a graduated glass tube with a wire section at the bottom. With this Klemm could measure the deposition of fibre on the wire section and noticed that pulp with higher degree of milling created a thinner and more compact fibre matrix (Swodzibnski & Doshi, 1986). Numerous methods have been developed during the years but two have been widely accepted, Canadian Standard freeness (CSF) and Schopper-Riegler (SR). The big difference between CSF and SR is how the results are interpreted, both measures the volume in the overflow beaker denoted Q_2 in Figure 1 and Figure 2 but the scale on SR is inverted. This means that when the pulp is exposed to a longer treatment, more fines will be created and the SR-value increased with less liquid collected in the overflow beaker.

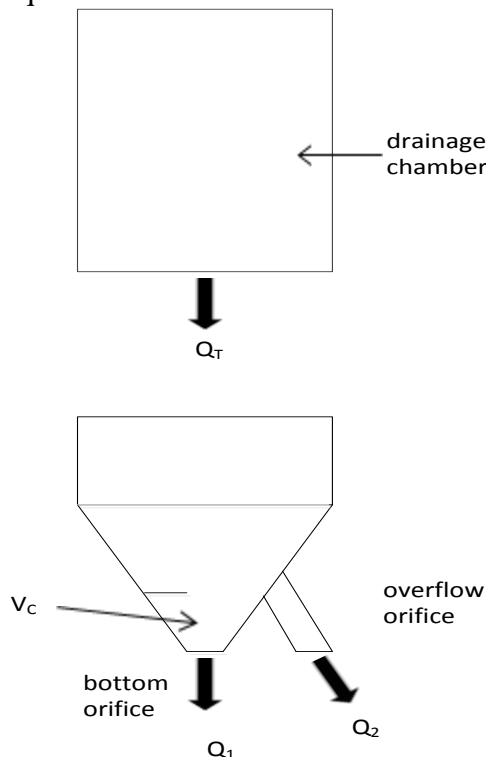


Figure 1: Schematic representation of a Canadian standard freeness equipment.

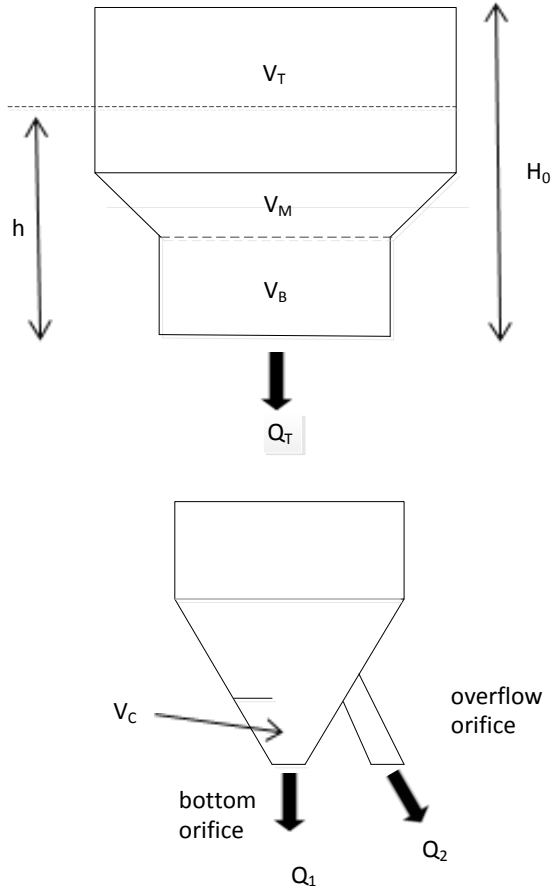


Figure 2: Schematic representation of a Schopper-Riegler equipment.

2.8.1 Structure difference between CSF and SR

When comparing structure between the two methods some significant difference can be observed. First the design of the drainage chamber; where CSF has a cylindrical shape of the drainage chamber with straight walls (Figure 1), the SR drainage chamber is a combination of a cone and a cylinder (Figure 2). The last significant difference is the sealing mechanism of the drainage chamber where CSF seals the screen from below so the pulp will have contact with the wire section. This can be compared to SR where a cone is lowered so the water is hinder from entering the drainage from above. Despite the differences a mathematical model for the freeness value can be derived in the same procedure with the different structural designs in mind.

2.9 Derivation of a model for CSF and SR

The drainage rate is assumed to be so high that the liquid level in the cone rise above the overflow instantly so liquid is collected from both orifices. This is a shared assumption for the derivation of the models. The structural difference can simply be treated during the derivation by divide the SR equipment in three different time intervals as cross sectional areas changes in the drainage chamber.

2.9.1 Canadian standard freeness

The model for Canadian standard freeness developed by El-Hosseiny and Yan is the one introduced in this part that, the complete derivation can be found in Swodzinski and Doshi (Swodzinski & Doshi, 1986). A schematic representation of a Canadian standard freeness equipment can be found in Figure 1.

First a total balance is performed over the cone after opening of the hatch. At this moment the cone are filled up above the overflow sprout.

$$\int_0^t Q_T dt = \int_0^t (Q_1 + Q_2) dt \quad [\text{Eq. 7}]$$

Q_T is the volumetric flow out of the drainage chamber, Q_1 the flow through the bottom orifice and Q_2 the flow through the overflow orifice. CSF is then defined as the collected volume from the overflow orifice (Q_2):

$$CSF = \int_0^{t_1} Q_2 dt = \int_0^{t_1} (Q_T - Q_1) dt - V_c \quad [\text{Eq. 8}]$$

where V_c is the volume of the cup (the volume between the bottom orifice and the overflow orifice) and t_1 is the time for the pulp to pass through the wire screen. Let the collected filtrate during the time span be signed as V_1 and in Eq. 8 this corresponds to the total filtrate from the drainage chamber.

$$V_1 = \int_0^{t_1} Q_T dt \quad [\text{Eq. 9}]$$

Further assume constant Q_1 during the time span t_1 along with combine Eq. 8 and Eq. 9 for formulating following expression:

$$CSF = V_1 - Q_1 t_1 - V_c \quad [\text{Eq. 10}]$$

The classic filtration equation (Eq. 4) can be used to express V_1 and t_1 in Eq. 10 which will give the generalized form developed by El-Hosseiny and Yan.

