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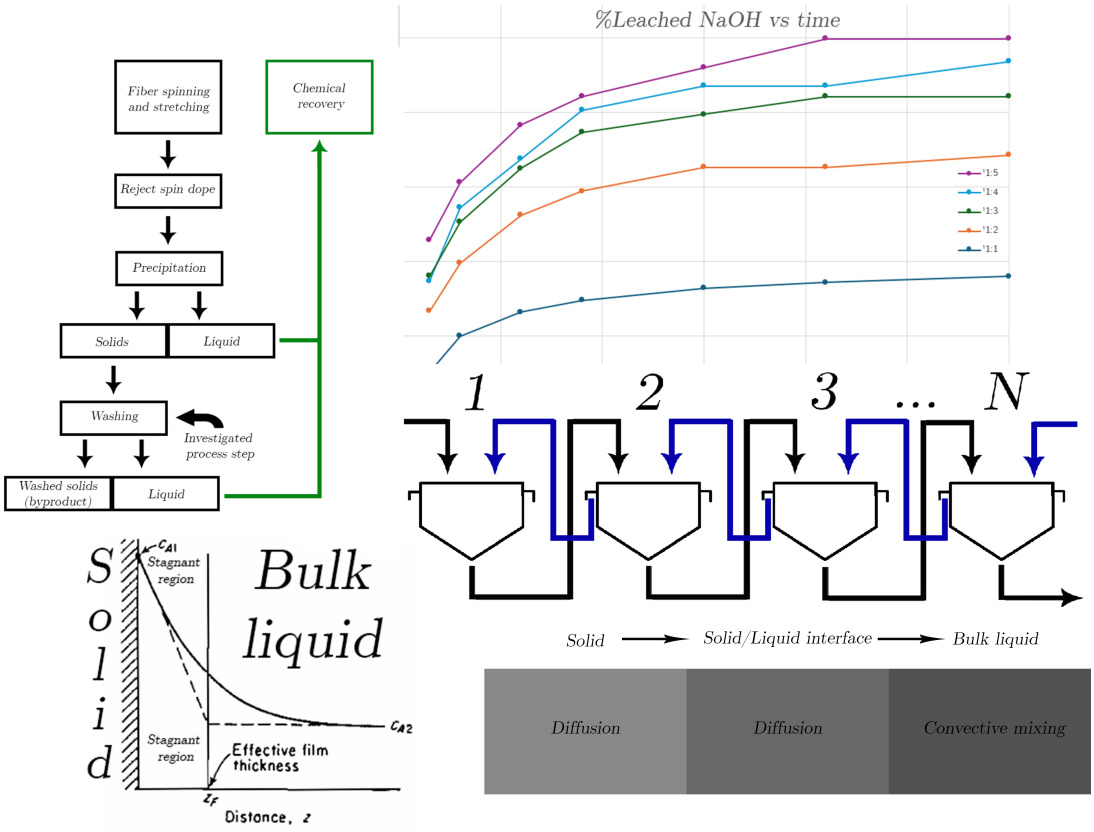

Optimization of Washing Strategies for Precipitated Cellulose from a Chemical Waste Stream

Bachelor's Thesis in Chemical Engineering

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Department of Chemistry and Chemical Engineering

CHALMERS UNIVERSITY OF TECHNOLOGY

Gothenburg, Sweden 2026

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COVER PHOTO: A mix of figures from the thesis.

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Abstract

TreeToTextile (TTT) is developing a new process for creating cellulose based fiber, using less harmful chemicals than the alternatives. The process generates reject spin dope, which currently becomes waste, resulting in chemical losses and disposal costs. To recover chemicals from the reject spin dope, the cellulose in the rejected dope is precipitated and filtered. However, the resulting cellulose precipitate retains a significant amount of liquid containing NaOH and heavy metals. This thesis investigates how washing this precipitated cellulose with water can recover chemicals.

Single stage batch washing experiments were conducted for solid to liquid S/L ratios 1:5, 1:4, 1:3, 1:2, and 1:1 and the washing performance was evaluated using wash curves and CSTR multi stage modeling. The results show that lower S/L ratios extract more solute but require more wash liquid and therefore more energy for subsequent evaporation. The apparent mass transfer coefficient was calculated for all S/L ratios and showed only minor variation, suggesting similar mass-transfer mechanisms for different S/L ratios. A four stage washing strategy using S/L ratio 1:2 is recommended, achieving an estimated 99.11% solute extraction.

Key words: Washing, Cellulose, Precipitate, Leaching, Fiber production, Chemical recovery, Mass transfer, Diffusion

Acknowledgement

I would like to express my sincere gratitude to TreeToTextile for giving me the opportunity to carry out this thesis at their demo plant in Nymölla. Being able to work so closely with an industrial process under development has been both inspiring and highly educational, and I am grateful for the trust and support shown throughout the project.

I especially want to thank my supervisors, Zaheer Mansoor and Nabin Karna, for their guidance, encouragement, and valuable discussions during the entire thesis period. I am also grateful for the insights and practical knowledge shared by the staff at TreeToTextile, whose contributions helped me understand the process beyond my thesis.

Finally, I would like to thank my teachers, classmates, and friends at Chalmers, whose support has made these three years both meaningful and memorable.

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Introduction

Modern chemical processes must be designed with both sustainability and profitability in mind. Reducing waste, lowering emissions and using resources in an effective manner are essential to meet sustainability goals, while efficient operation is necessary in terms of economics.

The practice of recovering and reusing chemicals in an industrial process is one of many ways companies can save money and lower emissions.

This thesis was conducted at TreeToTextile's(TTT) demo plant in Nymölla, Sweden. TTT is currently developing a new kind of process to produce fibers from wood-based cellulose pulp. The process is under development, meaning there are engineering challenges waiting to be solved. This study is a part of TTT's ongoing work in the chemical recovery sector. Specifically, chemical recovery from precipitated cellulose from a waste stream.

1.1 Background

The starting material for TTT's fiber is dissolving paper pulp, which consists of mainly cellulose. The process involves treating and dissolving the pulp, preparing a spinning solution, extruding and spinning the fiber, washing and cutting.

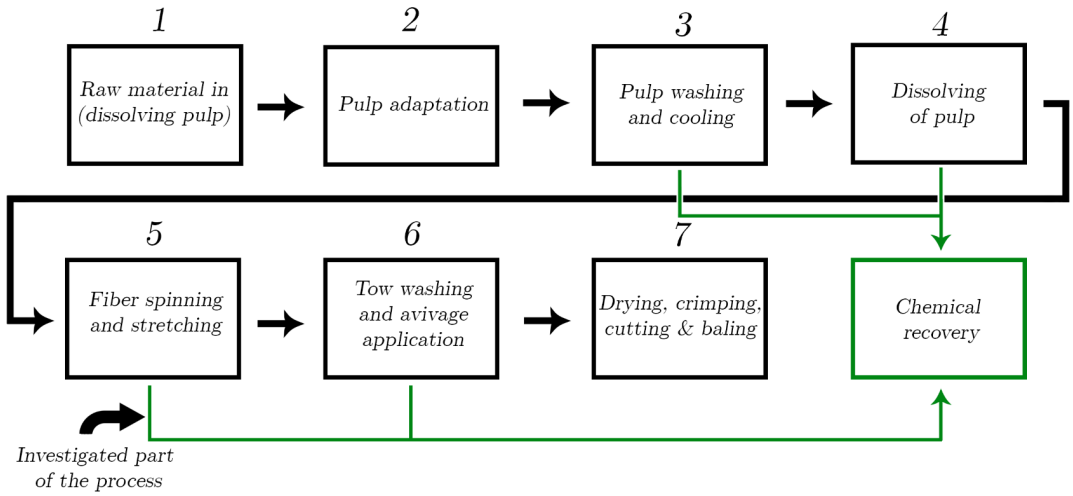

Figure 1.1: An overview of TTT's process. Adapted from [1]

The biggest difference from other man made cellulose fibers is the use of less harmful chemicals. In TTT's process, mainly NaOH and H_2SO_4 are used. By relying on less harmful chemicals and minimizing water use, the process aims to deliver a fiber that is more sustainable than existing alternatives.

Figure 1.1 provides an overview of TTT's process. To describe it simply: Step 1 is the influx of raw material. Step 2 and 3 prepares the pulp for step 4, where the cellulose is dissolved. The fiber is created in step 5, and further processed in step 6 and 7. The core of the process is step 4 and 5, while the surrounding steps are built around them.

1.1.1 Spin dope

Solution spinning is a method used to produce fibers by the means of dissolving and subsequently precipitating the polymer under controlled conditions. In this method, the spinning solution contains a polymer, which has been dissolved using a suitable solvent. The polymer is then extruded through a spinneret, which usually have multiple holes, to form fibers."[2] In figure 1.1, this process is part of step 5.

The polymer solution is often referred to as "spin dope" and can contain various chemicals depending on the specific fiber process. In TTT's process, cellulose is the dissolved polymer.

1.1.1.1 Reject spin dope and its precipitate

The properties of the spin dope (chemical composition, temperature, viscosity, etc) are important for the spinning process to work. If certain properties are incorrect, the dope might have to be rejected and thus discarded. Reject spin dope at TTT is currently sent to waste management, which results in lost chemicals and costly waste disposal. It is therefore necessary to develop a process which recovers valuable chemicals and brings down costs. Spin dope and reject spin dope are part of step 5 in figure 1.1.

To recover chemicals from the reject spin dope, the cellulose must first be removed from the solution. This is achieved by precipitating the cellulose and separating the solids by filtration. The resulting filtrate can then be sent to chemical recovery, while the cellulose precipitate remains.

In an ideal case, the precipitate would consist solely of cellulose, meaning all process chemicals could be sent to chemical recovery via the filtrate. In practice, however, the precipitated cellulose forms a highly hydrated, gel-like structure that retains a significant amount of liquid. This retained water contains the same dissolved chemicals as the filtrate, most notably NaOH and heavy metals, which therefore remain associated with the precipitate.

In an effort to recover the chemicals retained in the precipitate, this thesis investigates how washing the cellulose precipitate with water affects the removal of dis-

solved species, focusing on NaOH.

Overview of the future washing process:

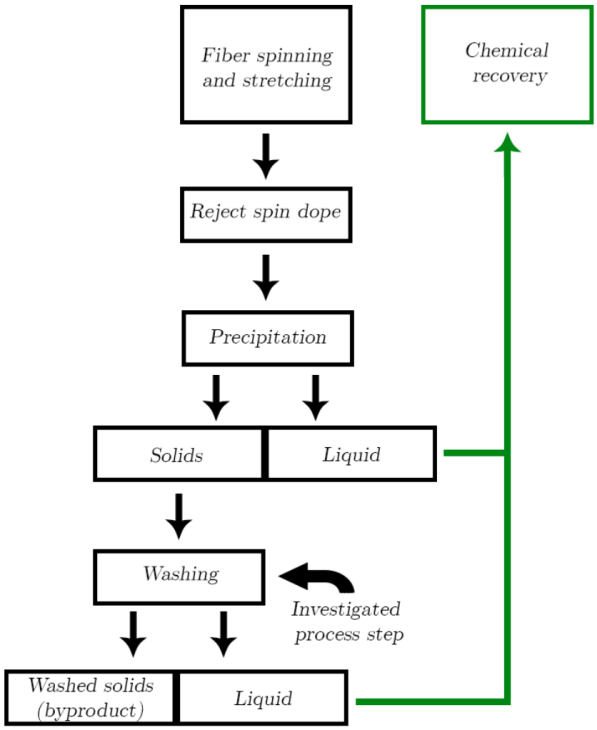

Figure 1.2: An overview of the investigated process.

