CHALMERS

Fouling of VSEP RO Membranes

A Pilot Plant Study of Black Water Recycling at Skogaberg

Examensarbete inom civilingenjörsprogrammet Kemiteknik
Master of Science Thesis in Chemical Engineering

EVA RUD

Department of Civil and Environmental Engnineering
Division of Water Environment Technology
CHALMERS UNIVERSITY OF TECHNOLOGY
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Department of Civil and Environmental Engineering
Water Environment Technology
Chalmers University of Technology
SE-41296 Göteborg
Sweden
Telephone: +46(0)31-7721000

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Preface

This report is a diploma work for the partial fulfilment of a Master of Science degree in Chemical Engineering at Chalmers University of Technology. This work has been performed at the Department of Sustainable Water and Waste, City of Göteborg and at the Department Civil and Environmental Engineering, Environment Technology during 20 weeks between September 2005 and January 2006.

First of all, I would like to thank my supervisors Britt-Marie Wilén and Mark de Blois for their encouragement, support and for all feed back.

I would also like to thank:

Pascal Karlsson for practical advice and for choosing me for this project.

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Carl-Henrik Hansson for all practical advice.

Hans Diechle for his support during the writing.

Marie-Catherine Coquin for all help in the initial part of this work.

Abstract

In this diploma work recycling of nutrients in black water by means of reverse osmosis (RO) was studied. A vibratory shear enhanced process (VSEP) RO was applied. The main focus of the study was to investigate the fouling on VSEP RO membranes.

The objectives were to (1) get information about what components the fouling is composed of; (2) to understand the causes of fouling and how the fouling affects the membrane surface and (3) to find ways to delay fouling

The following methods were used:

- Study different batches to look how different parameters influence permeate flow rate and the fouling phenomenon. The VSEP-stack was also opened to look how the fouling looked like on the membrane surface.

- Chemical analyses to identify which substances can be detected in the fouling, feed, concentrate and permeate in different operations mode and mass balance calculations.

- Light microscope, BET (Brauner, Emmet and Teller)-method and ESEM (Environmental Scanning Electron Microscope) – for study of the surface and the structure of the fouling.

The following conclusions can be drawn from the results:

1. The main part of the fouling consists of a biofilm with a lot of bacteria. These bacteria are strongly fixed to the membrane surface, the bacteria would not be removed totally by cleaning and there were many left even in turbulent zones. In the fouling there are also a lot of inorganic crystals. The main part of the crystals is composed of calcium carbonates and calcium phosphates.

2. Mainly the precipitations causes the decreasing of the permeate flow rate. The biofilm does not influence the flow rate in the same extend.

   How the fouling influences the surface is more difficult to say, the results show some change of surface structure but this does not seem to influence the flow rate. A long exposure time of bacteria on the membrane surface could probably destroy the surface and influence the membrane rejection and flow rate. In zones of turbulent flow, the surface look more smoother but it has not been possible to determine if that depends on a change of surface or a layer of biofilm.

3. It has been successful to add acid to delay fouling.

Sammanfattning

Syftet med detta examensarbete för civilingenjörsexamen i kemiteknik vid Chalmers Tekniska högskola är att undersöka foulingen på RO (omvänd osmos) membran i VSEP.

Målet är (1) att ta reda på vilka komponenter som foulingen består av, (2) förstå vad som orsakar foulingen och hur foulingen påverkar membranens yta och (3) metoder att motverka foulingen.

För att bestämma foulingen har följande metoder använts:

- Studie av olika batcher för att undersöka hur olika parametrar påverkar flödeshastigheten av permeatet och hur foulingen då beter sig samt öppnande av VSEP-stacken för att se hur foulingen ser ut på membranens yta.

- Kemiska analyser för att identifiera vilka substanser som finns i foulingen, inflödet, koncentratet och permeatet, vid membranfiltrering med olika inställningar samt beräkning av massbalanser.

- Ljusmikroskop, BET (Brauner, Emmet and Teller)-metod och ESEM (Environmental Scanning Electron Microscope) för att studera ytan och utseendet av foulingen.

Resultatet blev följande slutsatser:

1. Huvudsakligen består foulingen av en biofilm med mycket bakterier, men det finns även en hel del kristaller. Kristallerna består mest av kalciumkarbonater och kalciumfosfater Även i områden där turbulenta flöden förekommit finns bakterier vilket bevisar att de sitter hårt på ytan.

2. Det är utfällningen som ger upphov till det minskade permeatflödet. Biofilmen påverkar inte flödet i samma omfattning.

   Foulingens påverkan av membranens yta är svårare att bestämma. Resultaten visar att det finns förändringar på ytan men dessa verkar inte påverka flödeshastigheten. Dock kan det förväntas att bakterierna efter lång exponeringstid förstör ytan så mycket att detta påverkar både membranrejektionen och permeatflödeshastigheten. I zoner där turbulent flöde har förekommit har ytan ett mycket slätare utseende. Vad detta beror på har dock inte varit möjligt att bestämma, men det kan bero på en förändring av ytstrukturen eller att en tunn biofilm ligger på ytan.

3. Syratillsats har varit en lyckad metod för att motverka foulingen.

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1. Introductions

1.1 Background

1.1.1 The Black Water System at Skogaberg

Skogaberg is a new residential area on Hisingen in Göteborg. In this area the local house construction company, Egnahemsbolaget, and the Department of Sustainable Water and Waste, City of Göteborg, are collaborating on how to develop a sustainable system for the treatment of household sewage.

In Skogaberg two separate systems of pipes for wastewater have been built. In these two different pipes, black water (wastewater from toilets and food rests) and grey water (wastewater from the rest of the household) are separated. The grey water is going to the wastewater treatment plant in Göteborg (Gryaab), while the black water is undergoing a concentration process. The purpose of concentrating the black water is to recover valuable nutrients such as nitrogen, phosphorus, potassium and sulphur for agricultural use. The objective is to produce fertilizers that fulfil the farmer's requirements of purity, quality, and safety. It is also important that it does not consume too many resources and is easy to transport to the farmlands [*Karlsson (2004)*].

Previous feasibility studies and laboratory tests have given that some kind of membrane technique should be the best way to keep valuable nutrient in a concentrated form. The chosen membrane is VSEP, Vibratory Shear Enhanced Process, operated in a reverse osmosis mode [*de Blois (2004)*].

The black water is going to a pilot plant located close to Skogaberg. First the black water is sent through a drum sieve to remove larger particles. After that the filtrate is going to the VSEP and the more concentrated sludge fraction is intended to go to further treatment, e.g. digestion [*de Blois (2004)*].

1.1.2 Previous Diploma Works at Skogaberg

During the spring and summer 2005 three different diploma works were carried out at Skogaberg. One of the works was about the drum sieve and was carried out by Carlos Martinez, Borås University. The other two were about the VSEP system and were carried out by Shadab Ahmad and Marie-Catherine Coquin, both master students at Chalmers.

Shadab Ahmad has investigated the chemical processes occurring in the black water during concentration. Moreover his theoretical part included some fundamental theory about membrane, reverse osmosis and VSEP.

In his laboratory work the VSEP operated in a Laboratory mode (L-mode), i.e. only one membrane installed, was analysed. He examined feed batches with acid (initial pH = 6), antiscalant (10 ppm) and no dose. Also membrane cleaning analyses were carried out. The most important conclusions obtained were:

- There were no substantial differences in permeate flow between the three different experiments with acid, antiscalant or no dose to the feed.
- In all cases the permeate flow dropped after ca 80 % volume reduction. This indicates some fouling.
- The fouling was mainly organic.
- Cleaning agent with high pH gave best result [*Ahmad (2005)*].

Marie-Catherine Coquin also studied chemical reactions during concentration and estimated which compounds that theoretically could precipitate and compared this with the laboratory results. She also compared the results from the laboratory mode and the pilot mode. Furthermore, she wrote a very detailed description of the pilot plant and its operation including procedures to collect samples.

In her laboratory work she analysed the VSEP in a Pilot mode (P-mode), i.e. a stack with 38 membranes. She examined batches with acid (initial pH = 6), antiscalant (10 ppm) and no dose to the feed. Like Shadab Ahmad, she also studied cleaning processes. The main conclusions were:

- The results for the L-mode tests and the P-mode tests were similar.
- The acidified batch gave the highest concentration factor and the highest compound retention in the concentrate.
- The theoretical calculations gave that the most likely metal salts to precipitate are calcium carbonate, magnesium carbonate and calcium phosphate. The most important forms are hydroxyl apatite, calcite, tricalcium phosphate and aragonite.
- Acidification prevented the membrane from inorganic scaling of the membrane [*Coquin (2005)*].

1.1.3 Diploma Work Parallel with this Diploma Work

Parallel with this work, Torben Meins, University of Applied Science, Lübeck, has been examined how pressure and vibration, frequency and amplitude, should be adjusted for best operation to delay fouling.

1.2 Aim

The purpose of this diploma work was to examine fouling on the membranes with focus on chemical aspects. The objectives were to (1) get information about what components the fouling is composed of; (2) to understand the causes of fouling and how the fouling affects the membrane surface and membrane filtration and (3) to find ways to delay fouling.

From the earlier studies the following hypothesis could be delivered: the cause of the abrupt permeate flow decrease is due to precipitation of supersaturated, insoluble metal salts on the membrane surface. The volubility of this hypothesis has been examined in parallel with Torben Meins' work.

1.3 Experimental Approach

The following methods have been used:

- *Different operation modes* – study of different batches to investigate how different parameters influence permeate flow rate and the fouling phenomenon.

- *Visual analyses* – opening up the VSEP-stack to study what the fouling looks like on the membrane surface.

- *Chemical analyses* – identify which substances that can be detected in the fouling, feed concentrate and permeate in different operation modes.

- *Mass balance calculations* –to examine how different substances are distributed between concentrate, permeate and losses in the different batches.

- *Light microscopy* – to examine what the fouling looks like.

- *BET (Brauner, Emmet and Teller)-method* – to study if there are some changes in membrane structure before and after use.

- *ESEM (Environmental Scanning Electron Microscope)*– to study the membrane surface and see if there is any changes of structure and find out if there is some fouling on the surface after cleaning.

2. Literature Review

2.1 Black Water

The black water contains toilet water and food rests. This gives a very concentrated and nutrient rich sewage. The most important substances to recover are phosphorus, nitrogen, potassium and sulphur in the different forms (see table 1 below) [*de Blois (2004)*].

Table 1 Components in black water [*de Blois (2004)*].

Phosphorus	Phosphate	PO_4^{3-}, $H_2PO_4^-$, H_3PO_4, etc
	Phosphate (chemically bound)	$Ca_3(PO_4)_2$, $Mg_3(PO_4)_2$ etc
	Organically bound	bacteria
Nitrogen	Ammonium/Ammonia	NH_4^+/NH_3
	Ammonium (chemically bound)	NH_4MgPO_4
	Organically bound	Proteins, bacteria
	Nitrate	NO_3^-
	Nitrite	NO_2^-
Potassium	Dissolved	K^+
Sulphur	Sulphate	SO_4^{-2}
	Sulphite (chemically bound)	CaS etc
	Organically bound	Proteins, bacteria
Organic substances	Dissolved	
	Particles	

Black water has a very complex composition. It consists of water, salts (Na^+, Ca^{2+}, Mg^{2+}, Cl^-, SO_4^{2-}, and CO_3^-), complex carbohydrates (starches, sugars, proteins, and fats) and nutrients (nitrogen, phosphorus, and potassium). There are also bacteria and viruses [*Johnson, (2004)*]. All these components make a separation process very complicated.

2.2 Membrane

2.2.1 Different Types of Membranes

A membrane can be defined as: “an interphase separating two phases and selectively controlling the transport of materials between those phases”. Membrane technology is a rather new industrial process. Since the 1960s the development of synthetic materials for membrane processes have been very rapid and lead to more wide usage of membranes in industrial separation processes.

The classification of industrial membrane processes are based on the size range of materials which are to be separated and the driving forces used in the separation [*Coulson (2002)*]. There are many of different types of membrane separations processes. Mulder (1996) is dividing them in the following way:

Table 2: Some different membrane processes [*Mulder (1996)*].

Membrane process	Driving force Phases	Separation principle	Example of application
Microfiltration	ΔP L - L	Sieving mechanism	• Analytical application • Sterilisation • Production of ultra pure water
Ultrafiltration	ΔP L - L	Sieving mechanism	• Diary (milk, cheese making) • Food industry (potato starch, proteins) • Metallurgy (oil-water emulsion)
Nanofiltration	ΔP L - L	Solution-diffusion	• Desalination of brackish water • Removal of micropollutants • Wastewater treatment
Reverse Osmosis	ΔP L - L	Solution-diffusion	• Desalination of brackish water • Production of ultra pure water • Concentration of food juice and sugars
Piezodialysis	ΔP L - L	Ion transport (Coulomb attraction and electroneutrality)	• Salt enrichment
Gas separation	Δp G - G	Solution-diffusion	• H_2 or He recovery • CH_4 / CO • O_2 / N_2
Pervaporation	Δp L - G	Solution-diffusion	• Dehydration of organic solvents • Removal of organic compounds (alcohols, aromatics etc.) from water
Electrodialysis	ΔE L - L	Donnan exclusion mechanism	• Desalination of water • Desalination in food and pharmaceutical industry • Separation of amino acids
Dialysis	Δc L - L	Difference in diffusion rate, Solution-diffusion	• Hemodialys (removal of toxic substances from blood) • Alcohol reduction in beer
Diffusion dialysis	Δc L - L	Donnan exclusion mechanism	• Acid recovery from etching, pickling and metal refining • Alkali recovery from textile and metal refining processes
Membrane contactors	Δc L - L	Distribution coefficient	• SO_2, CO_2, CO, NO_X from flue gases • CO_2 and H_2S from neutral gas
	Δc/ΔP G - L		• Volatile bioproduction (alcohol, aroma component) • O_2 removal from water
	Δc/ΔP L - G		• Heavy metal • Fermentation production (citric acid, acetic acid, penicillin etc.)
Membrane distillation	ΔT/ΔP L - L	Vapour-liquid equilibrium	• Production of pure water • Removal of volatile organic compounds

L-liquid, G-gas

Applications of different membranes for different fractions are summarized according to figure 1. [*Mulder (1996)*]

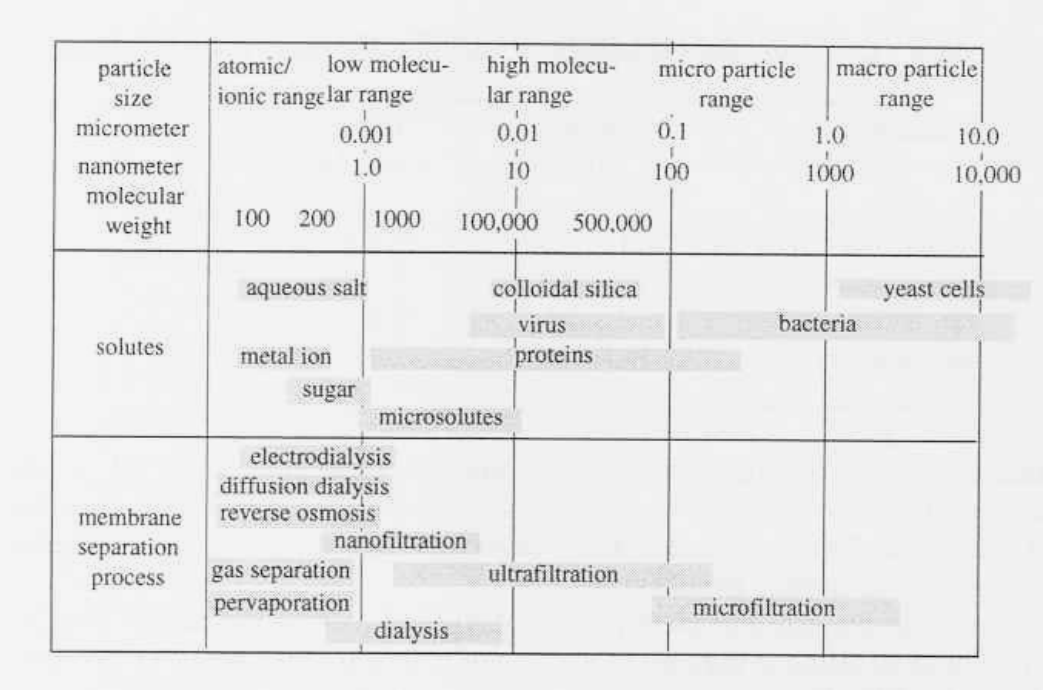


Figure 1. Summary of applications for different membranes [*Mulder (1996)*].

2.2.2 Membrane Applications for Waste Water Treatment

The role of membrane processes in wastewater treatment
Membrane technology has got an increased usage in wastewater treatment. The most common area is to clean wastewater to get a new water source, i.e. water recycling to obtain potable water from wastewater. Depending on what the water is going to be used for, different technology is necessary. The membrane applications to reuse municipal wastewater are used all over the world, at the end of 2004, 27 full scale installations were recorded. The development of membrane technology is one important way to secure the need of drinking water and other needs of water.

The higher the required quality of the cleaned water, the denser membrane is necessary to use. Microfiltration is able to remove suspended solids and larger microorganisms like protozoa and bacteria. Ultrafiltration also removes viruses and organic macromolecules down to a size of around 20 nm. Nanofiltration removes smaller organics and multivalent ions while if it is necessary to remove all dissolves species a reverse osmosis membrane has to be used [*Wintgens et al (2005)*].

How the membrane technology is used in wastewater treatments differs a lot. In Wollongong, Australia, low nutrient tertiary outlet from the ordinary sewage treatment plant, is passing a microfiltration and reverse osmosis membrane. The outgoing permeate (clean water) is going to the steel mill in the nearby Port Kembla and the industry does not need to use water from the drinking water source which is scarce [*Wintgens et al (2005)*].

Membrane technology is often combined with a bio reactor. In Japan this combination is often used in wastewater treatment. One reason to this is that a membrane bio rector takes small place and works well in small scale applications. This is necessary in densely built-up Japanese cities. It has been found that these membrane bio reactors have a lot of advantages over alternative biological treatment processes for water recycling, for example in process robustness [*Wintgens et al (2005)*].

Today it is not so common to produce drinking water directly from the wastewater. Often as in the example above from Australia, membrane treated water is replacing water use from a drinking water source which is not going to be used as drinking water. The main reason is public resistance to drink "wastewater" not due to technical limitations. But in Windhoek, Namibia, there is the world's first operation plant, which produces drinking water directly from wastewater. This plant is a complex treatment, including pre-ozonation, coagulation, dual media filtration, main ozonation, biological activated carbon adsorption and a two-stage granular activated carbon adsorption as well as ultrafiltration prior to chlorine disinfection [*Wintgens et al (2005)*].

Wastewater as alternative water source
In southern Italy the agricultures have problems with too small fresh water sources. A solution for this problem is to clean wastewater with membrane technology and use the permeate for irrigation.

The membrane module was made of a bundle of hollow fibres, with a pore size of 0.03 μm. Two different crops were studied, tomatoes and fennel and the effects were compared with ordinary water. During the test period no fertilizer or other agricultural chemicals were used. The result from membrane filtration show that the permeate has a quality similar to groundwater. There were no reminders founds of chemical or microbiological content in either soil or crops, so the study shows that this technology is very promising to use in the future [*Pollice et a (2004)*].

2.2.3 Reverse Osmosis

Reverse osmosis is used when low molecular weight molecules, such as inorganic salts and small organic molecules, are to be separated from a solution. The principle is that the membrane is permeable for the solvent (water) but not to the solute. To get the water to pass through the membrane it is necessary to apply a pressure [*Mulder (1996)*].

A simplified equation to describe the flux according Baker (2004):

$$J_i = A \cdot (\Delta p - \Delta \pi) \qquad (1)$$

J_i = *flux of water* $\left[\frac{m^3}{m^2 \times s}\right]$

Δp = *pressure diffrence across the membrane* $[Pa]$

$\Delta \pi$ = *osmotic pressure diffrence across the membrane* $[Pa]$

A = *Water permeability constant* $\left[\frac{m^3}{m^2 \times s \times Pa}\right]$

- At low pressure ($\Delta p < \Delta \pi$): Water goes from diluted to concentrated side.
- At equal pressure ($\Delta p = \Delta \pi$): No flux.
- At high pressure ($\Delta p > \Delta \pi$): Water goes from concentrated to diluted side [*Baker(2004)*].

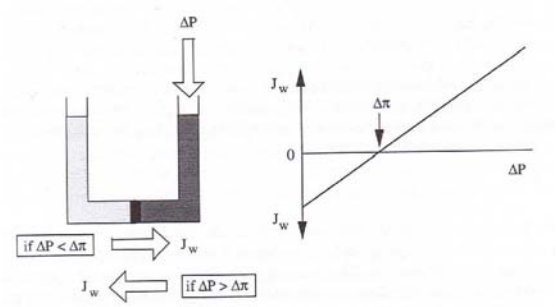


Figure 2. Overview of reverse osmosis principle [*Mulder (1996)*].

A simplified equation to describe the salt flux according Baker (2004):

$$J_j = B \cdot (c_{j0} - c_{jl}) \qquad (2)$$

J_j = *flux of salt* $\left[\frac{kg}{m^2 \times s}\right]$

c_{j0} = *concentration of salt on feed* $\left[\frac{kg}{m^3}\right]$

c_{ji} = *concentration of salt on permeate* $\left[\frac{kg}{m^3}\right]$

B = *salt permeablity constant* $\left[\frac{m}{s}\right]$

Usually $c_{jl} << c_{j0} \rightarrow J_j = B \cdot c_{j0}$ (3) this means that the salt flux is independent of the pressure. If the pressure increase, Δp and also (Δp-$\Delta \pi$) is increasing, this leads to a higher flux of water, while the salt flux still is similarly as before increasing the pressure. In other words the selectivity of the membrane increases when the pressure increases, due to the increased water flux at unchanged the salt flux [*Baker (2004)*].

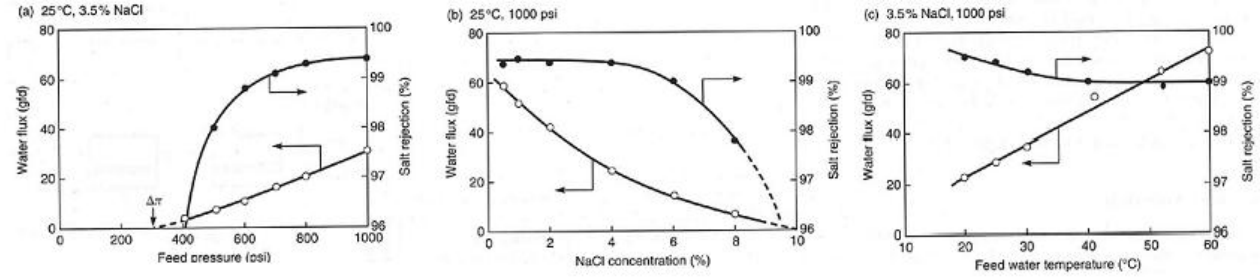
2.2.4 Selectivity

One way to describe the selectivity of a membrane is to use a salt rejection coefficient, R. It shows how different operation parameters affect the flux through the membrane [*Baker (2004)*].

$$R = \left[1 - \frac{c_{ji}}{c_{j0}}\right] \cdot 100\% \qquad (4)$$

$$c_{ji} = \frac{J_j}{J_i} \cdot \rho_i \qquad (5) \qquad \rho_i = \textit{density of water}$$

$$\textit{Equations } 1-5 \rightarrow \quad R = 1 - \frac{\rho_i \cdot B}{A \cdot (\Delta p - \Delta \pi)} \cdot 100\% \qquad (6)$$



• - Salt rejection o - Water flux

Figure 3. The effect of different operation parameters [*Baker (2004)*].

Higher pressure
- The flux of water increase proportional according to equation (1).
- R first increases fast whereupon it stabilises. This depends on equation (6), when Δp is small, a change of the pressure gives a large affect of R-value. At a large Δp, R approaches 100%.

Higher concentration
- The flux of water decrease, since a higher salt concentration gives a higher osmotic pressure and according to equation (1) this leads to a decreasing water flux.
- R is stable at first and then it will start to drop, this according to equation (6). At first a higher osmotic pressure does not influence so much, but the closer the applied pressure is the osmotic pressure the more influence it has on the salt rejection.

Higher temperature
- The flux of water increases proportionally, because the constant A is temperature dependent.
- R is marginally influenced, because both constants A and B are temperature dependent.

2.2.5 Concentration Polarisation in Pressure Driven Processes

One important phenomenon to understand in membrane processes is concentration polarisation. Concentration polarisation is occurring when the solute concentration gradually increases close to the membrane surface. This is because the feed contains many different components which have different diffusion rates. A concentration gradient will form on both sides of the membrane within the boundary layer generated by the applied cross flow [*Mulder (1996), Baker (2004), Coulson (2002)*].

A higher concentration at the surface generates a flow back into the bulk feed. In reverse osmosis this higher concentration layer leads to an increased osmotic pressure which gives a lower permeate flow. Also the solute rejection is decreased.