In Eq. 4 the pressure difference (ΔP) can be expressed as follow

$$\Delta P = \rho g h \quad [\text{Eq. 11}]$$

where

$$h = \frac{V_0 - V}{A}$$

h is the liquid height in the chamber, g the acceleration of gravity, ρ are the filtrate density, V_0 the initial slurry volume and V is the collected filtrate volume at time t . By combining Eq. 4, Eq. 11 followed with integration an expression for t_1 can be formed. The boundaries for the integration are from $V = 0$ to V_1 and $t = 0$ to t_1 with the result of an expression for t_1 .

$$t_1 = \frac{\mu}{\rho g} \left[\left(R_m + \frac{\alpha_{av} c V_0}{A} \right) * \ln \left(\frac{V_0}{V_0 - V_1} \right) - \frac{\alpha_{av} c V_1}{A} \right] \quad [\text{Eq. 12}]$$

By assuming constant Q_1 during the time span makes it possible to use Eq. 4 to formulate an expression for V_1 by substituting V with V_1 and solve for V_1 .

$$V_1 = \frac{A V_0 \rho g - Q_1 R_m \mu}{Q_1 \alpha_{av} c_0 \mu + A \rho g} \quad [\text{Eq. 13}]$$

Combining Eq. 10, 12 and 13 to form Eq. 14 completes El-Hosseiny and Yan's equation for Canadian standard freeness with a common simplification of assuming negligible screen resistance compared to filtration resistance.

$$CSF = (V_0 - V_c) - Y \left[\ln \left(\frac{V_0}{Y} + 1 \right) \right] \quad [\text{Eq. 14}]$$

where

$$Y = \frac{Q_1 V_0 c_0 \alpha_{av} \mu}{\rho g A}$$

2.9.2 Schopper-Riegler

The drainage chamber of the Schopper-Riegler can be divided into three regions. The top region correspond to the height for the starting level of the slurry down to where the cross sectional area starts to decrease. The region where the cross sectional area decrease is the middle region and the lowest one is the bottom region. A schematic representation of the equipment can found in Figure 2.

The same balance over the cone can be performed for SR as for CSF and will result in Eq. 15. SR is defined as follow

$$SR = V_1 - Q_1 t_{tot} - V_c \quad [\text{Eq. 15}]$$

where

$$t_{tot} = t_U + t_M + t_L$$

t_U is the corresponding time for liquid level decrease through the top region, t_M the time for the middle region and t_L for the lower region. The time variable for each region is derived separately with the top of each region acting as the start of the boundary during the derivation.

Upper region

The liquid level in the upper region can be expressed with the origin and the decrease in terms of collected amount of liquid in the beakers.

$$h = H_0 - V/A_1 \quad [\text{Eq. 16}]$$

H_0 is the initial height of the liquid level and A_1 the cross sectional area of the upper region.

Insert Eq. 16 into Eq. 4 give following general filtration equation for the upper region

$$Q = \frac{dV}{dt} = \frac{A_2(H_0 - V/A_1)\rho g}{\mu(R_m + \alpha_{av}C_0 V/A_2)} \quad [\text{Eq. 17}]$$

A_2 is the cross sectional area of the lower region which correspond to the filtration area. From Eq. 17 an expression for t_U is formulated by integrating from $t = 0$ to $t = t_{tot}$, $V = 0$ to $V = V_1$ and solving for t_{tot} , where t_{tot} is the total time expired so far. The system is assumed to start at top just before the liquid level decrease in the drainage chamber. The same assumption is made as before where the screen resistance is neglected.

$$t_{tot} = \frac{\mu}{\rho g A_2} \left[\frac{(A_1^2 \alpha_{av} C_0 H_0)}{A_2} \ln \left(\frac{A_1 H_0}{A_1 H_0 - V_1} \right) - \frac{A_1 \alpha_{av} C_0 V_U}{A_2} \right] \quad [\text{Eq. 18}]$$

To formulate an expression for V_1 , Q is assigned with Q_1 which is assumed constant during the whole time span t_{tot} , change V to V_1 in Eq. 4 along with solving for V_1 .

$$V_1 = \frac{A_1 A_2^2 H_0 \rho g}{Q_1 C_0 \mu \alpha_{av} + A_2^2 \rho g} \quad [\text{Eq. 19}]$$

Eq. 19 applies only as long as the liquid level is located in the upper region. By inserting Eq. 19 into Eq. 18 the expression for t_{tot} is complete.

Middle region

The cross sectional area in the middle region decrease with height and the therefore h need a expression to formulate the change in height.

$$h = H_B + r - r_2 \quad [\text{Eq. 20}]$$

H_B is the height of the bottom section, r the radius of the fluid surface at any height in the middle region and r_2 is radius of the lower section of the drainage chamber. Next step in derivation of model is to express volume of the middle as a truncated cone.

$$V_0 - V - V_B = \frac{\pi(r^3 - r_2^3)}{3} \quad [\text{Eq. 21}]$$

V_B is the volume of the bottom section. Next step is to formulate the expression for volumetric flow rate through the truncated cone.

$$Q = \frac{dV}{dt} = \frac{dV}{dr} \frac{dr}{dt} \quad [\text{Eq. 22}]$$

Combine Eq. 20, Eq. 21 and Eq. 22 give following expression.

$$\frac{A_2 \rho g}{\mu} \int_{t_U}^{t_{tot}} dt = \int_{r_1}^{r_2} \left(\frac{K_1 r^5 - K_2 r^2}{K_3 + r} \right) dr \quad [\text{Eq. 23}]$$

where

$$K_1 = \frac{\alpha_{av} C_0 \pi^2}{A_2 3}$$

$$K_2 = \frac{\pi \alpha_{av} C_0}{A_2} \left(V_0 - V_B + \frac{\pi}{3} r_2^3 \right)$$

$$K_3 = H_B - r_2$$

Integrating both sides gives following expression for t_{tot} , the time for the liquid level to descend through top and middle region.

$$t_{tot} = t_U + \frac{\mu}{A_2 \rho g} \left\{ K_1 \left[\frac{(K_3 + r)^5}{5} - 5K_3 \frac{(K_3 + r)^4}{4} + 10K_3^2 \frac{(K_3 + r)^3}{3} - 5K_3^3 (K_3 + r)^2 + 5K_3^4 (K_3 + r) - K_3^5 \ln(K_3 + r) \right] - K_2 \left[\frac{(K_3 + r)^2}{2} - 2K_3 (K_3 + r) + K_3^2 \ln(K_3 + r) \right] \right\} \Bigg|_{r_1}^r \quad [\text{Eq. 24}]$$

As in the earlier part of the derivation an expression for V is needed and as previous Q is assumed constant and assigned Q_1 along with assigning V with V_1 in Eq. 4. Through solving for V_1 following expression is reach for V_1 which is only viable for the term in Eq. 24 that represent the middle region.

$$V_1 = \frac{A_2^2 \rho g (H_B + r - r_2)}{Q_1 C_0 \mu \alpha_{av}} \quad [\text{Eq. 25}]$$

Lower region

As the shape of the filtration chamber change back to a cylinder the same procedure can be used as for the upper region. Start with express a change in height according to the volume.

$$h = \frac{V_0 - V}{A_2} \quad [\text{Eq. 26}]$$

Followed by forming the complete time expression for time expired by integrate the Eq. 4 with following boundaries, V_{U+M} to V_1 and t_{U+M} to t_{tot} , where (U+M) is the boundary between middle and bottom region.

$$t_{tot} = t_U + t_M + \frac{\mu \alpha_{av} C_0}{\rho g A_2} \left[\left(V_0 \ln \left(\frac{V_0 - V_{M+T}}{V_0 - V_1} \right) + (V_{M+T} - V_{tot}) \right) \right] \quad [\text{Eq. 27}]$$

In the lower region V_1 can be expressed as follow

$$V_1 = \frac{V_0 A_2 \rho g}{Q_1 \mu C_0 \alpha_{av} + \rho g A_2}$$

With an expression for each region for the Schopper-Riegler equipment complete the generalized model is completed where an expression for the t_{tot} in Eq. 16 have been formulated. As V_1 have been used as a variable in all three regions, each expression shall be used in each respectively place.