1.1.2 Previous research

Internal studies conducted at the R&D Center have identified the optimal precipitant and precipitation parameters. However, solid–liquid separation and subsequent washing steps have been found to significantly influence chemical carryover.

As the process is under development, many areas and parameters are likely still not fully understood and might change as the process matures.

1.2 Problem statement

Determine the optimal washing strategy and solid–liquid (S/L) ratio for precipitated cellulose such that:

- Residual chemicals in the solids are minimized.
- Dewatering efficiency is maximized to reduce volume and handling costs.
- Chemical recovery is improved to enhance process sustainability.
- The total washing water added does not disproportionately increase downstream OpEX (via evaporation load).

1.3 Objectives

The objective of this thesis has been to develop and validate a washing strategy for precipitated cellulose that optimizes chemical removal, minimizes precipitate volume, and limits downstream operational costs.

1.3.1 Sub-objectives

- Perform singe stage leaching experiment for S/L ratios (1:1, 1:2, 1:3, 1:4, 1:5)
- Quantify residual chemicals in the precipitate after each experiment.
- Model and evaluate the impact of multiple washing stages in series. Quantify extraction at each stage.
- Determine the impact of S/L ratios on precipitate volume change and liquid retention.

1.4 Goals

The goal of the work has been to investigate and identify a S/L ratio and washing strategy which satisfy chemical recovery and minimize down stream costs.

2
Theory

Mixing TTT's cellulose precipitate with water results in a slurry. In this slurry system, NaOH migrates from the solids to the water. In order to promote mass transfer, the slurry can be agitated. This chapter will therefore investigate mass transfer, washing theory and modeling with focus on an agitated slurry system.

2.1 Mass Transfer in Agitated Slurries

Convection and diffusion are the fundamental mass transfer mechanisms when forming and agitating a slurry for the purpose of performing washing on the solid phase. Diffusion is responsible for chemicals migrating out of the solid phase, into the surrounding liquid. Convection is responsible for transporting chemicals away from the solids, and creating a uniform concentration in the bulk liquid.

2.1.1 Convection

Convective mass transfer concerns the transport of dissolved compounds caused by the bulk fluid motion. Convection can looks different, depending on the system, but in an agitated slurry the dominant kind is forced turbulence. Agitation generates turbulent flow, which fundamentally consists of swirling "eddies" that interact, breaks up and reforms, producing rapid mixing in the system.[3]

At the macroscopic scale, large eddies travel significant distances and reduce large-scale concentration differences. Smaller eddies smooth out local concentration differences, while also creating many interfaces for diffusion.

2.1.2 Diffusion

Molecular diffusion concerns the movement of individual molecules through a substance, as a consequence of the thermal energy of the molecule. Simplified kinetics imagines a molecule traveling in a straight line (free path) until it collides with another particle which results in a change of it's velocity and direction. When a molecule diffuses the net distance traveled in a given direction is greater than zero. [3]

The driving force behind diffusion is a difference in concentration. Molecules within a substance of uniform concentration travel back and forth, while the net distance

traveled remains zero. In areas with a concentration gradient (difference in concentration), molecules moving towards a low concentration area have a longer free path than molecules moving towards a high concentration area. This leads to molecular diffusion from high to low concentration.

Diffusion is also present in the bulk liquid. Agitation creates eddies, which evens out the macroscopic concentration differences. However, these eddies do not result in a uniform concentration on microscopic scales. They do however, create and renew interfaces for diffusion to happen. This is called eddy, or turbulent, diffusion.

2.1.3 Film Theory

Many theories have been developed to describe the interfacial mass transfer mechanism (mass transfer between different inferfaces). One of the oldest and most intuitive theories is "film theory" and according to Treybal, the central idea is that a thin, stagnant "film" of liquid exists at the solid-liquid interface. Inside this film, fluid motion is negligible, meaning that molecular diffusion is the only governing mass transport mechanism (See figure 2.1). Beyond this film, bulk liquid is well mixed.[3]

The theory assumes:

- A stagnant film of constant thickness next to the solid
- No convection inside the film
- Diffusion across the film
- A well mixed bulk liquid

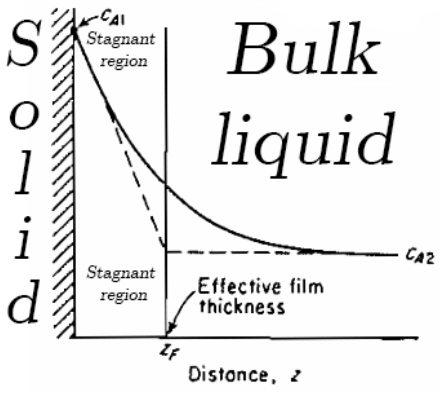

Figure 2.1: A figure depicting film theory. Adapted from [3].

2.1.4 Mass transfer framework

Mass transfer in an agitated slurry for washing the solid phase is assumed to occur in three main steps:

- Internal diffusion, where compounds are transported from inside the solid to its surface.
- Interfacial diffusion at the solid-liquid interface, where compounds are transported from the solid's surface to the surrounding bulk liquid.

- Convective mixing, where compounds are transported away from the solid, mixing with the rest of the bulk liquid.

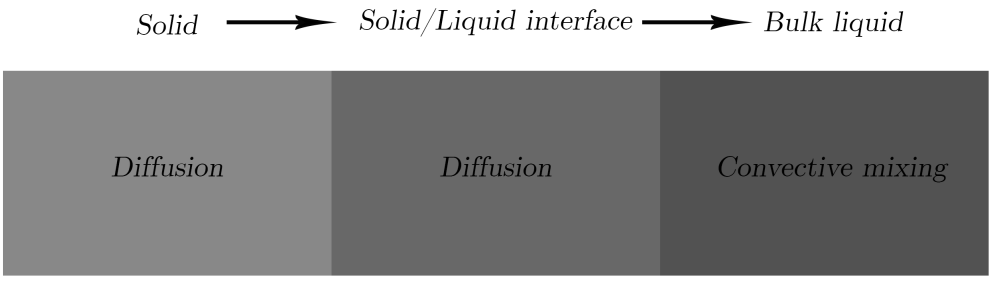

Figure 2.2: Mass transfer framework for the agitated slurry during washing of the solid phase.

Inside the solid phase, the transport mechanism is less well defined. The solids contain a high fraction of water, allowing dissolved compounds to diffuse out via the internal water phase. The structure is assumed to be porous as well, meaning that dissolved compounds also diffuse through water filled pores.

At the particle surface, there is an interfacial region between the solid and the surrounding liquid. Transport across this region is controlled solely by molecular diffusion, as described in Film Theory.

Lastly, convective mixing transports dissolved compounds away from the solid through turbulent eddies, creating a nearly uniform concentration in the bulk liquid. This ensures that the driving force for mass transfer is maintained at the solid–liquid interface.

2.1.5 Factors influencing mass transfer

Mass transfer during washing is influenced by several factors:

Residence time: Long enough contact time allows for the system to reach equilibrium, while too short residence time might limit extraction. Since the washing kinetics follows a first order approach to equilibrium, the effect of residence time is strongest at early times and diminishes as the driving force decreases.

Specific surface area: Smaller solids leads to higher interfacial area available and shorter diffusion paths. Smaller particles are beneficial for diffusion, but might create filtration challenges.

Solid phase structure: The internal structure of the solids influences how easily solute can migrate out. For example, a highly porous material could facilitate easier diffusion paths out of the solids compared to denser materials.

Solubility: The equilibrium concentration sets the upper limit for how much solute can be transferred into the wash liquid. Systems with low solubility reach equilibrium quickly, reducing the effectiveness of additional washing time or dilution.

Agitation: Higher mixing power generates stronger eddies and more frequent renewal of the liquid film surrounding the particles, which increases the convective mass-transfer. Insufficient agitation leads to thicker boundary layers and slower mass transfer.

2.2 Leaching

Solid-liquid extraction, commonly referred to as leaching, involves mass transfer between a solid and a liquid phase that are in contact. The solid phase contains substances that dissolve into the surrounding liquid. In an agitated slurry system, the leaching rate can be influenced by several mechanisms, including intraparticle diffusion, film diffusion, and bulk mixing.

2.2.1 Principles of Leaching

The fundamental mechanism behind leaching is diffusion, which in turn is driven by a concentration gradient. In order to run a successful leaching operation, this concentration gradient is of vital importance. Convection/advection also plays an important role by transporting away and renewing the liquid near the particle surface, and thereby maintaining the concentration gradient.

If no advective mechanism is present, such as in the slurry system, extraction of solute continues only as long as there is a difference in concentration between the solid surface and the surrounding liquid. As more solute is transferred to the bulk liquid, this difference becomes smaller, weakening the driving force behind leaching. Eventually, the system approaches the solubility limit, and a practical equilibrium is reached where no further mass transfer occurs. This practical equilibrium defines the maximum achievable extraction, under the given operating conditions. Replacing or adding solvent lowers the solute concentration in the liquid and restores the concentration gradient.

The physical state of the solids plays a major role as the solute must be accessible to the solvent. It might be beneficial and sometimes mandatory to prepare the solids before a leaching operation. Smaller particles results in faster leaching due to a shorter diffusion paths, but might create filtration challenges instead.

Temperature also affects the leaching process. Higher temperatures increase the thermal energy which promotes diffusivity and decrease the viscosity, which enhances both internal diffusion and transport across the solid–liquid interface.

2.2.2 Rate-Limiting Factors

Several mechanisms can limit the rate of leaching in an agitated slurry:

- Bulk mixing, where insufficient agitation slows the renewal of liquid around the particles.
- Film diffusion, where transport across the stagnant liquid layer becomes limiting.
- Intraparticle diffusion, where solutes move slowly through the the solid.
- Equilibrium limitations, where the bulk liquid approaches the solubility limit

2.3 Washing

Washing refers to the removal of soluble contaminants from wet solids. In this thesis, the terms washing and leaching are used interchangeably, since leaching solute from the solids is the main washing mechanism.

2.3.1 Fundamentals of Washing

Washing can fundamentally be done in two ways, either via dilution or displacement. Displacement aims to completely replace the liquid associated with the solids, while dilution lowers the concentration of soluble contaminants in the retained liquid. In classical pulp washing, displacement washing is preferably used as it requires less wash liquid. However, the precipitate contains mostly of water which makes it different from washing pulp fibers.

Displacement washing equipment generally requires the solids to form some kind of permeable filter mat, which allows the wash liquor to flow through. This is not the case for the precipitated cellulose and if pressed, which is common for certain displacement equipment, it would result in a gel.[4] Therefore, displacement washing is not applicable to the specific precipitate.

Furthermore, the main objective of the washing procedure is to leach solute out of the solids, which is done by letting the internal water phase reach equilibrium with the wash liquid. In other words, the solids are washed by adding water, which dilutes the internal water phase via diffusion.

