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Electro-assisted dewatering of cellulosic materials

A comparison between fibrillated materials

Master's thesis in Innovative and Sustainable Chemical Engineering

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Department of Chemistry and Chemical Engineering
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MASTER'S THESIS 2017:NN

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Abstract

The traditional paper mills shift more towards biorefinaries to develop new materials that can replace fossil based materials. One of these materials is MicroFibrillar Cellulose which often is disintegrated with a mechanical treatment to form small and robust fibers. With this mechanical treatment that makes these fibers smaller than the traditional cellulose fibers and also creates a larger surface area, these fibers becomes very difficult separate from the liquid, which requires new techniques in the process.

If the mechanical dewatering stage becomes less effective, the thermal drying stage will become even more energy demanding than it is today. Electro-assisted filtration is suggested as one option which can increase the filtrate flow rate of cellulosic materials for the mechanical dewatering stage to make it more efficient. The electro-assisted filtration is performed by applying an electrical field to the suspension, keeping the material from forming a filter cake and can therefore get a higher filtration flow rate. This operation is highly dependent on the properties of the material, like for example the charge of the fibers. Therefore in this thesis two fibrillated pulps are compared, with or without a TEMPO oxidation as a chemical treatment.

The electro-assisted filtration had a strong influence of the cellulosic materials, especially the chemical treated ones. Both of the materials for the electroosmotic dewatering and the fibrillated pulp for electro-assisted filtration was mathematically modeled to make sure the theory matched the experimental data. The specific energy demand is affected by the applied voltage to the system, but is significantly lower than the specific energy demand that thermal drying have. Both of the systems was highly dependent on the electrical resistance and choice of filter medium and electrode material. Ohmic heating did not affect the fibrillated dissolving pulp that much, although in the TEMPO oxidized pulp this effect was significant. This is due to the higher conductivity of this suspension.

Keywords: solid-liquid separation, filtration, dead-end filtration, electro-assisted filtration, electroosmosis, dewatering, TEMPO oxidation, MicroFibrillar cellulose

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Jessica Jakobsson, Gothenburg, June 2017

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List of Symbols

Greek letters

α	Specific filtration resistance $[m/kg]$
α_{avg}	Average specific filtration resistance $[m/kg]$
$\varDelta H_{vap}$	Enthalpy of vaporization $[kJ/kg]$
$\varDelta P$	Pressure drop $[Pa]$
$\varDelta p_E$	Electroosmotic pressure $[Pa]$
$\varDelta p_H$	Hydraulic pressure $[Pa]$
μ	Viscosity of the liquid phase $[Pa * s]$
ϕ	Solidosity $[m^3/m^3]$
ϕ_{avg}	Average solidosity $[m^3/m^3]$
ρ_s	Solid density $[kg/m^3]$
ε_0	Permittivity of vacuum $[F/m]$
ε_{rs}	Relative dielectric constant of the fluid $[-]$
ζ	Zeta potential $[V]$

Latin letters

$\dot{Q}$	Power $[W]$
A	Filter area $[m^2]$
c	Mass of solids per filtrate volume $[kg/m^3]$
E	Electrical field strength $[V/m]$
E_{cr}	Critical electrical field strength $[V/m]$
h	Height of the filter cake $[m]$
I	Current $[A]$
K	Permeability $[m^2]$
k	Kozeny-Carman constant $[-]$
P	Pressure $[Pa]$
R	Electrical resistance $[\Omega]$
R_c	Filter cake resistance $[m^{-1}]$
R_m	Filter medium resistance $[m^{-1}]$
S_p	Specific surface area $[m^2]$
t	Time $[s]$
V	Volume $[m^3]$
v_e	Electrophoretic velocity $[m/s]$
V_L	Filtrate volume $[m^3]$
v_{e0}	Electroosmotic velocity $[m/s]$
V_{solid}	Volume of solid material in the filter cake $[m^3]$
V_{total}	Volume of the filter cake $[m^3]$
z	Distance from the filter medium $[m]$

1

Introduction

1.1 Background

Renewable chemicals and low energy demanding processes are highly wanted in today's chemical industries. In the forest industry the traditional pulp mills will shift more and more towards biorefinaries and in addition to the traditional paper products, new products with wood as a raw material which may replace fossil based products will be produced. This leads to several changes in the process, where one of the challenges is the separation between liquids and some novel solid materials.

One new material that in small scale has started to be produced by biorefinaries is MicroFibrillated Cellulose (MFC) [Herrick et al., 1983, Turbak et al., 1983]. It is broken down by some sort of mechanical treatment, which makes the particles small and provides a high surface area. This mechanical process can however be time consuming and requires a large amount of energy, therefore a chemical treatment is performed on this material before the mechanical treatment [Brodin and Theliander, 2012]. One of the most common chemical treatments for cellulosic materials is TEMPO oxidation which increases the surface charges by adding carboxylate groups to the molecule and therefore the mechanical treatment becomes more efficient [Brodin and Theliander, 2012].

These new materials that is produced in biorefinaries are smaller fibers with a higher surface area than ordinary wood fibers, which lead to a higher filtration resistance. This makes the mechanical filtration step more time consuming,expensive or even impossible from a practical point of view.

That is why electro-assisted filtration is a suggested method to use for this process. Electro-assisted filtration is a filtration method that not only uses pressure as a driving force, it uses an electrical field as well. The electrical field takes advantage of the surface charge of solid materials to increase the filtration rate. It potentially can not only decrease the filtration time, it also reached a higher dewatering level in the mechanical drying stage which potentially can decrease the energy demand for the thermal drying stage [Weber and Stahl, 2002, Wetterling, 2014, Saveyn et al., 2005]. It has been showed that the electro-assisted filtration can be highly effective, have a low energy demand and also be used to avoid blockage of the filter medium [Yoshida, 1993].

1.2 Objective

The aim of this thesis is to study how different cellulosic materials are influenced by an electrical field during filtration. The materials that are being compared are fibrillated cellulose, either with or without undergoing a TEMPO oxidation pretreatment. The electroosmotic effects, Ohmic heating, specific energy demand and electrical resistance are evaluated. The following research questions are adressed:

- Can electro-assisted filtration improve the filtration rate of cellulosic materials compared to ordinary pressure filtration?
- What is the specific energy demand for electro-assisted filtration of the investigated cellulosic materials?
- How is electro-assisted dewatering influenced by the choice of filter medium as well as electrode material?

1.3 Outline

This thesis is divided into the following chapters: In chapter 2 the theory for all the necessary materials, operations definitions and correlations are presented, both for pressure filtration and electro-assisted filtration. In chapter 3 the different treatments made for the cellulosic materials are first presented, followed by a description of the characterization methods used for the different materials. In the last section of chapter 3 the filtration equipment is shown and described.

In chapter 4 the results from the study are shown and discussed. First the results from the characterization are presented, then the results from the experiments with the fibrillated dissolving pulp are shown. In the last section of chapter 4, the results for TEMPO oxidized pulp are presented and discussed. In chapter 5 the conclusions from the study are presented, and lastly in chapter 6 some future work that could be performed is mentioned.

2

Theory

In chapter 2 the theory behind this thesis is described. First in section 2.1 the two different materials are described, then in section 2.2 the pressure filtration are described. At last the electro-assisted filtration related phenomena and concepts are described in section 2.3

2.1 Cellulose materials

2.1.1 MicroFibrillar Cellulose

One of the new materials that may be produced in biorefineries is MicroFibrillar Cellulose (MFC). The first time that MFC was mentioned and investigated were in the works by Herricks et al. (1983) and Turbaks et al. (1983), with the aim to find a new biodegradeble material from wood. MFC is not only a renewable material that is biodegradeble, it also have some properties that makes it possible to replace other materials made from petrochemicals, like for example plastic. Properties like, mechanical strength, oxygen barrier and the fact that it is surviving both pH and temperature changes are properties that are necessary for this transformation of raw materials to work as an replacement for fossil based raw materials [Syverud et al., 2011].

MFC is broken down from cellulose in dissolved pulp to thinner fibers usually by a mechanical treatment. Since the mechanical treatment can have a large power demand and be time consuming, a chemical treatment is often made. One method that is used in this report is TEMPO-oxidation, where the fibers are first oxidized and then goes through a mechanical treatment [Syverud et al., 2011, Brodin and Theliander, 2012].

2.1.2 TEMPO oxidation

TEMPO oxidation has been used in many studies which are summarized in a recent review [Isogai et al., 2011]. TEMPO oxidation enables the primary hydroxyl groups of carbohydrates to oxidize to aldehyde and carboxylate groups. To perform this oxidation, TEMPO (2,2,6,6-tetramethylpiperidine-1-oxyl), NaBr and NaClO is reacting according to figure 2.1 at pH 10.