3 Experimental set-up

This chapter will cover the material and equipment and describe the methods used during the experiments.

3.1 Material

The material used in this study is a softwood pulp with a fibre length of 2.05-2.25 mm which is produced by Södra. The pulp is produced in a kraft process, i.e. a chemical pulping method at Södra's mill in Väröbacka and is denoted black R, which is the sales name. The sheets are delivered with a dry content of 93 %.

3.2 Equipment

3.2.1 Defibrator

In the sample preparation for the experiments, defibration plays an important role insuring constant conditions for each trial. Without defibration the sample extraction from each batch will contain a variation in concentration which will complicate the interpretation of the data. This can be solved with the use of a defibrator that will break up the entanglement through applied shear forces with a pitch blade impeller. To achieve total fibre separation, the sample is subjected to treatment in a defibrator according to SCAN-C 18:65. The sample is added to a beaker with deionized water to a total volume of 2 L. Depending on if the sample is milled or not the number of rotations differs where a untreated sample need 30 000 rotation according to SCAN-C 18:65 compared to already milled sample only need 10 000 rotations. The second reason for usage of a defibrator is to accomplish the same conditions as in paper machine, where the pulp is so diluted that no entanglements are present.

3.2.2 PFI-mill

To enable investigation of pulp with different dewaterability a PFI-mill is used. The PFI-miller consists of a bowl with an inner diameter of 250 mm and a roller with a corresponding diameter of 200 mm with 33 knives (50 mm high and 5 mm thick) evenly placed along the rollers. The rotation of each part is accomplished via two electrical engines, one on 0.5 hp for the bowl with a rotation velocity of 710 m/s and 1.5 hp for the roller with corresponding rotation velocity of 1460 m/s. With a leverage the roller is pressure the pulp against the bowl walls with a pressure of 3.4 kPa. All the design specifications on the PFI-miller are presented in SCAN-C 24:67 which also include the protocol on the preparation of samples.

3.2.3 Filtration equipment

When the pulp has been exposed to the second defibration the batch is ready for the filtration experiment where the sample added to the chamber. The chamber is well mixed since an electrical engine is attached with associated pitch blade agitator, the chamber is also baffled. To the stirred tank it is possible to attach equipment that will allow experiments to be performed at pressures up to 6 bars. As the experiment proceeds a filter cake built-up will occur in the chamber designed for dead-end filtration consisting of glass or plastic wall that allow visual observation during the experiment. The chamber has an inner diameter of 53 mm, a height of 50 mm and with an effective filtration area of $2.2 \times 10^{-3} \text{ m}^2$. The filter used in the investigation is built up by three layers, a support grid, a forming fabric and the separating media which is a Munktell analytical filter paper grade 5 with a diameter of 70 mm. The filter paper was used to avoid possible clogging of the wire screen. During the experiment the filtrate is

collected in a beaker that further stands on a scale which can sample the filtrate over time via a connection to a computer.

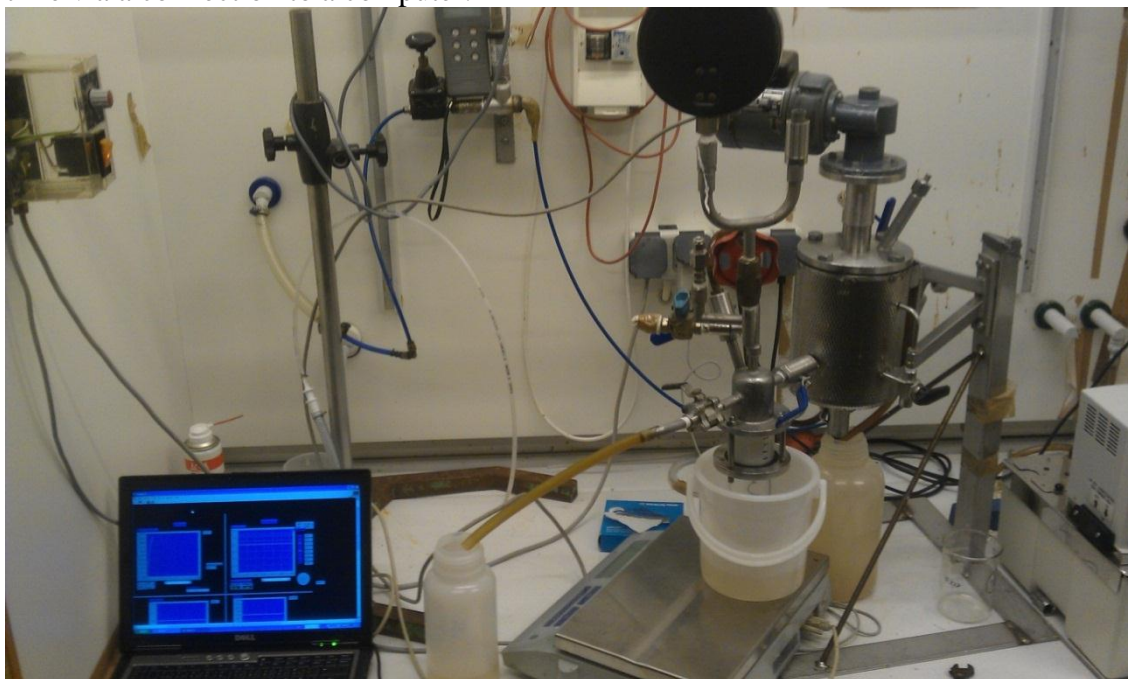


Figure 3: Experimental set-up of the filtration equipment.

On some filter cakes the wet weight was measured before drying of the cake to enable the evaluation of the filter cake porosity.

3.2.4 Freeness measurements

To measure freeness of the pulp at the different degree of milling two types of equipment was used; Schopper-Riegler and Canadian standard freeness. Both equipment use the same treatment procedure as mentioned in the early equipment sections and the freeness measurements are performed from the same batch as the filtration samples are collected from. From the design of the beaker where the flow through the overflow orifice is collected and the result explain the drainage resistance of the pulp and are conducted according to SCAN-C 19:65. Where Canadian standard freeness gives a value on the drainage rate and is conducted according to SCAN-C 21:65.