A key parameter when washing is liquid retention, which is defined as the amount of wash liquid associated with the dry solids. Retention limits washing efficiency because a fraction of the contaminated liquid cannot be removed by simple renewal of the bulk liquid. In this case, the retained liquid after washing is the sum of the internal water phase and the adsorbed surface liquid.

2.3.2 Wash ratios and Solid-to-Liquid Ratio S/L

Wash ratio describes the amount of fresh wash liquid added relative to the mass of solids. The solid to liquid ratio S/L describes the local mixture of solids and

liquid. In this thesis, a single washing stage was used in the experiment and the bulk liquid was filtered away, meaning that the wash ratio and S/L ratio are the same. In multi-stage washing, the two ratios differ.

Higher wash ratios generally improve washing performance because of reduced concentration of solutes in the bulk liquid. However, more wash-liquid increase water consumption and downstream processing efforts. As in most industrial operations, washing performance is therefore limited by economic and efficiency considerations.

2.3.3 Wash Curves

Wash curves describe how the solute concentration changes over time or across washing stages. In this thesis, washing curves are constructed from $NaOH/Na_2CO_3$ concentration measurements and are used to evaluate:

- Leaching behavior
- The influence of different S/L ratios

For this thesis, two different wash curves are constructed by plotting:

- Mass% of solute in the bulk liqud vs time
- %Solute leached to bulk liquid vs time.

2.3.4 Washing efficiency

Washing efficiency describes how effectively a solute is removed relative to the theoretical maximum. It is determined by several factors:

- Liquid retention
- Agitation
- Wash ratio and S/L ratio
- Mass transfer
- Number of stages
- Washing strategies

2.3.5 Washing strategies

There are a few different washing strategies to consider for the specific precipitate, although they are similar. Solids are mixed with wash liquid into a slurry which is, in some way, agitated. In all washing strategies described, the bulk liquid and solids must be separated between stages. This is essential for renewing the wash liquid and enabling further extraction. However, the practical methods for bulk-liquid removal and solids transfer were not investigated in this thesis, as the focus was placed on washing performance and stage modeling rather than mechanical handling.

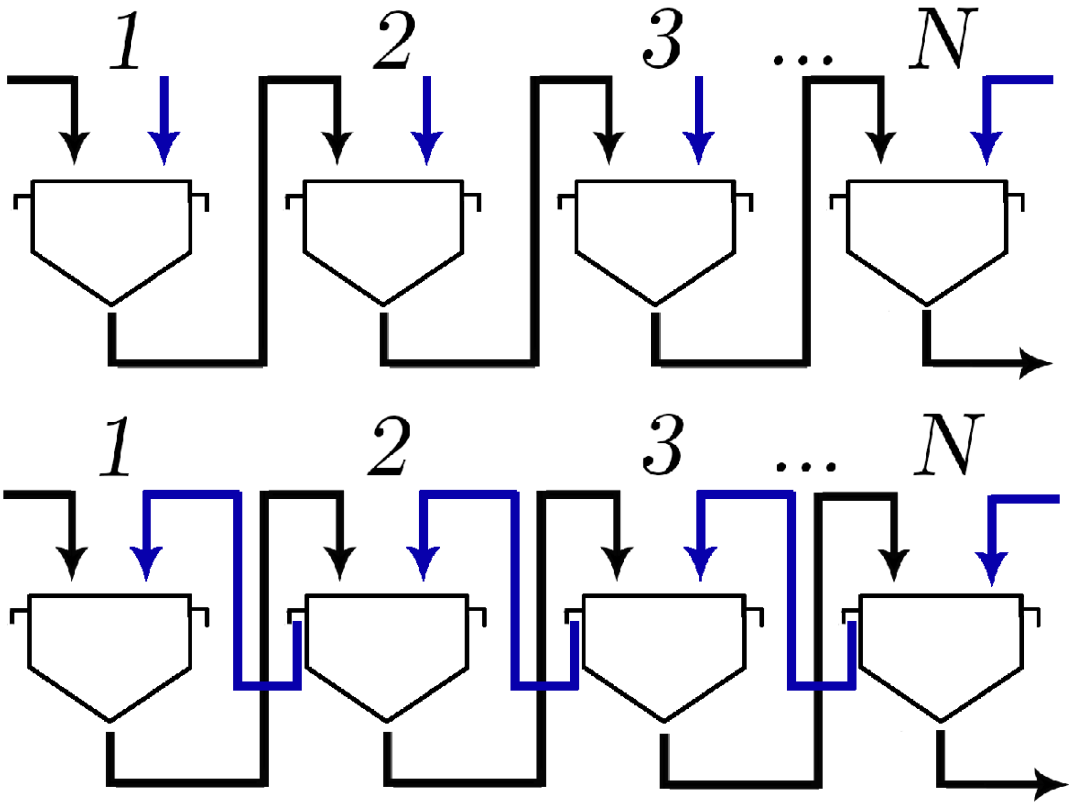

Figure 2.3: A schematic figure depicting batch and counter current washing. Black arrows indicate the movement of the solid phase, while blue arrows represent the flow of wash liquid. The top row illustrates N batch washing stages in series, where each step is supplied with fresh wash liquid. The bottom row illustrates N counter-current stages, where wash liquid is reused between the steps and flows opposite to the solids.

The three main washing strategies are:

- Mixed batch washing, which works by mixing solids and wash liquid and agitating the slurry. Once the system reaches equilibrium, the bulk liquid is removed. In a multi stage setup, the procedure is repeated (see figure 2.3). The system works purely through dilution. In this thesis, the washing experiment uses a single stage mixed batch strategy.

- CSTR (continuous stirred tank reactor), which operates via a steady inflow of wash liquid and solids and an outflow of slurry. The principle is similar to mixed batch washing, but a continuous operation. Residence time, agitation and tank volume determines washing performance.

- Counter current washing, which aims to maximize extraction while keeping wash liquid usage minimal. It is a multi stage strategy where fresh solids are washed by the most contaminated wash liquid, while the cleanest solids are washed with freshest wash liquid (see figure 2.3). This maintains a concentration gradient across all stages, which greatly improves washing efficiency.

2.3.6 Modeling of the washing process

In order to investigate differences between S/L ratios and the effect of multiple wash stages, modeling was performed by combining the apparent mass-transfer coefficient k with a CSTR in series equation. This allowed single stage kinetics to be translated into multi stage washing performance.

2.3.6.1 Mass-transfer coefficient

The rate at which solute is transferred to the bulk liquid assumes to follow integrated first order mass expression (see equation A.1). This approach treats the increase in bulk liquid concentration as a kinetic process governed by an apparent mass-transfer coefficient k.

2.3.6.2 CSTR

The experiments conducted in this thesis were performed using a stirred batch strategy. However, in order to estimate the performance of a multi stage washing system, the apparent mass transfer coefficient will be used in a equation for N CSTR stages in series(Equation A.3).

2.3.7 OpEX(operational expenditures) and CapEX(capital expenditures)

An important perspective when evaluating washing strategies are the operational expenditures, which are the costs associated with running the process on a daily basis, such as energy demands and water consumption. In this thesis, the cost of energy required to heat up and evaporate water will be the basis for OpEX calculation.

Capital expenditures are the costs associated with building the process. In this thesis, the cost of a mixing tank, agitator and pump for each stage will be the basis for CapEX calculation.

3
Laboratory work

In order to optimize washing strategies, the system which is to be optimized firstly needs to be understood. How much solute does the solid phase contain? How does the system behave over time? Does S/L ratios have an effect? To gain information about the system, single stage washing and dry solids experiments were set up.

3.1 Dry solids analysis

A property of the precipitate which has to be determined is the dry solid content. The dry solid content(%DS) is the percent of solids left after completely drying the precipitate which removes the retained liquid. The value is needed for various calculations.

3.1.1 Dry solids experimental procedure

In order to determine the required time to completely dry a sample, a drying test was conducted. Five glass petri dishes were weighted empty and marked. Roughly 5g precipitate was added to each petri dish and the total weight was noted. The dishes were put in to the oven, set and preheated to $105°C$. The removal times were chosen as: 1h, 2h, 3h, 4h, 5h. When the time was up, the sample were removed and let to cool. On a different occasion, 30min and 1.5h was also tested in the same way.

The experiment was conducted for two different precipitates produced at different times, bag 1 and bag 2. The precipitate in bag 1 was older and comparing the structure, precipitate from bag 2 composed of longer fibrous structures in contrast to grainy rise morphology in bag 1.

Values from equation A.8 could then be plotted against removal time.

3.2 Preliminary washing study

In order to understand the mass transfer kinetics, an experiment was set up to investigate how solute leaches out over time. The idea was to mix a slurry of solids and deionized water and study how the concentration of chemicals in the bulk liquid changes over time. The data could then be used to create wash curves, estimate the initial solute concentration and more.

A S/L ratio was chosen and the mass of solids and wash liquid was calculated. The solid phase and wash water was mixed to a slurry and agitated. After set time intervals, samples of the wash liquor were withdrawn and titrated for %NaOH/%Na_2CO_3.

3.2.1 Laboratory procedure

The following procedure was conducted for two different precipitates at five S/L ratios each (See table B.1 and B.2).

Cellulose precipitate and deionized water were weighed in separate plastic containers. The container with precipitate was placed on a hot plate, and a magnetic stir bar was added. The timer was started, the water was poured into the precipitate container, and stirring was turned on. The heating element was not used. The agitation speeds were: 1:5 – 650 RPM, 1:4 – 750 RPM, 1:3 – 1000 RPM, while for 1:2 and 1:1 the slurry was stirred manually using a pipette.

When each interval was reached (30s, 1min, 2min, 3min, 5min, 7min and 10min), liquid was pipetted from the water swirl for S/L ratios 1:5, 1:4 and 1:3, and for S/L ratios 1:2 and 1:1 the agitation was paused (2-4seconds), letting the solids sediment, followed by pipetting. 15ml of samples were withdrawn using a plastic 3ml pipette and put into test tubes.

The liquid content of the test tubes(solid sedimentation left out) were transferred to and weighed in measuring cups. Deionized water was added to the cups to reach a total volume of 60ml before titrating. The cups were placed in the titrator carousel, and the NaOH/Na_2CO_3 program was initialized. Results were given as mass% of NaOH and Na_2CO_3 of the sample.

3.2.2 Calculations

Calculations done for the preliminary washing study.

3.2.2.1 %Solute originally in precipitate

The last sample at 10 minutes of washing was used to estimate the concentration of solute in the precipitate. The wash liquid and the internal water phase of the precipitate were assumed to be in equilibri by then. Therefore, the total amount of solute in the system was approximated by:

$$(\text{internal water phase} + \text{wash liquid}) \times \text{concenration}_{\text{titration, 10min}}$$

This value was divided by the initial mass of precipitate to get the initial %solute orginally in (wet) precipitate. Which results in:

$$\%NaOH_{solids} = \frac{(m_{\text{wash liquid}} + m_{solids} \times (1 - \frac{\%DS}{100})) \times \frac{C_{\text{titration, 10m}}}{100}}{m_{\text{solids}}} \times 100 \quad \text{(A.11)}$$

3.2.2.2 %Solute leached

For each sample time the %leached solute can be described by the amount of solute in the wash liquid divided by the amount originally in the precipitate.

$$\%\text{leached NaOH} = \frac{C_{\text{titration}} \times m_{\text{wash liquid}}}{\frac{\%NaOH_{solids}}{100} \times m_{\text{solids}}} \times 100 \tag{A.12}$$

3.3 Bag filter washing

A duplex bag filtration system already at TTT's disposal was used to wash a larger quantity of solid phase. The filter is normally not used for this kind of process. The goal was to investigate if washing was possible and how such a system would behave. The duplex bag filter provides coarse filtration, meaning the solids are larger than the bag's pore size and are therefore retained inside the bag. It does not form a filter cake or dewater the solids, it simply holds the solids in place while the liquid drains through the bag when water is removed.