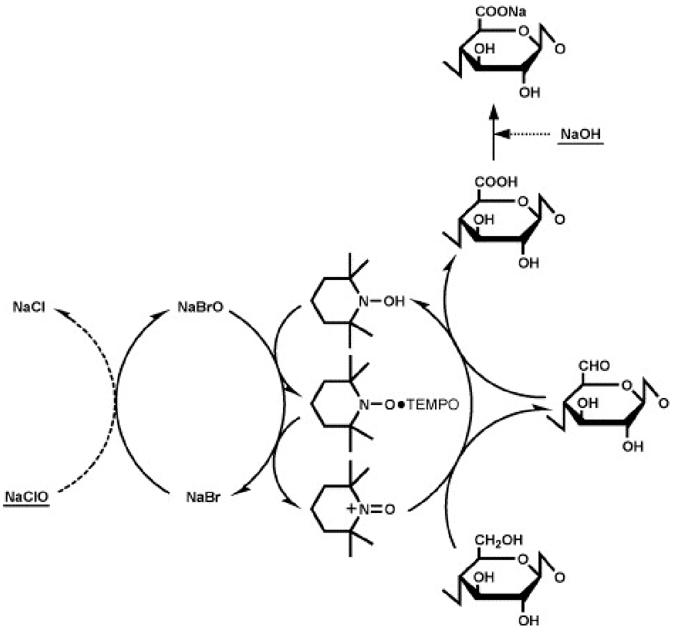

Figure 2.1: Reaction scheme of the TEMPO oxidation under alkaline conditions [Saito and Isogai, 2006]

For wood-deviated fibers this is the most common version of TEMPO oxidation and it provides a large amount of carboxylate groups and a lower level of aldehyde groups [Saito et al., 2006]. With these added carboxylate groups, the material goes faster to decrease the length and size of the fibers during the mechanical treatment. This also makes the material more charged and smaller fragments of the particles could occur in the suspension, which affects the filtration properties. This system is often performed at room temperature to achieve high selectivity and to not degrade the fibers [Brodin and Theliander, 2012].

2.2 Filtration

Filtration is a separation method used for solid-liquid separations. It is one of the oldest separation methods and it has many different applications [Matteson and Orr, 1987], one example of a process where filtration is used is in paper mills to dewater the cellulose before the thermal drying process. There are some different types of filtration, like deep bed filtration, cross flow filtration and dead-end filtration [Tien, 2006]. In this report, pressure will be used as one of the driving forces and a dead-end filtration, also know as cake filtration, will be used as a filtration technique. This will be more discussed in this section together with some important equations used to describe pressure filtration.

2.2.1 Cake filtration

In cake filtration a filter cake is formed above a filter medium: here the particles are captured by the filter medium and the fluid passes through the same medium, as seen in figure 2.2. The filtration rate is dependent on that a driving force that makes the liquid go thought the filter medium, these can be vacuum, centrifugal or pressure filtration [Ripperger et al., 2013]. The filtration operation can be illustrated as flow through a porous bed with a growing thickness during the cake filtration.

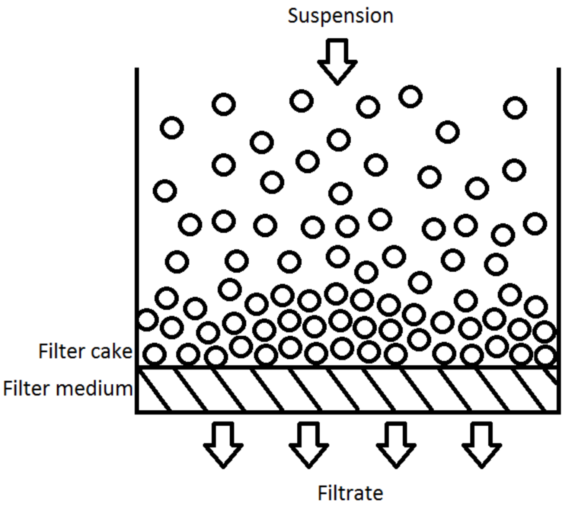

Figure 2.2: Schematic description of cake (dead-end) filtration

2.2.1.1 Flow through a porous bed

When a liquid is flowing though a porous bed there will be a pressure drop due to friction between the liquid and the solid material. This can be described by the Darcy equation [Darcy, 1856] which gives a relationship between the flow rate, pressure drop and the permeability of the porous bed, seen in equation 2.1:

$$\frac{dV}{dt} = -\frac{KA}{\mu}\frac{dP}{dz} \tag{2.1}$$

where K is the permeability of the porous bed (i.e. the filter cake), μ the viscosity of the fluid, A the flow area and $\frac{dP}{dz}$ the pressure drop in the direction of the filter medium.

2.2.1.2 Filtration resistance

The specific filtration resistance, α, depends on the surface area of the solid material and which type of porous structure that the cake forms [Ripperger et al., 2013]. The specific filtration resistance can be defined from the permeability and the solidosity, this is shown in equation 2.2:

$$\alpha = \frac{1}{K\rho_s\phi} \tag{2.2}$$

where ρ_s is the density of the solid material and ϕ the solidosity of the filter cake. The solidosity is defined as equation 2.3:

$$\phi = \frac{V_{solid}}{V_{total}} \tag{2.3}$$

where V_{total} is the total volume of the filter cake and V_{solid} the volume of solid material in the cake. The specific filter resistance and solidosity is only constant through a filter cake if the cake is incompressible. Most materials form compressible filter cakes where the solidosity increases with compressive pressure in the cake structure. This causes the solidosity to increase close to the filter medium. The local permeability is strongly influenced by the solidosity which Kozeny-Carman showed in equation 2.4 [Carman, 1937, Kozeny, 1927]:

$$K = \frac{(1-\phi)^3}{kS_p^2\phi^2} \tag{2.4}$$

where the empirical parameter k is known as the "Kozeny-Carman contant" and S_p denotes the specific surface area of the solid material.

2.2.2 Average filtration resistance

Since the total pressure drop is the sum of the filter medium resistance and the resistance of the filter cake equation 2.5 is introduced:

$$\varDelta P = (R_c + R_m)\mu\frac{1}{A}\frac{dV}{dt} \tag{2.5}$$

where R_m is the filter medium resistance and R_c the flow resistance over the filter cake. For incompressible filter cakes the flow resistance over the cake can be described by equation 2.6:

$$R_c = \int_0^h \frac{1}{K}dh = \alpha_{avg}\phi_{avg}\rho_s h \tag{2.6}$$

where α_{avg} is the average specific filtration resistance, ϕ_{avg} the average specific solidosity and h the height of the filter cake. One of the most classic models to use for cake filtration is the filter equation that Ruth (1935) derived is a combination of equation 2.5 and 2.6 shown in equation 2.7 [Ruth, 1935]:

$$\frac{dt}{dV} = \frac{\mu(\alpha_{avg}cV + R_m A)}{A^2 \Delta P} \tag{2.7}$$

where c is the mass of solids per filtrate volume. With this equation and experimental data, R_m and $\alpha_{avg} * c$ can be estimated for a certain filtration system. This equation can be used as long as $\alpha_{avg} * c$ is constant during the filtration to get a linear fitting to the system.

2.3 Electro-assisted filtration

In the following sections the correlations and definitions that are important for eletro-assisted filtration are presented and discussed. First the basic definitions from surface and colloidal chemistry are shown in section 2.3.1, then the phenomenas that influence the electrical field is introduced in section 2.3.2. Then the electrolysis reactions will be presented in section 2.3.3 and at last the model used for the evaluation of the data is shown in section 2.3.4

2.3.1 Definitions from surface and colloidal chemistry

2.3.1.1 Ionic Strength

Ionic strength is a measurement of the concentration of ions in a solution. This can be affected by adding different salts to the solution, changing the pH or increasing the electrolysis reaction that occurs during electro-assisted filtration. It is well known that positive ions are attracted by the cathode and negative ions are attracted the anode. This is also the case for charged particles or materials such as fibers.

2.3.1.2 Electric double layer

For charged particles in a suspension an electric double layer is formed due to attraction of ions with opposite charge. The solid material in a suspension has a surface charge which then attracts the ions of the solution with the opposite charge that is near the surface, outside this double layer a diffusive layer is formed around the rest of the material [Weber and Stahl, 2002], shown in figure 2.3. The ions that form the closest barrier to the solid surface of the electrical double layer, can be used as a way to determine the zeta-potential (ζ - potential).

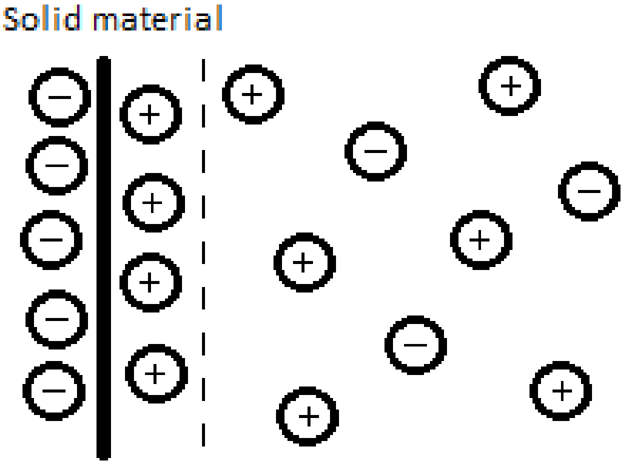

Figure 2.3: Illustration of the electric double layer

2.3.1.3 ζ -potential

The ζ- potential gives a measure of the electrical potential of the double layer. When a solid particle is moving around in the solution, a so called shearplane shapes around the particle. The ζ -potential is the difference between the potential on the shearplane and the bulk liquid in the solution [Weber and Stahl, 2002]. The ζ-potential is dependent on both the pH in the solution, the particles charge and the ionic strength [Mahmoud et al., 2010].

2.3.2 Influence of the electrical field

There are many phenomena that can influence the electrical field. Electrophoresis and electroosmosis are two of the most important phenomena when electro-assisted filtration is performed, they will therefore be further introduced in this section.

2.3.2.1 Electrophoresis

Electrophoresis describes how a charged particle in an electrical field is attracted and moves towards the electrode with the opposite charge. This correlation may be described by the Helmholtz-Smoluchowski equation shown in equation 2.8 [Hiemenz and Rajagopalan, 1997]:

$$v_e = \frac{\varepsilon_{rs}\varepsilon_0\zeta}{\mu}E \tag{2.8}$$

where v_e is the electrophoretic velocity in the direction towards the cathode, ε_{rs} the relative dielectric constant of the fluid, ε_0 the permittivity of vacuum and E the strength of the electrical field. Equation 2.8 is valid when the radius of the particle is much higher than the Debye length, which is a measure of the thickness of the electrical double layer.