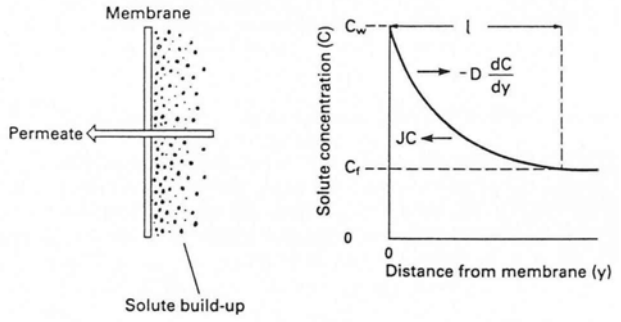


Figure 4. Overview of concentration polarisation [*Coulson (2002)*].

After a while, steady-state will be reached. At steady-state the flow rate through the membrane is equal to the flow from the bulk feed to the membrane surface minus the rate of back flow (from membrane surface to the bulk feed) according the following equation:

$$J(C - C_P) = -D\frac{dC}{dy}$$

C – solute concentration
C_p – permeate concentration

2.3 Vibratory Shear Enhanced Process, VSEP

Traditionally membranes are operated in a cross flow mode. This cross flow does not keep the membrane surface totally clean from particles and colloids and in the end the pores are plugged which leads to permeate flow reduction. To avoid this, New Logic® (Emeryville, Canada) developed a new kind of membrane system which is vibrated, Vibratory Shear Enhanced Process, VSEP [*http//www.vsep.com; Johnson, (2004)*].

In the VSEP it is possible to vary both frequency and amplitude to get the surface clean from suspended particulates and colloids. The sinusoidal shear waves of the membranes push the incoming particles from the surfaces and back into the bulk phase, resulting in a membrane surface clear for filtration. See figure 5. [*http//www.vsep.com; Johnson, (2004)*]

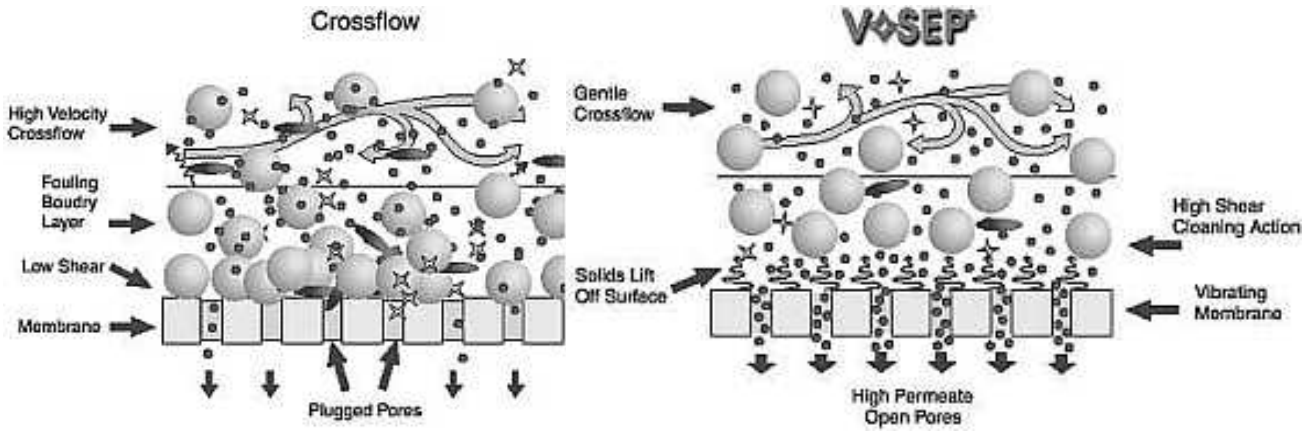


Figure 5. Comparison between traditional and VSEP membranes [*http//www.vsep.com*].

Figure 6 shows how the flow is going through the VSEP. The feed enters at the top of the membrane stack and passes the membranes as a cross flow. The concentrate is coming out at the bottom and the permeate at the top.

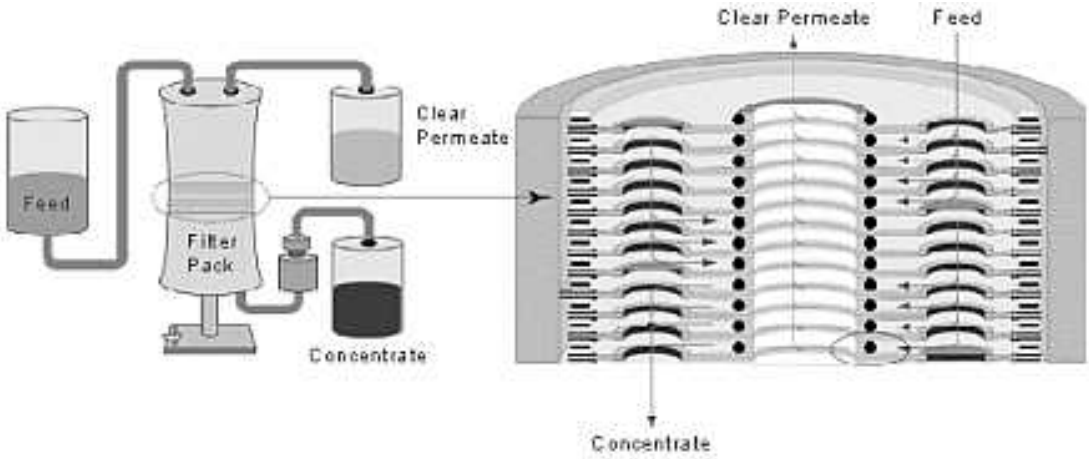


Figure 6. Scheme over flow in a VSEP [*http//www.vsep.com*].

In full-scale VSEP systems often a couple of VSEP units are used in a parallel, see figure 7.

Figure 7. VSEP in full-scale plant [*http//www.vsep.com*].

2.4 Fouling

Fouling occurs on and inside membranes by deposition, reaction, precipitation and/or microbiological processes [*Cardew (1998)*]. Fouling is the main cause of decreasing flux and lower quality of the desired product and therefore one of the most important parameters to control [*Baker (2004)*]. Many parameters will affect the fouling, such as concentration, temperature, pH, ionic strength, and specific interactions (e.g. hydrogen bonding dipole-dipole interactions) [*Mulder (1996)*].

2.4.1 Definition of Fouling

There are a lot of different definitions of fouling. According to Baker (2004) fouling can be divided into four types: scaling, silt (particles), bio fouling (bacteria) and organic fouling (grease, oil). Scaling gives most problems [*Baker (2004)*]. According to Mulder (1996) fouling can be divided in the following groups; organic precipitates (macromolecules, biological substances, etc), inorganic precipitates (metal hydroxides, calcium salts, etc) and particulates [*Mulder (1996)*].

Scaling is precipitation of metallic salts at the internal and/or external surface of the membrane [*Baker (2004)*, *Cardew (1998)*]. Scaling is a problem at high salt concentrations. The most common salts are: calcium carbonate ($CaCO_3$), calcium sulphate ($CaSO_4$), silica complexes, barium sulphate ($BaSO_4$), strontium sulphate ($SrSO_4$), and calcium fluoride (CaF_2). Calcium carbonate is the most common and silica complexes are most difficult to remove [*Baker (2004)*]. Also calcium phosphate ($Ca_3(PO_4)_2$) and iron hydroxide ($Fe(OH)_3$) are common salts that causes scaling [*Johnson, (2004)*]. It is the composition of the feed that influences which component is most common and gives most problems.

There is a method to calculate the risk of scaling:

$$\text{Recovery Rate} = \frac{\text{permeate flow rate}}{\text{feed flow rate}}$$

$$\text{Concentration Factor}\left(\text{CF}\right) = \frac{1}{1 - \text{Recovery Rate}}$$

If CF < 2 there is normally no scaling problem [*Baker (2004)*]. This value is low; in practical applications it is often necessary to get a higher concentration factor to use the resources in a economical way. Therefore it is important to find methods to delay fouling.

Silt is formed by suspended particles of all types that accumulate on the membrane surface. Some sources of silt are: organic colloids, iron corrosion products, precipitated iron hydroxide, algae or fine particles [*Baker (2004)*].

Bio fouling is growth of bacteria on the membrane surface. A lot of bacterial growth will lead to destruction of the membrane. Organic fouling depends on oil or grease onto the membrane surface [*Baker (2004)*].

2.4.2 Methods to Control Fouling

Operating parameters
Nowadays there are "low-fouling" membranes which have low surface energy and almost no charge at the surface. These prevent particles and molecules from adsorbing onto the membrane surface [*Johnson, (2004)*]. Other technical applications to avoid fouling are pre-filtration and cross-flow systems. It is also possible to avoid precipitation at the membrane through stopping the operation before solubility limits. [*Johnson, (2004)*].

pH
Fouling can be delayed by adding acid or alkali. The pH can prevent the precipitation of different compounds. For example the solubility of calcium carbonate will increase with lower pH-value. On the contrary silica has a higher solubility at a higher pH-value. A pH adjustment can also be necessary if the membrane is sensitive for high and/or low pH-values [*Cardew (1998)*].

Antiscalant
Antiscalant forms large complexes which prevent compounds from binding to the pores of the membrane. Some important antiscalants are substances with sulfonate, phosphonate or carboxylic acid as functional group. Antiscalant also leads to a higher solubility. However, it is important to remember that chemical additives make the system more complex and expensive. For example phosphonate-antiscalant can cause bio fouling since phosphorus increase the biological growth. Therefore non-chemical methods are most desirable [*Johnson, (2004)*].

Chelating agents
Addition of chelating agents slow down and neutralize fouling of precipitations. Already at low concentrations adsorption of ions and crystallization happens spontaneously at the surface of chelating agents, instead of at the surface of the membrane. Typical agents are carbon, alum and zeolites. [*Johnson, (2004)*].

Chlorination
To control bacterial growth, chlorination is necessary. Chlorine itself is a problem since membranes often are chlorine-sensitive [*Baker (2004)*].

2.4.3 Other Studies of Fouling

Six different studies of fouling
Rosenberger et al (2005) have compared results from six independent fouling projects in Europe. All projects are in the area of wastewater treatment with micro- or ultrafiltration membrane technology in different ways, either in effluent filtration for further polishing, or for direct sludge filtration in the membrane bio reactor concept. The scales of the projects differ from lab scale over pilot scale to full scale. One of the conclusions from these six projects is the importance of analysing the feed continuously, different substances influence the fouling and the membrane performance in different ways, and knowing the feed contents gives information about possible fouling.

Rosenberg et al (2005) concluded that to determine the composition of fouling, TOC, COD and polysaccharides are important parameters to analyse in the feed. These are

identified by photometric methods or if more detailed information is desired; size exclusion chromatography (SEC) and liquid chromatography - organic carbon detection (LC-OCD) are often used.

Today there are no standardised methods to analyse the fouling phenomenon, which complicates the comparison between different studies [*Rosenberger et al (2005)*].

One method for evaluation of fouling

In membrane bio reactor systems extracellular polymeric substances (EPS) are the major components in the fouling. To analyse the fouling mechanism Ye et al (2005) have used sodium alginate (a microbial polysaccharide) to study micro- and ultrafiltration. The examination was divided in two different parts, "short-term critical flux evaluation" and "long-term subcritical flux filtration". In the first part the membrane pore size, alginate concentration, cross flow velocity and the feed composition were studied. The conclusion from these tests were that for a more dense membrane the fouling was started earlier, a higher concentration of alginate gives a higher fouling rate, while a higher cross flow decrease the fouling rate and last the fouling is varying with the composition. During the second part the flux, pressure and the deposition of fouling over a longer time period were studied. After this the membranes were studied with FESEM (Field Emission Scanning Electron Microscopy), this shows that the alginate cake on the membrane surface differ in compactness after the different experiments.

The final conclusion is that instead of defining cleaning frequency for maximum transmembrane pressure value it should be better to base it on filtration time and fouling cake reversibility (the ability to dissolve the cake). So by using a model solution it was easier to determinate the roles of different parameters on the fouling phenomenon [*Ye et al (2005)*].

3. Materials and Methods

3.1 The Pilot Plant and Sample Collection

In figure 8 and table 3 the pilot plant is described. The black water is entering the pilot plant through a grinder feed pump (1). From the grinder feed pump the black water is passing a pre-treatment step, a drum screen (2) where the sludge is sent to the ordinary sewage system while the black water is sent to a stirred feed tank (3). The feed tank has two pH-meters, one conductivity meter and one level-tracer. A dosing pump (8), for dosing some additive (acid, antiscalant) from the dosing tank (9) is connected to the feed tank. From the feed tank the black water goes to the VSEP-unit (5). A feed pump (4) regulates the pressure and the cross-flow in the membrane stack by the feed pump frequency and a pressure valve (placed at the concentrate outlet of VSEP-unit). In the system there are also two smaller tanks, a cleaning tank (6) and a rinsing tank (7). These are used in the cleaning and flux test procedures.

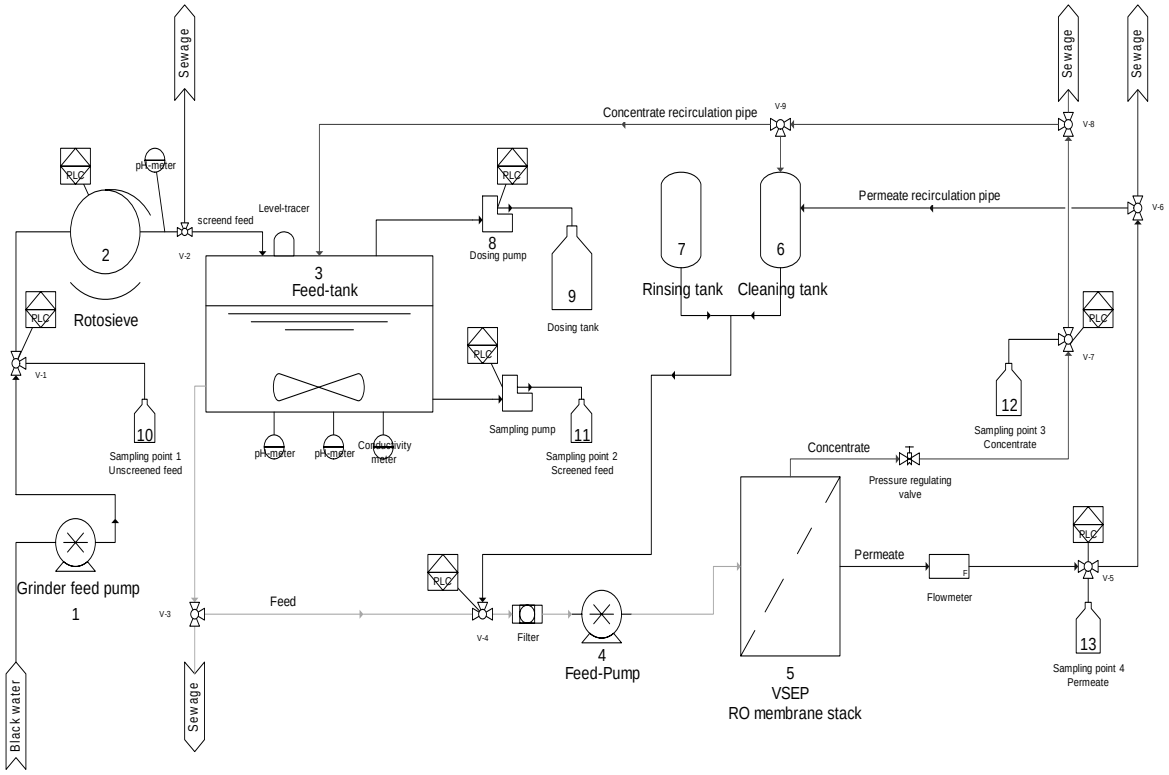


Figure 8. Outline of pilot plant [*Meins (2005)*].

Table 3: Description of pilot plant

In figure	Instrument	Specification
1	Grinder feed pump	Piraya S13-4D
2	Drum screen	Rotosieve; cut off level: 0.06 mm
3	Feed tank	600 litres
4	Feed pump	Hydracell
5	VSEP pilot unit	New Logic®, 38 RO membranes, LFC1, Hydranautics®
6	Cleaning tank	30 litres
7	Rinsing tank	30 litres
8	Dosing pump	ProMinent® gamma/L Solenoid Dosing Pump
9	Dosing tank	
10	Sampling point – feed	ProMinent® gamma/L Solenoid Dosing Pump
11	Sampling point – Screened feed	
12	Sampling point – Concentrate	
13	Sampling point – Permeate	
	pH-meter	ProMinent® Dulcometer DMT electrode PHER 112SE
	Conductivity meter	JUMO 640
	Flow meter	Promag 33A ENDRESS+HAUSSER
	Software	Programmable Logic Controll (PLC)

The samples were taken from the sample points (10-13 in figure 8) in different stages. Table 4 shows which samples which were taken in the different batches.

Fouling collection

After batch 1 and batch 4 fouling samples were collected (according chapter 3.4). After batch 4 some membranes were also allowed to soak in HCl, after cleaning manually and with NC4, to dissolve fouling that was stronger bound to the membrane surface. After batch 2, the washing liquid was analysed.

0

Table 4. Sampling points

F	feed	0	Start (at full feed tank)
C	concentrate	50	50% volume reduction*
P	permeate	75	75% volume reduction*
TSED	Final tank concentration	80	80% volume reduction*
f	filtrated (1 μm)	90	90% volume reduction*
A	average	M	Maximum volume reduction*

*With % volume reduction means how the volume in the tank has decrease compared to the start volume of feed.

Batch	**0**	**1**	**2**	**3**	**4**	**5**	**6**	**7**
F[1]	-	-	-	x	-	-	x	x
F-f[1]	-	-	-	x	-	-	x	x
F0	x	x	x	x	x	x	x	x
F0-f	-	x	x	x	x	x	x	x
C50	-	x	x	x	-	-	x	x
C50-f	-	x	x	x	-	-	x	x
C80	x	CM[2]	x	x	x	x	x	x
C80-f	x	CM-F[2]	x	x	x	x	x	x
C90	-	-	-	-	-	-	-	x
C90-f	-	-	-	-	-	-	-	x
CM	x	x	x	-	x	x	-	x
CM-f	x	x	x	-	x	x	-	x
TSED	-	x	x	x	x	x	x	x
TSED-f	-	x	x	x	x	x	x	x
P0	-	x	x	x	x	x	x	x
P50	-	x	x	x	-	-	x	x
P75	-	-	-	-	-	x	-	-
P80	-	PM[2]	x	x	x	x	x	x
P90	-	-	-	-	-	-	-	x
PM	x	x	x	-	x	x	-	x
PA	-	x	-	x	-	-	x	x

[1]Orginal black water before acid dosing

[2] For batch 1 the 80% volume reduction is the maximum volume reduction.

x: measured

-: not measured

3.2 LFC1- and LFC3-membranes

All information about the membranes is from the homepage of the membrane producer Hydranautics® in USA. [*http://www.hydranautics.com*]

LFC is an abbreviation for Low Fouling Composite. These membranes have a low surface charge and a hydrophilic membrane surface. The LFC3-membrane is made by Composite Polyamide, there is no information about LFC1, but probably it is made by the same polymer. The difference between LFC1 and LFC3 is that LFC3 is more hydrophilic, otherwise no more information can be found at the homepage.

According the product information at the homepage, LFC-membranes are used as follows:

LFC1	Municipal and industrial surface and waste water applications where low pressure is a priority
LFC3	Municipal and industrial surface and waste water applications where high rejection is required

Element Performance

Element Type	Min Salt Rejection, %	Nominal Salt Rejection, %	Permeate Flow m^3/d
LFC1	99.2	99.5	41.6
LFC3	99.5	99.6	35.96

The element performances are valid for a spiral wound membrane at the following conditions: 1500 ppm NaCl solution, 1.55 MPa applied pressure, operating temperature at 25°C, 15% permeate recovery and a pH-range of 6.5-7.

For general applications the following limitations are recommended for spiral wound membrane and can give a hint what limitation it is for a VSEP plant.

Maximum Applied Pressure, MPa	4.14
Maximum Operating Temperature, °C	45
Feedwater pH Range (at 45°C)	3.0-10.0
Maximum Chlorine Concentration, PPM	<0.1

3.3 Different Operations Mode

To examine how the fouling behaves during different operation mode 8 different batches were carried out.

Table 5. The settings for the different operation mode

Batch		Acid dosing	Amplitude [inch]	Initial pressure [bar]	Cross flow [L/min]
0	Drinking water	No	1/2	11.5	4.5
1	Higher cross-flow	No	1/2	11.5	6.0
2	-	No	1/2	11.5	4.5
3	Constant pH	Yes	1/2	11.5	4.5
4	Higher amplitude	No	3/4	11.5	4.5
5	Higher amplitude	No	1	11.5	4.5
6	Constant pH	Yes	1/2	11.5	4.5
7	Constant pH and Higher amplitude	Yes	3/4	18	4.5

More general information about the different batches is given in appendix A. For more detailed information about cleaning procedures, operation adjustment and problems with different batches, see Torben Meins' report [*Meins, 2005*].

3.4 Visual Analysis and Fouling Collection

To analyse what the fouling looked like and was composed of, the VSEP-stack was taken apart and afterwards assembled according to the routines in the manual of VSEP [*operator manual*]. Each of the 38 membranes was studied visually and photographed. The fouling was collected by taking each membrane plate and cleaning it with distilled water and with help of a bath sponge the fouling was taken away from the surface. The amount of distilled water was noted. After batch 1, fouling was collected from the whole stack and mixed and after batch 4, the fouling samples were divided in upper part (first 19 membranes) and a lower part (last 19 membranes). After batch 4, two membrane plates were exchanged because of a damage of the surface. The used membranes were used in the surface analyses, BET and ESEM.

The fouling samples were analysed on conductivity, pH and alkalinity. Samples for further analysis were also sent to ALcontrol AB Sverige. The fouling was also analysed in light microscope.

3.5 Chemical Analysis

3.5.1 Analyses at the Pilot Plant

At the pilot plant pH, alkalinity and conductivity were measured.

Table 6. Analytical instruments.

pH (in samples)	Shott,Handylab 1, pH-meter
pH (in feed tank)	Expandable ion analyzer EA 940, Orion Research
Conductivity	Shott, Handylab LF1, Conductometer

The alkalinity was calculated according to chapter 3.5.3.

3.5.2 Analyses at ALcontrol AB Sweden

The following analyses were carried out at ALcontrol Laboratories for all batches and fouling samples.

- pH
- Alkalinity (pH=5.5)
- Conductivity
- Suspended solids, SS
- Chemical Oxygen Demand, COD(Cr) and/or COD(Mn)
- Total Organic Carbon, TOC
- Fatty acids
- Total Phosphorus, P_{tot}
- Phosphate phosphorus, PO_4^{3-}-P
- Total Nitrogen, N_{tot}
- Ammonium, NH_4^+
- Potassium, K^+
- Calcium, Ca^{2+}
- Magnesium, Mg^{2+}
- Chloride, Cl^-
- Sodium, Na^+
- Total Sulphur, S_{tot}
- Sulphate, SO_4^{2-}

For some batches and fouling samples some additional analyses were carried out namely:

- Biological Oxygen Demand, BOD – for some fouling samples
- Nitrite + Nitrate and Nitrite nitrogen – for batch 5
- Dry solids, DS – fouling samples and for batch 6+7
- Red heating - for batch 6+7

For more information about analyse methods and inaccuracy in measurements, see appendix B.

3.6 Calculations

3.6.1 Concentration Factor, F

A concentration factor (CF) can be calculated in different ways, with concentrations and with volumes. The description of CF in chapter 2.5.1, is based on flow rate and this gives the same result as calculations based on volume.

The volume based concentration factor is obtained by dividing the start volume with the end volume, but it is also possible to take account of the produced permeate volume.

$$F = \frac{V_{start}}{V_{end}} \quad \text{or} \quad F = \frac{V_{start}}{\left(V_{start} - V_{permeate}\right)}$$

V_{start} – initial tank volume
V_{end} – tank volume after that the operation is finished
$V_{permeate}$ – total produced permeate volume

The concentration factor based on concentration is obtained by the *f*-factor:

$$F = \frac{1}{f}$$

The factor *f*, the fraction of original volume which is left after concentration, is calculated in the following:

$$f = \frac{\left(C_f - C_{P_a}\right)}{\left(C_{TSED} - C_{P_A}\right)}$$

C_f – feed initial concentration
C_{TSED} – final tank concentration
C_{PA} – final permeate concentration

The *f*-factor must be based on some compound which does not disappear from the system. In this case sodium and potassium are suitable elements to use.

3.6.2 Mass Balances

To determine the distribution of all substances, mass balances were calculated. A total mass balance is defined as:

Feed = Permeate + Concentrate + Loss

Loss includes precipitation in the system, leakage to air and influence of inaccuracy in sample collections and analyses.

With the *f*-factor it is possible to calculate a feed concentration based on the concentration in concentrate and in the permeate for all different compounds.

$$C_{f_{calc}} = f \times C_{TSED} + (1 - f) \times C_{P_A}$$

The mass balances can now be calculated in the following way:

- Amount in concentrate: $f \times \frac{C_{TSED}}{C_f}$
- Amount in permeate: $(1 - f) \times \frac{C_{P_A}}{C_f}$
- Losses (%): $\frac{(C_f - C_{f_{calc}})}{C_f} \times 100$

C_f – feed initial concentration
C_{TSED} – final tank concentration
C_{PA} – final permeate concentration
C_{fcalc} – feed concentration calculated

3.6.3 Alkalinity

The alkalinity is defined as: the amount of acid which is needed to decrease the pH to a certain pH. The alkalinity was calculated as follow:

$$\text{Alkalinity} = \frac{C_{HCl} \times V_{HCl}}{V_{sample}} \quad [\text{mmole/L}]$$

For a more mathematical description of the definition of alkalinity, see Marie-Catherine Coquin's report [*Coquin, 2005*].