3.2.5 Kajaani FS 3000

To investigate the fibre length distribution in a sample of milled pulp, KajaaniFS3000 was used as analysing equipment. Kajaani FS 3000 use a fast matrix camera and an image analyser. The fibres in the sample are led through a glass capillary where they are measured one by one. The equipment use polarization to detect the fibres and measure their fibre length and works for all type of cellulose (kraft pulp, thermo mechanical pulp (TMP), cotton etc.).

3.3 Experimental conditions

All batches were prepared according to the standards presented by Scandinavian pulp paper and board testing committee for the milling step and as needed for the freeness measurements. Each batch contains 30 g of dry fibres which are defibriated under 30 000 revolutions before the milling. To achieve a representative sample for the filtration experiments and suitable for the freeness analyse the milling pulp is further processed with 10 000 revolution to separate present flocculation. Each filtration experiment was carried out under a constant overpressure at 0.1 MPa with the same

concentration of 3 g/L where the fibres are dispersed into deionized water. Higher concentration was tested but from 4.5 g/L and above the possibility of clogging in equipment resulted in the decision of using 3 g/L as concentration for the experiments. A filtration pressure of 1 bar was used to avoid clogging of the pipes and sedimentation in the cell. The filtration was halted slightly before the liquid surface reach the filter cake surface in order to avoid air blowing. After completed filtration experiment the cake was recovered for measurement of dry mass of the filter cake.

4 Result and discussion

In this section the data collected from the experiments which have been performed during the study are presented. This section contains several subsections that will deal with characterization of the fibres and evaluation of the experiments. The first part will involve characterization of the fibres followed with evaluation of the freeness values. The second part deals with the influence of milling on the freeness values. The third part concerns the evaluation of the filtration experiments. In the fourth part the milling dependency on the specific filtration resistance is presented and discussed. In the last and fifth part an evaluation of the presented model for the CSF equipment is evaluated.

4.1 Characteristic of pulp fibres

During milling the fibres are exposed to shear forces; to understand how the treatment affects the fibre length distribution a Kajaani FS 300 was used. The analysis was performed on four different points of milling to see the change in fibre length with an increase in beating, the results from the measurements are presented in Figure 4, 5, 6 and 7.

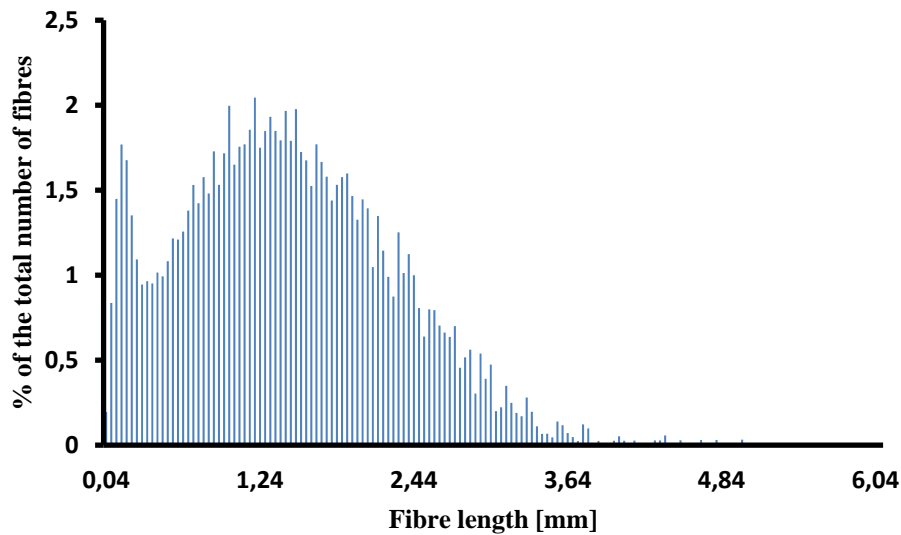


Figure 4: Fibre length versus percentage of total amount of fibres for an untreated sample.

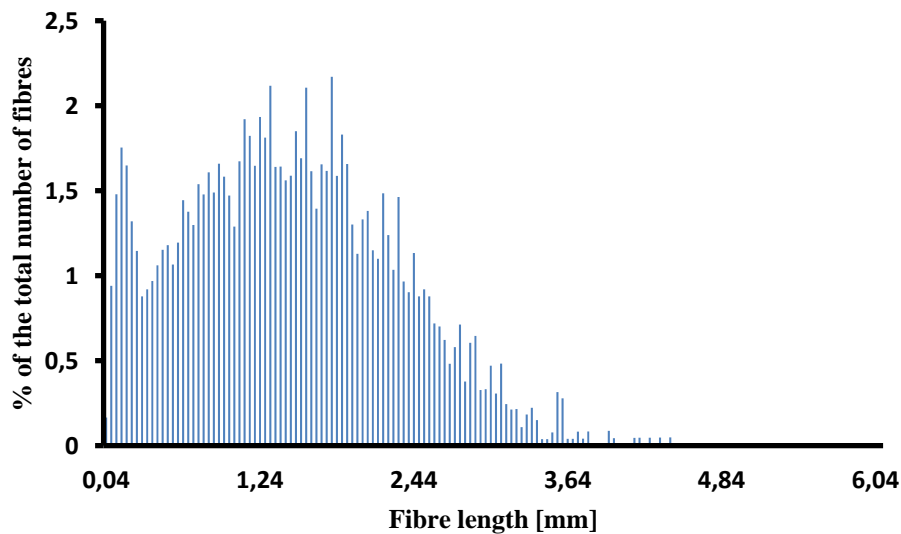


Figure 5: Fibre length versus percentage of total amount of fibres for a treated sample of 4000 revolutions.

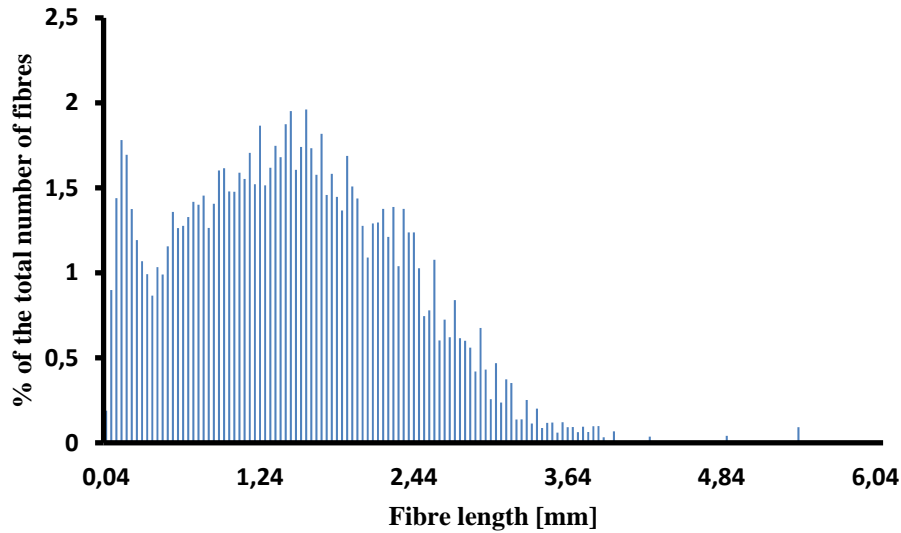


Figure 6: Fibre length versus percentage of total amount of fibres for a treated sample of 5000 revolutions.