2.3.2.2 Electroosmosis

Electroosmosis describes how the bulk liquid moves relative to the charge surface [Weber and Stahl, 2002]. This correlation is the opposite of the electrophoresis equation above, shown in equation 2.9 [Hiemenz and Rajagopalan, 1997].

$$v_{e0} = -\frac{\varepsilon_{rs}\varepsilon_0\zeta}{\mu}E \tag{2.9}$$

where v_{e0} is the electroosmotic velocity of the fluid in the direction towards the cathode. As for the electrophoresis equation, equation 2.9 is valid when the radius of the particle is much higher then the Debye length.

2.3.3 Electrochemical reactions in a electric field

When applying an electrical field to the water-based system there are electrolysis reactions that could occur at the elctrodes. These reactions was first introduced by Lockhart (1983b). At the cathode these reactions are:

$$\mathrm{2H_2O + 2e^- \longrightarrow H_2(g) + 2OH^-} \tag{C1}$$

$$\mathrm{M^{n+} + ne^- \longrightarrow M} \tag{C2}$$

where M can be any ionic substance in the system that can be reduced. At the anode the reactions that occurs looks like the following:

$$\mathrm{M \longrightarrow M^{n+} + ne^-} \tag{A1}$$

$$\mathrm{2H_2O \longrightarrow O_2(g) + 4H^+4e^-} \tag{A2}$$

These reactions does not only depend on the type of material, it is also affected by the ions of the electrolyte, which can cause some problems in the filtration step [Mahmoud et al., 2010]. Problems can be that a pH gradient forms inside the filter cake and this effect may damage the end product [Lockhart, 1983a,Lockhart, 1983b]. Moreover, the oxidation of the anode material causes a contamination of the filter cake which contaminate the product, in addition it also increases the operating costs since the anode material must be replaced frequently. This oxidation problem can be solved using a noble metal, such as platinum. Another problem is the production of gases, O_2 and H_2, can together be explosive, which causes a safety risk, although this can be eliminated by ventilation [Mahmoud et al., 2010].

2.3.3.1 Joule heating

When an electrical field is applied over a filter cake, a rise in temperature can occur. This is due to energy dissipation that occurs in the system. This was first discovered by Joule in 1841 [Joule, 1841]. He discovered that the nowadays well know correlation of the amount of heating that is produced, see equation 2.10:

$$\dot{Q} \propto I^2 R \tag{2.10}$$

where $\dot{Q}$ is the amount of heat produced per time unit, I the electrical current and R the electrical resistance of the conductor. Joule heating is also known as Ohmic heating since it correlates with Ohm´s law. This phenomena can be of great importance in electro-assisted filtration as an increasing temperature influence the viscosity of the fluid, which can change the rate of the filtration operation. However, if the temperature rises too a very high level, it can potentially damage the cellulostic material or end product, which is not desired.

2.3.4 Modelling of electro-assisted filtration

Yukawa (1976) introduced a model for electrofiltration that not only included the effect of electroosmosis, it also considered the effects of electrophoresis on the filter cake formation [Yukawa et al., 1976], shown in equation 2.11:

$$\frac{dV_L}{dt} = \frac{(\Delta p_H + \Delta p_E)A^2}{\mu(\alpha_{avg} c(\frac{E_{cr}-E}{E_{cr}})V_L + R_m A)} \tag{2.11}$$

where V_L is the filtrate volume, t the time, Δp_H the applied hydraulic pressure, Δp_E the electroosmotic pressure, A the filtration area and μ the dynamic viscosity. $\frac{E_{cr}-E}{E_{cr}}$ is called an electrophoresis coefficient where E_{cr} is the critical electric strength. This equation can also be used to model electroosmosis experiments, however then it needs to be modified since α_{avg} is assumed to be zero and Δp_H is zero for electroosmosis. The modified equation valid for electroosmosis is shown in 2.12

$$\frac{dV_L}{dt} = \frac{\Delta p_E A}{\mu R_m} \tag{2.12}$$

3

Methods

In this chapter the methods used for this thesis is shown. In section 3.1 the pretreatment for the different cellulostic materials is presented, in section 3.2 the different ways to characterize the materials are shown and in the last section 3.3 the filtration equipment is presented.

3.1 Preparation of model materials

The raw material used for the preparation of model materials is dried sheets of dissolving pulp from a Scandinavian sulfite mill. From these sheets 30 g dry mass are weighed in and are covered in deionized water to swell for at least 15 minutes before the disintegration. The pulp is diluted with 2 liters of deionized water and is then beaten up twice with a standard disintegrator (Noram model G3-13, Lorentzen and Wettre, Canada Inc.) that runs 30 000 revolutions per ∼10 minutes. After the disintegration the fibers were briefly treated in a ordinary household mixer in order to avoid lumps in the suspension. The suspension are then diluted to a solid content of 1 weight-%.

3.1.1 Mechanical treatment of the fibrillated dissolving pulp

The dissolving pulp suspension was subjected to mechanical treatment to get a fibrillated material. The suspensions were treated with an IKA Ultra Turrax ®T50 with a S50N-G45F dispersing element. A rotation speed of 10 000 rpm are used for 360 min in order to get a decreased fiber length and to increase the surface area of the cellulose particles [Wetterling, 2014]. The treatment are performed in a well mixed beaker with external cooling in order to limit the temperature rise during the treatment.

The treated suspension are left for at least 12 hours to get an even swelling of the fibers. In these 12 hours the suspension have a constant stirring to avoid sedimentation.

3.1.2 TEMPO oxidation

A chemical pre-treatment method are also used, called TEMPO oxidation, which is described in section 2.1.2. This method is used to oxidize the pulp so that the charge on the cellulose particle surface increases. TEMPO (2,2,6,6-tetramethylpiperidine-1-oxyl), NaBr and NaClO is added to the suspension according to the concentrations in table 3.1 [Brodin and Theliander, 2012]. The oxidation are performed at room temperature and the pH is kept constant at 9.8 ± 0.2 during the reaction by adding 0.5M NaOH when needed.

Table 3.1: Amount of chemicals used in the TEMPO oxidation treatment

TEMPO (mmol/g pulp)	NaBr (mmol/g pulp)	NaClO (mmol/g pulp)
0.1	1.0	4.0

The oxidation was considered to be finished when there are no change in the pH. Methanol was then poured in the suspension to make sure that all of the chloride ions are reacted. The pH in the suspension was then neutralized adding either 0.5 M HCl or 0.5 M NaOH to the suspension. After the oxidation, the suspension was washed, with displacement washing and re-suspension, with deionized water until the conductivity of the filtrate was below 100μS/cm (7-9 liter). The washed oxidized pulp was stored at 4°C until further treatment. The oxidized pulp was mechanically treated using IKA Ultra Turrax ®T50 with a S50N-G45F dispersing element and a rotation speed of 10 000 rpm for 120 min.

3.2 Characterization

To get a characterization of the dissolving pulp fibers and the effect of the treatments some different methods was used. The length of the fibers, both before, during and after the mechanical treatment, was measured using a Kajaani FS300. The length that was used was the centerline length and is reported as the average length. The particle size distribution was measured using laser diffraction with a Mastersizer 2000. This techniques gives a measure of the length and width of the fibers.

A Scanning Electron Microscopy (SEM) are used to see the structure of the fibers before and after the mechanical treatment. This is to get an idea about how the fibers surface has been changed when the mechanical treatment has been made. The SEM are performed using a LEO Ultra 55.

3.3 Filtration experiments

3.3.1 Filtration equipment

The filtration experiments were performed using a bench-scaled filter press that are designed to measure the local filter properties [Johansson and Theliander, 2003]. To be able to perform electro-assisted filtration experiments this equipment are modified [Wetterling, 2017], a schematic drawing is shown in figure 3.1.

The suspension are poured into a cell with a hose, this cell have a height of 175mm and an internal diameter that is reduced from 50mm to 30mm closest to the filter medium due to a supporting rack and are made from Plexiglas. The cell is designed with a platinum-electrode in the middle of it. The platinum electrode has a distance of 1 cm between the wires and has a constant distance from the filter medium at 25mm.

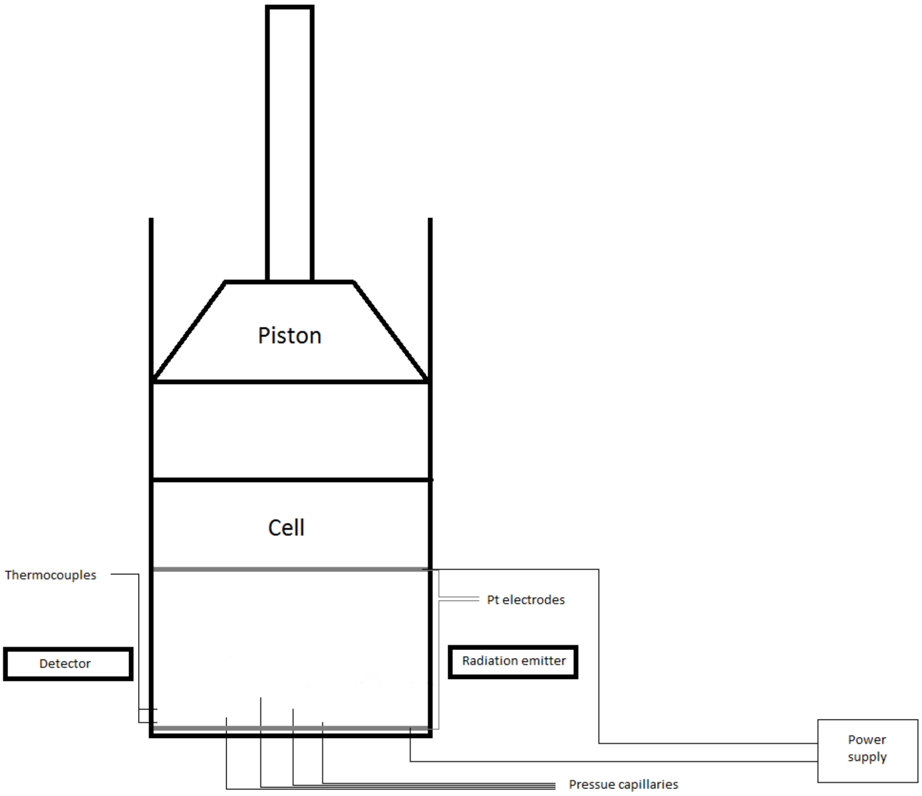

Figure 3.1: Schematic description of the filtration equipment used in the experiments

The bottom plate has four pressure capillaries that measures the liquid pressure at different distances from the filter medium. It is equipped with a platinum-mesh (Unimesh 300), placed on the bottom plate just below the filter medium. The electrodes are connected to a DC power supply (EA-PSI 5200-02 A Elektro-Automatik) that records the voltage, power and current of the suspension every 2 seconds.