3.7 Light Microscopy

Samples of fouling were analysed in a light microscope (Olympus BX40) in the laboratory at the wastewater treatment plant in Göteborg (Gryaab). Both frozen and un-frozen fouling samples from batch 1 and un-frozen samples from batch 4 were analyzed. Also some silt collected from the membrane surface was analysed. No preparations were carried out. To have some biological aggregates to compare with also activated sludge from the plant was analysed.

3.8 BET (Brauner, Emmet and Teller) - Method

To examine if there are any changes of the structure or on the surface area of the membrane the BET (Branauner, Emmet and Teller)-method was used. By this method the specific area and pore size distributions of the membrane were determined. The measurements were done by nitrogen adsorption-desorption at 77 K with a Tristar 3000 instrument (Micromeritics, Norcross, Georgia, USA) at the

department of Chemical Engineering – Applied Surface Chemistry, Chalmers University of Technology.

The BET surface area determinations were based on five experiments at relative pressure (p/p_0) of nitrogen in the range 0.05-0.20. The used cross-section area of the nitrogen adsorbate was 0.162 nm^2. Pore size distribution were determined from numerous points on both the adsorption and the desorption isotherms according to the BJH method [*Barett et al 1951*] using an adsorbate property factor of 0.953 nm.

The method is based on the knowledge about pressure and added volume of nitrogen gas. By knowing what area one nitrogen molecules occupied the total area can be calculated. BET-theory is based on the following equation:

$$\frac{p}{V \times (p_0 - p)} = \frac{1}{V_m \times C} + \frac{(C-1)}{V_m} \times \frac{p}{p_0}$$

V – amount of gas adsorbed at pressure p
V_m – amount of gas for a monolayer at surface
p_0 – saturation pressure at adsorption temperature
C – parameter (dimensionless) that describes the degree of interactions between adsorbed molecules and adsorbent. If the results correspond with the theory it should be a value between 1 and 100-300.

Then a plot with $\frac{p}{V \times (p_0 - p)}$ against $\frac{p}{p_0}$ gives the intercept as $\frac{1}{V_m \times C}$ and the slope as $\frac{(C-1)}{V_m}$ and from it is possible to calculate V_m. By knowing the specific area of one nitrogen molecules the total specific area can be calculated.

The BET-theory use some approximations.
- The surface of adsorbent is homogenous and impenetrable for the gas.
- The adsorption happens without dissociation of adsorbed molecules or interaction between adsorbed molecules.
- The heat of adsorptions is constant and independent of degree of coverage.

Even if these conditions are not fulfilled in the BET-theory, it has been shown that BET method gives very correct values for the specific area for most materials [*Coulson (2002), Löwendahl*].

Two samples were analysed, new LFC-3 membrane and a used and manually cleaned membrane. For preparation the membrane was cut in small pieces with a maximum diameter of 7 mm until a weight for 5 grams.

3.9 ESEM (Environmental Scanning Electron Microscope)

Analyses with Environmental Scanning Electron Microscope (ESEM) were done at the department Physics – Microscopy and Microanalyses, Chalmers University of Technology. The determination was made by FEI Quanta 200 ESEM FEG with an operation voltage 6 kV and 0.76 torr, spot size 3 and a spatial resolution of 10 nm. The gun was a field emission gun (FEG). During mapping and line scanning the operation voltage was 7 keV, spot size 5 and aperture size 100 μm.

The principle for elemental mapping and line scanning is that a laser beam with specific energy shoots electrons from the inner scales of the atoms. These electrons have specific energy for different atoms. By varying the energy of the laser beam it is possible to detect different elements. For specifications of different atoms see table 7.

Table 7. Energy peaks for different elements [*Petrova*]

Atom	**Energy peak [keV]***
Oxygen	0.52
Sodium	1.04
Phosphorus	2.01
Sulphur	2.31
Chlorine	2.62
Potassium	3.31
Calcium	3.63

*the width of the peaks is ±0.10-0.20 keV.

The only preparations of the samples were that the membranes were cut in small pieces and glued onto a special holder.

The following samples were analysed:

- *New LFC3 membrane* – to see what the surface structure the of an un-used membrane looks like.

- *LFC1 membrane from L-mode tests* – since it was no possibility to get a new LFC1-membrane, a used and cleaned membrane from the L-mode tests was analysed to see if there were any difference in structure between LFC1 and LFC3.

- *Used (after batch 4) and manually cleaned (most of the fouling with was removed by NC2(alkaline) and NC4 (acid)) membrane*– to determinate if there is any fouling left after cleaning and what it consists of.

- *"Turbulent zones"* – to control if the turbulence has affected the membrane surface.

- *"A grey spot"* – to determinate what the surface structure looked like at these spots which were occasionally observed on used membranes. These "grey spots" were also seen on new membranes.

4. Results

4.1 Different Operations Mode

A summary of the operational mode for different batches is shown in table 8. For more detailed information see appendices A and C.

Table 8. Short summary for all batches

Batch	Operation mode	Total residence time [hours]	Permeate flow ratio at 80% volume reduction [ml/(min·bar·°C)]	Final permeate flow ratio at maximum volume reduction [ml/(min·bar·°C)]	F (Na)
0	Drinking water	8.8	3.086	2.737	-
1	Higher cross-flow	15.9	0.316	0.307	5.9
2	No acid dosing	14	0.424	0.251	8.2
3	Constant pH=6	10.7	0.874	No reliable value*	9.4
4	Higher amplitude (3/4 inch)	14.1	1.204	0.215	15.7
5	Higher amplitude (1 inch)	13.3	0.357	0.223	8.1
6	Constant pH=6	12.8	1.267	0.850	17.0
7	Constant pH=6 and higher amplitude (3/4 inch)	11.5	1.174	0.853	11.4

*In batch 3 there was an operation problem at the end, and the latest reliable result is around 80% volume reduction.

Permeate flow ratio is calculated by divide the permeate flow rate with both feed pressure and feed temperature, to get a value that is independent of temperature and pressure. All presented flows are mean values of 5 numbers close to 80% respective final flow.

The results show that for batches with acid dosing at a constant pH of 6.0, permeate flow is still high at the end of test, giving the highest concentration factors and having the shortest total residence time.

4.2 Chemical Analysis

4.2.1 Fouling Analyses and calculations

The following calculations have been carried out for the different analysed components in the fouling:

$$\frac{\text{amount in fouling}}{\text{amount in feed}} = \frac{(\text{concentration of component in fouling}) \times (\text{total fouling sample volume})}{(\text{concentration of component in feed}) \times (\text{total fouling feed volume})}$$

$$\frac{\text{amount in fouling}}{\text{TSED}} = \frac{(\text{concentration of component in fouling}) \times (\text{total fouling sample volume})}{[\text{conc of comp in } (\text{un–filtr TSED} - \text{filtr TSED})] \times (\text{total TSED volume})}$$

TSED – precipitated amount in tank left over

$$\frac{\text{amount in fouling}}{\text{membrane area}} = \frac{(\text{concentration of component in fouling}) \times (\text{total fouling sample volume})}{(\text{area per membrane plate}) \times (\text{number of membrane plates})}$$

The area for each membrane is calculated to 0.0462 m^2. In the VSEP-stack there are 38 membranes. The sample volume is 3 litres if nothing else is given.

All titration curves for the fouling samples are found in appendix G.

Fouling after batch 1 (higher cross flow)
In tables 9a-b and figures 9-11 below the results from fouling analyses and calculations for batch 1 are presented.

Table 9a. Results from analyse of fouling after batch 1

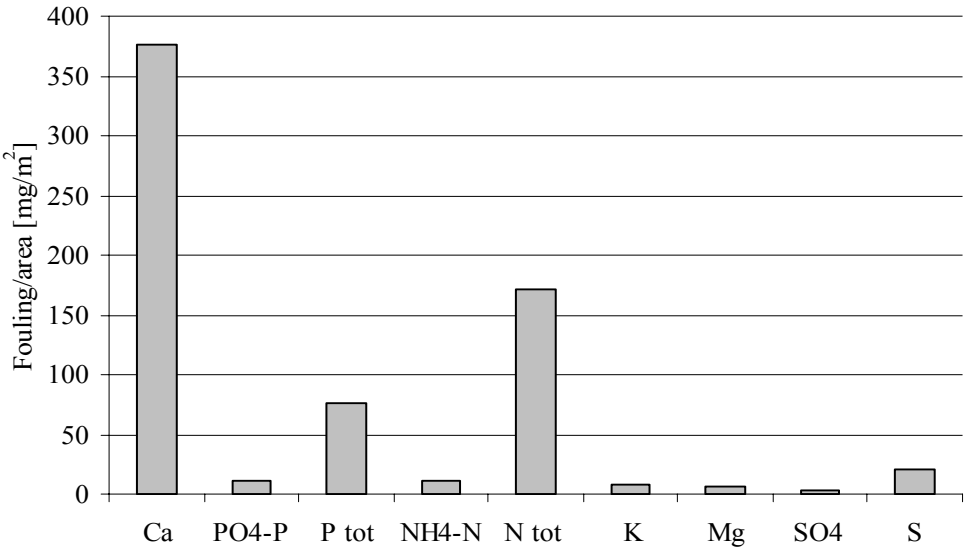
	BOD_7	**COD (Cr)**	**TOC**	**Fat**	**DS**	**SS**
Fouling,un-filtrated [mg/L]	1300	2400	410	140	0,18	2300
Fouling,filtrated [mg/L]	200	630	88	20	0.047	77
fouling/feed [%]	-	1.085	1.038	0.682	-	2.386
fouling/delta TSED [%]	-	13.793	17.672	2.438	-	6.721
fouling/area [mg/m2]	2223	4105	701	239	0.31	3934

*TSED – final tank concentration; delta TSED is the difference between TSED un-filtrated and TSED filtrated

Table 9b. Results from analyse of fouling after batch 1

	Ca	**PO_4-P**	**P tot**	**NH_4-N**	**N tot**	**K**	**Mg**	**SO_4**	**S**
Fouling,un-filtrated [mg/L]	220	6.4	45	6.6	100	4.2	4	2	12
Fouling,un-filtrated [mmole/L]	5.50	0.21	1.45	0.47	7.14	0.11	0.17	0.02	0.38
Fouling,filtrated [mg/L]	42	3.8	10	0.7	38	2.5	1.6	2.5	4.5
Fouling,filtrated [mmole/L]	1.05	0.12	0.32	0.05	2.71	0.06	0.07	0.03	0.14
fouling/feed [%]	3.570	0.368	1.424	0.035	0.253	0.055	0.396	0.040	0.345
fouling/delta TSED [%]	15.172	-	7.759	0.379	1.815	-	4.598	0.345	4.138
fouling/area [mg/m2]	376.27	10.95	76.96	11.29	171.03	7.18	6.84	3.42	20.52

*TSED – final tank concentration; delta TSED is the difference between TSED un-filtrated and TSED filtrated



Figur 9. Amount in fouling on the area of membrane surface.

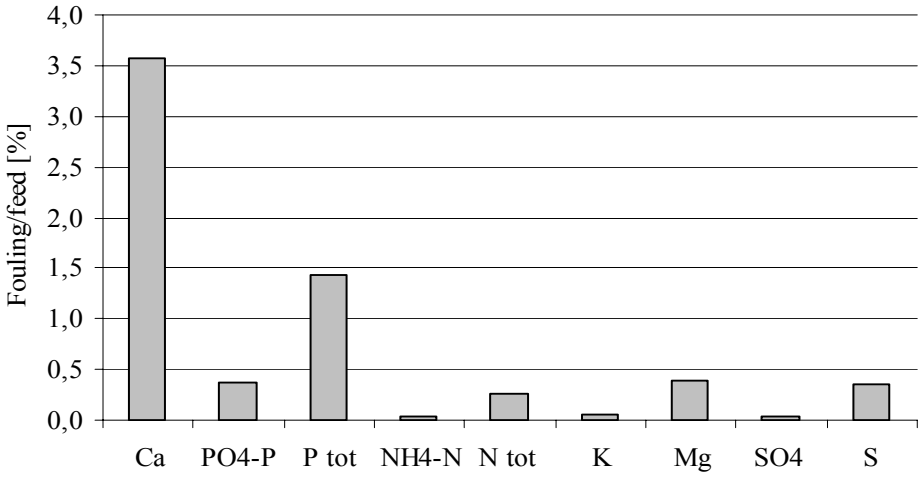


Figure 10. Percentage of feed found in the fouling .

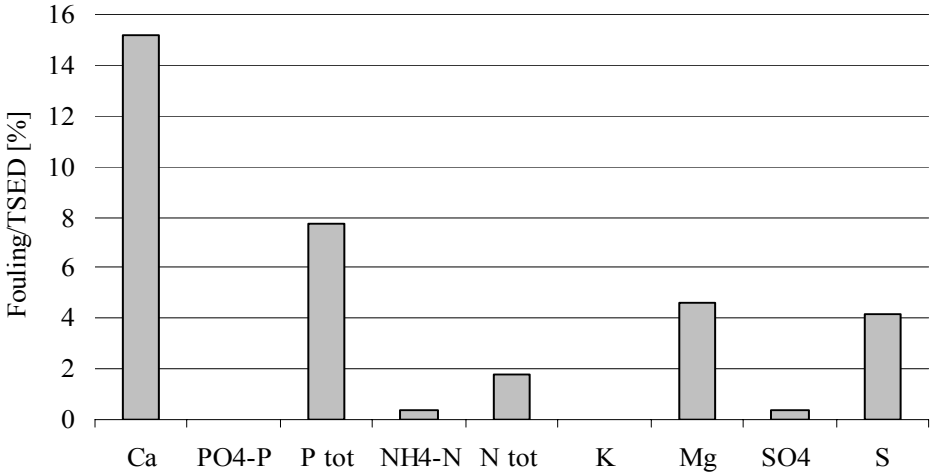


Figure 11. Percentage of amount in tank sediment found in the fouling.

From the inorganic fraction of the fouling the nutrients Ca, P_{tot} and N_{tot} are the main components, where calcium is the most common substance. The calculation of the percentage of the concentration in the feed found in the fouling shows that 3% of the calcium and 1.5% of total phosphorus are found in the fouling. If looking at the percentage of the particle bound amount in the tank left-over at the end of the concentration period as much as 15% of calcium and almost 8% of total phosphorus is found in the fouling. Also there are rather large amounts of total sulphur and magnesium in the fouling.

Notable is that the fouling contains a large amount organic matter. Approximately 1% of the COD and TOC in the feed get attached as fouling on the membrane surface. The high ratio between for COD and TOC in fouling and tank sediment must be an error in the analyse of TSED.

Fouling after batch 4 (higher amplitude)
In tables 10a-b and figures12-13 the results from fouling analyses after batch 4 are presented.

Table 10a Results from analyse of fouling after batch 4

	COD (Cr)	TOC	Fat	DS	SS
Filtrated first 19 [mg/L]	240	41	6,2	0.02	58
Un filtr. first 19 [mg/L]	830	130	72	0.07	530
fouling/area (mg/m2)	946	148	82	0.08	604
Filtrated last 19 [mg/L]	430	68	13	0.14	120
Un filtr. last 19 [mg/L]	1400	250	110	0.05	1100
fouling/area [mg/m2]	1596	285	125	0.06	1254
Total fouling/feed	1.086	1.145	0.768	-	1.587
Total fouling/delta TSED	2.212	3.167	1.091	-	2.310

*TSED – final tank concentration; delta TSED is the difference between TSED un-filtrated and TSED filtrated

Table 10b Results from analyse of fouling after batch 4

	Ca	PO_4-P	P tot	NH_4-N	N tot	K	Mg	SO_4	S
Filtrated first 19 [mg/L]	25	11	13	2.2	15	2.5	2	2	1.8
Filtrated first 19 [mmole/L]	0.625	0.355	0.419	0.157	1.071	0.064	0.083	0.021	0.056
Un filtr. first 19 [mg/L]	65	19	31	2.7	41	2.5	4.3	2	3.1
Un filtr. first 19 [mmole/L]	1.625	0.613	1	0.193	2.929	0.064	0.179	0.021	0.097
fouling/area [mg/m2]	74.11	21.66	35.35	3.08	46.75	2.85	4.90	2.28	3.53
Filtrated last 19 [mg/L]	49	19	27	3.9	32	2.5	4.1	2	3
Filtrated last 19 [mmole/L]	1.225	0.613	0.871	0.279	2.286	0.064	0.171	0.021	0.094
Un filtr. last 19 [mg/L]	140	28	66	9.8	73	3.9	9	2	5.7
Un filtr. last 19 [mmole/L]	3.5	0.903	2.129	0.7	5.214	0.1	0.375	0.021	0.178
fouling/area [mg/m2]	159.63	31.93	75.25	11.17	83.23	4.45	10.26	2.28	6.50
Total fouling/feed	3.327	2.975	3.411	0.082	0.515	0.101	1.336	0.169	0.293
Total fouling/delta TSED	5.891	78.333	6.124	-	4.750	2.667	2.639	-	7.333

*TSED – final tank concentration; delta TSED is the difference between TSED un-filtrated and TSED filtrated
? some error in the results from Alcontrol

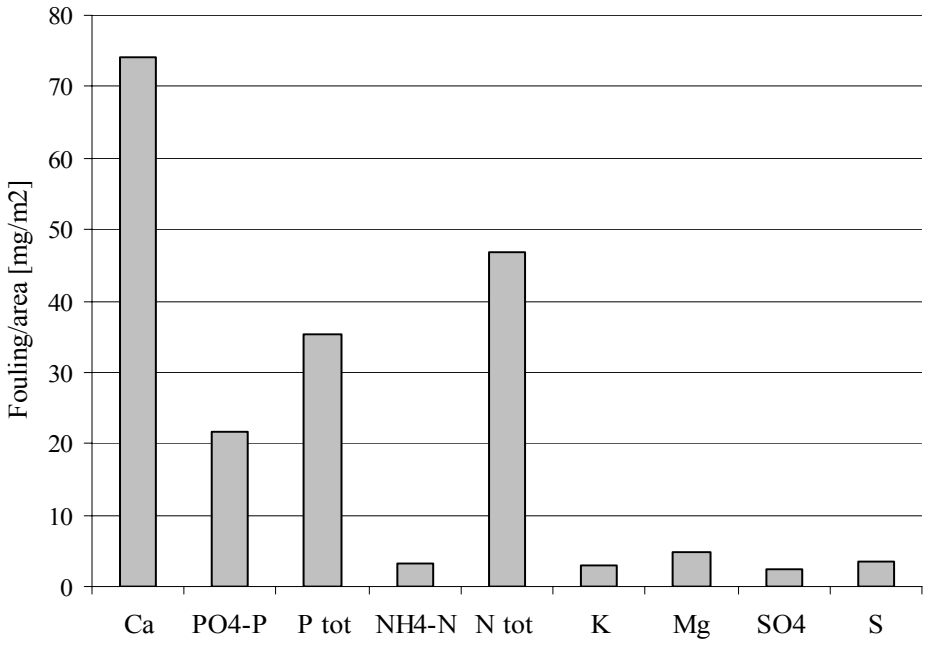


Figure 12a. Amount in fouling on the area of membrane surface in first 19 membranes.

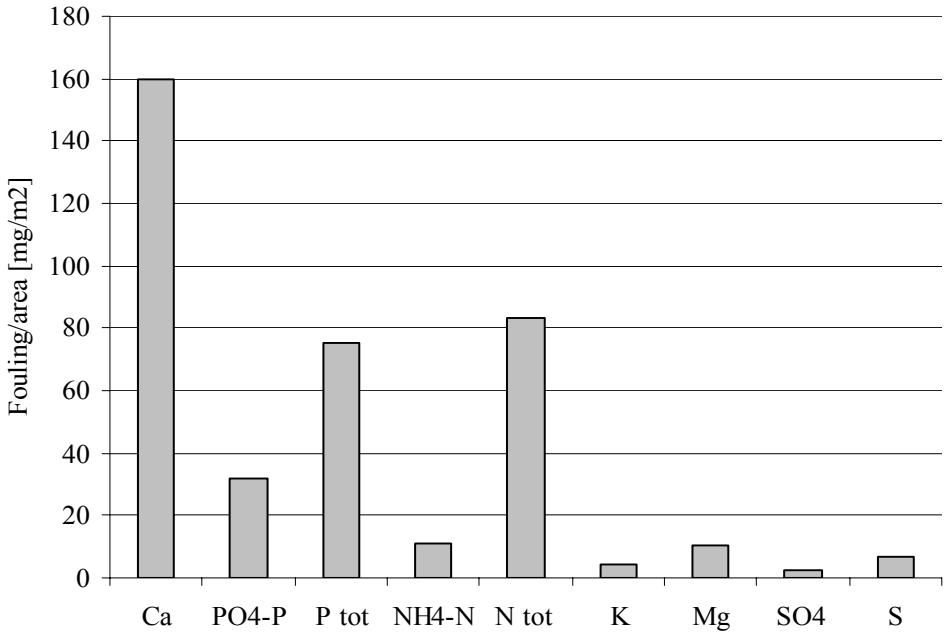


Figure 12b. Amount in fouling on the area of membrane surface in last 19 membranes.

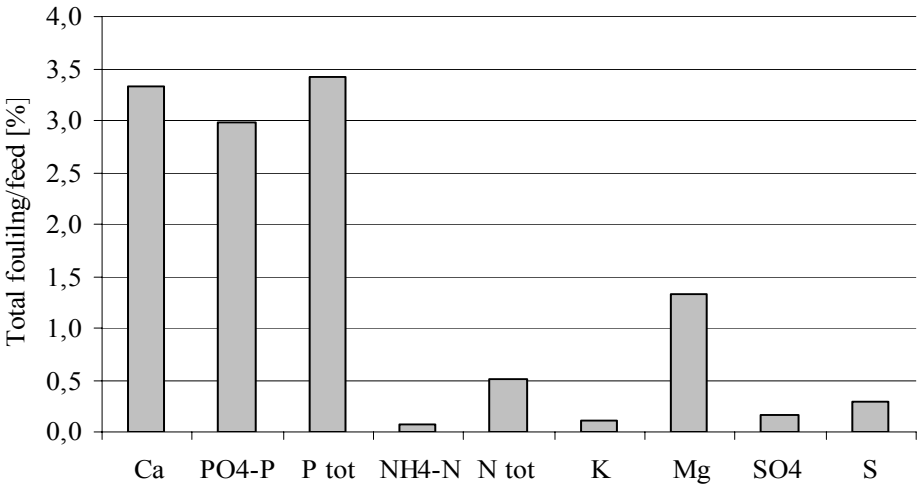


Figure 13. Percentage of feed found in the fouling .

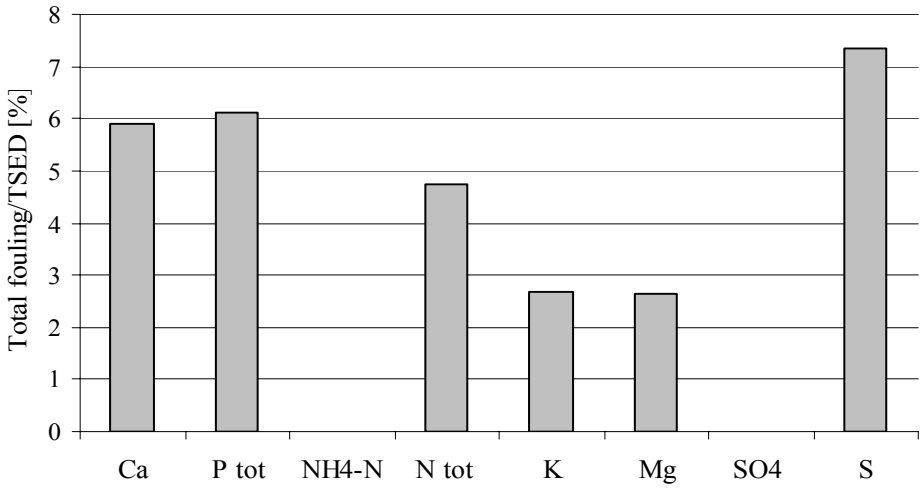


Figure 14. Percentage of the amount in tank sediment found in the fouling.

As for batch 1 the major substances of the fouling are Ca, P_{tot} or N_{tot}. However, in this sample the amount of total phosphorous was larger than after batch 1. Calcium in the fouling is still around 3%, of the total calcium amount in the feed.

Distribution of fouling into the VSEP stack
In table 11a-b the results from the examination of distribution of different components of fouling in VSEP-stack are presented.

Table 11a Distribution between the upper and the lower part of VSEP stack

	COD (Cr)	TOC	Fat	DS	SS
% in last 19	62.8	65.8	60.4	41.7	67.5
% in first 19	37.2	34.2	39.6	58.3	32.5

Table 11b Distribution between the upper and the lower part of VSEP stack

	Ca	PO_4-P	P tot	NH_4-N	N tot	K	Mg	SO_4	S
% in last 19	68.3	59.6	68.0	78.4	64.0	60.9	67.7	50.0	64.8
% in first 19	31.7	40.4	32.0	21.6	36.0	39.1	32.3	50.0	35.2

The ratios between different substances are approximately the same in the upper and lower part. The distribution of fouling between the upper and lower part of the VSEP-stack is in general that approximately 1/3 is in the upper part and 2/3 is in the lower. This is natural because the feed enters at the top and the flow gets more and more concentrated further down in the stack, and therefore also the probability for precipitation to occur increases.