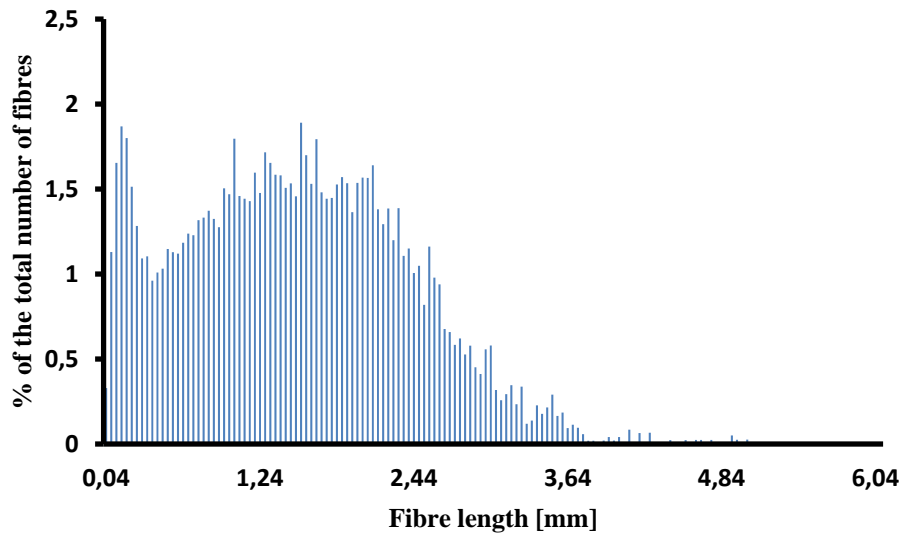


Figure 7: Fibre length versus percentage of total amount of fibres for a treated sample of 8000 revolutions

From the figures above (Figure 4 to 7) it becomes clear that the milling does not induce any fines. The lack of formed fines has an underlying reason in the design of the PFI-mill. The mill is specially designed to avoid formation of fines and focus on the internal defibrillation which results in keeping the original fibre length. This was confirmed by Somboon (Somboon, 2011) by comparing sample that have been expose to two different refiners, an industrial refiner (Voith refiner-LR40) and a PFI-miller. By analysing the fine Somboon found that the industrial refiner created more fines compared to the PFI-miller, similar to the observed behaviour of the PFI-miller used in the study.

4.2 The influence of milling on freeness values

A analyse on the relationship between milling and freeness values was performed and the result is presented in Figure 8 and 9. With freeness measurements gather over a wide

range of milling levels, indications of how milling affects the dewaterability and filtration resistance can be obtained.

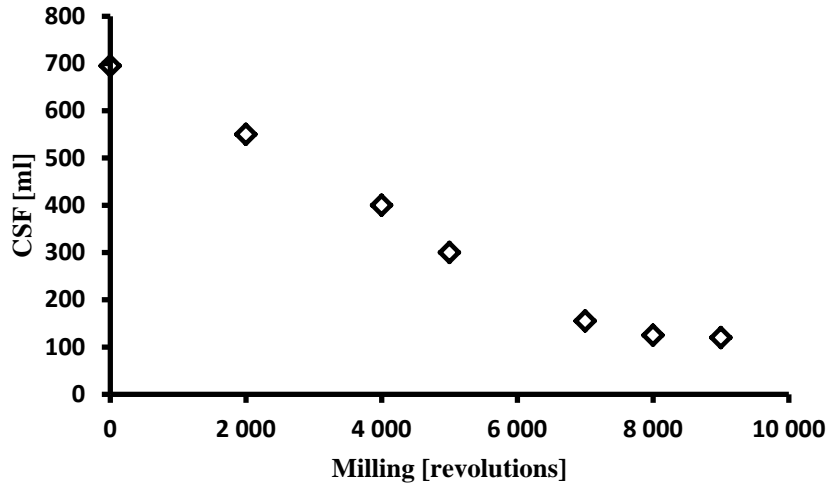


Figure 8: A milling curve for Canadian standard freeness.

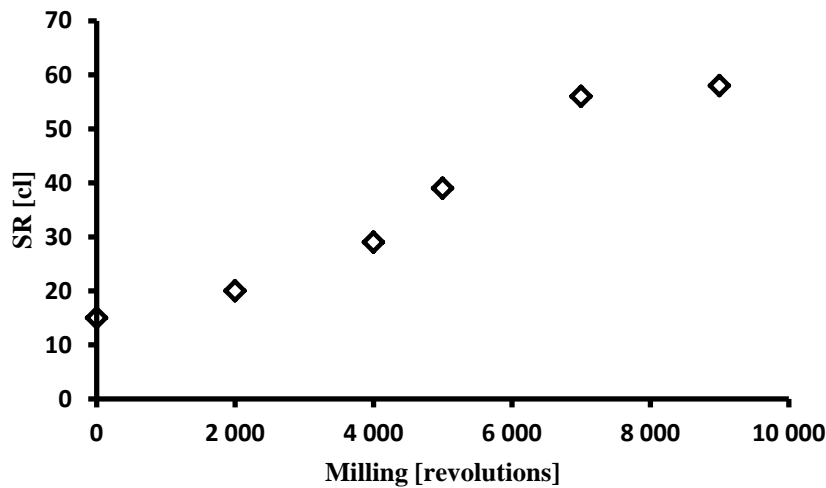


Figure 9: A milling curve for Schopper-Riegler.

From Figure 8 and 9, a few conclusions can be drawn. The initial applied shear forces have smaller effect on the SR value but the same effects cannot be observed for the CSF value. This could indicate on the presence of possible entanglements despite the following defibration after the milling. As the entanglements are broken up; more of the shear forces are used to beat the fibres and the freeness value start to exponential increase. The increase will eventually decline towards a constant value due to the design of the SR and CSF equipment. Despite further treatment some of the filtrate will always be collected through the overflow orifice. This because during the initial formation of the filter cake enough filtrate will pass through to rise the liquid level in the cone above the overflow orifice. In this state further treatment of the fibres will not affect the freeness value but will result in a denser fibre matrix for the formed filter cake.