3.3.1 Procedure

The data that was collected in this experiment:

- Mass of precipitate
- Volume before/after washing
- Mass of washing liquid in the end
- Flow rate
- Bulk liquid NaOH/Na_2CO_3 concentration over time
- %DS

Approximately 7,37kg of cellulose precipitate was added to a 200 micron bag filter(height and diameter measured) and placed in the filter housing. Water was pumped upwards from the bottom and the liquid was recirculated. At 30s, 1min, 2min, 3min, 5min 7min and 10min, a test tube was filled by intercepting the stream of returning wash liquid. After 10min, a bucket was filled with water while a timer was running in order to calculate flow rate. The bag filter was removed(height and diameter measured) and the wash liquid weigh before discarding.

3.4 Washing study

In order to gather better data for the different S/L ratios, a new experiment was planned and executed. The principle was the same as in the preliminary washing study, but scaled up and modified in several ways. A larger container and a mechanical stirrer were used, which should lead to more even agitation and reduce the impact of measuring and sampling errors. An improved sampling system with larger sample sizes was also implemented, where each sample would be extracted by submerging a test tube in the slurry. The collected sample would then be filtered immediately to remove the largest particles. This sampling approach was intended to produce samples that represent the slurry composition, thereby keeping the S/L

ratio in the slurry constant.

For the experiment, the following data were gathered:

- Water temperature before washing.
- Water weighed before washing.
- Solid phase weighed before and after washing.
- Solid phase volume before and after washing.
- pH of the slurry at each sample time.
- %NaOH and $\%Na_2CO_3$ of the bulk liquid at each sample time.

3.4.1 Laboratory Procedure

1. Solids were weighed in a measuring cup (weight and volume noted) and transferred to the washing container.
2. Deionized water was added to a different measuring cup (volume/mass and temperature noted).
3. Washing container, mechanical stirrer and pH meter were mounted in place. The pH meter was turned on.
4. In sequence: water was poured from the measuring cup into the washing container, agitation was started (600RPM, stopwatch starts on agitator).
5. For each of the timestamps, a sample was withdrawn and pH was noted prior to each sampling.
   (a) A sample was collected by dipping a test tube (55mL) in the slurry.
   (b) The content of the test tube was poured over a strainer(with a filter cloth) into a plastic cup.
   (c) The plastic cup and used filter cloth were removed and placed to the side.
   (d) The next sample was prepared by placing a new plastic cup with the strainer and a new filter cloth on top.
6. After the last sample was withdrawn, agitation was stopped.
7. The bulk liquid was strained off, and the filtered solids stuck to the fiber cloths were added back. The solids were transferred and weighed in a measuring cup (mass and volume noted). A sample of 10g was saved before discarding the rest (%DS).
8. Each sample was filtered via a 5µm filter paper using pressurized air.
9. The samples were titrated using the program for $NaOH/Na_2CO_3$.

3.4.2 Calculations and modeling

Calculations and modeling done for the preliminary washing study.

3.4.2.1 Alkali equivalence

All Na_2CO_3 present is assumed to be a product from NaOH reacting with CO_2 in the air. To express the total alkalinity as NaOH, the measured Na_2CO_3 is converted to its NaOH equivalent using a correction factor. This factor (0.7548) comes from the stoichiometry and the corresponding molar masses (see equation A.6). This value

is referred to as alkali (eq), meaning total alkalinity expressed as NaOH equivalent.

$$Alkali(eq) = \mathrm{Na_2CO_3} \times 0,7548 + NaOH \tag{A.7}$$

3.4.2.2 %Solute originally in precipiate and %solute leached

As was done in the preliminary washing study, % of solute originally in precipitate was calculated via equation A.11. and %leached over time was calculated via equation A.12.

$$\%NaOH_{solids} = \frac{(m_{washliquid} + m_{solids} \times (1 - \frac{\%DS}{100})) \times \frac{C_{\text{titration, 10m}}}{100}}{m_{\text{solids}}} \times 100 \tag{A.11}$$

$$\%\text{leached NaOH} = \frac{C_{\text{titration}} \times m_{\text{wash liquid}}}{\frac{\%NaOH_{solids}}{100} \times m_{\text{solids}}} \times 100 \tag{A.12}$$

3.4.2.3 Mass transfer coefficient (k)

The apparent mass transfer coefficient, k, was calculated, which is a measure on how fast solute is transported out of the solids. In order to solve the k value, the experimental data gathered in the washing study needs to be fitted to equation A.1. In order to do this, C_0, C_{eq} and C_t for all samples needs to be calculated.

$$C(t) = C_{eq} + (C_0 - C_{eq})e^{-kt} \tag{A.1}$$

All of these concentrations refer to the amount of solute in the solids with regards to the retained water phase. C_0 is the initial concentration in the solids, C_{eq} is the concentration when equilibrium is reached and C_t is the concentration at each sample time, such as:

$$C_t = \frac{\text{Mass of total solute originally in system} - \text{Mass of solute leached to bulk liquid}}{\text{Mass of water retained in solids}}$$

Which looks like:

$$C_t = \frac{m_{solids \times \frac{\%NaOH_{\text{solids}}}{100}} - m_{\text{wash liquid} \times \frac{C_{\text{titration, t}}}{100}}}{m_{solids} \times (1 - \frac{\%DS}{100})} \tag{A.10}$$

Using equation A.10, C_{eq} was calculated using the titration value from 10 minutes, as equilibrium is assumed by then. C_0 was calculated via equation A.9, which is the same as A.10 without any solute leached to bulk liquid.

The next step was to give k a placeholder value and calculate C_t via equation A.1

using C_0 and C_{eq}. By varying k, the modeled C_t values changes, and the next step was to find the "best" k which generates values closest to the experimental C_t values from equation A.10.

In excel, "data solver" was used to vary k via the GRG nonlinear algorithm. The objective was set to minimize the sum of squared errors(SSE) between the calculated experimental C_t values (Equation A.10) and the model prediction (Equation A.1).

3.4.2.4 Water usage, OpEX(Operational expenditures), CapEX(Captal expenditures) and TotEX(Total expenditures)

Water usage is calculated by multiplying the (L/S) ratio with the amount of stages(See equation A.13).

To calculate the energy required to heat up and evaporate the wash liquid, equation A.14 was used.

$$Q_{\text{total/kg precipitate}} = c_p(T_b - T_0) + \Delta H_{\text{vap}} \tag{A.14}$$

The cost for evaporation energy was calculated via equation A.15 which results in OpEX costs for one year.

$$\text{OpEX/yr} = \frac{Q_{/\text{kg water}} \times (L/S) \times 5000 \times 365 \times 0,1831}{3,6 \times 10,93} \tag{A.15}$$

CapEX was calculated by researching and selecting a mixing tank, agitator and pump with public prices. The cost associated with a single stage is the sum of these three multiplied by N stages.

$$\text{CAPEX/stage} = N \times (€_{tank} + €_{agitatior} + €_{pump}) \tag{A.16}$$

TotEX is the sum of OpEX/yr and CapEX.

3.4.2.5 % extracted solute as a function of washing stages

Using the k value previously determined, a residence time τ was calculated using equation A.4. The number of stages N was chosen and equation A.3 was used to calculate C_{end}, which subsequently was used to calculate %NaOH extracted via equation A.5.

$$\%Removed = \frac{C_0 - C_{end}}{C_0} \times 100 \tag{A.5}$$

3.4.2.6 Liquid retention

Liquid retention was calculated by dividing the mass of liquid associated with the solids by the mass of dry solids.

$$R = \frac{m_{solids} \times (1 - \frac{\%DS}{100})}{m_{solids} \times \frac{\%DS}{100}} \tag{A.2}$$

3.4.2.7 Volume change

In order to calculate the change in volume, the difference in volume before and after was divided with the initial volume.

$$\%V_{difference} = \frac{V_{before} - V_{after}}{V_{before}} \times 100 \tag{A.17}$$

4

Results

In this chapter, results from the various experiments will be provided and discussed. The raw data can be found in Appendix 2.

4.1 Dry solids experiment

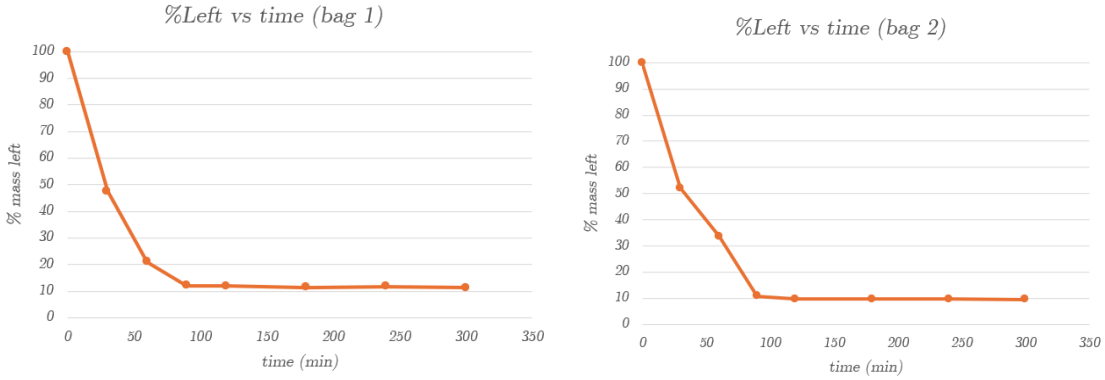

Figure 4.1: % of dry solids as a function of time

From figure 4.1 it can be noted that bag 1 graph shows rapid mass loss until 1h mark graph, where it begins to level out. After the 2h mark, there is no further mass loss. Bag 2 graph shows similar results, but no smooth leveling out as bag 1.

Complete drying was accomplished after 2 hours at 105°C, which resulted in $\%DS_{\text{bag 1}} = 11,89$ and $\%DS_{\text{bag 2}} = 9,88$. This time and temperature were used for all %DS values in the thesis.

The dry-solids experiment was initially performed once for bag 1 and once for bag 2, using removal times 1h, 2h, 3h, 4h, and 5h. Complete drying was already achieved after 2h, so a second round of experiments was conducted to obtain a more detailed drying curve. In this second round, additional measurements were taken at 30min and 1.5h only.

As shown in Figure 4.1, bag 1 displays a smooth, curved drying trend, while bag 2 shows a more irregular pattern. This is likely due to bag 2 being recently produced when the first round of experiment was conducted, meaning moisture was still adsorbed on the particle surfaces. Before the second round, this adsorbed moisture

had accumulated at the bottom of bag 2. In contrast, bag 1 precipitate did not contain noticeable surface or accumulated moisture in either experiment.