The piston can add an external pressure up to 6 MPa, the position of the piston is measured every 2 seconds. The temperature is also recorded every 2 seconds using two PFA-coated K-type thermocouples that are located 5 mm and 20 mm from the filter medium. Last there is a scale below the filter equipment that collects the filtrate from the filtration, this is also recorded every 2 seconds.

For viscous suspensions the standard platinum anode is too mechanically weak. Therefore another electrode with more mechanical strength were needed to be introduced. The criteria where that the grid were not too close, so the suspension easy can pass through. These criteria were fulfilled with an electrode in stainless steel with 0.36 cm between the wires, which is polished between each experiment to start with the same conditions, since stainless steel can oxidize during electrolysis reactions.

3.3.2 Filter Medium

Four different filter mediums are used in order to evaluate the influence of the filter mediums on electro-assisted dewatering. Three of them are of the same material but with different pore sizes and one are of a different material, see table 3.2. The different pore sizes are investigated to see how the results differ, both in filtration rate, power demand and other filtration properties. The different materials of the filter mediums are investigated to see if the material of the medium influence the filtration properties. In all the experiments a support filter paper, Munktell Grade 5, is used.

Table 3.2: Difference between filter mediums used in the experiments, the pore sizes are according to the product specifications

Brand	Material	Pore size (µm)
Pall Corporation, Supor ®	Hydrophilic Polyethersulphone	0.10
Pall Corporation, Supor ®	Hydrophilic Polyethersulphone	0.45
Pall Corporation, Supor ®	Hydrophilic Polyethersulphone	0.80
Sartorius Stedim Biotech (RC)	Regenerated Cellulose	0.45

4

Results and Discussion

The filtration and electro-assisted filtration behavior of the two different cellulostic material are discussed in chapter 4. This chapter is divided into three different parts, first the characterization of the two cellulosic materials are discussed in section 4.1. Then the filtration behavior of the fibrillated dissolving pulp is discussed in section 4.2, both for pressure filtration, electroosmosis, electro-assisted filtration, filter medium and the electrode material. At last the TEMPO oxidized pulps filtration behaviour is discussed in section 4.3, both for the pressure filtration and electroosmosis experiments.

4.1 Characterization

4.1.1 Fibrillated dissolving pulp

The effects of the mechanical treatment on the fiber length is shown in figure 4.1. The length of the fibers decrease significantly during the early stages of the treatment, after this only a limited decrease is observed. The same behaviour is observed in the particle size distribution at different times during the mechanical treatment, shown in figure 4.2. The size of the fibers decreases rapidly until about 240 minutes, then the size of the fibers evens out.

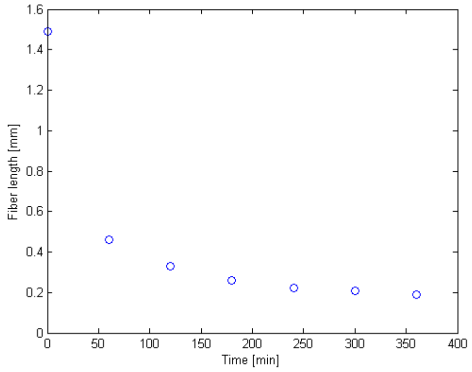

Figure 4.1: Shows how the fiber length decreases over time

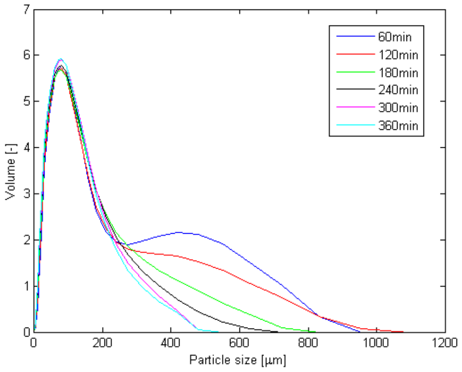

Figure 4.2: Shows the size distribution of the fibers

Even though the decrease of the size of the fibers is rather modest at longer times, the fibers are subjected to shear forces which increases the surface area [Wetterling, 2014]. The effect of the mechanical treatment on the surface structure of the material is shown in figure 4.3 and 4.4. In figure 4.3 the material before the mechanical treatment is shown. The fiber surface is quite smooth with small fragments on it. Figure 4.4 shows how the fibers surface structure looks like after the mechanical treatment. The surface is fibrillated and the fiber looks damaged, the surface area has thus increased as a result of the mechanical treatment.

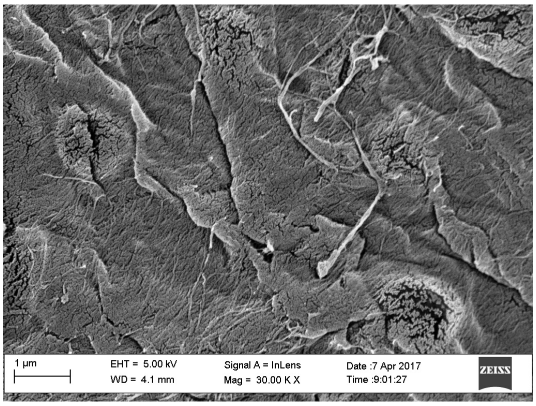

Figure 4.3: A microscopic image of the cellulose fibers before the mechanical treatment

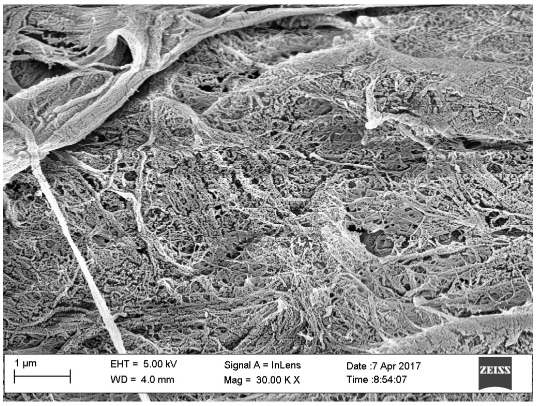

Figure 4.4: A microscopic image of the cellulose fibers after 360 minutes of the mechanical treatment

4.1.2 TEMPO oxidized pulp

The preparation of the TEMPO oxidized pulp is described in section 3.1.2. The pulp is first washed and after each liter the conductivity of the filtrate is measured to make sure that the suspension is clean enough to perform electro-assisted filtration on. After 7 liters 60 μS/cm is reached, which is considered low enough, see figure 4.5.

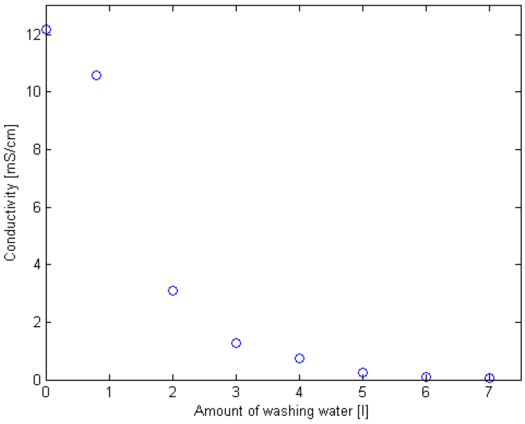

Figure 4.5: The conductivity during the washing of the TEMPO oxidized pulp

The washed TEMPO fibers were fibrillated using a mechanical treatment. The effect on the fiber length is shown in figure 4.6. The fiber length is decreasing rapidly until about 60 minutes of the mechanical treatment. If this figure is compared to 4.1 and 4.2, it can be seen that the length and size for the TEMPO oxidized pulp decreases faster and stabilized quicker than for the fibrillated dissolving pulp. However, the average fiber length is higher than for the fibrillated dissolving pulp discussed in section 4.1.1.