Fouling dissolved in HCl
The manually cleaned membranes from batch 4 are first cleaned with distilled water and then with NC4, to analyse the substances that are strongest bound to the membrane surface. Five membrane plates were soaked in a HCl solution at a starting pH of 2.50 for one hour. To get pH 2.50, 16.15 mmol of HCl was added to 6 litres of distilled water. After 30 min the pH of the liquid had increased to 2.58 and therefore adjusted with 2.45 mmol of HCl back to a pH of 2.50. The final value of pH was 2.54.

The results of these analyses are presented in table 12 and figure 15.

Table 12 Analyses of fouling dissolved in HCl

	Ca	PO_4-P	P tot	NH_4-N	N tot	Mg	SO_4	S	TOC
Dissolved foul.in HCl [mg/L]	4	1.1	1.6	0.05	0.54	0.2	2	0.14	1
Dissolved foul.in HCl [mole/L]	0.100	0.035	0.052	0.004	0.039	0.008	0.021	0.004	-
fouling/area (mg/m2)	28.986	7.971	11.594	0.362	3.913	1.449	14.493	1.014	7.246

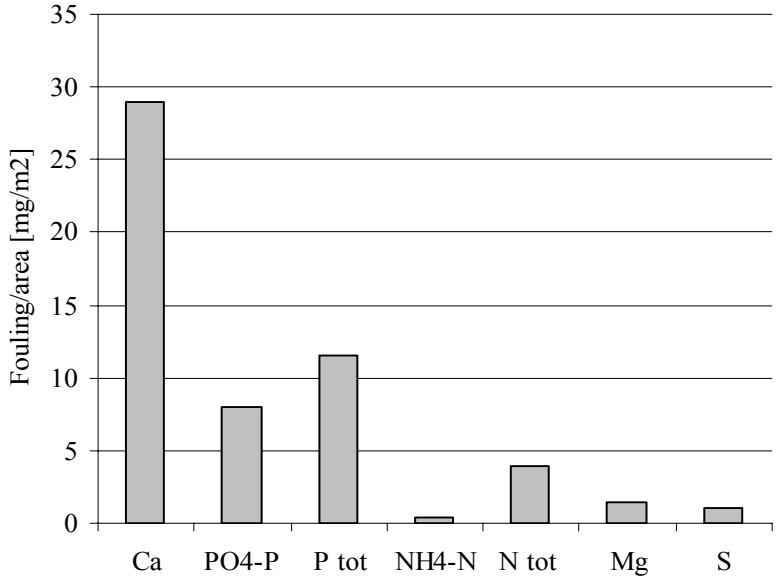


Figure 15. Distribution of different components in fouling on membrane surface.

Cleaning Liquid

After batch 2 the cleaning liquids were analysed. In tables 13a-b and figure 16 below are the results.

Table 13a. Analysis of which components are dissolved in washing liquid.

	pH	Conductivity [mS/m]	Alkalinity [mg/L]	BOD7 [mg/L]	COD(Cr) [mg/L]	TOC [mg/L]	Fat [mg/L]	SS [mg/L]
NC2 without fouling	12	150	640	<3	1900	150	380	7
NC2 with fouling	12	150	680	58	2200	180	380	43
difference	0	0	40	55	300	30	0	36
fouling/area (mg/m2)	-	-	-	941	5131	513	0	616
NC4 without fouling	<3.0	280	<1	<3	670	110	62	16
NC4 with fouling	<3.0	190	<1	<3	590	110	36	22
difference	0	-90	0	0	-80	0	-26	6
fouling/area (mg/m2)	-	-	-	0	-1368	0	-444.7	102.62

NC2 – high pH, NC4 – low pH and based on phosphoric acid

Table 13b. Analysis of which components are dissolved in washing liquid.

	Ca	**PO_4-P**	**P tot**	**NH_4-N**	**N tot**	**K**	**Mg**	**SO_4**	**S**
NC2 without fouling [mg/L]	24	0.91	0.8	0.07	40	42	2.3	25	79
NC2 with fouling [ml/L]	28	1.2	1.6	0.2	97	43	2.4	27	89
difference [mg/L]	4	0.29	0.8	0.13	57	1	0.1	2	10
difference [mole/L]	0.100	0.009	0.026	0.009	4.071	0.026	0.004	0.021	0.313
fouling/area (mg/m2)	68.41	4.96	13.68	2.22	974.87	17.10	1.71	34.21	171.03
NC4 without fouling [mg/L]	22	450	450	0.32	13	15	2.5	<10	43
NC4 with fouling [mg/L]	30	430	430	0.33	120	16	3.1	<10	39
difference [mg/L]	8	-20	-20	0.01	107	1	0.6	0	-4
difference [mole/L]	0.200	-0.645	-0.645	0.001	7.643	0.026	0.025	-	-0.125
fouling/area (mg/m2)	136.82	-342.06	-342.06	0.17	1830.02	17.10	10.26	0.00	-68.41

NC2 – high pH, NC4 – low pH and based on phosphoric acid

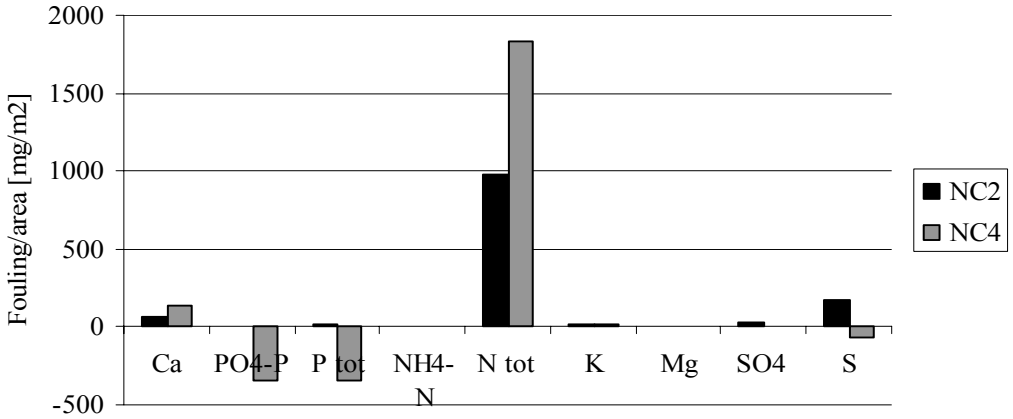


Figure 16. Amount of different components in fouling that was washed away with NC2 (high pH) and NC4 (low pH).

The washing procedure started with NC2 and thereafter with NC4. The negative values for PO_4-P and P_{tot} for NC4 are explained by the fact that NC4 contains phosphorus from the beginning and the small difference between before and after washing cannot be measured accurately. The composition of the washing liquids shows that fouling apart from organics contains a lot of calcium, nitrogen and sulphur.

NC2 was the starting cleaning liquid but the results show that it necessary to use NC4 to get away most calcium and nitrogen. NC2 takes away the organic substances as expected and NC4 is better in taking away the calcium scaling.

4.2.2 Mass Balances

For batches 2 and 4 only the amount in concentrate was calculated because there were no values for PA. For batch 5 a PA-value was estimated in the following way:

$$\left(\frac{P_0 + P_{75}}{2}\right)\times 0.75 + \left(\frac{P_{75} + P_{80}}{2}\right)\times 0.05 + \left(\frac{P_{80} + P_M}{2}\right)\times (0.2 - 0{,}105)$$

P concentration of a substance in the permeate

0 Start (at full feed tank) *80 80% volume reduction**

*75 75% volume reduction** *M Maximum volume reduction**

*With % volume reduction means how the volume in the tank has decrease compared to the start volume of feed.

In tables 14-17 the results from the calculations of mass balances are shown.

Table 14. Feed composition [mg/L]

	1	2	3	4	5	6	7
Suspended solids	610	650	750	650	630	1200	750
COD(Cr)	1400	1700	1600	1300	1500	2400	1500
COD(Mn)	190	260	220	230	180	380	
TOC	250	310	310	210	270	570	370
Fatty acids	130	170	160	150	140	330	120
Ptot	20	23	21	18	23	30	19
PO_4-P	11	18	10	10	11	21	14
Ntot	250	200	530	140	460	350	260
NH_4^+	120	190	150	97	150	140	120
N_{tot}−(NH_4-N)	130	10	380	43	310	210	140
K^+	48	55	50	40	50	51	49
Ca^{2+}	39	40	40	39	39	50	36
Mg^{2+}	6.4	6.9	8	6.3	7.3	8.5	6.1
Cl^-	150	110	120	82	95	95	210
Na^+	67	93	93	65	76	77	86
Sulfur	22	19	21	19	24	24	25
SO_4^{2-}	32	26	13	15	19	18	40
Alkalinity [3.5]*	0.0112	0.0162	0.013	0.0106	0.0152	0.0152	0.012
Alkalinity [5.5]*	0.007	0.01	0.0078	0.006	0.0096	0.008	0.0074
Alkalinity [3.5-5.5]*	0.0042	0.0062	0.0052	0.0046	0.0056	0.0072	0.0046

* (mmole/L)

For the analyses of the filtrated feed samples see appendix D1.When comparing the feed composition between the different batches it can be seen that the amount of total nitrogen and ammonium nitrogen differs a lot. Also the amount of sulphate differs for the different batches. It was also clear that batch 6 was more concentrated, especially for the organic substances, than the other batches.

Table 15. Amount in concentrate [%]

	1	2	3	4	5	6	7
Suspended solids	44.3	30.1	100.9	63.7	12.4	68.7	128.1
COD(Cr)	37.4	47.4	86.6	73.5	81.5		
TOC	52.6	78.8	123.8	100.1	100.6	66.1	118.0
Fatty acids	35.0	40.2	86.6	67.9	97.0	55.3	393.1
Ptot	59.1	63.7	81.2	70.8	75.2	76.5	105.7
PO_4-P	29.1	43.4	89.5	33.1	22.5	86.9	106.1
Ntot	67.5	262.7	52.3	100.1	120.8	89.2	80.6
NH_4^+	80.1	64.3	68.9	43.3	81.5	71.5	53.9
N_{tot}–(NH_4-N)	55.8	4031*	45.7	228.1	139.8	100.9	103.6
K^+	91.4	77.7	87.4	74.8	84.0	80.8	89.1
Ca^{2+}	69.2	67.2	103.9	75.1	88.7	77.7	111.6
Mg^{2+}	84.4	63.7	97.3	79.9	88.0	90.0	101.7
Cl^-	47.2	68.9	230.9	63.7	15.6	285.1	145.6
Na^+	93.2	77.5	83.7	77.4	86.1	84.1	89.4
Sulfur	84.4	115.7	91.4	80.5	87.5	81.0	87.4
SO_4^{2-}	110.7	150.4	237.8	0.8	31.2	235.5	87.4
Alkalinity [3.5]*	73.1	70.9	30.7	67.0	67.4	30.4	29.1
Alkalinity [5.5]*	72.3	68.4	4.1	57.3	65.0	4.4	1.2
Alkalinity [3.5-5.5]*	74.3	74.9	70.7	79.6	71.7	59.3	74.1

* (mmole/L)

*the differens between Ntot and NH4 in the feed is small and therefore the strange results.

Table 16. Amount in permeate [%]

	1	2	3	4	5	6	7
Suspended solids	0.0		0.0		0.0	0.0	0.0
COD(Cr)	1.8		3.1		3.0		3.0
TOC	1.4		5.2		3.9	4.6	3.5
Fatty acids	0.8		18.4		1.4	0.3	3.0
Ptot	0.3		4.7		1.7	3.8	2.0
PO_4-P	0.0		2.4		2.5	2.3	2.7
Ntot	6.0		18.5		8.3	8.3	6.7
NH_4^+	11.1		14.3		18.5	16.8	12.2
N_{tot}–(NH_4-N)	1.3		20.2		3.4	2.7	2.0
K^+	5.9		17.3		12.5	17.0	11.2
Ca^{2+}	0.8		3.4		1.2	2.8	1.5
Mg^{2+}	1.3		4.5		1.9	3.3	1.5
Cl^-	7.8		58.1		22.1	67.4	20.9
Na^+	6.8		16.3		13.9	15.9	10.6
Sulfur	2.0		5.1		4.6	4.3	1.9
SO_4^{2-}	5.2		13.7		23.1	10.5	4.6
Alkalinity [3.5]*	12.2		0.0		21.9	8.7	6.5
Alkalinity [5.5]*	13.7		3.9		25.6	7.1	3.7
Alkalinity [3.5-5.5]*	9.9		-5.8		15.6	10.5	10.9

* (mmole/L)

Table 17. Losses [%]

	1	2	3	4	5	6	7
Suspended solids	55.7		-0.9		87.6	31.3	-28.1
COD(Cr)	60.9		10.3		15.5		
TOC	46.0		-29.0		-4.5	29.3	-21.5
Fatty acids	64.1		-5.0		1.6	44.4	-296.1
Ptot	40.7		14.1		23.2	19.7	-7.8
PO_4-P	70.8		8.1		75.1	10.8	-8.8
Ntot	26.5		29.2		-29.2		
NH_4^+	8.8		16.8		0.0	11.7	34.0
N_{tot}−(NH_4-N)	42.9		34.1		-43.3	-3.6	-5.5
K^+	2.7		-4.7		3.5	2.2	-0.3
Ca^{2+}	29.9		-7.3		10.2	19.5	-13.1
Mg^{2+}	14.3		-1.7		10.1	6.6	-3.2
Cl^-	45.0		-189.0		62.3	-252.5	-66.5
Na^+	0.0		0.0		0.0	0.0	0.0
Sulfur	13.6		3.5		7.9	14.7	10.8
SO_4^{2-}	-15.9		-151.5		45.7	-146.0	8.1
Alkalinity [3.5]*	14.7		69.3		10.6	60.9	64.4
Alkalinity [5.5]*	14.0		92.0		9.4	88.5	95.1
Alkalinity [3.5-5.5]*	15.8		35.1		12.7	30.3	15.0

* (mmole/L)

In general acidified batches give higher yield than batches without acid apart from nitrogen and ammonium nitrogen where the amount in the concentrate was somewhat lower. Furthermore there are no clear relationships between the difference in feed compositions and difference in concentrate fractions. Notable is that batches 3 and 6 (with the same operating parameters) did not give the same yield of organic substances, where batch 6 gave a smaller amount in concentrate. For the other substances the values were in the same order of magnitude.

For detailed information for the compositions in feeds, concentrates, permeates and mass balances see appendices D1-3 and E.

Batch 0 – drinking water

In table 18 the result from the concentration of drinking water is shown.

Table 18. Result from the batch with drinking water

Batch 0	F0	PM	C0	C80	C80-f	CM	CM-f	TSED80	TSED80-f
Ca^{2+} (mg/l)	25	3.3	31	170	140	750	660	130	130

These results shows that the membrane also leaks calcium when drinking water is concentrated. An important conclusion is therefore that calcium carbonates starts to precipitate at 80% up-concentrations also in a feed with only drinking water, see also table 8.

4.2.3 Precipitation in Feed Tank

In figures 17 and 18 and in table 19 the results from the analyses of how the precipitations occur at different volume reduction levels and how this influences the permeate flow.

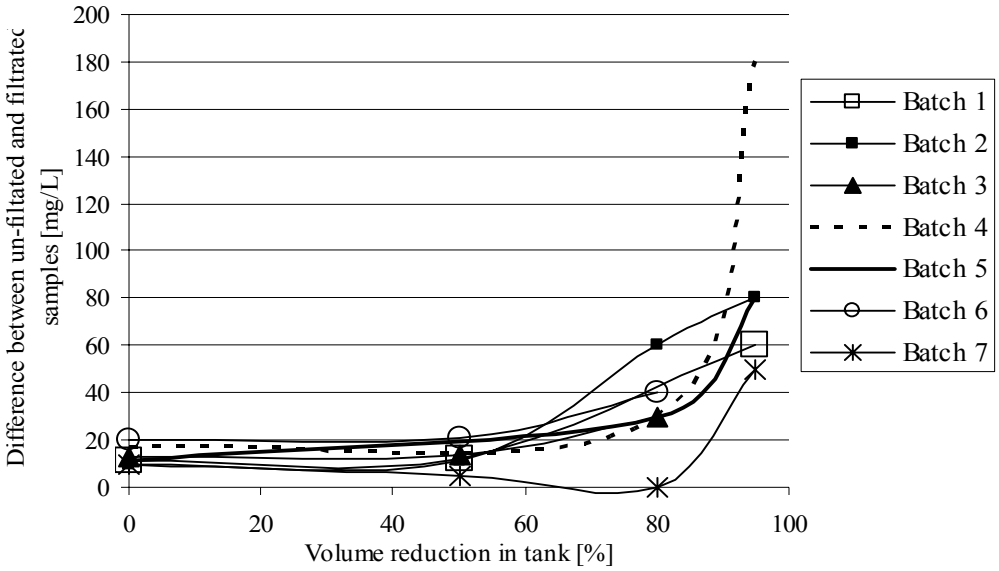


Figure 17. Precipitation of calcium at different volume reductions in the feed tank.

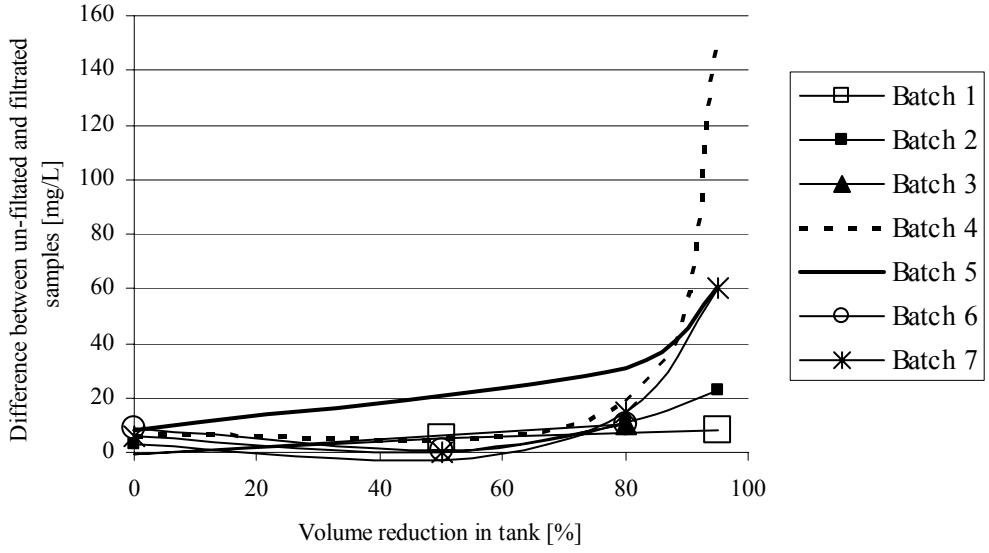


Figure 18. Precipitation of phosphate at different volume reduction in the feed tank.

Table 19. Permeate flow rate [ml/min] during operation.

Volume reduction in tank	Batch 1	Batch 2	Batch 3	Batch 4	Batch 5	Batch 6	Batch 7
0	920	820	670	910	570	470	800
50	440	668	649	745	1202	656	722
80	127	195	601	514	134	716	572
95	118	112	671	108	120	630	600

It is very clear that when the amount of precipitation in the tank increases the permeate flow rate decreases. For the acidified batches (3,6,7) the amount of precipitations is not so high as in the other batches (1,2,4,5). Notable is that there was relatively little precipitation in batch 4 up to a high volume reduction whereupon it suddenly increased very fast.

Data for all un-filtated and filtrated samples are shown in appendix F

In table 20 the results of the comparison between the amount of calcium, magnesium and phosphates are summarized.

Table 20. Ratio between some substances (molar ratio).

	80% volume reduction		**Maximum volume reduction**	
Batch	**Ca^{+} - PO_4-P**	**Mg^{2+} - PO_4-P**	**Ca^{+} - PO_4-P**	**Mg^{2+} - PO_4-P**
1	-	-	17,77	2,93
2	12,92	1,78	8,24	2,72
3	7,11	0,78	-	-
4	3,95	0,43	2,86	0,94
5	2,29	0,50	3,11	1,22
6	9,48	0,78	-	-
7	-	-	1,97	-

The theoretical molar ratio between calcium and phosphate ($Ca_3(PO_4)_2$), and magnesium and phosphate ($Mg_3(PO_4)_2$) is 1.5. In the result above ratio is much higher, which indicates that there probably is a lot of calcium carbonate ($CaCO_3$) in the precipitations. A high ratio indicates much calcium carbonate while low ratio indicates much calcium phosphate.

4.2.4 Permeate Quality

Table 21 shows the Swedish National requirement for the most important parameters when wastewater is to be discharged into receiving waters. The lower values is applied for large wastewater treatment plant or for sensitive recipients. It is interesting to know if it is possible to discharge the permeate directly or whether further treatment is necessary.

Table 21. Set effluent limits for discharge into water [*Naturvårdsverket*].

Parameter	**Maximum concentration allowed (mg/l)**
Total Nitrogen	15/-
Total Phosphorus	0.5/0.3
COD(Cr)	70
BOD_7	15/10
TOC*	23

*value calculated for comparison purposes with the ratio between COD(Cr) and TOC from results from ALcontrol. The factor was calculated to 3.1

The result from the analysis of permeate is presented in table 22.

Table 22. Average values for permeate.

	COD (Cr)	**BOD7**	**TOC**	**P-tot**	**N-tot**
Batch	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*
1	<30	-	4.2	0.07	18
2	-	-	35	-	-
3	55	-	18	1.1	110
4	-	-	20	-	54
5	52	-	12	0.43	44
6	-	90	28	1.2	31
7	-	50	14	0.42	19

The concentration of COD(Cr) is below the set effluent limits for all batches. For TOC it is very close to the limits and for batch 2 and batch 6 the content in permeate is more than is allowed to discharge. For nitrogen is the content in the permeate is much over the limitations for all batches and the content must decrease to fulfil the demands. For total phosphorus the concentration is close to the limitation for batch 5 and 7 and whereas it is much higher for batch 3 and 6.

In Table 23 the concentrations in the permeate at the start of the experiments are shown.

Table 23. Start values for permeate.

	TOC	**P-tot**	**N-tot**
Batch	*mg/l*	*mg/l*	*mg/l*
1	4.1	-	-
2	19	-	-
3	17	-	95
4	10	-	12
5	11	0.37	20
6	20	0.72	18
7	10	0.26	10

The results show that the nitrogen concentration is over the limit already at the beginning Of the experiment. Also TOC and total phosphorus concentrations were relatively high already at the beginning. Therefore also the average value is over the limit since the higher concentration of concentrate gives decreasing quality in the permeate, see figures 19-21 below.

One factor to examine is the ratio between permeate concentration (P) divided by concentrate concentration (C). When this ratio increases it indicates that fouling occurs and fouling contributes to decreasing permeate quality. See appendix H for more values.

In figure 19-21 shows how the permeate quality change with the volume reduction.

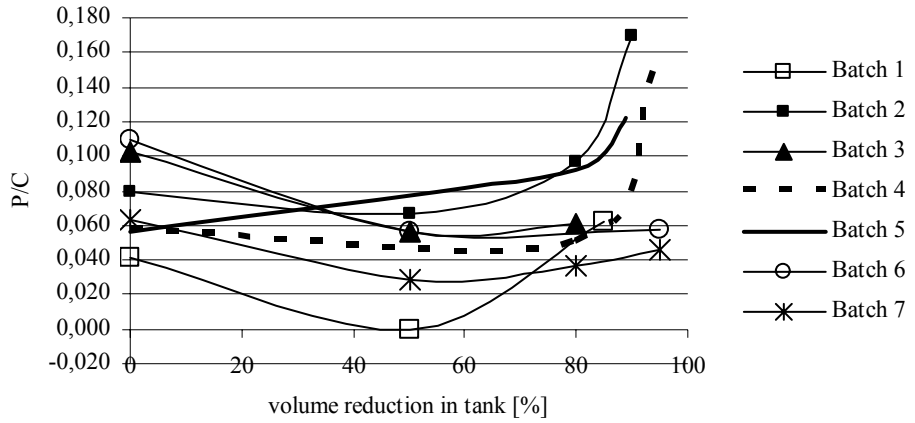


Figure 19. Permeate-concentrate ratio of potassium.

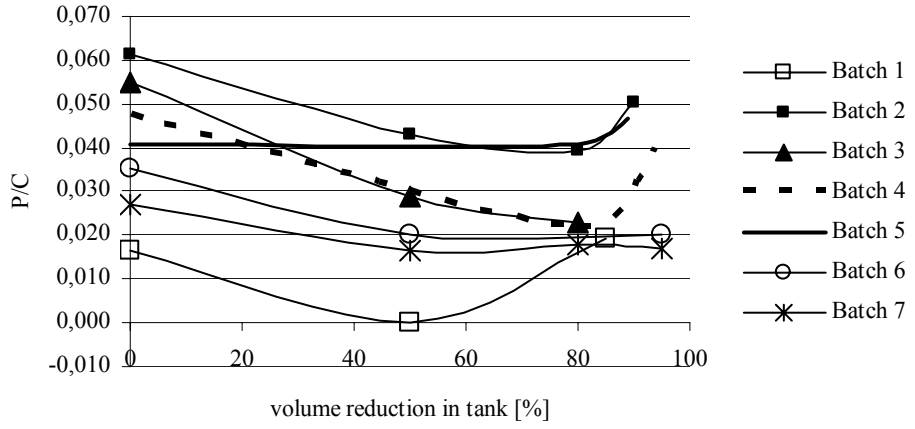


Figure 20. Permeate-concentrate ratio of TOC.