4.2 Preliminary washing study

Results from the preliminary washing study. Titration concentration over time, %NaOH originally in the precipitates and %leached NaOH will be shown here.

4.2.1 %NaOH in bulk liquid over time

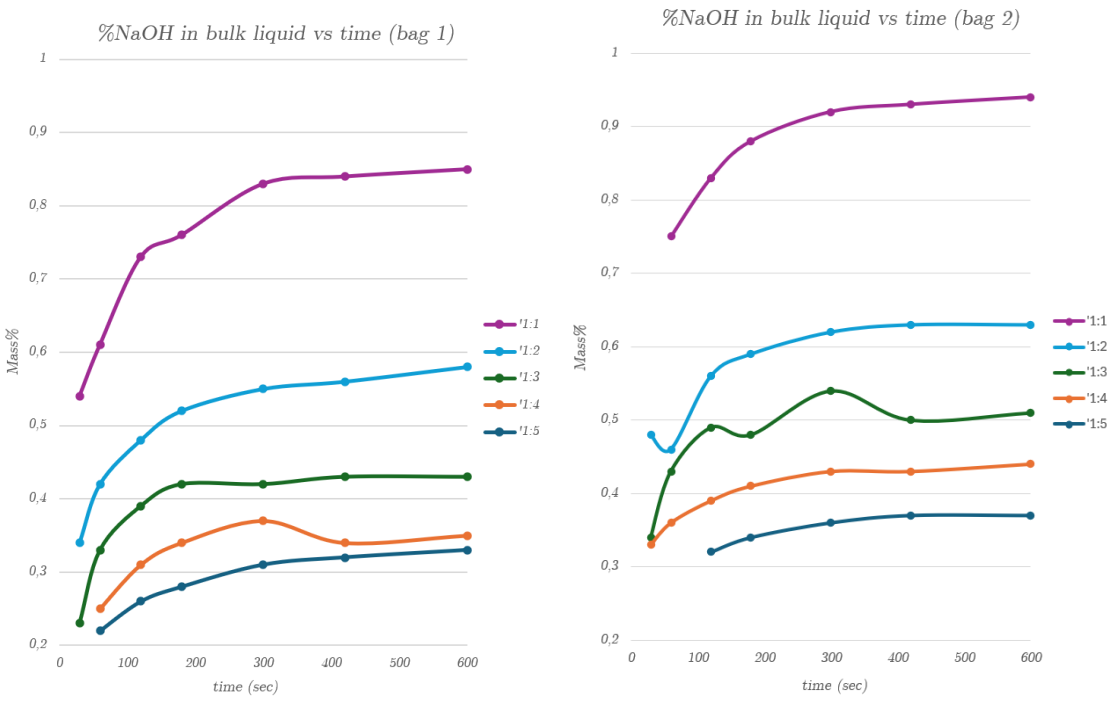

Figure 4.2: %NaOH in bulk liquid over over time from samples with with different S/L ratios

Figure 4.2 shows that concentrations of NaOH in the bulk liquid generally increased rapidly in the beginning and leveled out towards the end. Higher S/L ratios resulted in higher concentrations in the bulk liquid, which is reasonable as there's more solute in relation to the amount of wash liquid.

Many values don't follow a clear trend, which results in fluctuation over time. It is not reasonable that bulk liquid concentration goes down after time, which points to unreliable data (see appendix B.3). The most reasonable explanation is insufficient sample sizes for the sampled concentration range. The sampling was also uneven, as pipetting took anywhere from 10-30seconds, which could further contribute to the irregularity. Titration data for Na_2CO_3 is not provided as most samples gave no result.

4.2.2 %NaOH originally in the precipitate

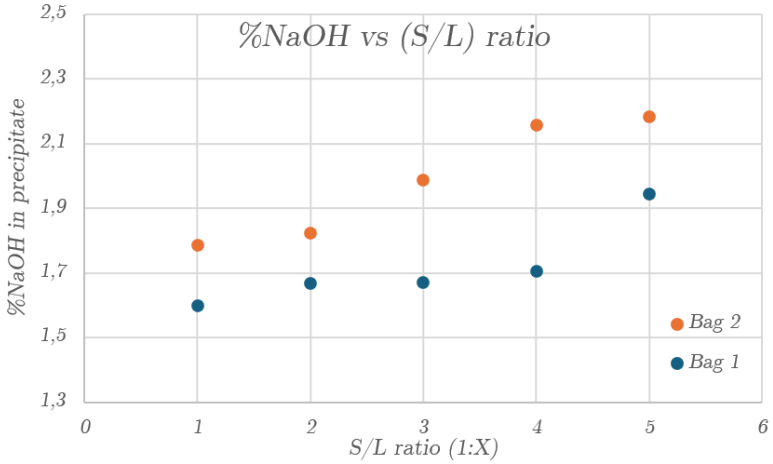

Figure 4.3: %NaOH originally in precipitate as a function of S/L ratio

Figure 4.3 shows that calculated %NaOH originally in precipitate is higher for lower S/L ratios and subsequently that bag 2 data resulted in higher values compared to bag 1. The same solids should reasonably result the same calculated %NaOH originally in precipitate, the large difference might be because of the unreliable data.

The difference between the solids from bag 1 and bag 2 is that they were produced on different dates. Visually, there were slight differences between the bags. Solids from bag 2 were longer, while bag 1 contained a larger fraction of fine particles. These are differences which originate from the precipitation step.

The different results could stem from variation in the waste spin dope composition which the solids were produced from. Another reason could be that more NaOH has reacted with CO_2 in the air, resulting in more Na_2CO_3.

4.2.3 %Leached NaOH

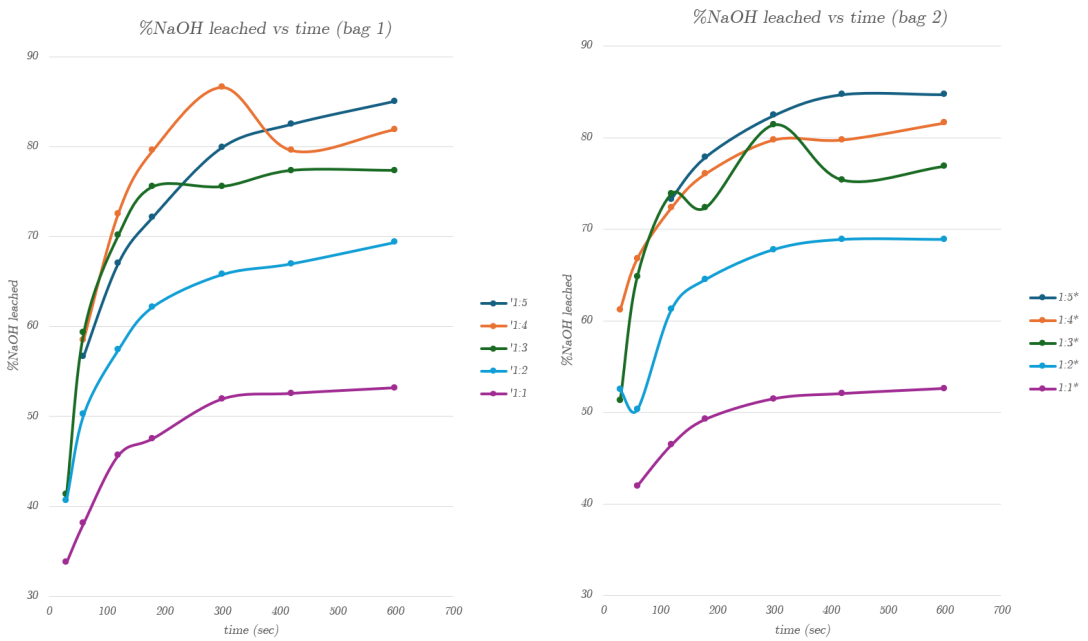

Figure 4.4: %total NaOH leached as a function of time for different S/L ratios

Figure 4.4 shows that lower S/L ratios result in a larger fraction extracted at 10 min in a single stage because the higher amount of bulk liquid relative to solute maintains a larger concentration gradient for longer, providing a stronger driving force for mass transfer.

Fluctuation can be seen in the data, which is expected since the calculations rely on titration values that were unreliable due to small sample sizes and uneven sampling (Appendix B.3).

4.3 Bag filter washing

The experimental setup worked and was running without any issues. However, none of the samples produces titration results, likely due to the concentrations in the bulk liquid being below the method's detection limit. Calculated S/L ratio was 1:9,4, which would explain the low concentrations. The volume was reduced from roughly 9,3L to 8,2L, which is a 12% loss. Flow rate was calculated at 0,232kg/s and 69,5kg of bulk liquid was weighed at the end of the experiment.

4.4 Washing study

In this chapter, the results from the washing study will be provided and discussed.

4.4.1 %Solute in bulk liquid over time

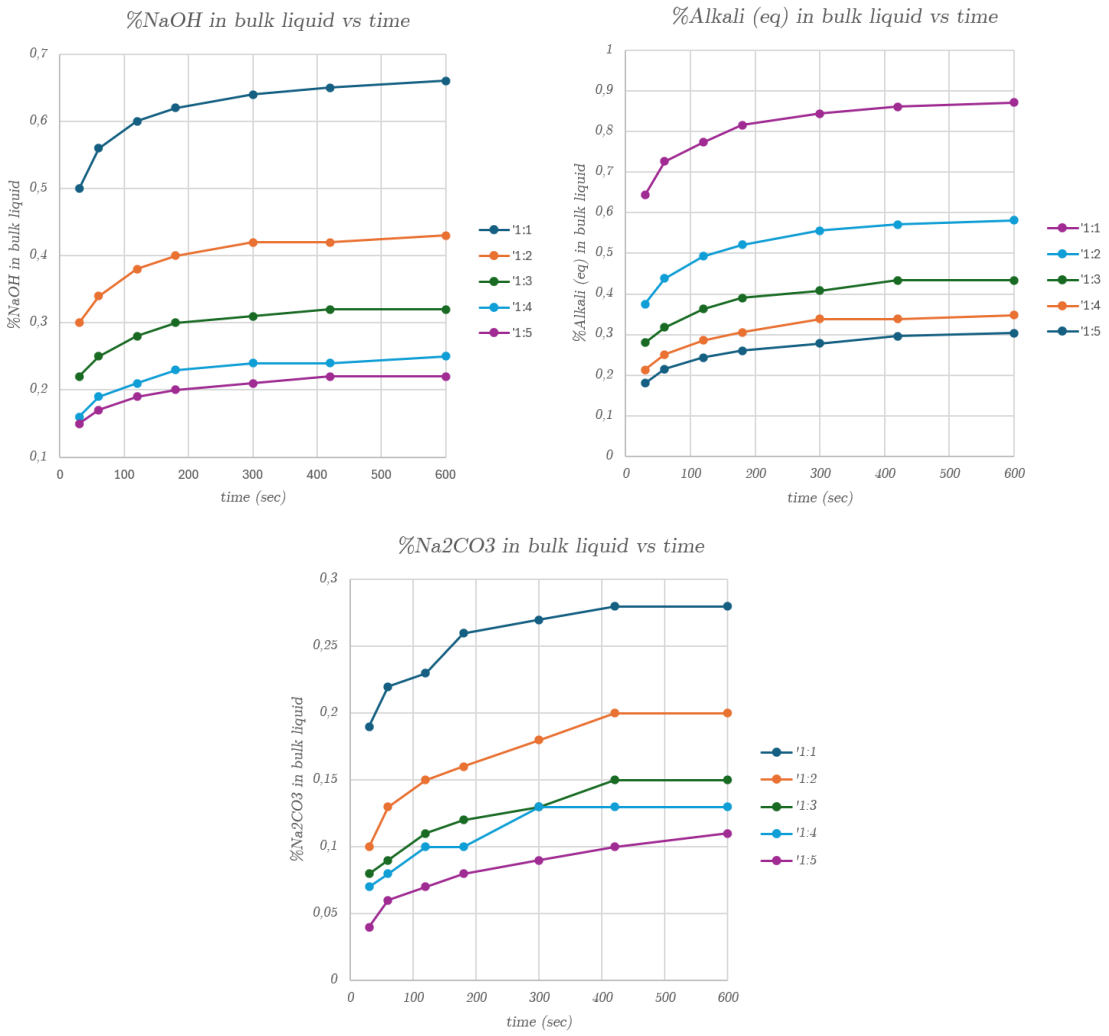

Figure 4.5: Concentration of NaOH, alkali (eq) and Na_2CO_3 in bulk liquid as a function of time for different S/L ratios

Figure 4.5 shows three graphs of solute concentration in the bulk liquid over time: NaOH, alkali (eq), and Na_2CO_3. All graphs show an initial rapid increase in concentration that gradually levels off as equilibrium is approached. The NaOH data follow a clear and smooth trend, whereas the Na_2CO_3 curve appears more irregular. This is likely due to the limited sensitivity of the titration method at low concentrations and because the increase in Na_2CO_3 per sampling interval is very small.