4.3 Evaluation of filtration experiments

The results from the filtration experiments were evaluated via estimation of the specific filtration resistance through usage of Eq. 4. As presented in the chapter 2; the filtration equation is limited to incompressible filter cakes. Organic materials have a tendency to

form compressible filter cakes while inorganic materials often form incompressible filter cakes. As the cake starts to compress the result will have smaller effects compared to if the filter cake was thicker and therefore Eq. 4 is assumed to still be valid.

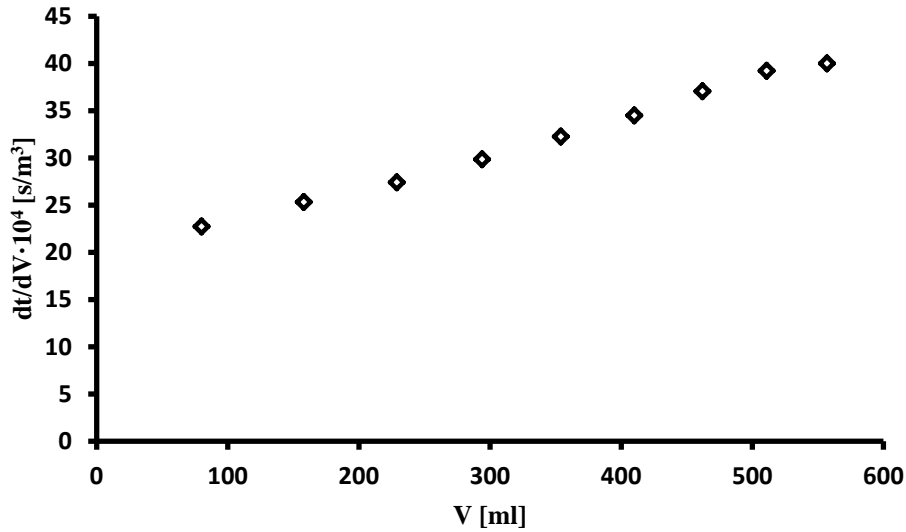


Figure 10: An example over a data series where the inverted flow rate is plotted against the collected filtrate volume.

The data from the experiments is plotted with the collected filtrate volume versus dt/dV . The shape of the data tells the conditions during the experiments and can be used to validate if the conditions during the experiments correspond to the ones for the filtration equation. If the data form a straight line then the Eq. 4 can be used to explain the filtration process. Figure 10 represent a filtration experiment where the data points form a straight line, which implies that the chosen filtration equation can be applied during experimental time.

The data points do not form a straight line immediately and the reason is the filtrate flow change from a constant flow to droplets. The size of the droplets varies and also the interval with they leave the wire screen. The consequence is that during certain time steps the no increase in mass will be registered and other time steps will registered higher amount of mass than it should be. This fluctuation can be removed by a simple average over a suitable range of data points.

The filtration experiments were followed by an investigation on the porosity of the formed cake. The wet weight was measured on five filter cakes (SR of 39 and CSF of 300 ml) before drying. Following porosity was calculated from the wet and dry weight, 6.25 %, 3.95 %, 4.73 %, 5.09 % and 3.95 %. The difference in porosity for filter cakes with the same freeness value might be the reason for the distribution of specific filtration resistance performed on each freeness level. The underlying reason for this variance might be due to variations in the slurry concentration that passes the inlet to the filtration cell (see Appendix 1 Table 2). This is also reflected in the cake height of the formed filter cake. The change was less than half a centimetre but in order to the original size it was significant.

4.4 The effect of millings on the specific filtration resistance

As the data have been collected with a very small time interval (2 seconds) a mean average was used enable an estimation of the specific filtration resistance. The times span for the mean average varied depending on the number of available data points in

the series, all from 12 seconds up to 30 seconds. In the three figures bellow specific filtration resistance is compared with level of milling and freeness values.

The magnitude of the specific filtration resistance can be calculated by using Eq. 4 and solving for the specific filtration resistance, where the slope of the data points is proportional to the specific filtration resistance (as in Figure 10). In Figure 11, 12 and 13 the specific filtration resistance is plotted against grade of milling and freeness values.

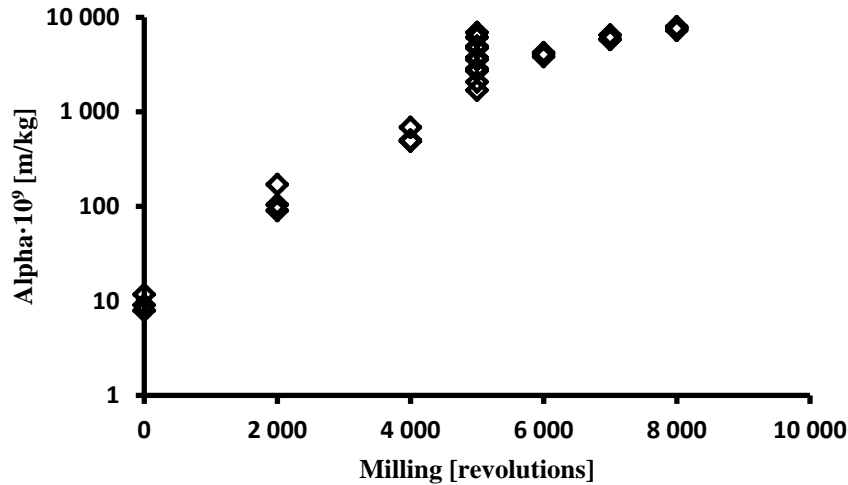


Figure 11: Milling versus specific filtration resistance.

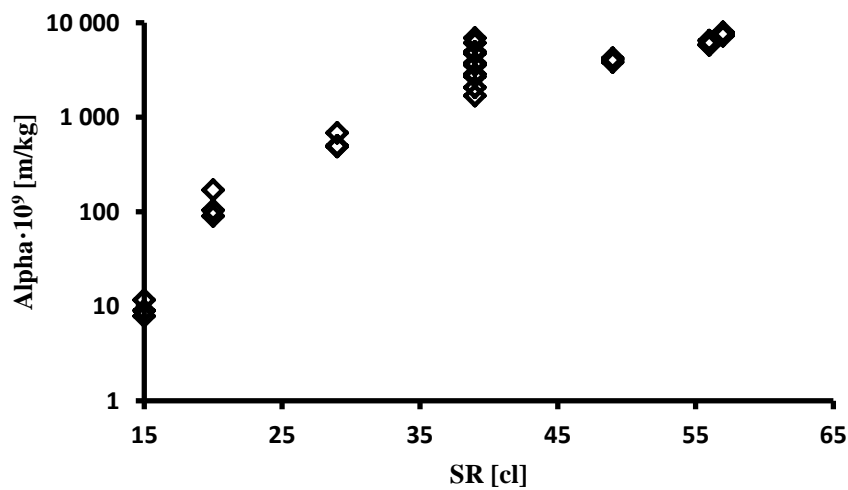


Figure 12: Schopper-Riegler versus specific filtration resistance.