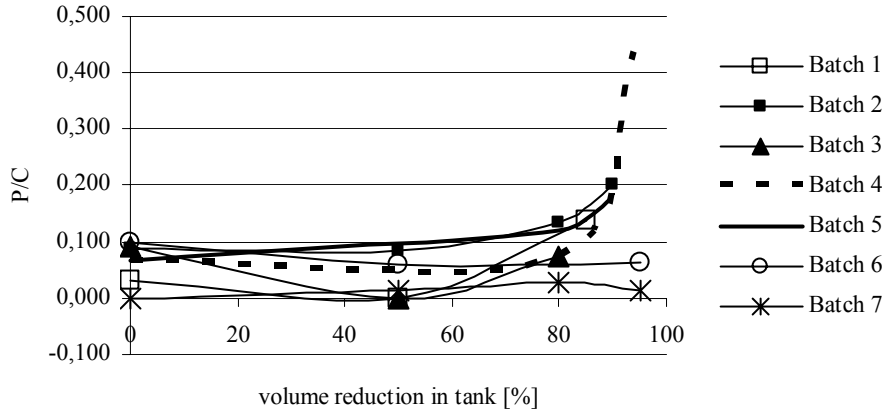


Figure 21. Permeate-concentrate ratio of ammonium.

4.2.5 Red Heating and Dry Solids

In table 24 and 25 is the results of red heating and dry solids from ALcontrol.

Table 24 Results from read heating analyse

	Red heating rest	**Red heating loss**	**Red Heating rest**
	% of DS	*% of susp*	*% of susp*
Batch 6			
TSED	-	83	17
Batch 7			
F	42.4	>95	<5
F0	40.2	>95	<5
C50	-	>95	<5
C80	38.7	>95	<5
C90	34.8	93	7
TSED	23.9	>95	<5

Red heating loss is a measure of the amount of the organic substances, and red heating rest is the amount that is left after burning, i.e. the inorganic frcation. This means that suspended solids in the feed consists of more than 95% of organic substances. In dry solids the amount of inorganic substances should be approximately the same during the whole operation though the analyses indicate a decreasing trend with higher concentration degrees. An explanation could be that there are precipitations of salts on walls and at the bottom of the tank or in the pipes or at the membranes.

Table 25. Dry solids concentration in some samples of batch 6 and batch 7.

	Dry Solid
	mg/L
Batch 6	
TSED	27100
Batch 7	
F	1110
F0	1120
C80	440
C80-F	5980
C90	14600
C90-F	10000
TSED	20000

The result for C80 is probably wrong since the concentration in the filtrated sample is higher than in the un-filtrated. Otherwise the results are reasonable by the fact that the purpose of the membrane operation is to remove water from the sewage to get it more concentrated.

4.3 Visual Analysis

The pictures from the membrane sheets from the stack which was taken apart the first time after batch 1 are shown in figure 22-26. The stack has been soaking in water for three days and before opening of the stack the membranes were flushed with water.

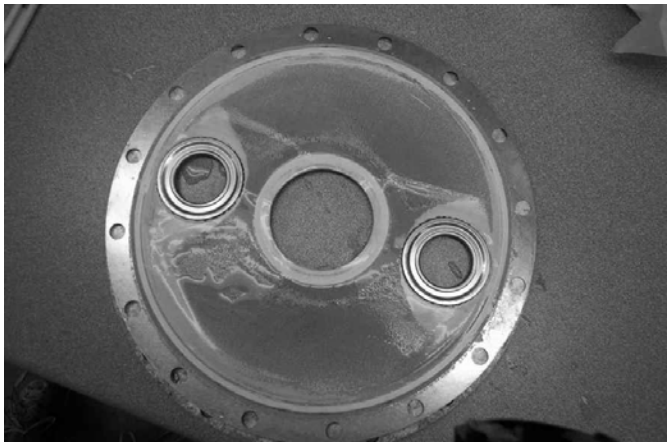


Figure 22. Most common appearance of the membranes.

Most of the bottom membranes are looking like this. Near the inflow the turbulence is higher and therefore there is no fouling.

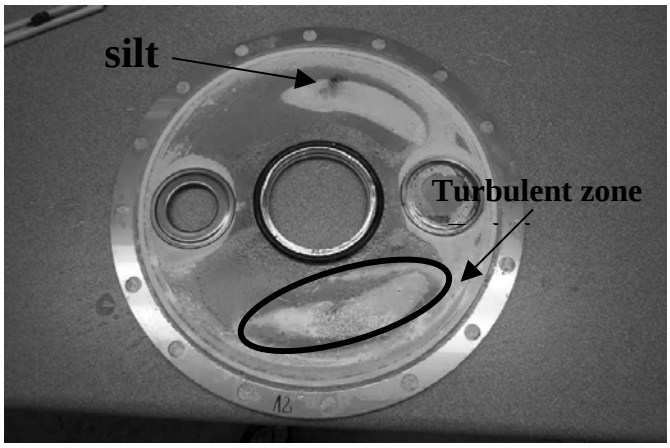


Figure 23. Areas with turbulent zones.

This is an upper membrane. It shows that turbulent zones have less fouling. The darker greyish areas show some kind of silt.

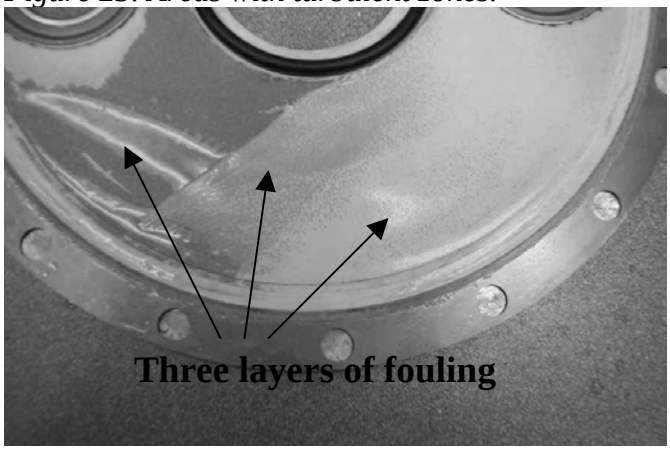
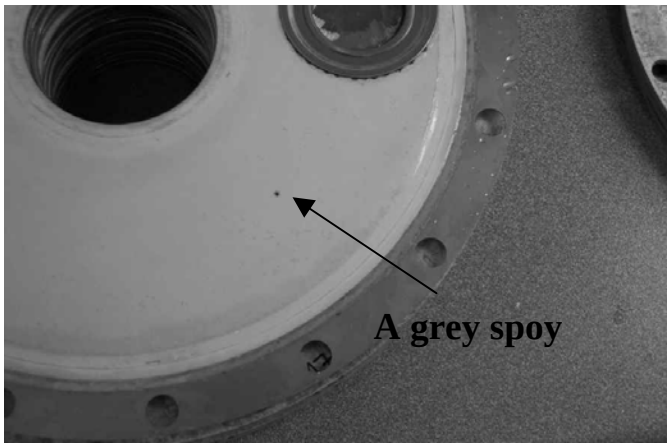


Figure 24. Fouling in three different layers

Here it is very clear how the fouling is built up. First there is a loose layer. After that, there are two layers which are more strongly bound to the membrane surface. This is the real fouling which can only be removed by cleaning and can perhaps be avoid by preparation of the feed.



This kind of grey spots occurred on the surface of about 5 of the tested membranes. They could also be observed on completely new membrane plates.

Figure 25. Grey spot on membrane surface.

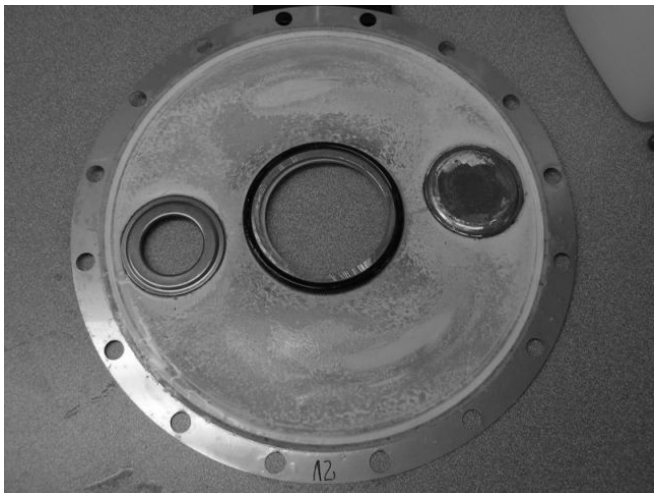
On washed membranes these light grey zones, “turbulent zones” are visible and indicates that the membrane surface has been altered.

Figure 26. Turbulent zones give change in colour on membrane surface.

In figure 27-30 pictures from the stack taken apart the second time, after batch 4 are shown. This time the membrane stack was opened directly after the operation and just quickly flushed with water after running. Figure 31 and 32 show the membrane sheets after an operation with constant pH (pH = 6).

Here there is just a very thin loose layer on the membrane surface compared to the first time the VSEP stack was opened up.

Figure 27. Most common appearance of the membranes.

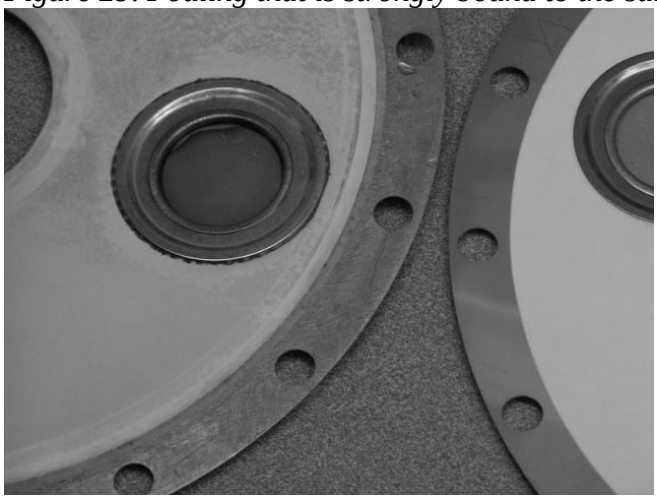


Also this time there are areas where the flow was more turbulent with no visible fouling.

Figure 28. Area with turbulent zones.

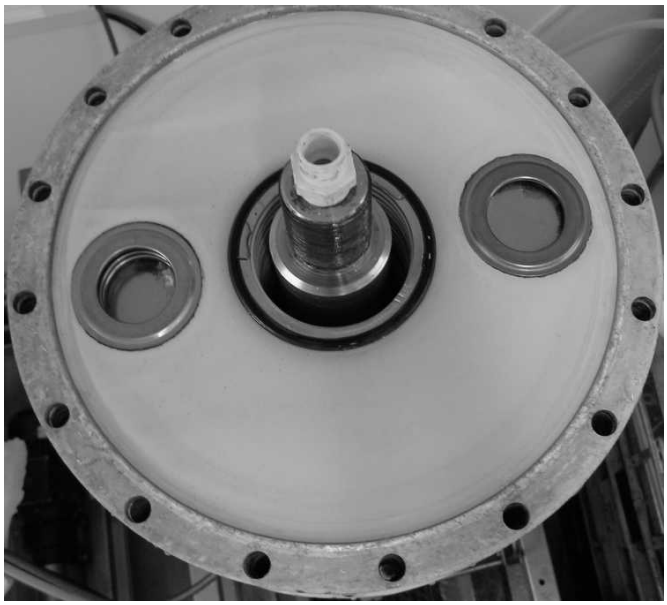
The fouling layer close to the surface is strongly bound and difficult to remove.

Figure 29. Fouling that is strongly bound to the surface.



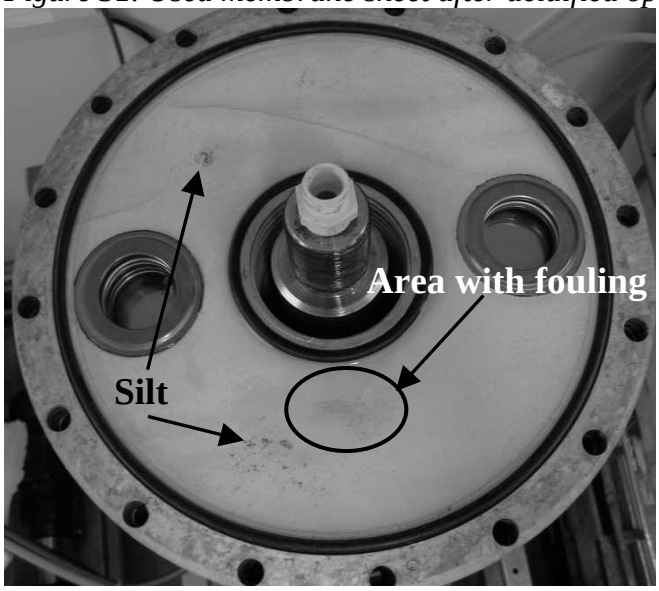
To the right on the picture, a new membrane plate is shown, which is much whiter than the used one to the left.

Figure 30. Used membrane to the left and new membrane to the right.



After an acidified operation almost all visible fouling disappeared.

Figure 31. Used membrane sheet after acidified operation.



At some membrane sheets there is still some fouling and silt on the membrane surface.

Figure 32. Used membrane sheet after acidified operation.

4.4 Analyse by Light Microscope

The light microscope images show that the fouling was composed of biomass with a similar appearance as bioflocs. It contained a lot of bacteria that were swimming around. Unfortunately the bacteria were not so well visible on the pictures, but in figure 33 the bacteria are marked with white rings. The picture shows a lot of flocs of organic material.

To have something to compare with, an activated sludge sample from the Rya wastewater treatment plan was also analysed. The pictures from the different samples look very similar (figures 34 and 35).

When observing frozen samples the difference between organic and inorganic substances were clearly visible, but it did not show any bacterial activity since the bacteria probably died during the storage in the fridge.

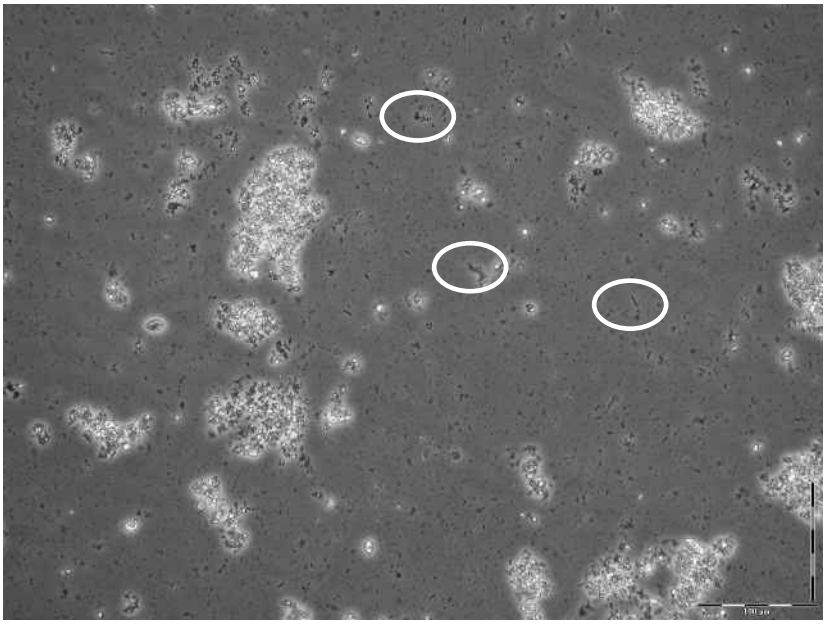


Figure 33. (20x) Fouling with bacteria (pointed out with white rings).

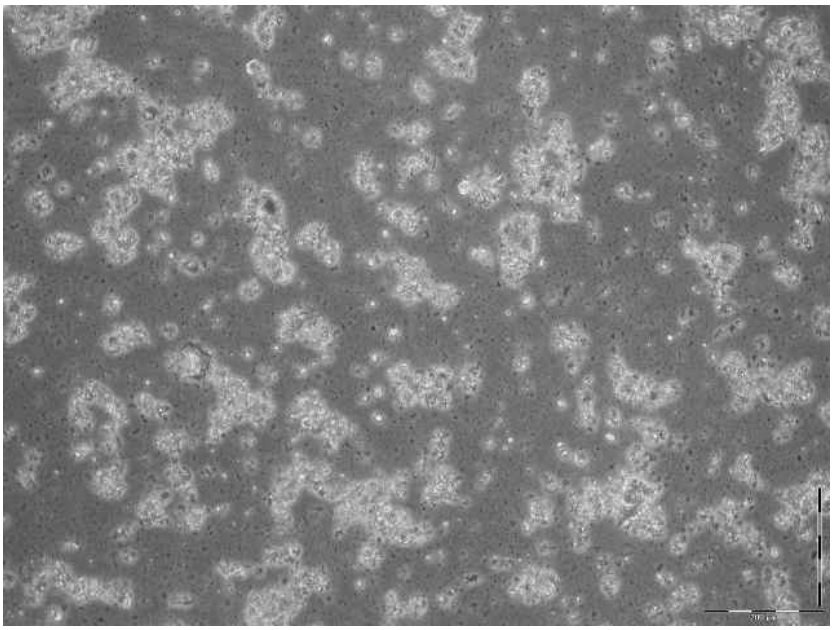


Figure 34. (10x) Fouling with a similar appearance as biofilm or activated sludge flocs.

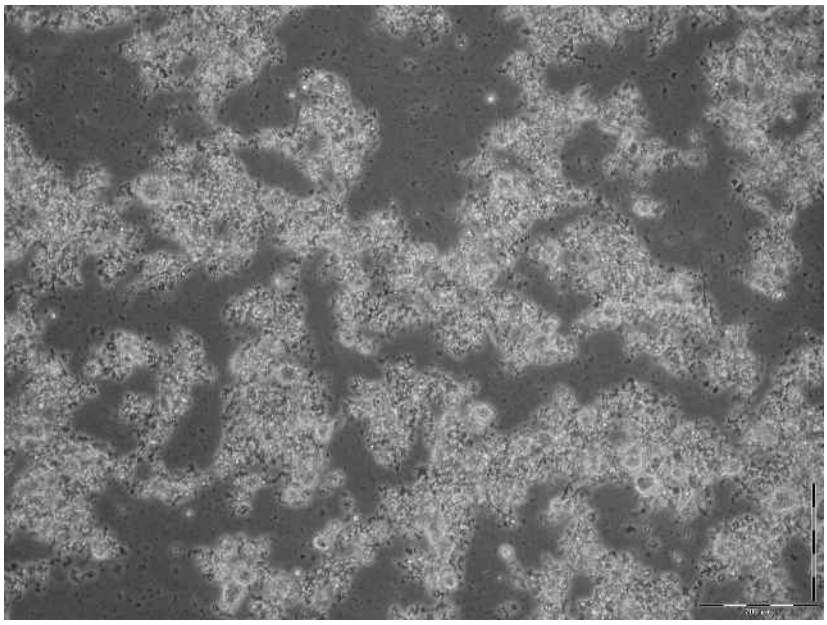


Figure 35. (10x) Activated sludge from the Rya wastewater treatment plant.

The inorganic crystals are visible as dark spots (figures 36 and 37). At higher magnification the inorganic crystals are seen very clearly (figure 38). As compared to the sludge from the Rya wastewater treatment plant, the fouling contained a larger amount of inorganic particles (figure 37).

The silt that is found at the membrane surface are composed of crystals in many different colours, but also some organic substances are attached to the inorganic material (figure 39). The crystals are not soluble in hydrochloric acid, either at a concentration of ~0.25 or ~1.0 M. This indicates that these are sand particles (silicates).

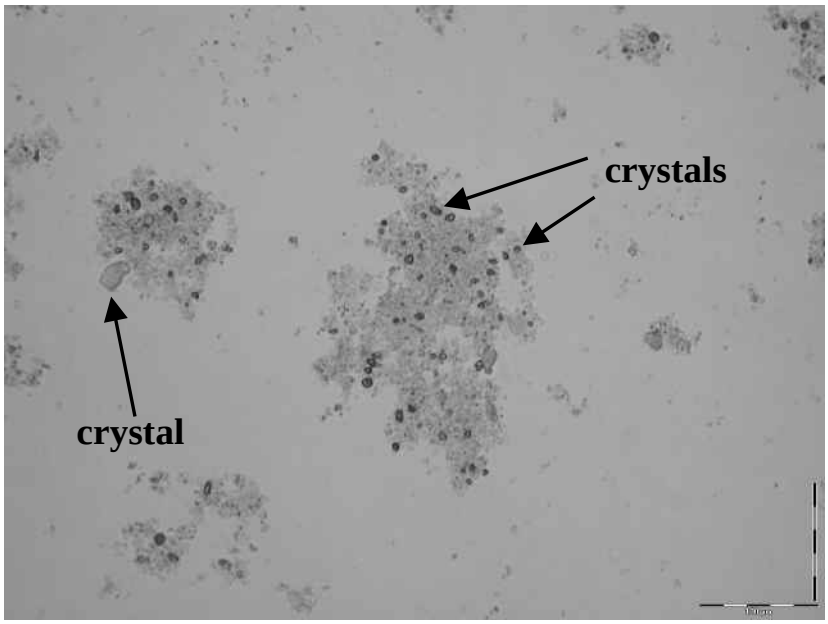


Figure 36. (20x) Fouling, dark spots are inorganic crystals.

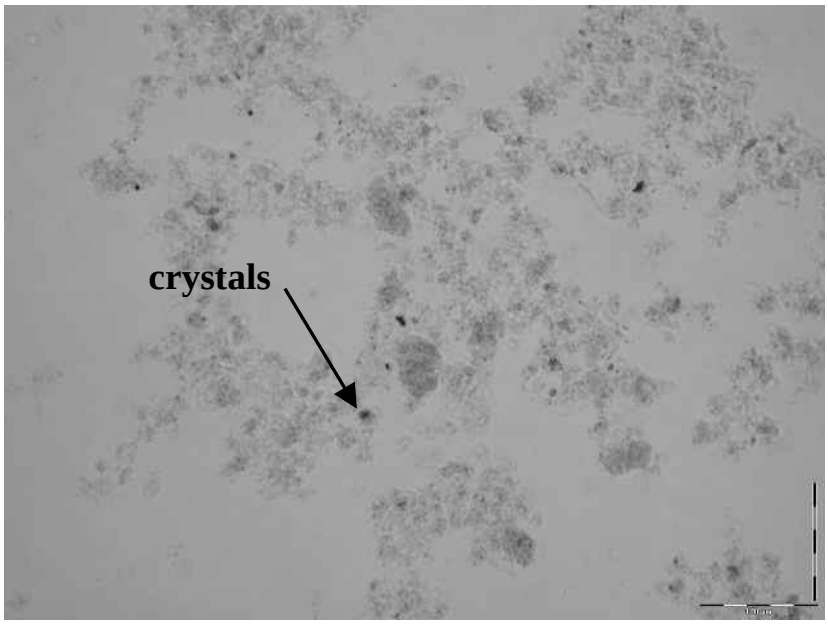


Figure 37. 20x) Sludge from the Rya wastewater treatment plant.

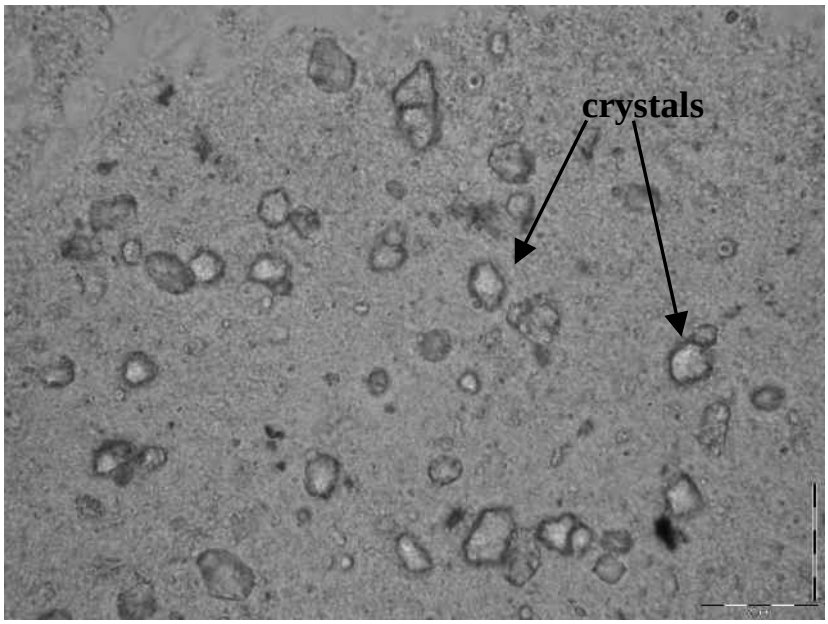


Figure 38. (100x) Fouling, inorganic crystals.