4.4.2 %Leached solute over time

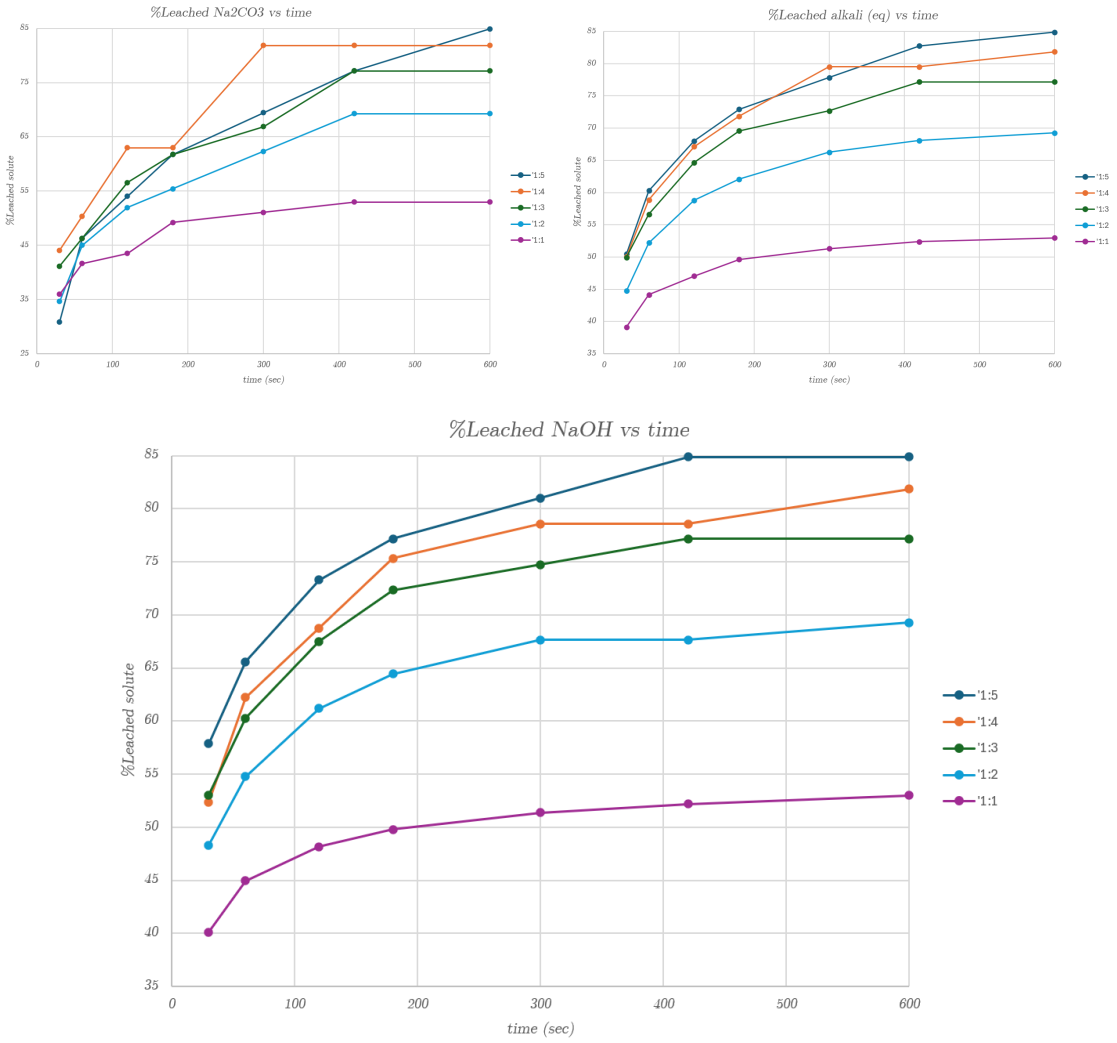

Figure 4.6: %total NaOH, alkali (eq) and Na_2CO_3 leached as a function of time for different S/L ratios

Figure 4.6 shows how much of the total solute is transferred from the solids to the bulk liquid over time. NaOH follows a smooth trend, compared to Na_2CO_3 which is due to the irregular concentration data. Lower S/L ratios show a better total extraction for a single stage, which is reasonable as the driving force is larger.

The Na_2CO_3 graph is irregular, even more so than the concentration data. The only clear trend is rapid mass transfer in the beginning, which evens out towards the end.

4.4.3 %Solute originally in the precipitate

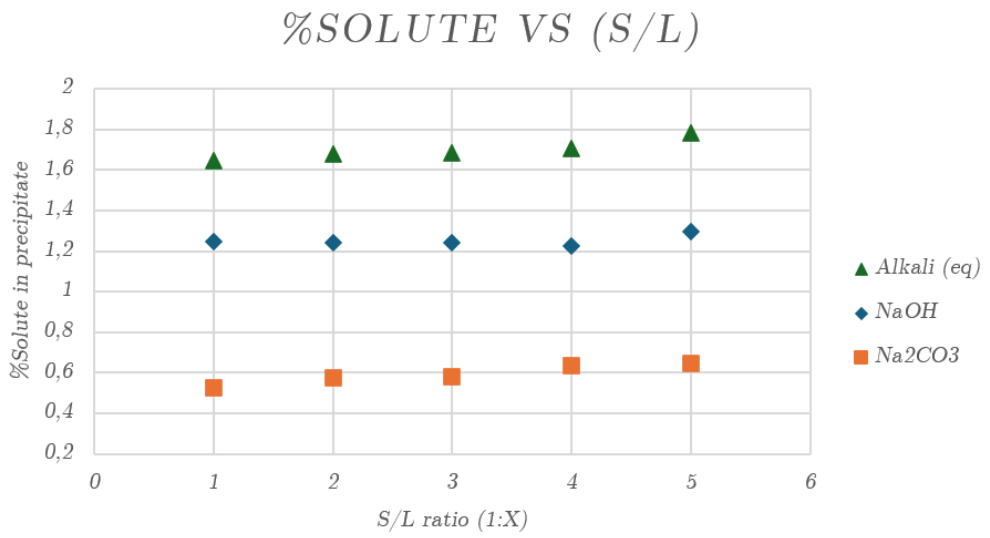

Figure 4.7: Estimated %NaOH, $\%Na_2CO_3$ and alkali (eq) the Solids as a function of S/L ratio

An increased percentage of solute originally in the precipitate is observed at lower S/L ratios from figure 4.7. The differences between S/L ratios are smaller than in the preliminary study, but the trend remains. The underlying cause is unclear. One possibility is that small calculation errors become amplified at lower S/L ratios, making the results appear higher. Another possibility is that lower S/L ratios approach apparent equilibrium faster, meaning that more time would be required to reach equilibrium for higher S/L ratios.

This difference in %solute originally in the precipitate may influence the subsequent calculations, which should be kept in mind. A possible solution to this problem would be to determine a more reliable initial concentration through longer equilibrium experiments and base all calculations on that value.

4.4.4 Apparent mass transfer coefficient k

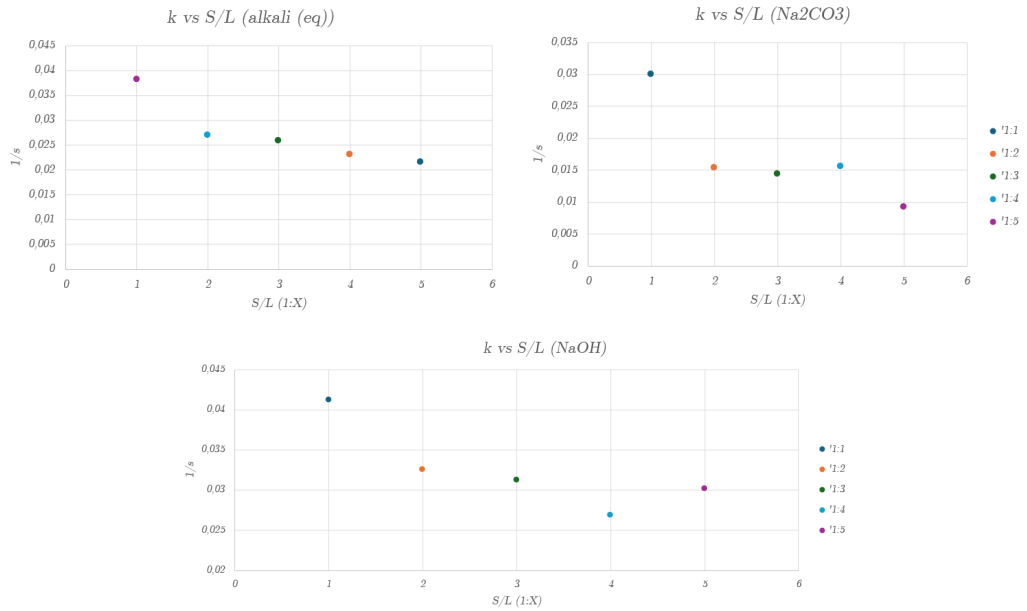

Figure 4.8: Approximated k as a function of S/L ratio

The apparent mass-transfer coefficient k was obtained in Excel by fitting the precipitate's internal concentration data (C_0, C_t and C_{eq}) to the integrated first-order model (equation A.1), where k was taken from the fit that minimized the sum of squared errors (SSE).

As seen in figure 4.8, the apparent mass transfer coefficient k did not vary substantially between the S/L ratios. The k value calculated for S/L ratio 1:1 seems to be an outlier of all the values. If 1:1 is ignored, the difference in k is small enough to assume the mass transfer is constant between S/L ratios 1:2-1:5.