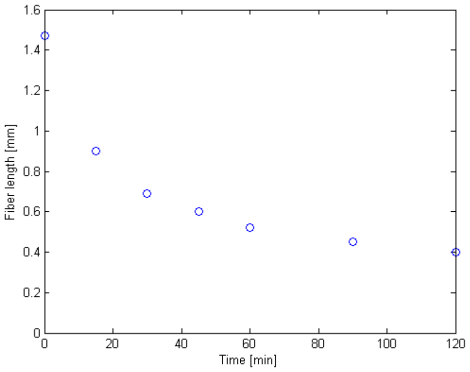

Figure 4.6: Shows how the fiber length decreases over time

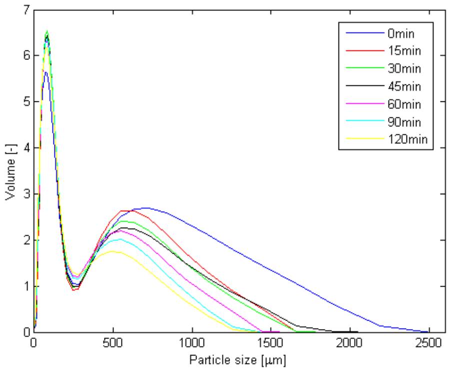

Figure 4.7: Shows the size distrubution of the fibers over time

The particle size distribution is shown in figure 4.7. For the TEMPO oxidized pulp a fraction of the fibers has a smaller size than the fibrillated dissolving pulp fibers observed in figure 4.2. TEMPO oxidation has therefore contributed to disintegration of the fibers. The size of the larger size fraction of fibers can also be seen to decrease in size much faster than the fibers in the dissolving pulp suspension. This is most likely a combined effect of the added carboxylate groups and decoupling of the carbohydrate network which makes the fibers decrease in size faster [Brodin and Theliander, 2012]. However, a fraction of larger fibers is observed in the TEMPO oxidized pulp, this could be an effect of a insufficient mixing when the mechanical treatment is performed, as the TEMPO oxidized pulp suspension is more viscous than the fibrillated dissolved pulp are discussed in section 4.1.1.

4.2 Dewatering of fibrillated dissolving pulp

The experiments on the fibrillated dissolving pulp may be divided into some different parts. First the pressure filtration is investigated, then the focus shifts towards electrofiltration, and finally electro-assisted filtration was stated. After that the filter medium and electrode material effect are investigated and discussed.

4.2.1 Filtration

The volume of filtrate over time for pressure filtration experiments with 3 bar pressure are shown in figure 4.8. These filtration experiments were repeated to obtain information regarding the experimental errors. The filter medium resistance and average specific filtration resistance for the system was determined by using equation 2.7, see in table 4.1. All three experiments are in the same range and thus show a good repeatability.

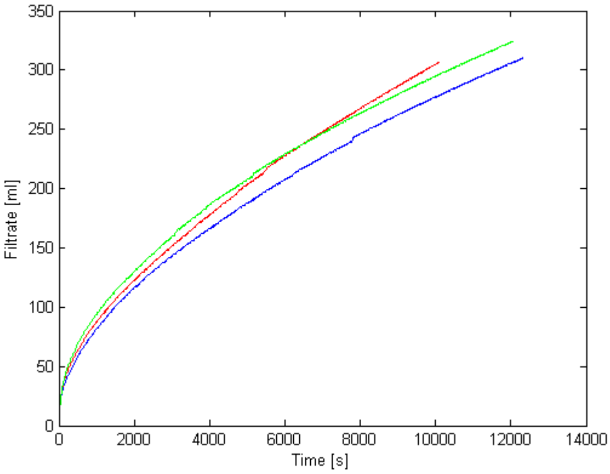

Figure 4.8: Repeated experiments for 3 bar filtration on the fibrillated dissolving pulp with Supor100 as the filter medium

Table 4.1: The $\alpha_{avg}*c$ and R_m for the repeated experiments for fibrillated dissolving pulp

Experiment	$\alpha_{avg} * c[1/m^2]$	$R_m[m^{-1}]$
1	$2.00 * 10^{14}$	$7.94 * 10^{12}$
2	$2.03 * 10^{14}$	$7.70 * 10^{12}$
3	$2.17 * 10^{14}$	$7.00 * 10^{12}$
Average	$2.07 * 10^{14}$	$7.55 * 10^{12}$

4.2.2 Electroosmosis

Electroosmosis experiments with an applied voltage of 25 V, 75 V and 150 V are shown in figure 4.9. The electroosmosis experiments had no added pressure and used Supor100 as a filter medium. The results in figure 4.9 shows that a higher applied voltage results in an increased electroosmosis flow rate. The dewatering rate seems to be constant during the early stages, then it decreases. This behaviour could be an effect of the electrolysis reactions lowering the pH of the filter cake, and thus the surface charge of the cellulose particles [Wetterling, 2017].

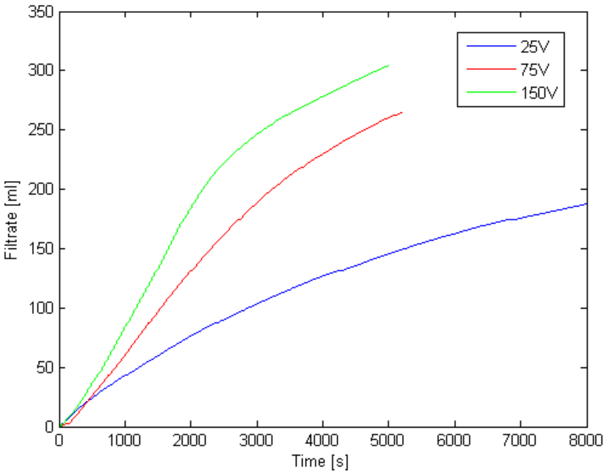

Figure 4.9: The volume for the filtrate over time for the electroosmosis experiments

The inverse electroosmotic flow rate is shown in figure 4.10. In the 25 V experiment the inverse flow rate is increased during the operation. This could be due to an increasing flow resistance during the filtration. For the 75 V and 150 V experiments the filtrate flow rate is constant during the early stages of the filtration operation. This behaviour was described by the fitted Yukawa equation, see equation 2.12. The average filter medium resistance from pressure filtration experiments given in table 4.1 is used. The values for the fitted electroosmotic pressures are shown in table 4.2 and the elecroosmotic pressure is shown to increase with higher applied voltages. At the end of the filtration operation the inverse flow rate increases, an effect of the electrolysis reactions decreasing the pH in the filter cell, thereby neutralizing the charge of the fiber surfaces.

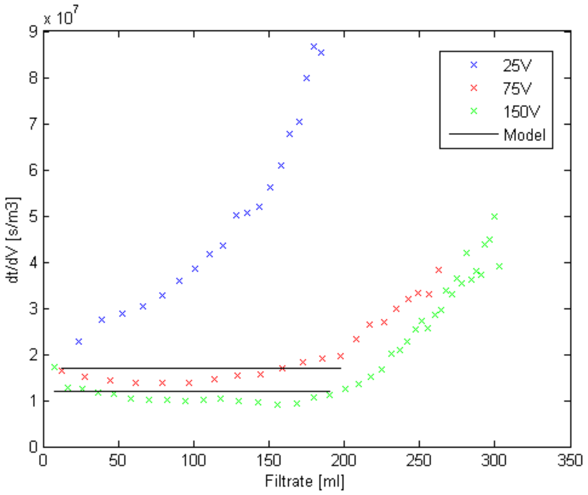

Figure 4.10: The modelled inverse flow rate for the electroosmosis experiments

Table 4.2: The electroosmotic pressue for the different experiments

Experiment	Δp_E [bar]
75 V	2.72
150 V	4.21

When electorosmosis experiments are performed, power needs to be added to form the electrical field. The specific energy demand during the electroosmotic dewatering is shown in figure 4.11. The specific energy demand increases with the increasing voltage applied to the system. The higher voltage applied to the system requires a higher current intensity and thus leads to higher losses through Ohmic heating [Larue et al., 2006]. The specific energy demand can also be seen to increase during the experiments. This is an effect of a decreasing electrical resistance, see figure 4.12. As the electrical resistance is decreasing a higher current intensity is required in order to maintain the applied voltage. However, if the specific energy demand is compared to thermal drying, this demand is low even for the 150 V experiment since ΔH_{vap} for water is 2257 kJ/kg.

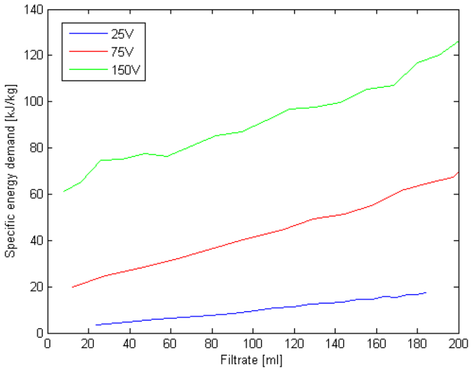

Figure 4.11: The specific energy demand for electroosmosis experiments

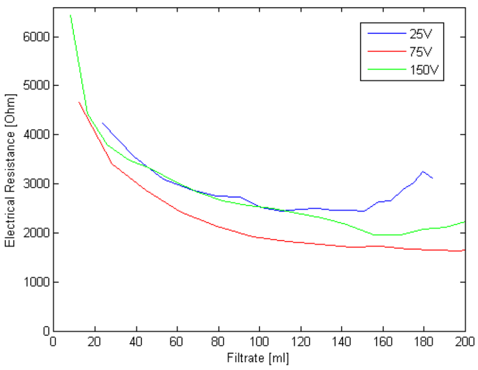

Figure 4.12: The electrical resistance for electroosmosis experiments

4.2.3 Electro-assisted filtration

Electro-assisted filtration experiments are performed with a pressure of 3 bar and a voltage of either 75 V or 150 V. In figure 4.13 the volume of filtrate versus time for the experiments is shown. These are also compared to the pressure filtration experiments with 3 bars pressure. The difference in filtrate volume over time between the 150 V and 75 V is clearly noticeable and the 150 V experiments is faster. The electro-assisted experiments are also very much faster than the experiment without an electrical field applied.