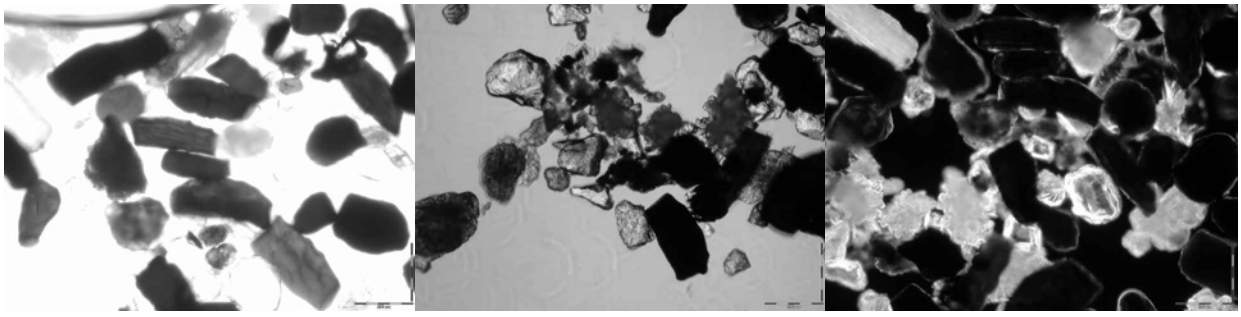


Figure 39. 10x Silt, crystals in many different colours (red, green, white or transparent).

4.5 BET (Brauner, Emmet and Teller) - Method

There is a difference in surface area between two measurements of the same membrane samples. However, the surface area is always significant smaller for the used membrane than for the new one. The difference is 7.6% respective 6.3%, so it is in the same range.

Table 26. BET results from duplicator test of the same samples

Sample	**BET Surface Area (m^2/g)**	**BET Surface Area (m^2/g)**
New LFC3-membrane	4.5493 ± 0.0123	4.7636 ± 0.0184
Used LFC1-membrane	4.2017 ± 0.0102	4.4654 ± 0.0107

The curves in appendix I show that the distribution of pore size is large. In the desorption curves there is a weak peak around 250 Å for the new membrane and around 300 Å for the used membrane. The wide distribution is probably depending in the fact that the membrane has a more porous structure on the back side that could influence the results.

4.6 ESEM (Environmental Scanning Electron Microscope)

Pictures of the membrane surface

Comparison between LFC 1 and LFC 3.

The structure of the LFC 1 and LFC 3 membranes are very similar, but the LFC 3 membrane looks more coarse. It is assumed that the similarity is sufficient to use the new and un-used LFC3-membranes as reference image to see if there is some change in structure after usage (figure 40a,b). In figure 41 it is shown very clearly that the cleaned membrane was not totally clean, white rings indicate small crystals and a larger crystal can also be seen. These crystals are situated relatively far apart and should probably not affect the permeate flow through the membrane but they could potentially act as seeds for fouling.

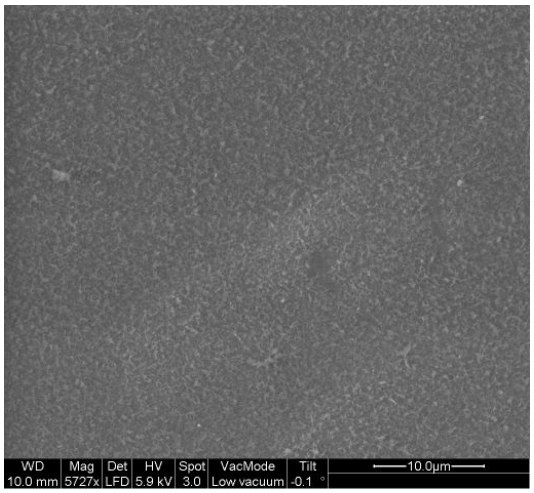


Figure 40a. Image of LFC 1 membrane.

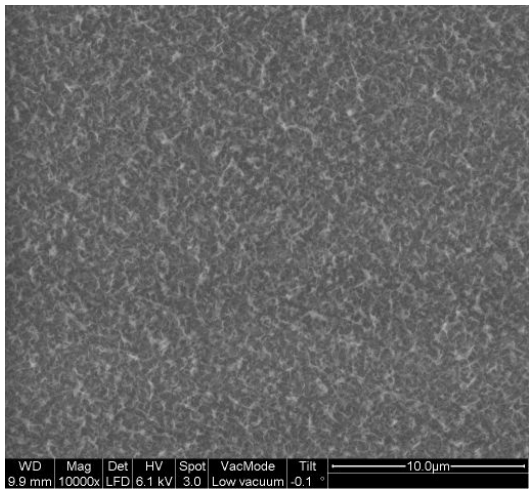


Figure 40b. Image of LFC 3 membrane.

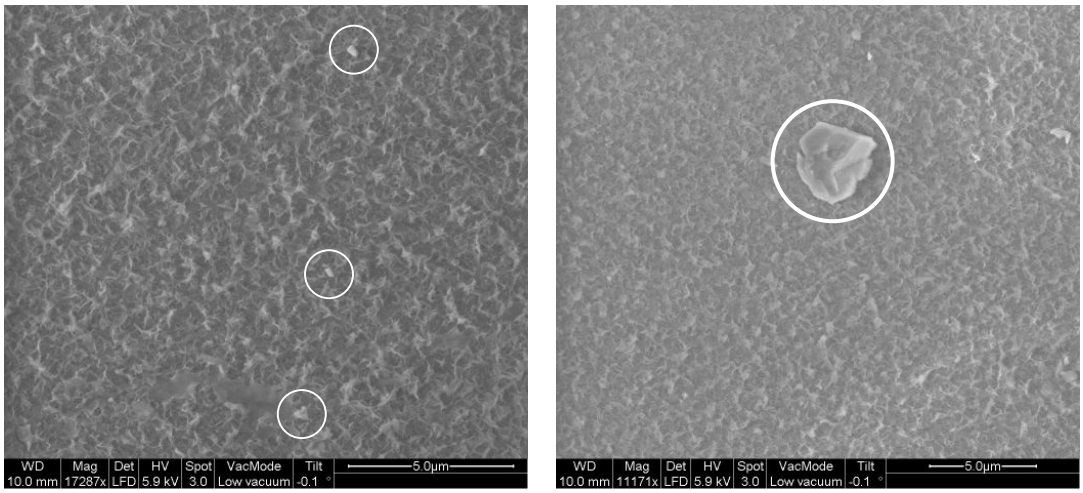


Figure 41. "Clean" LFC1-membrane (from L-mode experiments) with small crystals and a large crystal.

Used membrane in P-mode tests

The surface of the used membrane (after batch 4) looks smoother than the original membrane surface (figure 42). There are a lot of bacteria at the surface, which indicates that the smoother surface is a thin bio film. The white rings point out some of the bacteria, while the whiter spots in the white squares presumably are dust particles that have contaminated the membrane after operation. Figure 43 shows a larger magnification of the bacteria and shows a hole in the biofilm and some small inorganic crystals were also found between the bacteria. The bacteria are pointed out with white broken rings in the figure. Figure 44 shows a hole in the membrane surface.

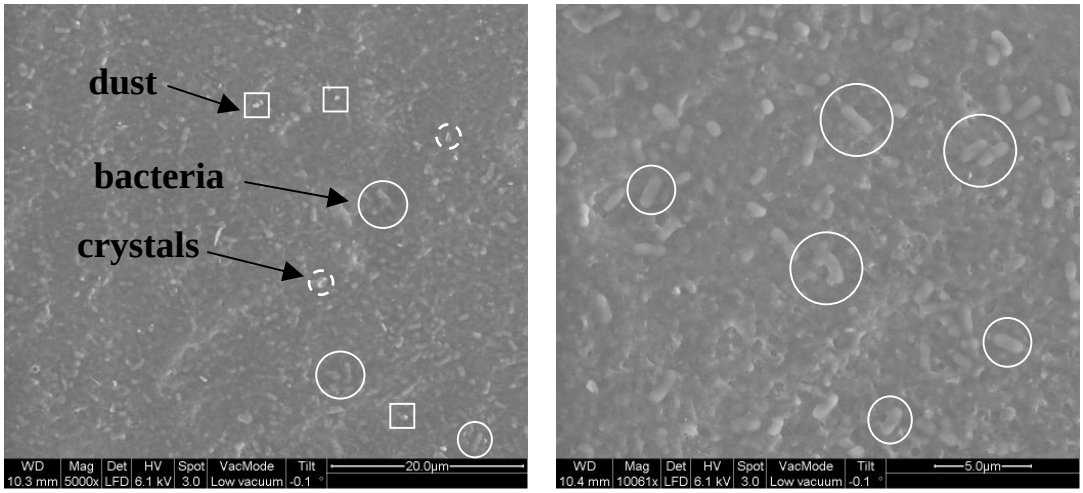


Figure 42. Used and manually cleaned LFC1 membrane (after batch 4) the most loosely bound fouling has been taken away.

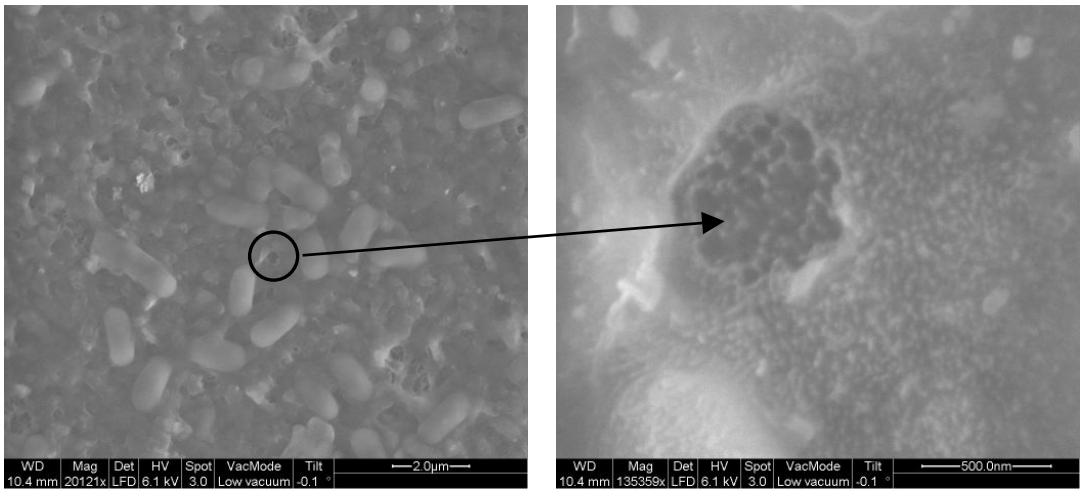


Figure 43. Used and manually cleaned LFC1 membrane (after batch 4). Bacteria and a hole in the membrabe can be seen.

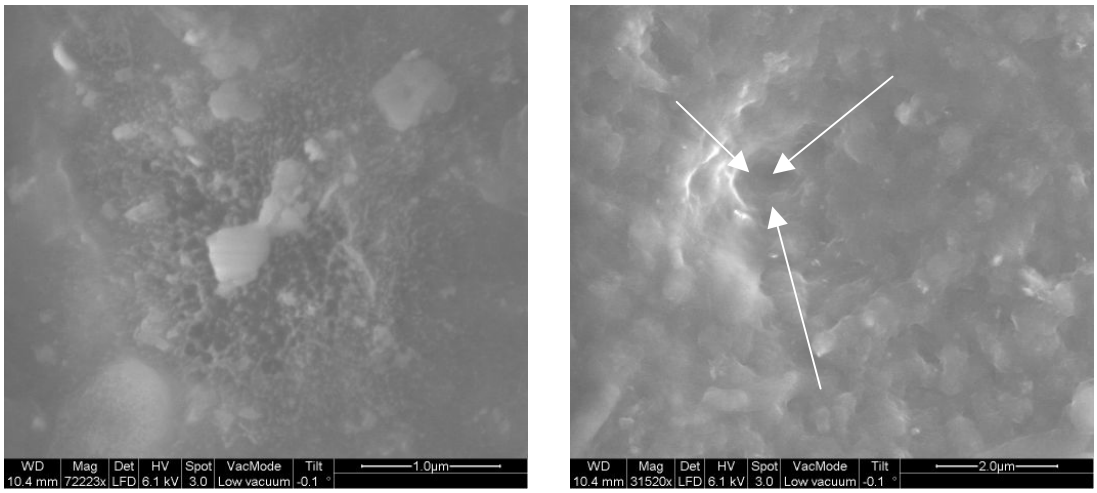


Figure 44. Used and manually cleaned LFC1 membrane (after batch 4); bacteria and a hole can be seen.

Turbulent zones

In the area exposed to turbulent flow the surface has a totally different structure (figure 45 a-b). It is difficult to see if there is some kind of film attached to the surface or if there is a change in surface structure due to erosion of the membrane. In some places in the turbulent zone there are still a lot of bacteria attached (figure 46). This shows that the bacteria are strongly bound to the membrane.

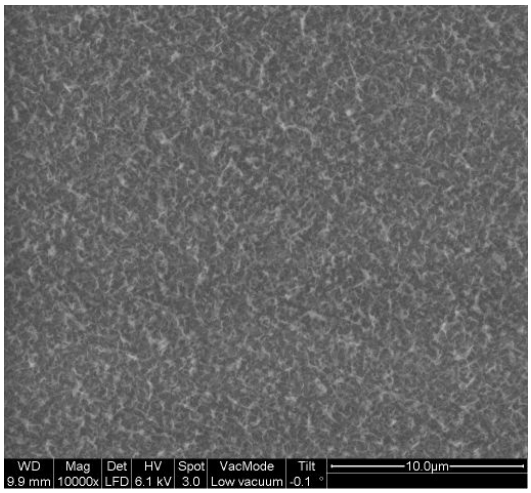


Figure 45a LFC 3 – reference surface

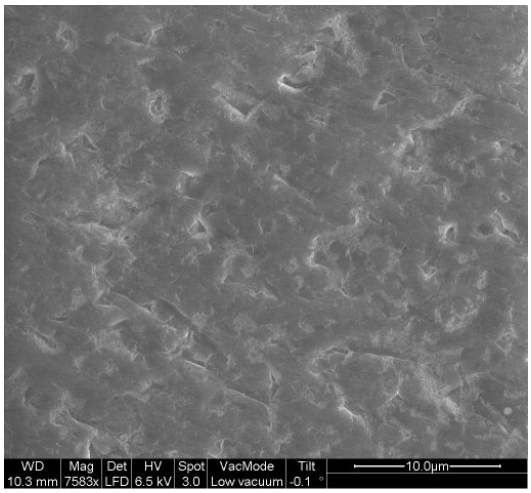
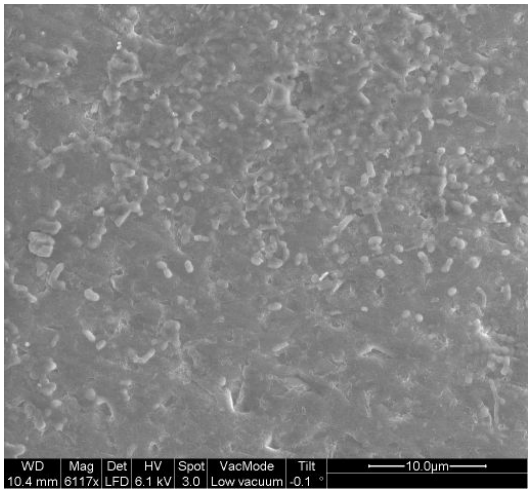


Figure 45b "Turbulent zone"



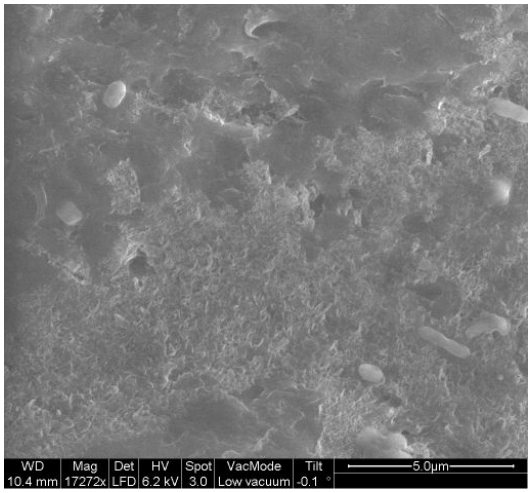


Figure 46"Turbulent zone"

Grey spots

On some new and used membranes grey spots were observed. These grey spots are holes or cavities (figure 47). Figure 48 is taken at the edge of the hole, and shows three different levels of the surface (the white lines lay at the edge). However, it does not look like a hole through the membrane.

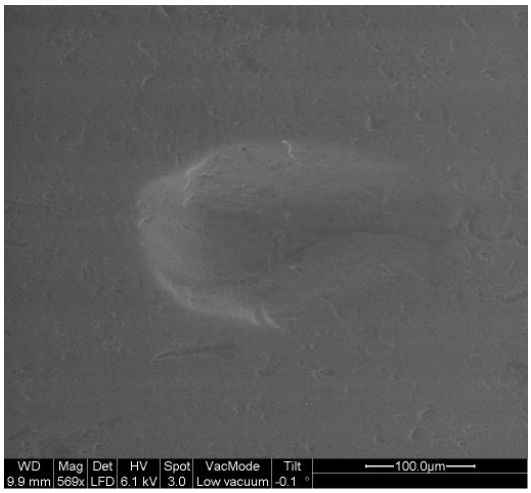


Figure 47 The grey spot is a hole.

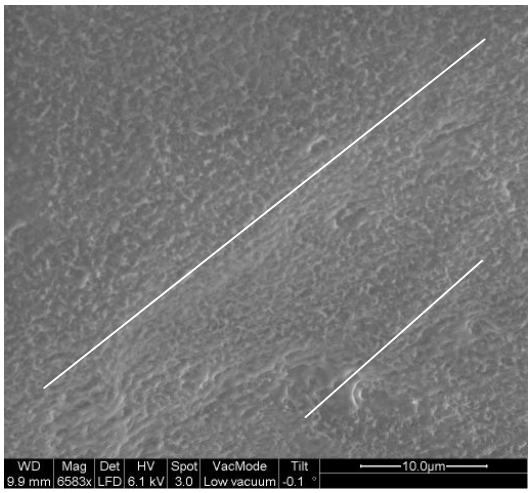


Figure 48 The edge of the hole.

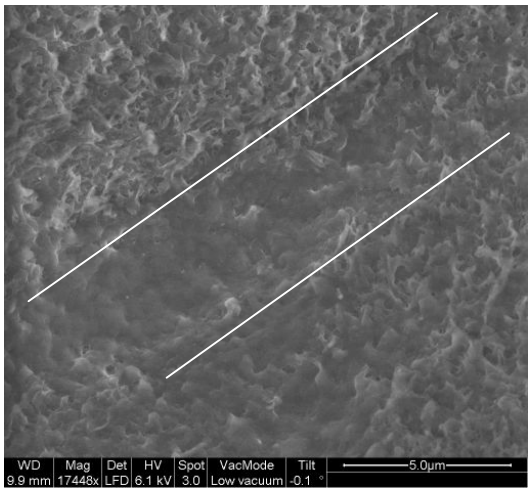


Figure 49. The edge of the hole.

Mapping

In figure 50 examples of mapping analyses from another study [*Petrova*] for some metallic samples are shown. Here the difference in composition at different parts of the sample is seen very clearly, the lighter area indicates what substance it is, for instance the middle figure shows Al as light area, whereas the right figure shows Ni as light dots.

The mapping of the membrane samples (figure 51) did not give any conclusive result regarding composition on the surface. As shows in figure 52 all pictures are homogenous in the greyscale and no substance is indicated.

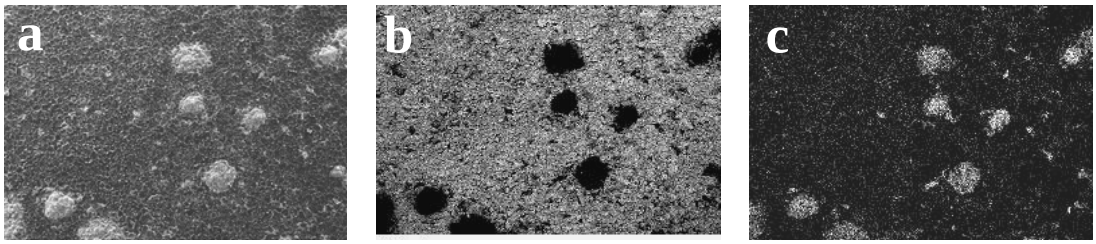


Figure 50. Example of mapping analyses for some metallic samples (a: original ESEM picture, b: light area = Al, c: light area = Ni) [Petrova]

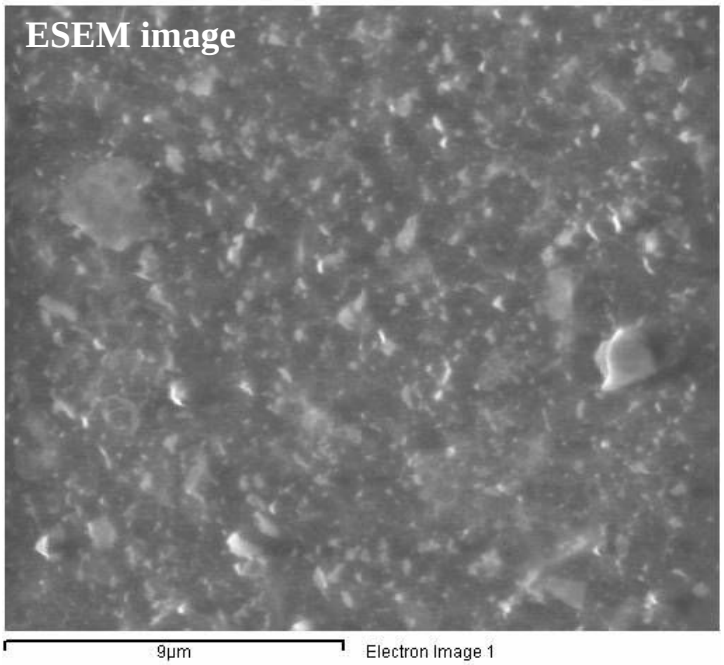


Figure 51. Electron image of the part of the membrane that was analysed.

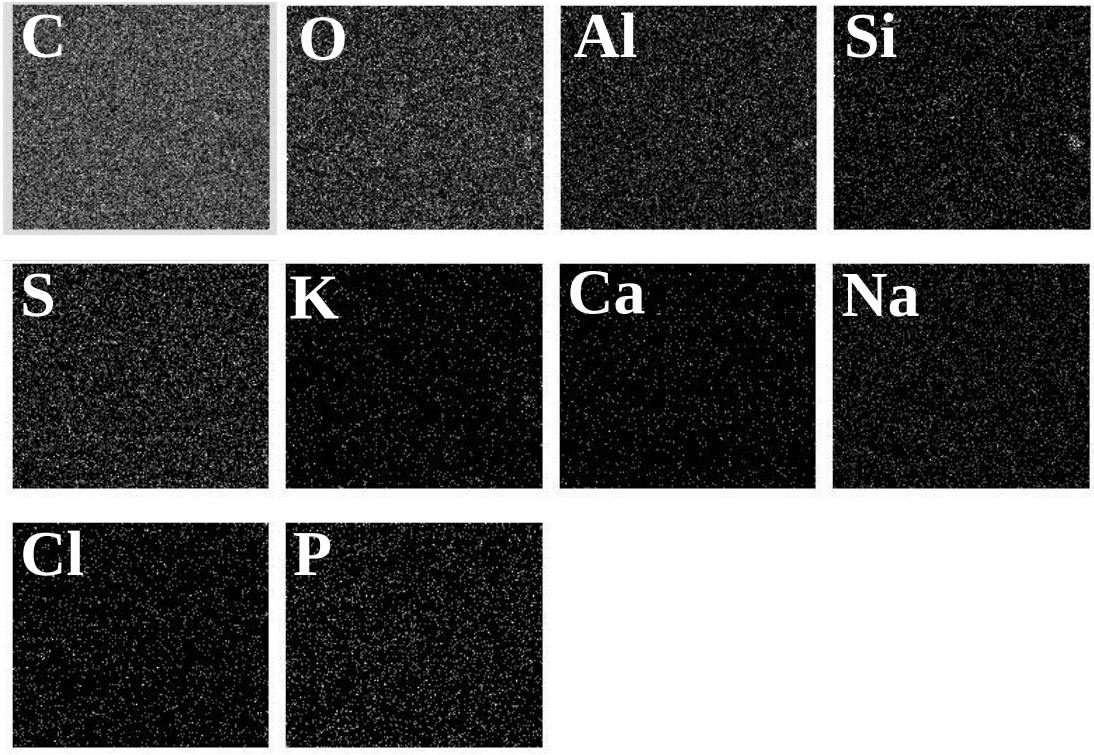


Figure 52. Results from mapping analyses, every picture shows different components, but no detection was successful.

Line scanning
Instead of mapping it is possible to choose some points or lines and analyse the composition there. This can be an advantage because it is possible to choose interesting points that seem different from the surface around them.

Two points were selected, one beside a crystal (spectrum 1) and one on the crystal (spectrum 2), see figure 53. Also a line between two crystals was analysed. The scanning over the points shows that the crystals contain K and P but not the surface beside the crystals. It also shows that there is a lot of S and O on the surface. See more in the discussion.