Important to note is that k is a lumped mass-transfer coefficient that combines all transport mechanisms present in the system. It is therefore not a fundamental constant, but a value specific to this experimental setup. In practice, k reflects how quickly solute moves from the liquid inside the solids to the bulk liquid. Because mixing affects how efficiently fresh liquid contacts the solids, k is also influenced by the level of agitation.

4.4.5 Multiple stages and associated costs

TotX(total expenditures) is defined as the sum of OpEX(operational expenditures) for a year, which is calculated via equation A.14 and CapEX(capital expenditures), the costs associated with building the process, which is calculated via A.16. The values can be found in appendix B.5.

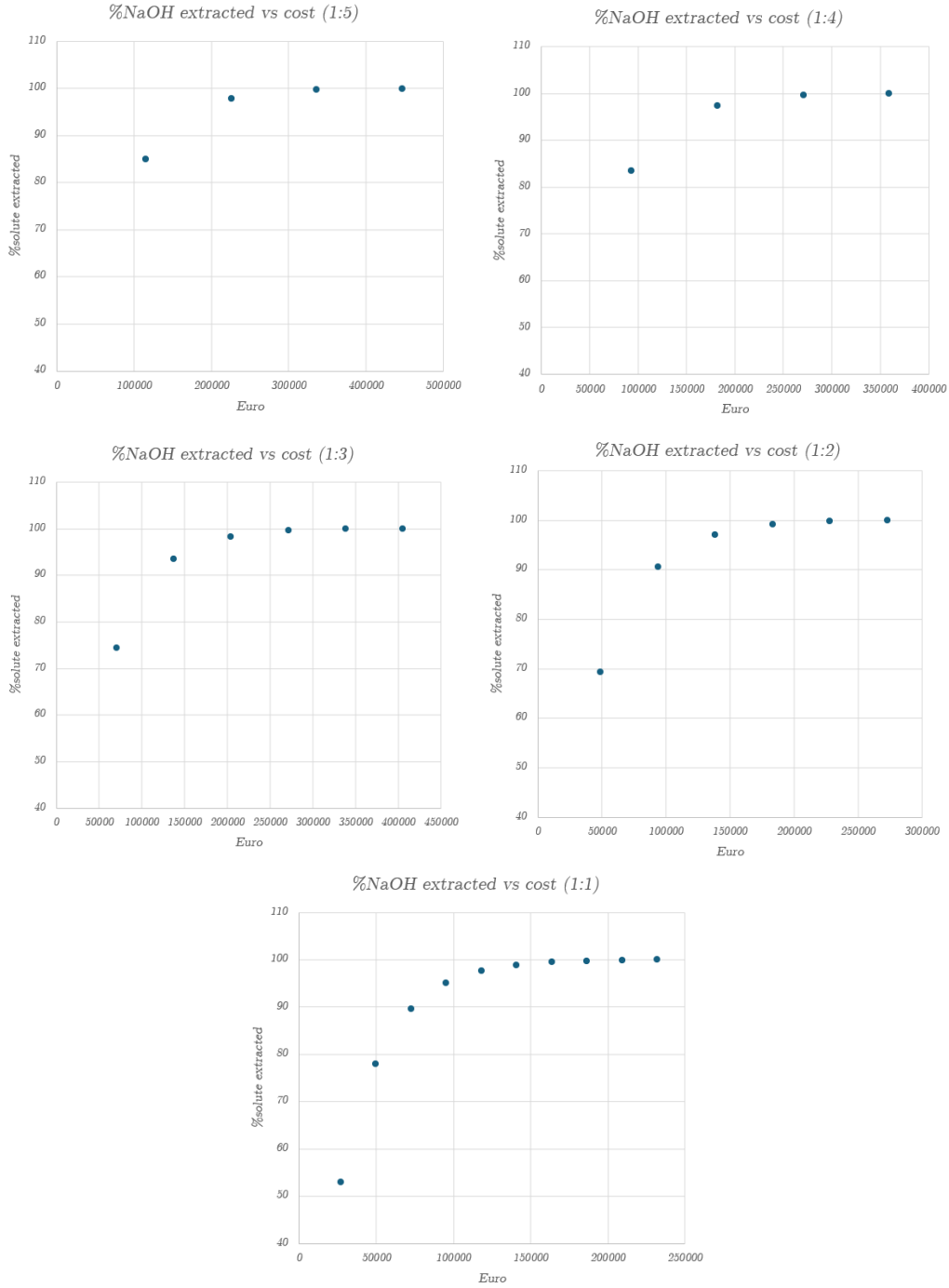

Figure 4.9: %NaOH extracted as a function of total expenditure (EUR)

In figure 4.9, the dot furthest to the left in each graph represents one stage, the second dot represents a second stage, etc. Multiple stages leads to more total %NaOH extracted, but each subsequent stage removes less solute. Lower S/L ratios require fewer stages to reach a certain %NaOH extracted. However, lower S/L ratios consumes more wash liquid per stage.

The data (Appendix B.5) shows that OpEX is the largest cost, which increase for

each subsequent stage and is higher per stage for lower S/L ratios. Important to note is that the price for water is not included in the OpEX calculation, and would in reality be higher.

CapEX is low compared to OpEX and for for all S/L ratios and N stages. In reality, CapEX would be higher as pipes and other equipment would need to be installed as well. The cost of modifying the tank to fit the specific process requirements are also not calculated. Even if the true CapEX would be a few times higher, it would still not influence TotEX substantially.

Figure 4.9 shows that all S/L ratios move closer to 100% extraction with each additional stage, although the gain per stage becomes progressively smaller. Identifying where the curves begin to level off, together with the associated cost at those stage numbers, forms the basis for the recommendation.

4.4.5.1 Liquid retention and Volume change

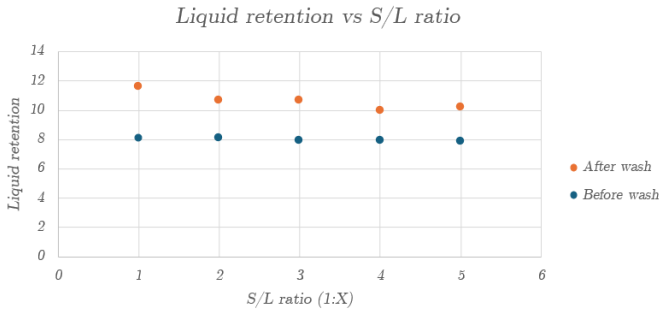

Figure 4.10: Liquid retention before and after washing as a function of S/L ratio.

Figure 4.10 shows liquid retention for different S/L ratio before and after washing. For all S/L ratios, more liquid is associated with the solids after washing. The data also indicates that slighty more liquid is retained for lower S/L ratios.

The % of DS before washing would need further investigation as the precipitate had been stored and allowed to dry before analysis. The %DS after washing was done with precipitate sampled immediately after washing experiment. The washed precipitate had water adsorbed on the surface, and this would be the case for freshly precipitated cellulose as well. Comparing the %DS of freshly produced precipitate and %DS of freshly washed precipitate would be preferred.

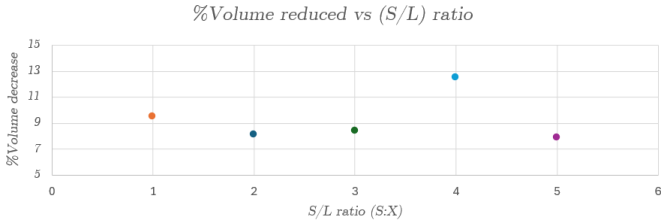

Figure 4.11: Solid phase volume decrease (%) as a function of S/L

Comparing the % of volume reduced for different S/L ratios after washing shows varied results, as seen in 4.11. The %of volume decrease in the conducted experiments, including the bag filter experiment, shows a range between 7.9-12.5%.

The volume before and after washing is measured using containers marked in 20 mL increments. Depending on how the measurement is performed and how the sample settles in the container, the recorded value can vary slightly. Although all samples are handled using the same procedure, considerable uncertainty is associated with these measurements. The volume data should therefore be viewed as indicative rather than exact.

4.5 Additional Considerations for Multi-Stage Washing

This thesis evaluates washing performance based on idealized stage models, but the practical handling of solids and liquid between stages was not investigated experimentally. In a real multi-stage washing system, the bulk liquid must be removed after each stage and the solids transferred forward. Because the precipitated cellulose does not form a permeable filter cake and collapses into a gel when pressed, liquid removal would rely on filtration of the bulk slurry or decantation. The internal water phase remains associated with the solids and is carried into the next stage, which defines the effective wash ratio and limits the achievable extraction. Although the mechanical design of underflow, pumping, or settling equipment was not evaluated in this work, these considerations are important for future scale up and should be evaluated in continued development of the washing process.

4.6 Recommendations

In order to remove a significant amount of solids, a multi stage set up is necessary. The primary cost of washing the cellulose precipitate is the energy required to evaporate the wash liquid. It is necessary to minimize the wash liquid consumption in order to create an efficient process.

Important to note is that a counter current washing strategy would, in theory, consume less wash liquid. As OpEX is the largest cost by far, it is recommended that this strategy is further researched and modeled.

Based on the experimental data and the assumptions made, a three stage washing strategy with S/L ratio 1:2 is recommended. The calculations show that 97.09% of NaOH would be extracted with a yearly cost of 138 631 EUR. If more extraction is desirable, a fourth stage could be implemented which would result in 99.11% NaOH extracted at a yearly cost of 183 421 EUR. This gives a good balance between number of stages, water usage and extraction.

4.7 Future work

The titration measurements would have benefited from having higher resolution, as varying titration values by +-0.01, a noticeable impact was noted on the wash curves. A titrator with higher sensitivity and decimals would therefore have been valuable at these concentration ranges, especially for lower S/L ratios.

Some things would be beneficial to investigate further, in order to verify the results and deepen the understanding of the washing kinetics. Future work could include:

- Counter current modeling.
- Scaled up experiments.
- Higher temperatures.
- Muli stage counter current washing trials.
- Improved sampling and analysis.
- Freshly produced precipitate.

5
Conclusions

Single-stage stirred batch washing experiments with cellulose precipitate showed that lower S/L ratios extract a larger fraction of solute, but at the cost of increased wash-liquid usage and subsequent evaporation energy. The apparent mass-transfer coefficient k was similar for all S/L ratios except 1:1, indicating that washing performance is governed primarily by dilution of the internal water phase rather than by differences in mass transfer kinetics.

A CSTR-in-series model was used to estimate multi-stage washing performance. The analysis showed that both S/L ratio and the number of stages have a strong influence on operational expenditures, which is dominated by the energy required to heat and evaporate wash liquid. In contrast, capital expenditures contributes only a small fraction of the total cost, highlighting the importance of minimizing wash-liquid usage rather than minimizing equipment count.

Based on the combined extraction performance and cost evaluation, the recommended washing strategy is a four stage strategy using S/L ratio 1:2. This set up achieves 99.11% extraction with a total cost of 183 421 EUR for one year.