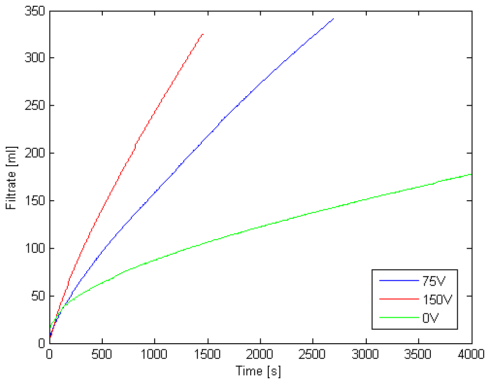

Figure 4.13: The effect for the volume of filtrate over time at 3 bar filtration pressure and various applied electrical field strengths

Figure 4.14 shows the inverse filtrate flow rate versus the filtrate volume of the electrofiltration experiments as well as the reference without an applied electrical field. For a filtration without an electrical field the filtration resistance increases during the operation due to cake formation. For the electro-assisted filtration experiments the change to the flow rate is less pronounced, the electrical field thus influence the cake formation. Increasing the applied voltage to the system can also be seen to increase the filtrate flow rate.

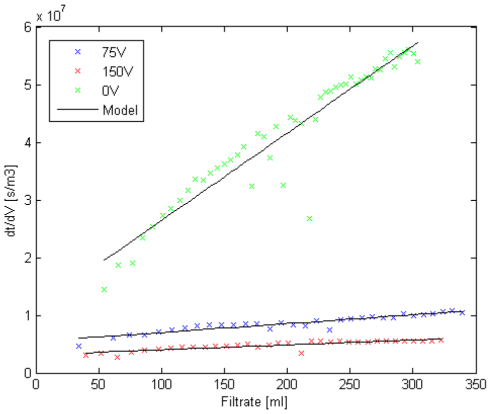

Figure 4.14: The inverse filtration flow rate of the electro-assisted filtration modelled with Yukawas eqation

The effects of the electric field, shown in figure 4.14, are described by the Yukawa equation 2.11, and the modelled values are shown as black lines in the figure. The average values of filter cake resistance and filter medium resistance in table 4.1 is used. The fitted parameters for the Yukawa equation is shown in table 4.3. The effect of the electrical field on filter cake formation is expressed by $\frac{E_{er} - E}{E_{cr}}$ and is similar for both electro-assisted filtration experiments. The electroosmotic pressure on the other hand increases with increased voltage applied to the system. This is the same trend as is observed in table 4.2 for the electroosmotic dewatering experiments without an applied filtration pressure.

Table 4.3: The electroosmotic pressue and electrical field strength for the different experiments

Experiment	Δp_E [bar]	$\frac{E_{cr} - E}{E_{cr}}$
0 V	0	1
75 V	2.27	0.20
150 V	3.64	0.19

The specific energy demand of the experiments, shown in figure 4.15, is higher for the 150 V then for the 75 V experiments. The same trends are shown here as in figure 4.11, the main difference is that an applied pressure is contributing to the filtrate flow in the electro-assisted filtration experiments and the specific energy demand is therefore lower for these experiments.

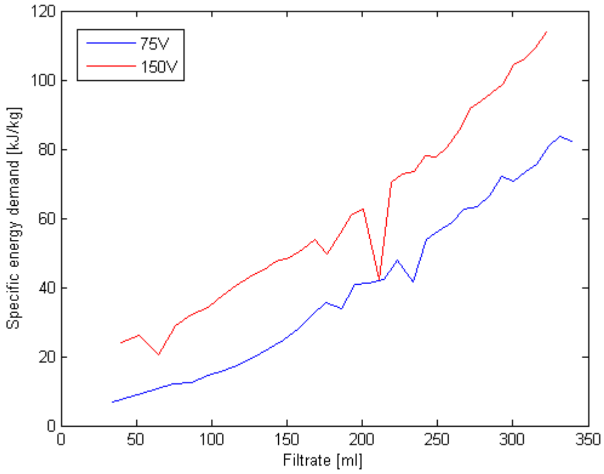

Figure 4.15: The specific energy demand for electro-assisted filtration

Ohmic heating may result in a significant rise in temperature in the filter cell system, and as a consequence influence the filtration behaviour through the viscosity of water. How the temperature was changed for the different applied voltages is shown in figure 4.16. As can be seen in the figure the temperature rises a few degrees for

150 V while for 75 V it barely rises at all and as expected in the 0 V experiment there is no noticeable rise in temperature. This temperature rise is not an effect that will damage the cellulose but the viscosity decreases if the temperature is rising which contributes to a faster filtrate.

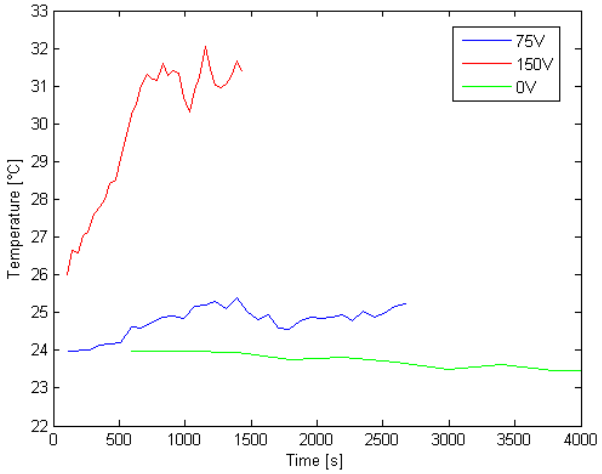

Figure 4.16: The temperature profile for electro-assisted filtration, 5 mm from the filter medium

4.2.4 Filter medium

The effect of the filter medium on the electro-assisted filtration behavior was investigated to determine which filter medium is the most suitable for the system and the suspended material used. The filter mediums used in this study are described in section 3.3.2. The different filter mediums was tested with pressure filtration at 3 bar pressure and at 150 V and 75 V for electroosmosis experiments.

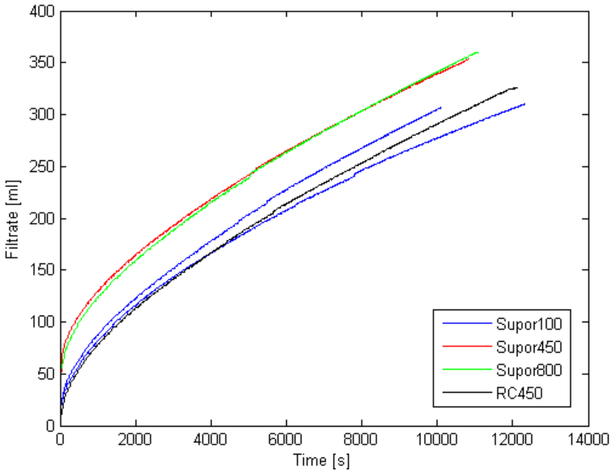

Figure 4.17: The volume of the filtrate over time for 0 V, 3 bar experiments

Figure 4.17 shows the volume of filtrate over time for pressure filtration experiments without an applied electrical field. The flow resistance of the filter medium is calculated using equation 2.7,and the results are shown in table 4.4. The filters with the highest resistances are Supor100 and RC450. Supor100 has the smallest pore size whereas the RC450 filter medium has been shown give a high flow resistance due to skin formation for cellulose materials [Mattson et al., 2012].

Table 4.4: The flow resistances for different filter mediums

Filter Medium	$R_m [m^{-1}]$
RC450	$8.17 * 10^{12}$
Supor100	$7.70 * 10^{12}$
Supor450	$7.20 * 10^{12}$
Supor800	$6.89 * 10^{12}$

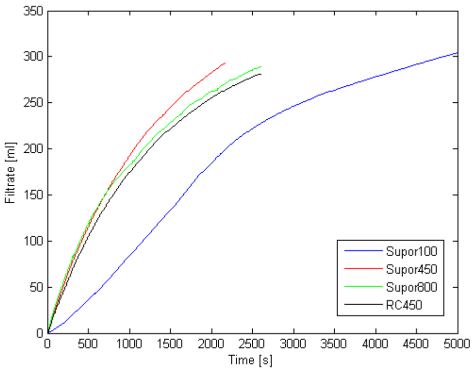

Figure 4.18: The volume of the filtrate over time for 150 V experiments

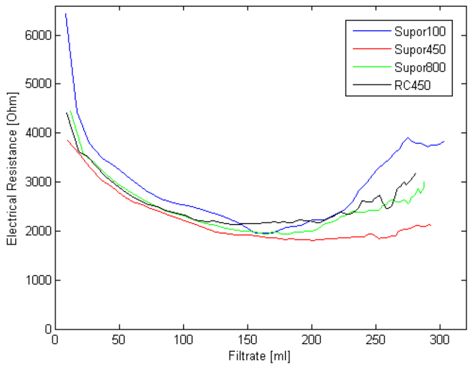

Figure 4.19: The electrical resistance for 150 V experiments

The results of the investigation of different filter mediums for electroosmosis experiments are shown in figure 4.18. For these experiments Supor100 gives a lower electroosmotic dewatering rate than all the other investigated filter mediums, including the RC450 filter medium. The increased flow resistance for the RC450 filter medium compared to Supor450 and Supor800 can not be observed when an electrical field is applied. In figure 4.19 the electrical resistance of the system is shown. For Supor100 the electrical resistance is higher than the other filter mediums during the entire experiment, thus having a detrimental effect on the electroosmotic dewatering. For electro-assisted filtration processes a filter medium with a low filtration resistance and low electrical resistance is desired. However, these properties increase with decreasing pore sizes, which is needed in order to separate small particles.

4.2.5 Electrode

A platinum anode have been used in all the experiments for the fibrillated dissolving pulp. This electrode was compared to a stainless steel electrode that has a higher mechanical strength and a closer mesh size, both of these electrodes are described in section 3.3.1. Since stainless steel is not a noble metal, the electrode is susceptible to oxidation when used as an anode. Supor450 is used as a filter medium during these experiments.