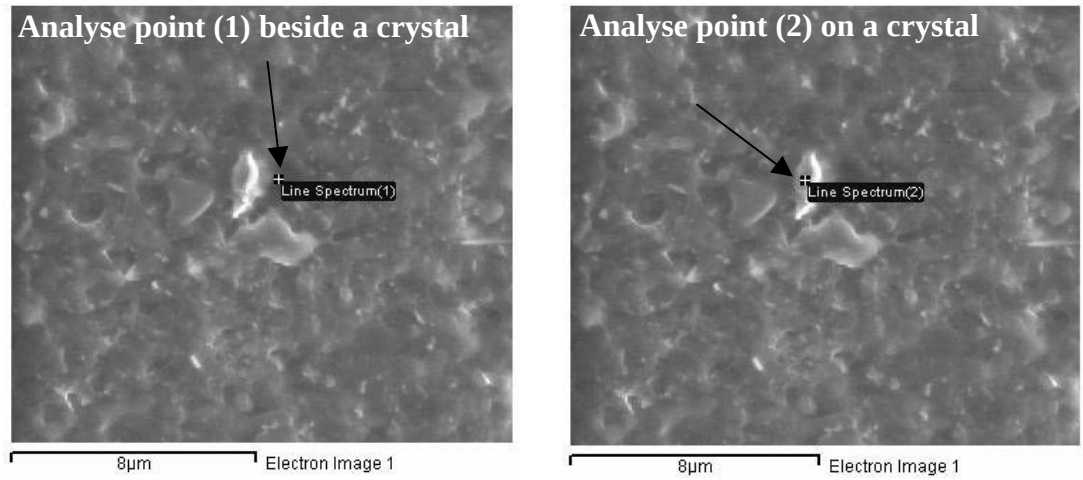


Figure 53. Analyse points.

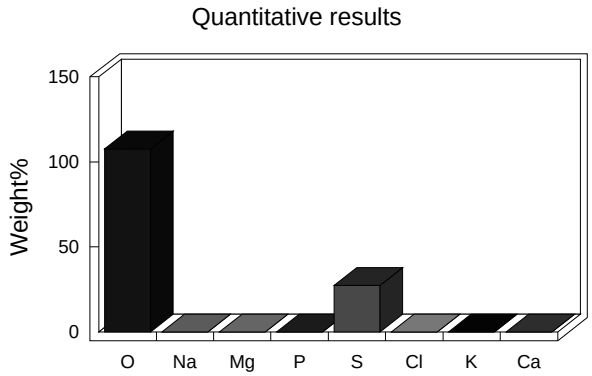


Figure 54a. Components in point 1 in fig 51.

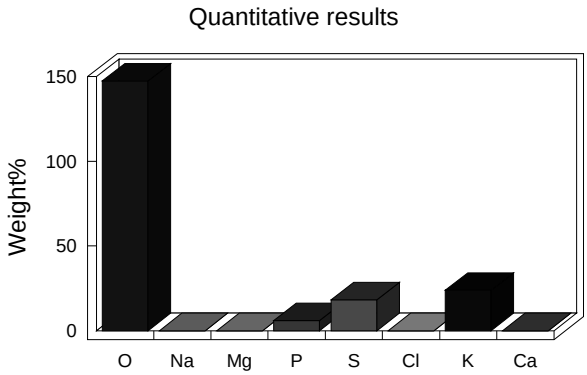


Figure 54b. Components in point 2 in fig 51.

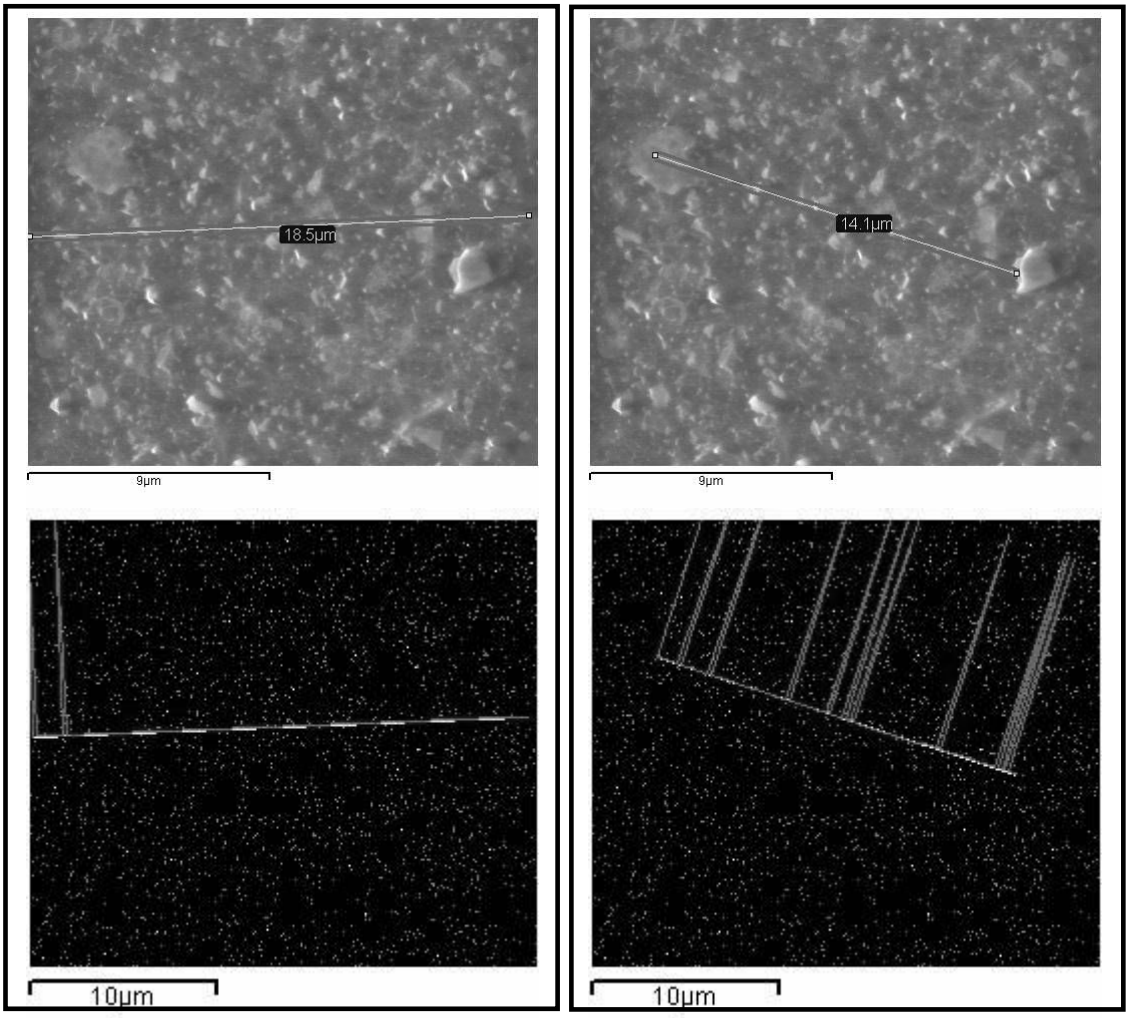


Figure 55. The lines indicate presence of calcium.
Upper pictures - chosen area; lower pictures – signal of calcium.

Line scanning between two points (figure 55) shows that if the line is in contact with crystals it indicates presence of calcium but if only the line beside the crystals is scanned, the signal is weaker, i.e. there is not so many peaks in the detection beside the crystals as when also the crystals is detected. This indicates that the crystals are build up of calcium containing compounds.

5. Discussion

5.1 Definition of Fouling

According to chapter 2.4.1 there are at least two different classifications of fouling. In this report Baker's definition is used. In the following way the classifications are used: *scaling*, precipitation of salts on the membrane surface; *silt*, particles of silicate, sand, lays loose on the membrane surface; *bio fouling*, bacteria and biofilm and *organic fouling*, oil or grease. Sometime also scaling and silt are called inorganic substances and bio fouling and grease is called organic substances.

5.2 Different Operations Mode

When looking at the concentrations factors, operation time and the permeate flow it is very clear that batches with acid dosing gives the most effective operation. Acidification delays the degree of saturation, hence also precipitation and therefore it also delays fouling. The hydrogen in hydrochloric acid reacts with the carbonates and prevents the production of calcium carbonates.

5.3 Chemical Analyses

When comparing the results from ALcontrol AB it is important to remember that no consideration has been taken to the inaccuracy in measurement. This can explain some incongruous values in the results. Other factors that influence are for example sample collection methods and feed and/or concentrate left in the system. No duplicate samples have been analysed, otherwise that would have given results with higher accuracy.

All sulphate results from ALcontrol differ a lot and therefore no clear conclusion from these results can be drawn.

5.3.1 Fouling Analyses

Comparison between fouling from batch 1 and batch 4
The feed in batch 4 has a higher concentration of organic substances than the feed in batch 1. However, the fouling from batch 4 has a smaller amount if organic substances compared to the fouling from batch 1. It could be that the higher amplitude (3/4 inch against 1/2 inch) lifts the organic substances from the surface and prevent fouling. The hydrodynamic layer along the membrane surface gets different depending on amplitude and cross flow. Another explanation is that the shear effect of the vibration is decreased by a higher cross-flow for batch 1. This should also influence the fouling, but in this examination work no detailed studies of this has been done.

For the inorganic substances the feed contents for the batches are similar. But the fouling as well as the feed for batch 4 contains a higher amount of phosphate and phosphorus. Potassium, magnesium, and sulphur have a higher fouling-to-feed ratio

in batch 4 compared to in batch 1. Also the higher concentration factor in batch 4 influences the higher amount of inorganic content in fouling.

Since the fouling in batch 1 contains more organic matter than the fouling in batch 4, it can be hypothesized that the organic layer in the fouling of batch 1 prevents from scaling directly onto the membrane surface. The scaling is embedded in the organic scaling and can be removed more easily than if it had been attached directly onto the membrane surface.

If scaling occurs directly onto the membrane surface (batch 4) organic fouling apprears to attach easier at the membrane surface. In the acidified batches, scaling was avoided and at same time no bio- and organic fouling could be observed. In other words, the inorganic and organic fouling appears to affect each other.

Distribution of fouling in the membrane stack
In the membrane stack about 1/3 of the fouling is found in the upper part and the rest (2/3) is found in the lower part of the stack. This is because the feed is entering at the top of the stack. During the way through the stack the black water is getting more and more concentrated the probability for precipitation of inorganic salts increases. As discussed before inorganic fouling also gives organic and bio fouling.

Fouling dissolved in HCl
In the results from ALcontrol the sulphate content is higher than the total sulphur, which should not be possible, and therefore no consideration for this result has been taken.

The results and calculations show that the main precipitates on the membrane surface are calcium carbonate and calcium phosphate, and maybe a small amount of magnesium phosphates. The relatively high amount of nitrogen is probably rests of proteins and bacterial cells on the surface.

Analyse of cleaning liquid
An analysis of NC2 (alkaline) and NC4 (acid) show that it is necessary to use both the cleaning agents, but the NC2 removes all organic substances. The high amount of nitrogen can depend on cells from bacteria that have been fixed on the membrane. There can also be an inaccuracy in the analyses that influences the results. To secure those results are correct it would be necessary to take duplicate samples and also samples from different cleaning operations.

Otherwise the analysis of cleaning liquid supports the theory that calcium compounds are the main part of the inorganic fouling.

5.3.2 Mass Balances

In the two batches where the fouling were collected, the composition in the feed is very similar except for the total nitrogen

By looking at the concentrate it is very clear that batch 4 has a higher concentration factor than batch 1. This influences the fouling since a higher concentration gives a higher amount of precipitates and there is a higher risk for fouling.

A general comparison of the feed for all batches show that all batches except batch 6 have a very similar composition, batch 6 is more concentrated. There is one exception; total nitrogen differs a lot between the batches, which could complicate the comparison of the fouling analyses.

In some of the batches there is an unexpected large difference between the total nitrogen and ammonium and also for total phosphorus and phosphate. This probably depends on the high contents of food rests in the black water, that has not been degrade during the short residence time in the pipes from the households to the pilot plant. Usually wastewater is transported in long pipes with much more time to ammonify/hydrolyse big organics molecules like proteins and fat molecules.

The concentrate composition for the different batches differs depending on the concentration factor. For the acidified batches almost 100% of the organic substances are refund in the concentrate. Also the concentration of the inorganic substances is larger in the concentrate for the acidified batches, except for batch 6. Probably this depends on the higher concentration of the feed or less precipitation in the system.

By the fact that there are no values for the losses and the permeate for batch 2 and 4 it is complicated to draw conclusions from the results. But general there are smaller losses for the acidified batches. For the permeate it is difficult to see any clear trend.

5.3.3 Precipitations in the Tank

By analysing the precipitations in the tank a relation between the amount of precipitates, fouling and permeate flow decrease can be found. The amount of precipitation increases dramatically with a high volume reduction which depends on the high degree of supersaturation.

The calcium compounds (calcium carbonate and calcium phosphate) are the most common inorganic substances in the fouling and precipitations. Though it is very difficult to identify specific compounds, a comparison of the chemical composition of the concentrates with the results from theoretical calculations carried out in a previous diploma work [Coquin, 2005] showed that the results agree very well. Examples of possible precipitation compounds are: calcium phosphate, calcium hydrogen phosphates, hydroxyl apatite, aragonite, calcite, huntite, dolomite and magnesite.

5.3.4 Permeate Quality

The permeate quality is not good enough to discharge to recipient without further treatment. The reason to the low quality of permeate is that the membrane is not dense enough.

When fouling occurs, the ratio between permeate and concentrate increases and the quality of permeate decreases. One way of getting a higher permeate quality is to collect the permeate in a tank and let it pass through the VSEP once again. Another way is to prevent too much membrane leakage is to use a denser membrane with a higher membrane rejection

5.4 Visual Analyses

When opening the VSEP stack it is shown clearly that the fouling looks very different in both cases. The first time the fouling was build up by three layers. Probably only the two layers close to the surface are the most problematic types of fouling (scaling).

A lot of silt was also found on the membrane surface. During the vibration these particles can cause mechanical wear on the membrane surface. This can eventually lead to the formation of holes on the surface and leakage through the membrane plate. It is therefore very important to protect the system from silt.

The second time the membrane package was opened it had been exposed to higher amplitude which seems to have lifted the loose fouling more from the membrane surface. But on the other hand the layer of fouling close to the membrane surface was more strongly bound to the surface. These results are in agreement with the results from the chemical analyses if the fouling.

The second time the membrane was opened no silt was found. Probably this is because at the higher amplitude the silt is lifted from the surface and flushed out of the system. Another explanation is that the influent was free from silt.

At the opening after an acidified operation there were almost no fouling found. This shows that the preventionof scaling also reduces the formation of bio fouling and organic fouling.

5.5 Analyse by Light Microscope

The fouling is a biofilm with a high content of inorganic crystals. The higher concentration of inorganic substances depends on the supersaturation of the concentrated black water with respect to insoluble metal salts like calcium carbonates and phosphates.

The silt was made up of very colourful, green, red, white and transparent, crystals.

5.6 BET (Brauner, Emmet and Teller) - Method

The result shows that the specific surface area of the membrane gets smaller for a used membrane. The smaller area can be explained by plugged pores or that the pores have become larger i.e. holes in the membrane surface have been formed. Another factor that can influence the results is that the used membrane has been cleand in NC4 that has a low pH, which can open up the pores. To avoid this influence on the results, it would have been an idea to clean the unused membrane as well. But presumably the low pH does not open up the pores as much as 6% difference as the results show.

The pore size distribution was very large. This depends on the fact that the membrane is built up of a carrier material on the back side of the membrane that is much more porous than the reverse osmosis membrane surface on the top. All the small samples pieces of the membrane were random placed in a flask. Therefore is it difficult to draw any conclusions from this result.

5.7 ESEM (Environmental Scanning Electron Microscope)

Pictures of the surface
The ESEM photographs show that there are a lot of bacteria and a small amount of crystals also on a manually cleaned membrane. This shows that it easy to take away fouling of crystals by cleaning and harder to take away bio fouling i.e. bacteria. These bacteria could probably influence the surface and membrane filtration when they are fixed to the surface for a long time. Therefore it is important to find a method that prevents the membrane surface from contamination of bacteria.

At areas of turbulent flow the surface is influenced in some way. It is very difficult to say if there is some change in the surface structure or if there is a biofilm close to the membrane surface. Maybe it is a combination of both. If the biofilm is fixed on the surface and then moves around caused by the turbulent flow this can wear out the membrane surface. It is recommended to analyse the surface properties of the membranes used.

Mapping and line scanning
Mapping of elements on the membrane surface was found to be more complicated than expected, and gave no relevant results. The problem with the mapping analysis can be explained by the fact that the energy peaks of calcium and potassium are very close to each other and therefore difficult so separate. Also sulphur and phosphate have energy peaks close together. The biofilm is probably another reason to why the mapping got so complicated; the electrons from the elements are not able to transport through the biofilm to the detector. Another problem could be that electrons stay at the surface and the surface gets charged and no signal is sent to the detector. The material of the membrane can also be sensitive to too strong signals and burns up.

With line scanning the results are somewhat more relevant but even here the same problem as with mapping was encountered.

5.8 Summary of Fouling Results

The following types of fouling have been identified: silt was laying on the membrane surfaces when the VSEP stack was opened. Both light microscopy and ESEM showed scaling as crystals in the fouling. The results from ALcontrol showed that the most common precipitations are calcium carbonate and calcium phosphates and the high amount of fats indicates organic fouling. Bio fouling could be identified in the light microscope and ESEM analyses where biofilm and a lot of bacteria were found.

It seems that the scaling influences the permeate flow rate in a strong abrupt way. The bio fouling and organic fouling is probably more porous and loose and that it is permeable for liquid and therefore the permeate flow rate is not influenced to a large

degree. However, Torben Meins' results show that the organic and bio fouling give a gradual decrease of the permeate flow directly from the start.

From the results three cases have been found to occur.: (1) when inorganic fouling, scaling starts it also gives organic and bio fouling; (2) if organic fouling appears first the scaling is imbedded and therefore easier to remove; and finally (3) if inorganic fouling can be avoided, organic and bio fouling becomes much less as well.

An important parameter that influences the fouling is the feed. A problem with sewage is that this feed has a varying composition with time that makes the system very complicated. To simplify the study of effects of different operational conditions a synthetic black water with a constant composition could be used.

6. Conclusions

The following conclusions can be drawn from the results:

1. Fouling on VSEP RO membranes consists of:
    a. *Bio fouling* – a biofilm with a lot of bacteria. These bacteria are strongly fixed to the membrane surface, the bacteria were difficult to remove and they were even left in turbulent zones after washing.
    b. *Organic fouling* – the results from ALcontrol shows high amounts of fats.
    c. *Inorganic fouling* – scaling, a lot of inorganic crystals which are mainly composed of calcium carbonates and calcium phosphates.
    d. *Silt* – insoluble particles on the membrane surface.

    Fouling occurs in three different ways: (1) inorganic fouling, when scaling starts also organic and bio fouling will start; (2) organic fouling in which inorganic fouling is imbedded and therefore easier to remove; and finally, (3) if inorganic fouling can be avoided, organic and bio fouling is also much less.

2. The fouling is caused by three factors:
    a. Supersaturation of metal salts that precipitate onto the membrane.
    b. Growth of bacteria.
    c. Accumulation of organic substances, especially when scaling occurs.

    How the fouling influences the surface is more difficult to establish, the results show some change of surface structure but this does not seems to influence the flow rate. In zones of turbulent flow, the surface looks more smooth but it has not been possible to determine if that depends on a change of surface structure or due to a layer of biofilm.

3. The addition of acid was found to be a successful method to delay fouling.

7. Outlook

Another type of membrane
In this laboratory work only one type of membrane has been tested, LFC1 (Low Fouling Composite). It has been shown that a lot of bacteria easy can be fixed at the surface and that they are difficult to remove. It would be interesting to test membranes with other surface properties to see if bacteria attach to a different degree onto them. Furthermore the permeate quality does not fulfil the limitations. It would therefore be interesting to find a denser membrane which gives higher rejections so that it does not leak ions and should hence give higher permeate quality. This membrane should also have a surface that is more bacteria repelling.

Another acid
Hydrochloric acid dosing gives undesired chloride addition to the product. But other inorganic acids also give a lot of disadvantages; sulphuric acid and phosphoric acid give precipitations of calcium sulphate respectively calcium phosphate, and nitric acid is expensive and by adding nitrogen the relation between phosphorus and nitrogen gets too small for an attractive product.

Therefore an organic acid should be recommended, one advantage with organic acids is that they also have little antiscalant effect i.e. forms complexes with calcium and delay the degree of saturation. A disadvantage is that with too long residence time, the bacteria will break down the acids.

Two suitable organic acids are acetic acid and citric acid; these acids are some weaker and therefore a larger amounts have to be dosed. One advantage is that more gas is produced (?). One disadvantage is that these acids are more expensive than hydrochloric acid.

Synthetic sewage
To analyse how fouling is dependent on feed composition it would be interesting to use a synthetic black water, as described in chapter 2.4.3. In that way more controlled conditions can be obtained and it is easier to study how different operation parameters or changes in feed contents influence the fouling. This would be very interesting to do with different types of membranes to get a better understanding for what compounds bind to the membrane surface.

Sedimentation step
One way to delay fouling is to have a sedimentation step between the drum screen and VSEP. Some of the precipitations and suspended solids in the feed would sink to the bottom of the tank and the feed would then contain lower concentrations of precipitations and suspended solids, hence the fouling would occur later.

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Appendix

A. General comparison for all batches

B. Insecurity in measuring

C. Parameter evolution

D. 1: Feed composition, 2: Final concentrate composition, 3: Permeate composition

E. Mass balances

F. Concentration and concentration difference in filtrated and un-filtrated samples

G. Ratio between permeate and concentrate concentrations

H. Results from BET analyses

I. All results from Alcontrol

Appendix A: General comparison for all batches

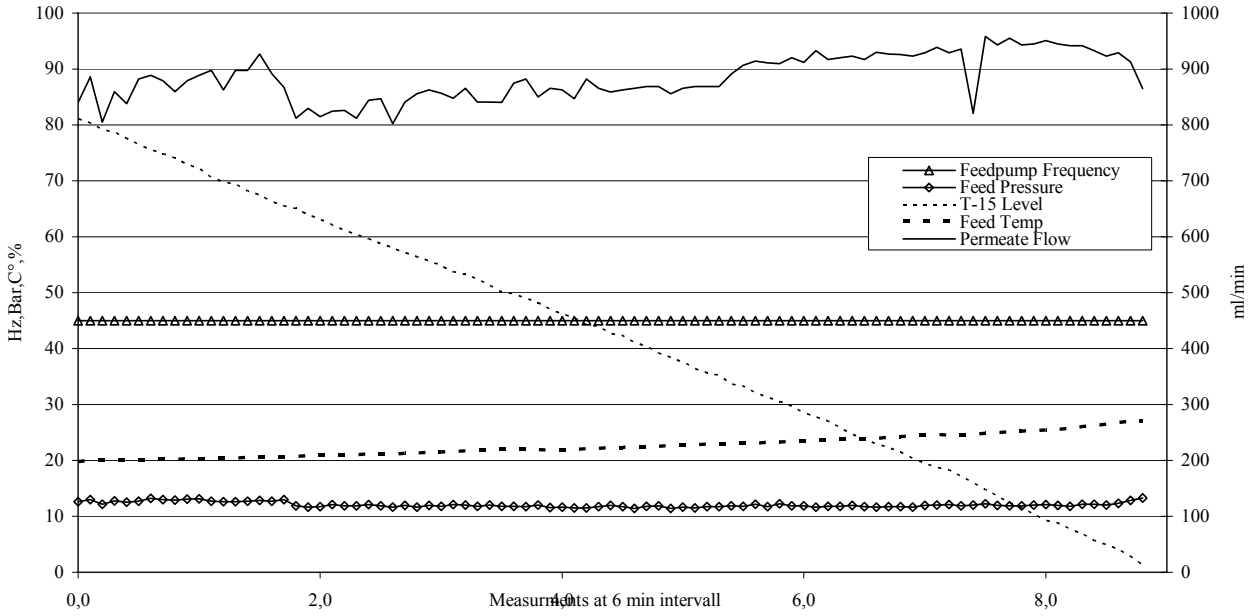
	Batch 0	**Batch 1**	**Batch 2**	**Batch 3**	**Batch 4**	**Batch 5**	**Batch 6**	**Batch 7**
	Drinking water	Higher cross-flow	No dose	Constant pH = 6.0	Higer amplitude	Higer amplitude	similar as batch 3	const pH + higher ampl
Tank volume, start (litre)	487	472	481	470	476	480	474	474
Tank volume, max concentration (litre)	8	87	63	55	31	72	30	30
Total volume permeate		381	413	417	442	421	443	435
Concentration factor, f								
# F (Vend/Vstart)	60.9	5.4	7.6	8.5	15.4	6.7	15.8	15.8
# F ((Vstart-Vpermeate)/(Vstart)		5.2	7.1	9.0	13.9	8.1	15.3	12.3
# F (Na)		5.9	8.2	9.4	15.7	8.1	17.0	11.4
# F (K)		5.8	8.1	9.9	15.1	7.8	16.5	11.5
Total residence time (hours)	8.8	15.9	14	10.7	14.1	13.3	12.8	11.5
Feed pump frequency (Hz)	45	60	45	45	45	45	45	45
Amplitude (inch)	1/2	1/2	1/2	1/2	3/4	1	1/2	1
Initial pressure (bar)	12.6	11.5	11.5	11.5	11.5	11.5	11.5	18
Final pressure (bar)	13.3	12.3	1	19.5	16.5	18	35	28
Initial permeate flow (ml/min)	840	920	820	670	910	570	470	800
Final permeate flow (ml/min)	865	118	112	671	108	120	630	600
Initial feed temperature (°C)	19.8	20.8	20.5	20.6	20.3	19.6	17	16
Final feed temperature (°C)	27	31.6	26.4	26.1	31.8	30	26	26
Initial pH								
# feed		7.5	7.7	6	7.4	7.2	7.4	7.3
# permeate		6.6	7.6	6	6.8	6.7	6.4	6.2
Final pH								
# feed		8	8	6.1	8	8.1	6	5.8
# permeate		8.6	8.5	6.3	8.5	8.7	6.3	6.4