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A

Appendix

Integration of First Order Washing Model

The governing differential equation is [3]:

$$N = k_L(c - c^*)$$

Re-written:

$$\frac{dC}{dt} = k\,(C_{\text{eq}} - C)$$

Separating variables:

$$\frac{dC}{C_{\text{eq}} - C} = k\,dt$$

Integrating both sides:

$$\int \frac{dC}{C_{\text{eq}} - C} = \int k\,dt$$

$$-\ln(C_{eq} - C) = kt + C_1$$

Exponentiating:

$$C_{eq} - C = Ae^{-kt}$$

Applying the initial condition $C(0) = C_0$:

$$A = C_{eq} - C_0$$

Final expression:

$$C(t) = C_{eq} + (C_0 - C_{eq})e^{-kt} \tag{A.1}$$

Liquid retention equation

$$R = \frac{m_{solids} \times (1 - \frac{\%DS}{100})}{m_{solids} \times \frac{\%DS}{100}} \tag{A.2}$$

CSTR related equations

[5]:

$$kC_{An} = k\frac{C_{A0}}{(1+\tau k)^n}$$

Re-written:

$$C_{end} = \frac{C_0}{(1+k\tau)^N} \tag{A.3}$$

[5]:

$$\tau = \frac{1}{k}(\frac{X}{1-X})$$

Re-written:

$$\tau = \frac{1}{k}(\frac{C_0}{C_{eq}} - 1) \tag{A.4}$$

$$\%Removed = \frac{C_0 - C_{end}}{C_0} \times 100 \tag{A.5}$$

NaOH equivalence conversion

A conversion factor for converting Na_2CO_3 to NaOH is calculated by comparing molar masses and amount of sodium atoms in each molecule:

$$\frac{M_{NaOH}}{M_{\mathrm{Na_2CO_3}}} \times 2 = \frac{40}{105,99} \times 2 = 0,7548 \tag{A.6}$$

$$Alkali(eq) = \mathrm{Na_2CO_3} \times 0,7548 + NaOH \tag{A.7}$$

Dry solids equation

$$\%DS = \frac{m_{dried}}{m_{initial}} \tag{A.8}$$

Internal solute concentration of originally retained liquid

The equations below are described for NaOH, but applies for Na_2CO_3 and NaOH(eq) as well.

$$C_0 = \frac{m_{solids} \times \frac{\%NaOH_{\mathrm{solids}}}{100}}{m_{solids} \times (1 - \frac{\%DS}{100})} \tag{A.9}$$

$$C_t = \frac{m_{solids} \times \frac{\%NaOH_{\mathrm{solids}}}{100} - m_{\text{wash liquid}} \times \frac{C_{\mathrm{titration,\ t}}}{100}}{m_{solids} \times (1 - \frac{\%DS}{100})} \tag{A.10}$$

%of solute originally in precipitate and %of solute leached to bulk liquid

$$\%NaOH_{solids} = \frac{(m_{washliquid} + m_{solids} \times (1 - \frac{\%DS}{100})) \times \frac{C_{\text{titration, 10m}}}{100}}{m_{\text{solids}}} \times 100 \quad \text{(A.11)}$$

$$\%\text{leached NaOH} = \frac{C_{\text{titration}} \times m_{\text{wash liquid}}}{\frac{\%NaOH_{solids}}{100} \times m_{\text{solids}}} \times 100 \quad \text{(A.12)}$$

Water usage and cost equations

OpEX=Operational expenditures
CapEX=Capital expenditures

Precipitate per day = 5000kg KWh/MJ = 3,6
SEK/EUR = 10,93[9] USD/EUR = 0,87[9] 0,1831 öre/Kwh
$c_p = 4.18$ kJ/(kg K), $T_b = 100°C$, $\Delta H_{\text{vap}} = 2257$ kJ/kg

$$m_{water} = N_{stages} \times (L/S) \quad \text{(A.13)}$$

$$Q_{/\text{kg water}} = c_p(T_b - T_0) + \Delta H_{\text{vap}} \quad \text{(A.14)}$$

$$\text{OpEX/yr} = \frac{Q_{/\text{kg water}} \times (L/S) \times 5000 \times 365 \times 0,1831}{3,6 \times 10,93} \quad \text{(A.15)}$$

$$\text{CapEX/stage} = N \times (€_{tank} + €_{agitatior} + €_{pump}) \quad \text{(A.16)}$$

1000L tank + agitator = 4176 EUR[6][9] Pump = 799,53 EUR[7][9]

Volume change equation

$$\%V_{difference} = \frac{V_{before} - V_{after}}{V_{before}} \times 100 \quad \text{(A.17)}$$

B

Appendix

Preliminary washing study wash liquid and precipitate amounts

Figure B.1: Bag 1 precipitate initial masses

S/L ratio	m solids (g)	m wash liquid (g)
1:5	109,811	550
1:4	140,369	560
1:3	189,66	570
1:2	270	540
1:1	550	550

Figure B.2: Bag 2 precipitate initial masses

S/L ratio	m solids (g)	m wash liquid (g)
1:5	110,022	550
1:4	140,037	560
1:3	190,146	570
1:2	270,746	540
1:1	550	550

Preliminary study titration results

Bag 1		30	60	120	180	300	420	600
1:5	NaOH	0	0,22	0,26	0,28	0,31	0,32	0,33
	Na2CO3	0	0	0	0	0	0	0
1:4	NaOH	0	0,25	0,31	0,34	0,37	0,34	0,35
	Na2CO3	0	0	0	0	0	0,11	0,12
1:3	NaOH	0,23	0,33	0,39	0,42	0,42	0,43	0,43
	Na2CO3	0	0	0	0	0,11	0,13	0,14
1:2	NaOH	0,34	0,42	0,48	0,52	0,55	0,56	0,58
	Na2CO3	0	0	0,1	0,13	0,16	0,19	0,2
1:1	NaOH	0,54	0,61	0,73	0,76	0,83	0,84	0,85
	Na2CO3	0,13	0,16	0,24	0,26	0,31	0,33	0,35
Bag 2		30	60	120	180	300	420	600
1:5	NaOH	0	0	0,32	0,34	0,36	0,37	0,37
	Na2CO3	0	0	0	0	0	0	0
1:4	NaOH	0,33	0,36	0,39	0,41	0,43	0,43	0,44
	Na2CO3	0	0	0	0	0	0	0
1:3	NaOH	0,34	0,43	0,49	0,48	0,54	0,5	0,51
	Na2CO3	0	0	0	0,11	0	0,14	0,15
1:2	NaOH	0,48	0,46	0,56	0,59	0,62	0,63	0,63
	Na2CO3	0	0	0	0,14	0,16	0,17	0,19
1:1	NaOH		0,75	0,83	0,88	0,92	0,93	0,94
	Na2CO3	0	0	0,1	0,13	0,15	0,18	0,18

Figure B.3: Titration values from preliminary washing study

Raw data from washing study

(S/L) ratio	$\%DS_0$	$m_{solids,0}$ (g)	$V_{solids,0}$ (ml)	m_{water} (g)	T_{water} (°C)	$m_{solids,1}$ (g)	$V_{solids,1}$ (ml)	$\%DS_1$	Bag nr
1:5	10,994	497	760	2483	19,9	587	700	7,907	1
1:4	10,994	593	960	2381	19,8	683	840	8,557	1
1:3	11,212	743	1060	2228	20,0	785	980	8,547	2
1:2	11,212	987	1480	1974	20,0	1116	1360	9,118	2
1:1	11,246	1471	2100	1471	20,0	1588	1900	8,914	3

pH:

(S/L)	30s	1m	2m	3m	5m	7m	10m
1:5	12,38	12,53	12,64	12,69	12,73	12,75	12,77
1:4	12,47	12,64	12,75	12,79	12,82	12,83	12,84
1:3	12,66	12,80	12,88	12,92	12,94	12,95	12,96
1:2	12,83	12,92	13,0	13,02	13,05	13,06	13,06
1:1	12,96	13,06	13,12	13,15	13,17	13,17	13,18

%NaOH titration:

(S/L)	30s	1m	2m	3m	5m	7m	10m
1:5	0,15	0,17	0,19	,20	0,21	0,22	0,22
1:4	0,16	0,19	0,21	0,23	0,24	0,24	0,25
1:3	0,22	0,25	0,28	0,30	0,31	0,32	0,32
1:2	0,30	0,34	0,38	0,40	0,42	0,42	0,43
1:1	0,50	0,56	0,60	0,62	0,64	0,65	0,66

%Na_2CO_3 titration:

(S/L)	30s	1m	2m	3m	5m	7m	10m
1:5	0,04	0,06	0,07	0,08	0,09	0,10	0,11
1:4	0,07	0,08	0,10	0,10	0,13	0,13	0,13
1:3	0,08	0,09	0,11	0,12	0,13	0,15	0,15
1:2	0,10	0,13	0,15	0,16	0,18	0,20	0,20
1:1	0,19	0,22	0,23	0,26	0,27	0,28	0,28

Figure B.4: Raw data from washing study

Impact of multiple stages stages data

S	L	Stages	%extracted	Water/kg	MJ/kg	OPEX/year	CAPEX	TOTEX
1	5	1	84,88	5	12,96	110061,0247	4975,53	115036,5547
1	5	2	97,71	10	25,91	220037,1258	5775,06	225812,1858
1	5	3	99,65	15	38,87	330098,1505	6574,59	336672,7405
1	5	4	99,95	20	51,83	440159,1752	7374,12	447533,2952
1	4	1	83,52	4	10,37	88065,80449	4975,53	93041,33449
1	4	2	97,29	8	20,73	176046,6853	5775,06	181821,7453
1	4	3	99,55	12	31,1	264112,4898	6574,59	270687,0798
1	4	4	99,93	16	41,46	352093,3707	7374,12	359467,4907
1	3	1	74,37	3	7,77	65985,66064	4975,53	70961,19064
1	3	2	93,43	6	15,55	132056,2449	5775,06	137831,3049
1	3	3	98,32	9	23,32	198041,9056	6574,59	204616,4956
1	3	4	99,57	12	31,1	264112,4898	7374,12	271486,6098
1	3	5	99,89	15	38,87	330098,1505	8173,65	338271,8005
1	3	6	99,97	18	46,65	396168,7348	8973,18	405141,9148
1	2	1	69,25	2	5,18	43990,44043	4975,53	48965,97043
1	2	2	90,55	4	10,37	88065,80449	5775,06	93840,86449
1	2	3	97,09	6	15,55	132056,2449	6574,59	138630,8349
1	2	4	99,11	8	20,73	176046,6853	7374,12	183420,8053
1	2	5	99,73	10	25,91	220037,1258	8173,65	228210,7758
1	2	6	99,92	12	31,1	264112,4898	8973,18	273085,6698
1	1	1	52,98	1	2,59	21995,22021	4975,53	26970,75021
1	1	2	77,89	2	5,18	43990,44043	5775,06	49765,50043
1	1	3	89,6	3	7,77	65985,66064	6574,59	72560,25064
1	1	4	95,11	4	10,37	88065,80449	7374,12	95439,92449
1	1	5	97,7	5	12,96	110061,0247	8173,65	118234,6747
1	1	6	98,92	6	15,55	132056,2449	8973,18	141029,4249
1	1	7	99,49	7	18,14	154051,4651	9772,71	163824,1751
1	1	8	99,76	8	20,73	176046,6853	10572,24	186618,9253
1	1	9	99,89	9	23,32	198041,9056	11371,77	209413,6756
1	1	10	99,95	10	25,91	220037,1258	12171,3	232208,4258

Figure B.5: Stages and subsequent OpEX/CapEX (EUR)

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