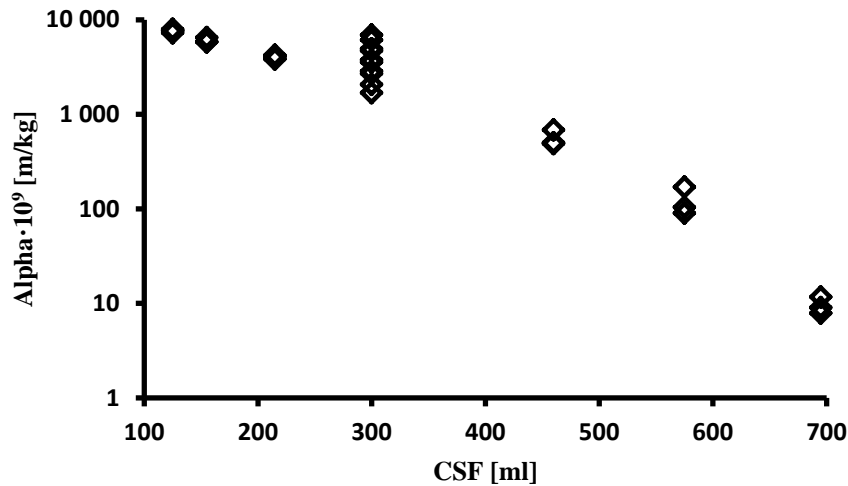


Figure 13: Canadian standard freeness versus specific filtration resistance.

By comparing Figure 11 and 12, the specific filtration resistance shows a similar relation to both level of milling and Schopper-Riegler. As expected the relation between Canadian standard freeness and specific filtration resistance shown in Figure 13 is similar to the one in Figure 12 but inverted. Through examination of slope of the data in Figure 12, the specific filtration resistance keep having an exponential growth over the measured freeness interval which follows the trend in the first part of Figure 9. While the specific filtration resistance keep an exponential growth the SR stagnates eventually at a constant value. The reason for the stagnation towards a constant value for SR and CSF is the design of the equipments where some liquid will always pass through the wire screen before a substantial filter cake has been formed. In the transition from an exponential growth to a levelling towards a constant value, a short linear region takes form. The linear region is probably a result from the exponential increase in specific filtration resistance is counteracted by the decrease in SR due to the design discussed previous.

Further comparing the specific filtration resistance with Canadian standard freeness as in Figure 13, the data points show an inverted slope as expected compared to the one in Figure 8. The specific filtration resistance values from this study can be compared to results in the literature performed on softwood fibres. Chellappah (Chellappah et al. 2009) conducted filtration experiments on dissolved tissue paper in deionized water with an overpressure of 0.45 bar. The article does not mention any freeness values for the tissue paper but Chen (Chen et al. 2006) suggest a range of the Canadian standard freeness values for tissue paper to be between 500 and 700 ml. With an approximate CSF value on the tissue paper used by Chellappah et al. the specific filtration resistance (9.4×10^{13} m/kg) that correspond to the tissue paper can be compared with the one used in this study (1×10^{11} m/kg) with similar CSF value of 575 ml. Tissue fibres are typically shorter compared to the original softwood fibres from a kraft process, as the ones used in this thesis. With shorter fibres a more compact filter cake will be formed and could be a contributing factor to the difference in filtration resistance.

Chellappah observed a significant decrease in specific filtration resistance with the use of salt solution (CaCl_2) instead of pure deionized water as dispersing medium for the fibres. In the article no definitively reason for the decrease in specific filtration resistance was stated but some suggestions are charge, packing and/or swelling effects. With an increased swelling of the fibres the structure of the filter cake will change with

an increase in porosity with result in a lower specific filtration resistance. The magnitude the effect of the salt solution had on the specific filtration resistance was a decrease with a factor 100 (Chellappah et al., 2009). The results from this study could also be compared to what Roux (Roux and Rueff, 2011) found during the study of a pilot paper machine. For a pulp with a Schopper-Riegler of 55 cl and a concentration of 3.8 g/L, the formed filter cake on the wire section had a specific filtration resistance close to 10^{10} m/kg at an overpressure of 0.25 bar. This can be compared to a specific filtration resistance of 5×10^{12} m/kg for a pulp used in this study with similar concentration and the same SR value. Roux does not mention the condition of the suspension which could influence the specific filtration resistance.

Another explanation for the difference in specific filtration resistance could be difference in filtration. The higher filtration pressure compresses the filter cake to a large extent, resulting in a higher specific filtration resistance due to low porosity.

In Figures 11, 12 and 13 at the milling level of 5000 revolutions a larger variation of the results can be seen. At this level specific filtration resistance is spread over a wide range (1.7×10^{12} to 6.9×10^{12} m/kg). A possible explanation for these fluctuations could be a clogging of the pipe from the mixing chamber to the filtration chamber. Why the pulp behaves like this for just at 5 000 revolutions and not for the others is difficult to explain. If the PFI-mill would have produced fines during the beating the behaviour could be explained with a special mixture of long fibres and fines. As the amount of fines increase with still a great portion of long fibre still present, the fines could fill up the cavities between the long fibres and form an even more solid matrix.

4.5 Evaluation of the derived CSF-model

A analyse of Eq. 14 show how the specific filtration resistance of the filter cake formed on the wire screen will change along with CSF. In Figure 14 the result from the evaluation is presented. This figure is constructed by specifying the specific filtration resistance and then calculating the CSF. In the article where the derived model is presented (Swodzinski & Doshi, 1986) no valid range over which the equation is valid for the specific filtration resistance is presented. Through an evaluation of the result presented in Figure 14, Eq. 14 seems to have a limited valid range. As discussed earlier CSF value can't reach the extreme values (0 and 1 000 ml) due to the fact that design of the equipment makes it impossible to collect all of the filtrate either through the overflow orifice or the bottom orifice.

By comparing the specific filtration resistance values from Eq. 14 and the ones from this study and the one performed by Chellappah, it seems that Eq. 14 underestimate specific filtration resistance. The values for specific filtration resistance calculated in this study are slightly more than a factor 100 larger than the ones predicted from Eq. 14.

One factor that could in least in part be sued to explain this difference is the higher filtration pressure used in this study. The higher pressure for the filtration experiments will lead to a higher specific filtration resistance and the higher pressure will compress the filter cake, resulting in even higher specific filtration resistance. The pressure of the vacuum boxes is closer to ambient pressure then the 1 bar over pressure used in this study, in the range of 15-40 kPa (Norman, 2000).