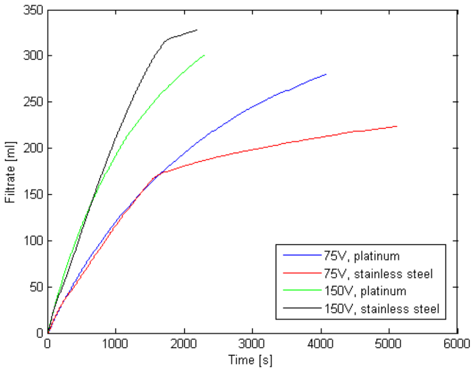

Figure 4.20: A comparison between the platinum and stainless steel electrode for 0 bar

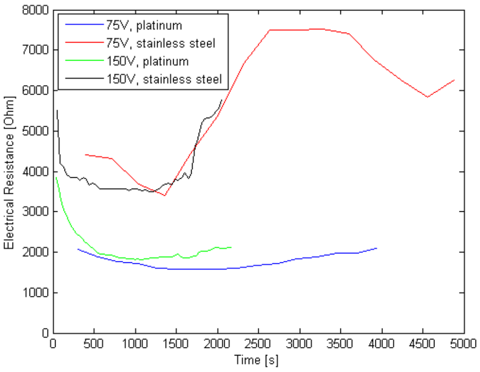

Figure 4.21: A comparison between the platinum and stainless steel electrode in electrical resistance

Electroosmosis experiments are performed using both electrode materials, shown in figure 4.20. The electroosmotic flow rate is unaffected by the electrode design in the early stages of the experiments. For the experiments using the stainless steel electrode an abrupt decrease in flow rate is observed at about 1500 seconds for both applied voltages. The corresponding electrical resistance, shown in figure 4.21, is then increasing drastically. During the cake dissection a discoloring on the top of the filter cake was found, originating from oxidation of the stainless steel electrode. The oxidation was thus likely the cause of the decreasing electrical contact between the electrodes. Platinum electrodes are therefore preferable for repeated use.

4.3 Dewatering of TEMPO oxidized pulp

The experiments that were made using the TEMPO oxidized pulp are mostly electroosmosis experiments and some pressure filtration experiments at 3 bar pressure. All the experiments are performed with Supor100 as a filter medium, as there are smaller particle fragments in the TEMPO oxidized pulp. The stainless steel electrode is used for all TEMPO oxidized pulp experiments as the mechanical strength in the platinum electrode was insufficient for the viscous suspension.

4.3.1 Filtration

In figure 4.22 the volume of filtrate versus time for the TEMPO oxidized pulp is compared to the fibrillated dissolved pulp for a pressure filtration experiment at 3 bar pressure. As can be seen, the TEMPO oxidized pulp has a much higher filtration resistance than the fibrillated dissolving pulp.

That the TEMPO oxidized pulp is so much slower than the fibrillated dissolving pulp can explained with $\alpha_{avg} * c$ and R_m, shown in table 4.5, which shows that $\alpha_{avg} * c$ is almost 100 times larger for the TEMPO oxidized pulp. R_m is also 100 times as large for the TEMPO oxidized pulp, indicating that the calculated filter medium resistance includes a flow resistance from particles deposited on the filter medium. This could also possibly be an effect of partially blinded filter medium and/or skin formation.

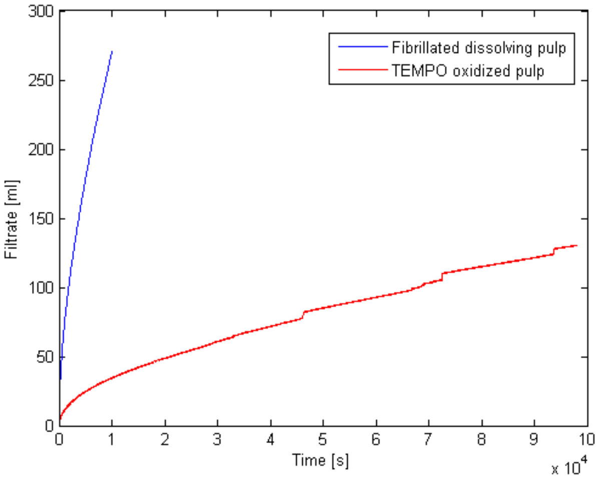

Figure 4.22: The volume of filtrate over time for 3 bar pressure filtration

Table 4.5: The $\alpha_{avg} * c$ and R_m for fibrillated dissolving pulp and TEMPO oxidized pulp

Material	$\alpha_{avg} * c[1/m^2]$	$R_m[m^{-1}]$
Fibrillated dissolving pulp	$2.01 * 10^{14}$	$7.70 * 10^{12}$
TEMPO oxidised pulp	$1.55 * 10^{16}$	$2.63 * 10^{14}$

4.3.2 Electroosmosis

Figure 4.23 shows the volume of filtrate versus time for the electroosmosis experiments. With higher voltage applied the electroosmotic flow rate increases. This trend is very similar to the observed behaviour for the fibrillated dissolving pulp in section 4.2.2. The electroosmotic flow rate is much faster than the pressure filtration for TEMPO oxidized pulp in shown in figure 4.22, showing the advantages of electro-assisted dewatering for this material. The dry content after the electroosmotic dewatering experiments was found to be between 10-14 weight-%.

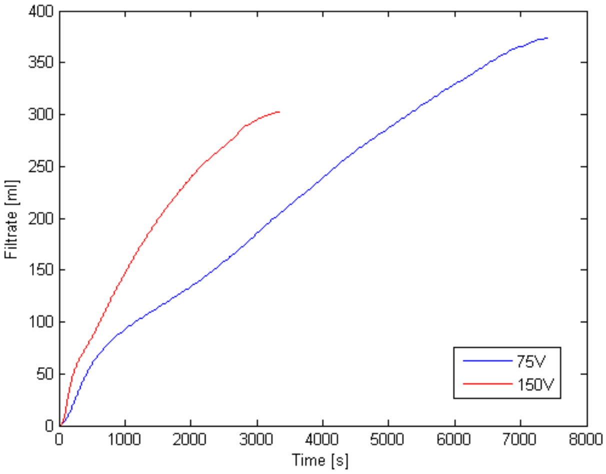

Figure 4.23: The volume of filtrate over time for electroosmosis experiments with the TEMPO oxidized pulp

The inverse electroosmotic flow rate is shown in figure 4.24. The flow rate is high during the early stages of the experiment but is gradually decreasing to a leval where it is a fairly constant flow rate. This constant level is modelled with the Yukawa equation, see equation 2.12, and the corresponding electroosmotic pressure is given in table 4.6. The filter medium resistance used is the value for fibrillated dissolving pulp, shown in table 4.1 in order to obtain electroosmotic pressure that can be used in a comparison between the cellulosic materials. The electroosmotic pressures is of the same order of magnitude, but during the early stages of the operation the dewatering rate of the TEMPO oxidized pulp is significantly higher.

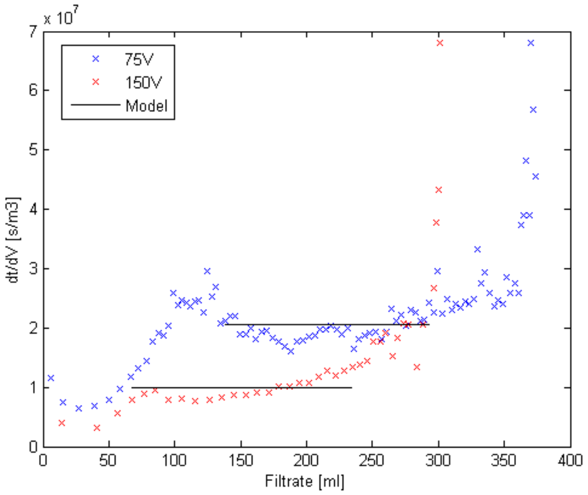

Figure 4.24: The modelled inverse flow rate for the electroosmosis experiments

Table 4.6: The electroosmotic pressure for the different experiments

Experiment	Δp_E [bar]
75 V	2.24
150 V	5.21

Figure 4.25 shows how the electrical resistance of the system is changing during the electroosmosis dewatering experiments. During the early stages the electrical resistance of the system decrease, which corresponds to the region with the higher flow rate in figure 4.24. This is the same behaviour that was observed for the fibrillated dissolving pulp experiments, shown in 4.12. After that region the electrical resistance increases, this effect can be explained by either a formation of a cellulose layer with a high electrical resistance or by oxidation of the anode, or a combination of them.

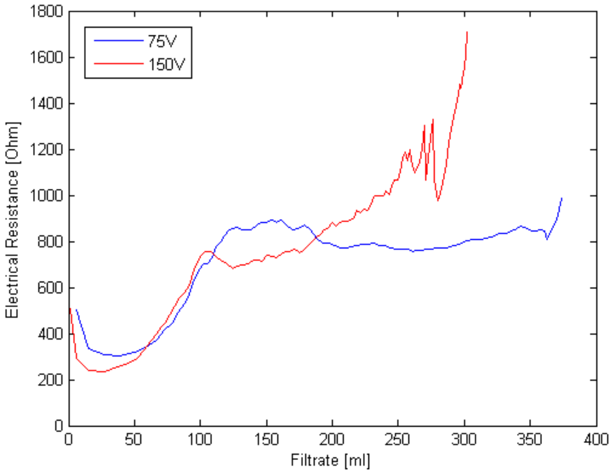

Figure 4.25: The electrical resistance for experiments with the TEMPO oxidized pulp

The specific energy demand of the electroosmotic dewatering of TEMPO oxidized pulp is shown in figure 4.26. For both of the experiments the specific energy demand is fairly constant during the electroosmotic dewatering but increases when the dewatering operation is finished. The specific energy demand of the TEMPO oxidized pulp is higher than for the fibrillated dissolving pulp in figure 4.11, a result of the lower electrical resistance of the system. The higher current intencity the higher Ohmic heating, which leads to a a significant temperature rise, see figure 4.27.