Table 1: The data used in Eq. 14 to evaluate the relation between specific filtration resistance and CSF.

V_0	1000 ml	μ	$8,94 \times 10^{-4} Pa \times s$
V_C	24,2 ml	ρ	$1 g/cm^3$
Q_{10}	8,8 ml/s	g	$981 cm/s^2$
C_0	3 g/L		
A	0,0082 cm ²		

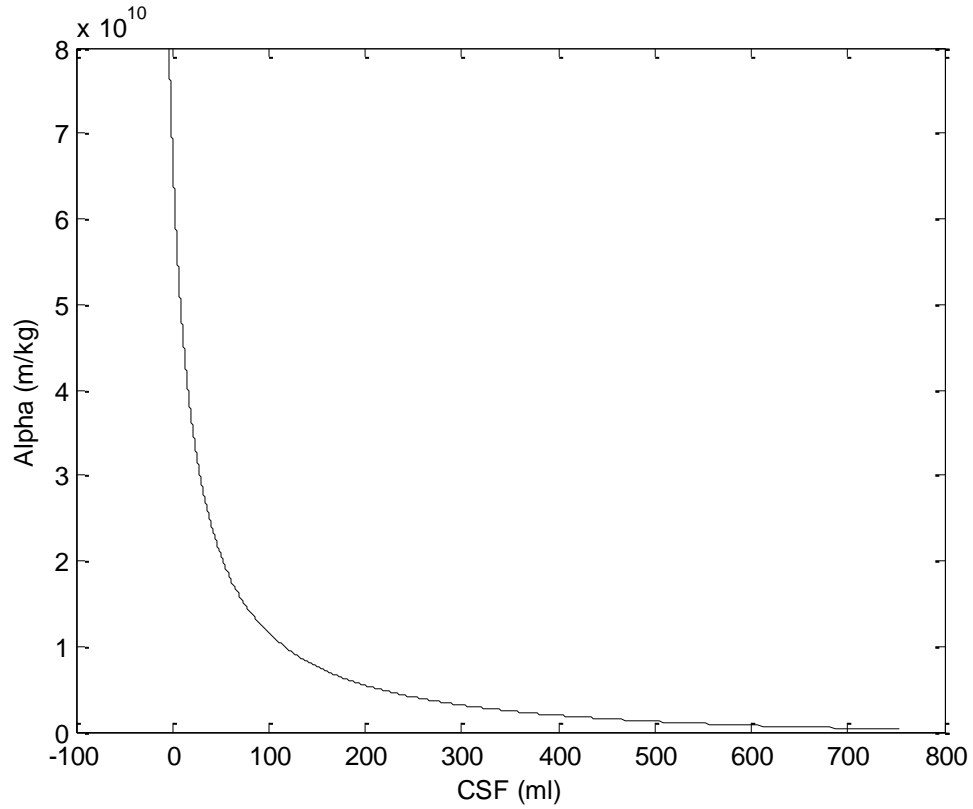


Figure 14: How the Canadian freeness values change with the specific filtration resistance

5 Conclusions

- The filtration experiments on milled softwood fibres with a fibres length of 2.05 to 2.25 mm gave specific filtration resistance from 9×10^9 to 7.7×10^{12} m/kg at an over pressure of 1 bar . This corresponded to a CSF range of 695 ml to 125 ml and a SR range of 15 to 57 cl.
- A comparison of the results from the evaluation of the CSF-model and the result from this study, indicate on lower values on the specific filtration resistance for the CSF-model. An important explanation for this difference is the difference in prevailing pressure and the following difference in compression of the filter cake.
- With the use of current filtration equipment introduce a challenge to keep the slurry concentration constant in inlet flow to the filtration cell.

6 Future work

To be able to make a better evaluation of the correlation between the freeness and the filtration resistance, experiments at lower filtration pressure should be performed. Using this kind of data the freeness equations (Eq. 14 and 27) could be used to estimate the specific filtration resistance on the wire section at filtration equipment by the freeness values. This would be adventitious since instead of performing filtration experiment that could take up to 30-60 minutes; the same result could be reached by using a freeness equipment where the time needed for one experiment is around a minute.

7 Nomenclature

A	filtration area [m^2]
A_1	cross sectional area of the upper section in the SR-equipment [m^2]
A_2	cross sectional area of the the lower section in the SR-equipment [m^2]
CSF	Canadian standard freeness value [ml]
c	the mass of solid per unit filtrate volume [kg/m^3]
c_s	concentration of solids in the slurry [kg/m^3]
g	acceleration of gravity [m/s^2]
h	height [cm]
J_R	correction factor
K	permeability [m^2]
k	Kozeny Carman constant
P_s	solid compressive pressure [Pa]
P_c	pressure drop over filter cake [Pa]
Q_1	volumetric flow rate through the bottom orifice [cm^3/s]
Q_2	volumetric flow rate through the side orifice [cm^3/s]
Q_T	volumetric flow rate out of the drainage chamber [cm^3/s]
R_m	filter media resistance [m^{-1}]
S_p	specific surface [m^2]
SR	Schopper-Riegler value [cl]
u	superficial flow velocity [m/s]
t_{tot}	total filtration time [s]
t_U	time for drain the upper section of the SR-equipment [s]
t_M	time for drain the middle section of the SR-equipment [s]
t_L	time for drain the lower section of the SR-equipment [s]
V	volume of filtrate [m^3]
V_0	initial volume of pulp slurry in the drainage chamber [m^3]
V_1	volume of filtrate collected during the time span [m^3]
V_c	volume of the fluid between the bottom orifice and the overflow orifice [cm^3]
V_U	volume of the filtrate out of the drainage chamber for SR at time t_U [cm^3]
v	superficial velocity along the drainage chamber [m/s]
v_s	superficial velocity of solids [m/s]
v_{sed}	settling velocity of a particle [m/s]
w	surface weight [kg/m^2]
w_c	surface weight of the filter cake [kg/m^2]

7.1 Greek letters

α	local specific filtration resistance [m/kg]
α_{av}	average specific filtration resistance [m/kg]
μ	viscosity of fluid [$\text{Pa}\cdot\text{s}$]
ρ	filtrate density [kg/m^3]
ρ_s	solid density [kg/m^3]
\emptyset	local volume fraction of solids

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9 Appendix I

Table 2: A compilation of all the C used in the filtration equation.

SR(cl)	C (g/L)
15	2,4
15	2,32
15	1,77
20	2,28
20	2,18
20	2,6
29	2,04
29	2,13
29	2,27
29	2,21
39	1,65
39	1,52
39	1,63
39	1,6
39	1,74
39	2,25
39	1,63
39	1,36
39	1,54
39	1,64
49	2,16
49	1,81
49	1,91
56	1,7
56	1,7
56	1,67
57	1,83
57	1,66
57	1,94
57	1,88