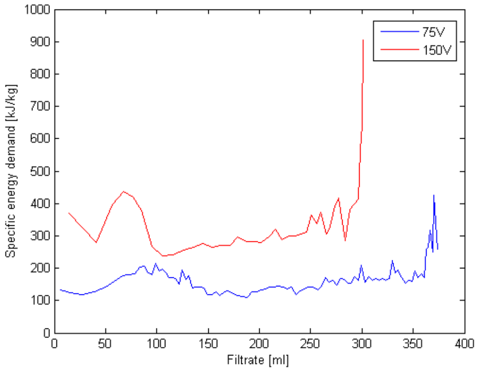

Figure 4.26: The specific energy demand for the experiments with the TEMPO oxidized pulp

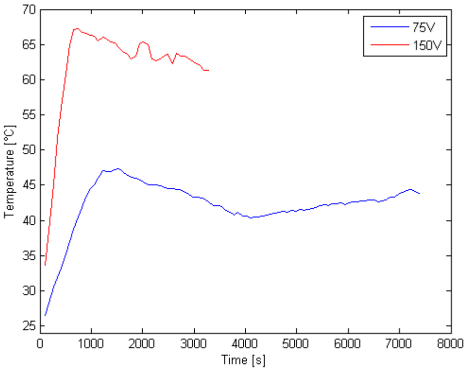

Figure 4.27: The temperature profile for experiments with the TEMPO oxidized pulp, 5mm over the filter medium

5

Conclusions

- Electro-assisted filtration decreases the time for the filtration for both the fibrillated dissolving pulp and the TEMPO oxidized pulp.
- The specific energy demand for electroosmotic dewatering is significantly lower than the ΔH_{vap} value for water, both for the fibrillated dissolving pulp and the TEMPO oxidized pulp. The specific energy demand is however highly dependent on the electrical resistance of the suspension.
- The use of a stainless steel anode during electro-assisted dewatering results in oxidation and thus contamination of the filter cake. This may have a detrimental effect on the dewatering operation.
- A filter medium with low filtration resistance and a low electrical resistance is desired for electro-assisted filtration operations. These properties are however, in contrast with the need for small pore sizes, in order to retain fine particles.
- The TEMPO oxidized pulp is harder to filtrate due to a higher specific resistance of the filter cake compared to the fibrillated dissolving pulp. Both of the materials is strongly influenced by the applied electrical field, resulting in increasing filtrate flow rate. The TEMPO oxidized pulp has an overall higher specific energy demand, which implies a higher rise in temperature, a result of a higher electrical conductivity of the suspension.

6

Future Work

The dewatering rate of the TEMPO oxidized pulp was found to be strongly influenced by an electrical field applied to the system. This needs to be investigated further: especially with an applied electrical field combined with a mechanical pressure. For this an electrode with high mechanical strength made from a noble metal needs to be used to avoid oxidation and destruction of the filter cake and electrode.

The filtrate flow rate was shown decrease during electroosmotic dewatering operation in this thesis. This behavior can be explained by the electrolysis reactions lowering the pH in the filter cell. To study the electrical field without interference of these reactions requires modification of the experimental equipment. This can be achieved either by using buffered suspensions or an equipment that allows electrode flushing.

Bibliography

[Brodin and Theliander, 2012] Brodin, F. and Theliander, H. (2012). Absorbent materials based ob kraft pulp: Preparation and material characterization. *BioResources*, 7(2):1666–1683.

[Carman, 1937] Carman, P. (1937). Fluid flow through granular beds. *Transactions of the Institution of Chemical Engineers*, 15:150–166.

[Darcy, 1856] Darcy, H. (1856). Les fontaines publiques de la ville de dijon : exposition et application des principes à suivre et des formules à employer dans les questions de distribution d'eau.. / par henry darcy. *V. Dalmont*.

[Herrick et al., 1983] Herrick, F., Casebier, R., K., H., and Sandberg, K. (1983). Microfibrillated cellulose: Morphology and accessibility. *Journal of Applied Polymer Science*, 37:797–813.

[Hiemenz and Rajagopalan, 1997] Hiemenz, Paul, C. and Rajagopalan, R. (1997). *Principles of colloid and surface chemistry*. Dekker, New York; Basel; Hong Kong, 3. ed., rev. and expanded edition.

[Isogai et al., 2011] Isogai, A., Saito, T., and Fukuzumi, H. (2011). Tempo-oxidized cellulose nanofibers. *Nanoscale*, (3):71–85.

[Johansson and Theliander, 2003] Johansson, C. and Theliander, H. (2003). Measuring concentration and pressure profiles in deadend filtration. *Filtration*, 3(2):114–120.

[Joule, 1841] Joule, J. (1841). Xxxviii.on the heat evolved by metallic conductors of electricity, and in the cells of a battery during electrolysis. *Philosophical Magazine Series 3*, 19(124):260–277.

[Kozeny, 1927] Kozeny, J. (1927). Über kapillare leitung des wassers im boden. *Mathematisch-Naturwissenschaftliche*, 136:271–306.

[Larue et al., 2006] Larue, O., Wakeman, R., Tarleton, E., and Vorobiev, E. (2006). Pressure electroosmotic dewatering with continuous removal of electrolysis products. *Chemical Engineering Science*, 61(14):4732–4740.

[Lockhart, 1983a] Lockhart, N. C. (1983a). Electroosmotic dewatering of clays, ii. influence of salt, acid, and flocculants. *Colloids and Surfaces*, 6(3):239–251.

[Lockhart, 1983b] Lockhart, N. C. (1983b). Electroosmotic dewatering of clays, iii. influence of clay type, exchangeable cations, and electrode materials. *Colloids and Surfaces*, 6(3):253–269.

[Mahmoud et al., 2010] Mahmoud, A., Olivier, J., Vaxelaire, J., and Hoadley, A. (2010). Electrical field: A historical review of its application and contributions in wastewater sludge dewatering. *Water Research*, 44(8):2381–2407.

[Matteson and Orr, 1987] Matteson, M. J. and Orr, C. (1987). *Filtration: principles and practices.* M. Dekker, New York, 2. ed., rev. and expanded edition.

[Mattson et al., 2012] Mattson, T., Sedin, M., and Theliander, H. (2012). On the local filtration properties of microcrystalline cellulose during dead-end filtration. *Chemical Engineering Science*, 72:51–60.

[Ripperger et al., 2013] Ripperger, S., Gösele, W., Alt, C., and Loewe, T. (2013). *Ullmann's encyclopedia of industrial chemistry: Filtration, 1. Fundamentals.* VCH Verlag, Germany.

[Ruth, 1935] Ruth, B. (1935). Studies in filtration iii. derivation general filtration equations. *Industrial and Engineering Chemistry*, 27(6):708–723.

[Saito and Isogai, 2006] Saito, T. and Isogai, A. (2006). Introduction of aldehyde groups on surfaces of native cellulose fibers by tempo-mediated oxidation. *Colloids and Surfaces A: Physicochemical and Engineering Aspects*, 289(1-3):219–225.

[Saito et al., 2006] Saito, T., Nishiyama, Y., Putaux, J.-L., Vignon, M., and Isogai, A. (2006). Homogeneous suspensions of individualized microfibrils from tempo-catalyzed oxidation of native cellulose. *Biomacromolecules*, 7(6):1687–1691.

[Saveyn et al., 2005] Saveyn, H., Van der Meeren, P., Hofmann, R., and Stahl, W. (2005). Modelling two-sided electrofiltration of quartz suspensions: Importance of electrochemical reactions. *Chemical Engineering Science*, 60:6768–6779.

[Syverud et al., 2011] Syverud, K., Xhanari, K., Chinga-Carrasco, G., and Res, J. N. (2011). Films made of cellulose nanofibrils: surface modification by adsorption of a cationic surfactant and characterization by computer-assisted electron microscopy. *Journal of Nanoparticle Research*, 13:773–782.

[Tien, 2006] Tien, C. (2006). *Introduction to cake filtration: analyses, experiments and applications.* Elsevier, Amsterdam;Boston, 1st; edition.

[Turbak et al., 1983] Turbak, A., Snyder, F., and K., S. (1983). Microfibrillated cellulose, a new cellulose product: Properties, uses and commercial potential. *Journal of Applied Polymer Science*, 37:815–827.

[Weber and Stahl, 2002] Weber, K. and Stahl, W. (2002). Improvement of filtration kinetics by pressure electrofiltration. *Separation and Purification Technology*, 26:69–80.

[Wetterling, 2014] Wetterling, J. (2014). *The influence of particle properties on cake filtration: surface structure and surface charge.* Chalmers University of Technology, Gothenburg, Sweden.

[Wetterling, 2017] Wetterling, J. (2017). *Filtration of Microcrystalline Cellulose: Electro-assisted filtration and the influence of surface properties.* Chalmers University of Technology, Gothenburg, Sweden.

[Yoshida, 1993] Yoshida, H. (1993). Practical aspects of dewatering enhanced by electro-osmosis. *Drying Technology*, 11(4):787–814.

[Yukawa et al., 1976] Yukawa, H., Kobayashi, K., Tsukuf, Y., Yamano, S., and Iwata, M. (1976). Analysis of batch electrokinetic filtration. *Journal of Chemical Engineering of Japan*, 9(5):396–401.