Appendix B: Insecurity in measuring

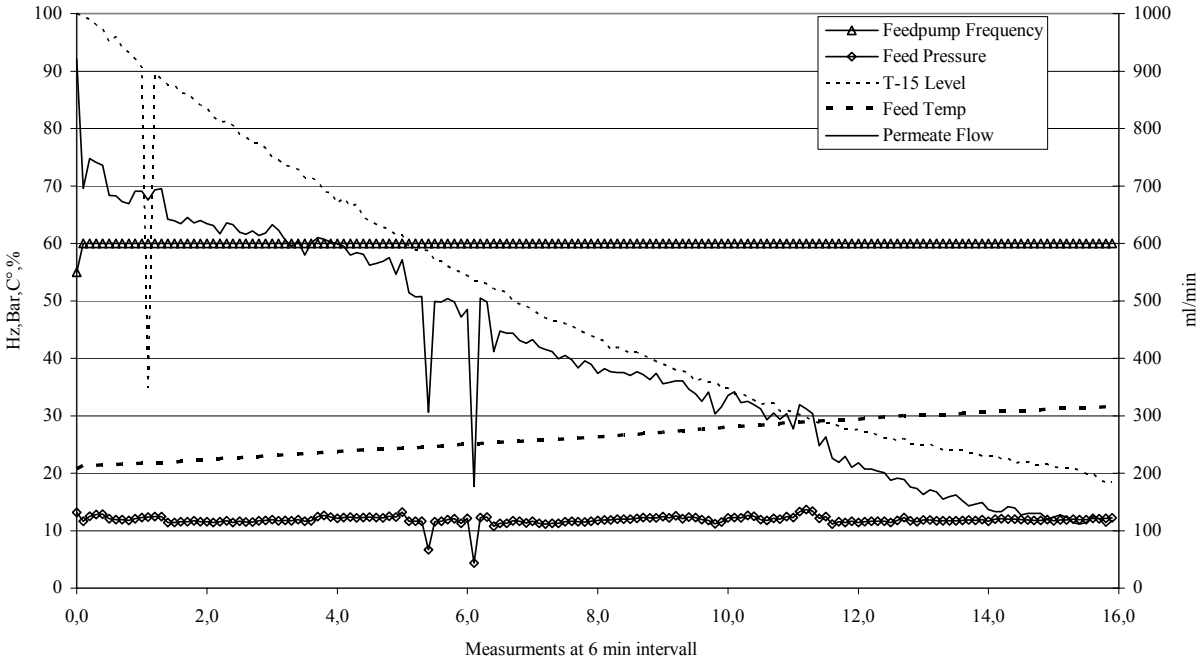
Analyse of	Unit	Insecurity in measuring	Method
pH	-	± 5%	SS028122-2,mod
Alkalinity	mg/l	± 10-15%	SS-ISO9963-2, mod
Conductivity	mS/m	± 5%	SS-EN 27888, mod
BOD7	mg/l	± 20%	SS-EN 1899-1
COD(Cr)	mg/l	± 15-20%	Merck Nova 60, ampull
TOC	mg/l	± 10%	SS-EN 1484
Fatty acid, totally	mg/l	± 25%	SS028103 mod (perklor)
Dry Solid	mg/l	± 10%	SS028113-1
Suspended solids	mg/l	± 5-25%	SS-EN 872
Phosphorus totally, P	mg/l	± 10-20%	SKALAR Meth.503-SO4
Phosphate phosphorus	mg/l	± 10-30%	TRAACS Appl.No J004-88-1
Nitrogen totally, N	mg/l	± 10-15%	TRAACS Appl. No J-002-88
Ammonium nitrogen	mg/l	± 10-15%	TRAACS appl.No GB-352-87
Potassium, K	mg/l	± 20%	SS-EN ISO 11885-1
Calcium, Ca	mg/l	± 20%	SS-EN ISO 11885-1
Magnesium, Mg	mg/l	± 20%	SS-EN ISO 11885-1
Chlorine, Cl	mg/l	± 15%	SS-EN ISO 10304-1
Sulphate, SO4	mg/l	± 15%	SS-EN ISO 10304-1
Sulphur	mg/l	± 10%	SS-EN ISO 11885-1
Sodium, Na	mg/l	± 10%	SS-EN ISO 11885-1
Nitrate + Nitrite, N	mg/l	± 10-20%	TRAACS Appl.No J002-88-1
Nitrite nitrogen	mg/l	± 10-20%	TRAACS Appl.No J002-88-1
Red Heated loss	% av susp	± 25%	SS028112-3
Red Heated rest of susp.	% av susp	± 25%	SS028112-3
Red Heated rest	% av TS	± 15%	SS-EN 12879-1

Appendix C: Parameter evolution

Summary concentration of drinking water

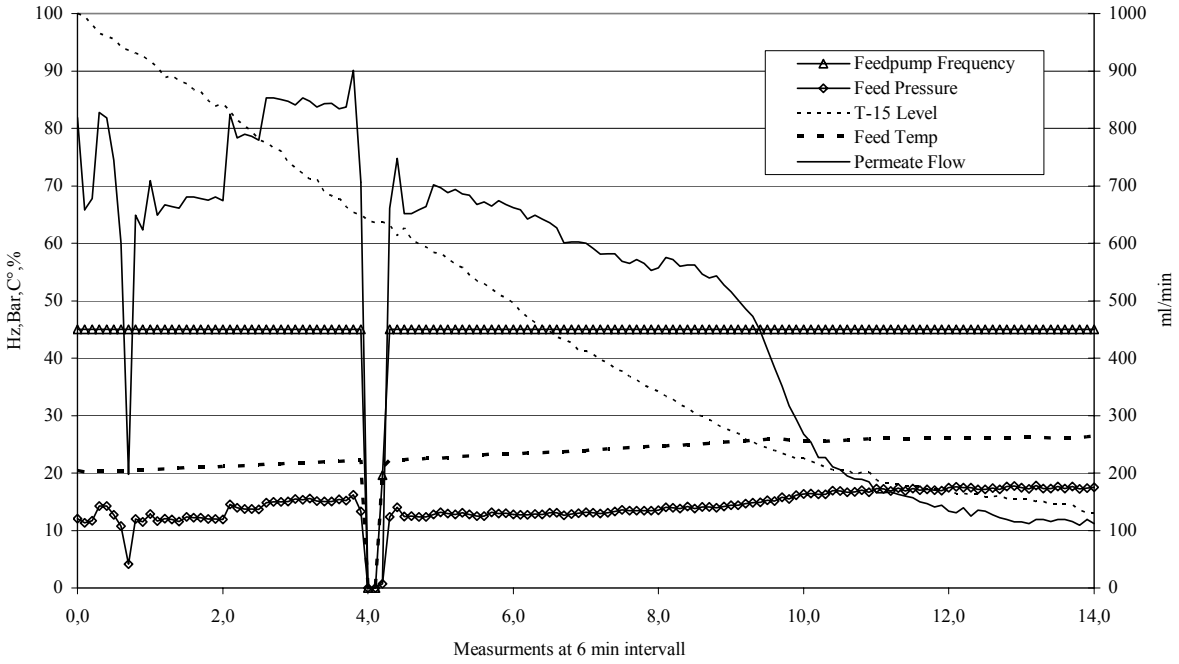


Summary Batch 1

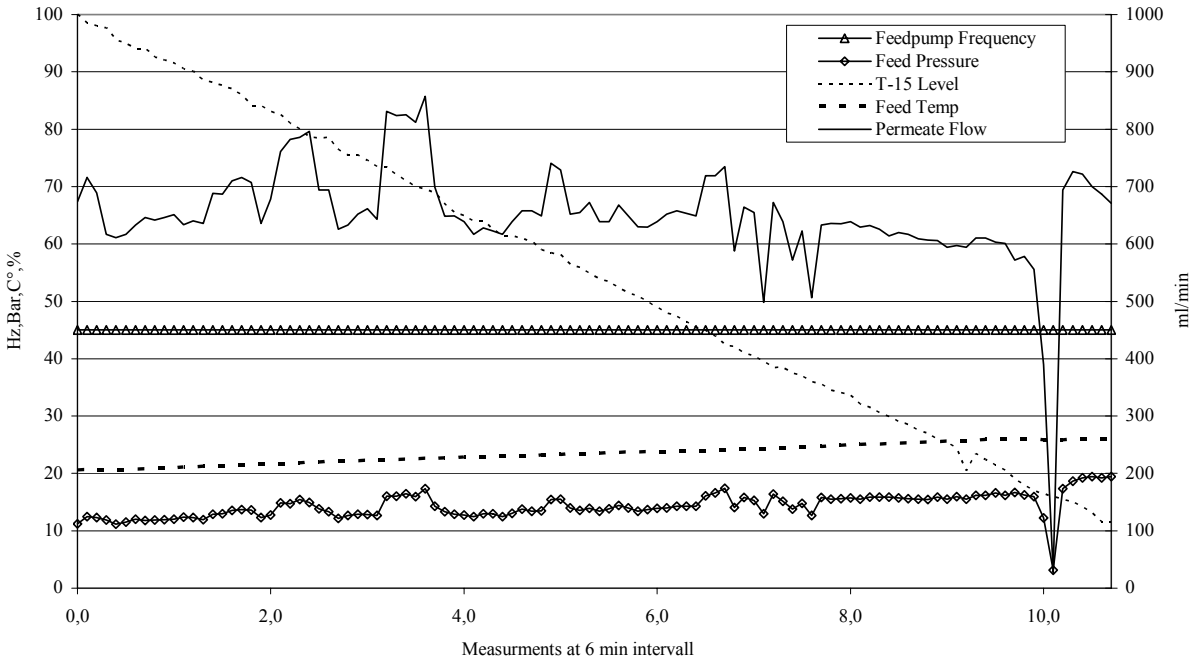


Appendix C: Parameter evolution

Summary Batch 2

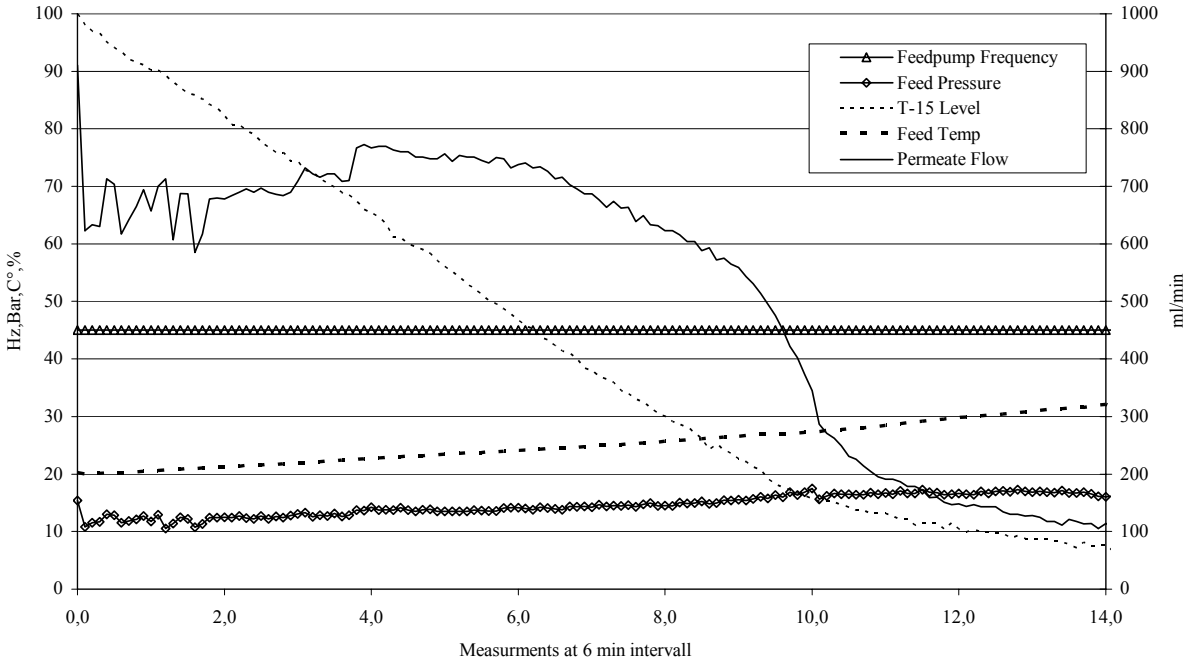


Summary Batch 3

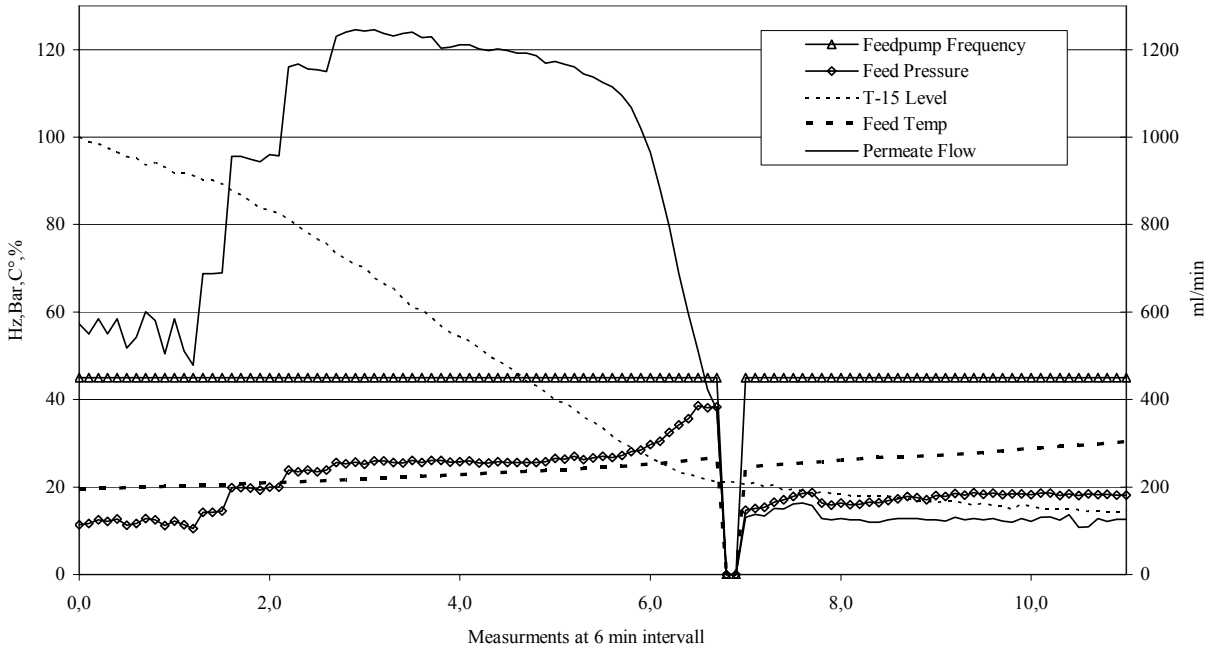


Appendix C: Parameter evolution

Summary Batch 4

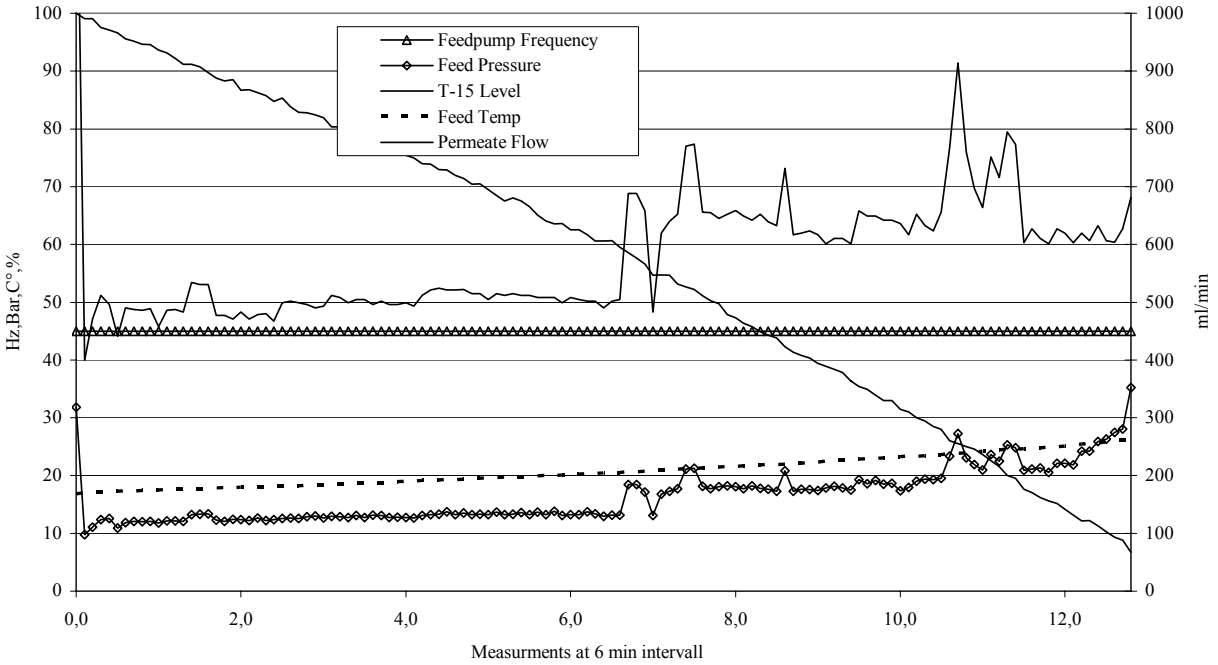


Summary Batch 5

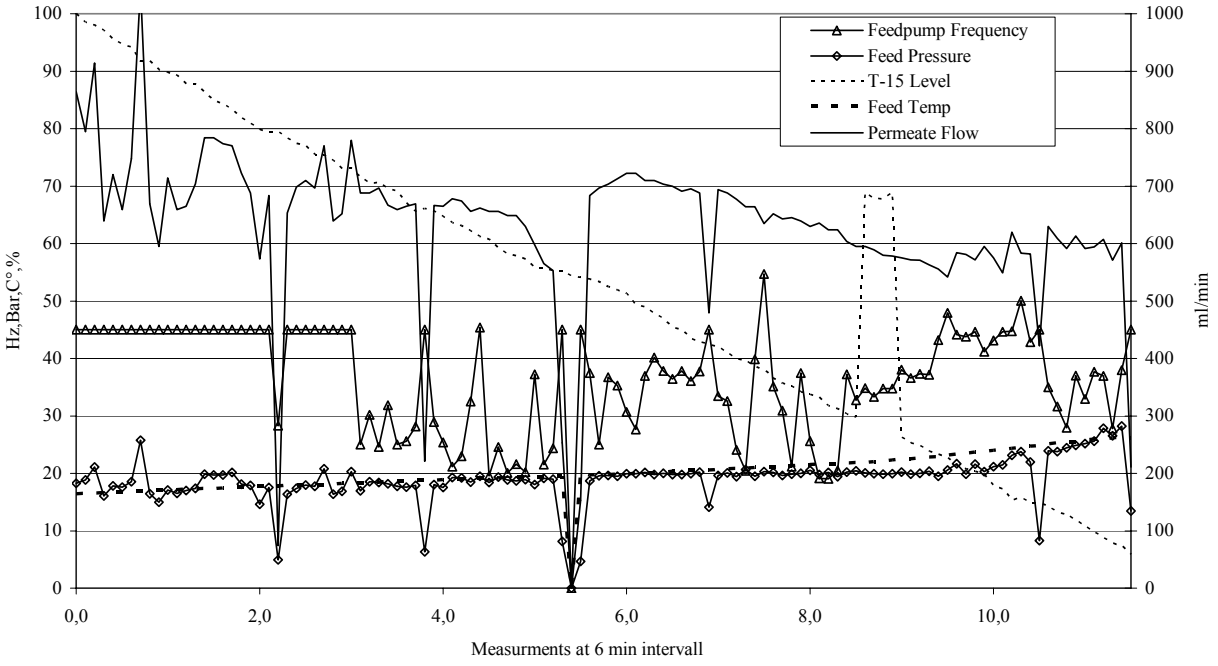


Appendix C: Parameter evolution

Summary Batch 6



Summary Batch 7



Appendix D1: Feed composition

Parameter	Batch 1		Batch 2		Batch 3				Batch 4	
					origin		*pH = 6*			
	un-filtrated	filtrated	un-filtrated	filtrated	un-filtrated	filtrated	un-filtrated	filtrated	un-filtrated	filtrated
pH	7.5		7.7		7.4		6.6		7.4	7.7
Alklinity (pH=??) (meq/L)	540		770		620		330		450	490
Conductivity (µs/cm)	150		190		160		170		120	
Suspended solids (mg/L)	610	72	650	30	750	13	640		650	41
COD(Cr) (mg/L)	1400	490	1700	680	1600	590	1500	660	1300	490
COD(Mn) (mg/L)	190	95	260	110	220	100	220	100	230	70
TOC (mg/L)	250	150	310	230	310	170	410	210	210	170
Fatty acids (mg/L)	130	15	170	21	160	24	170	18	150	15
Ptot (mg/L)	20	13	23	19	21	14	19	15	18	12
PO_4-P (mg/L)	11	12	18	15	10	11	9,5	12	10	9,8
Ntot (mg/L)	250	250	200	270	530	410	450	390	140	100
NH_4^+ (mg/L)	120	130	190	170	150	130			97	93
K^+ (mg/L)	48		55		50				40	
Ca^{2+} (mg/L)	39	28	40	30	40	27	43		39	22
Mg^{2+} (mg/L)	6.4	5.5	6.9	5.7	8	6.9			6.3	5
Cl^- (mg/L)	150		110		120	130			82	
Na^+ (mg/L)	67		93		93	90			65	
Stot (mg/L)	22	20	19	24		21	20	20	19	18
SO_4^{2-} (mg/L)	32	41	26	41	13	34		34	15	26

Appendix D1: Feed composition

Parameter	Batch 5		Batch 6				Batch 7	
			origin		*pH = 6*			
	un-filtrated	filtrated	un-filtrated	filtrated	un-filtrated	filtrated	un-filtrated	filtrated
pH	7.2	7.6	7.4	7.6	6.6	7	7.3	7.8
Alklinity (pH=??) (meq/L)	630	620	560	610	250	430	590	550
Conductivity (µs/cm)	160		160	160	170	170	150	
Suspended solids (mg/L)	630	64	1200	100	1100	100	750	42
COD(Cr) (mg/L)	1500	640	2400	1200	2400	950	1500	580
COD(Mn) (mg/L)	180	15	380					
TOC (mg/L)	270	200	570	270	560	300	370	170
Fatty acids (mg/L)	140	23	330	34	330	35	120	29
Ptot (mg/L)	23	15	30	21	32	22	19	13
PO_4-P (mg/L)	11	8.1	21	20	23	21	14	12
Ntot (mg/L)	460	390	350	280	380	300	260	300
NH_4^+ (mg/L)	150	150	140	120			120	
K^+ (mg/L)	50		51	48	51	50	49	
Ca^{2+} (mg/L)	39	28	50	30	52	37	36	26
Mg^{2+} (mg/L)	7.3	6.1	8.5	7.2	8.5	7.7	6.1	5.2
Cl^- (mg/L)	95		95		260		210	
Na^+ (mg/L)	76		77		77		86	
Stot (mg/L)	24	23	24	20	22	21	25	22
SO_4^{2-} (mg/L)	19	37	18	26	<2.0	12	40	43

Appendix D2: Final concentrate composition

Parameter	Batch 1				Batch 2				Batch 3			
	TSED		CM		TSED		CM		TSED		CM	
	un-filtrated	filtrated	un-filtrated	filtrated	un-filtrated	filtrated	un-filtrated	filtrated	un-filtrated	filtrated	un-filtrated	filtrated
pH	8	8.2	8	8.2	8	8.1	8	8.1	6.1	6.1		
Alklinity (pH=??) (meq/L)									1000	750		
Conductivity (µs/cm)					970	920	1000	990	1180	1190		
Suspended solids (mg/L)	1600	420	1600	220	1600	130	2000	250	7100	150		
COD(Cr) (mg/L)	3100	2500	3400	2400	6600	3700	5800	4300	13000	5400		
COD(Mn) (mg/L)	600	430	580	410	1000	600	860	740	1900	890		
TOC (mg/L)	780	700	830	740	2000	1500	1900	1600	3600	2100		
Fatty acids (mg/L)	270	72	240	66	560	120	450	160	1300	230		
Ptot (mg/L)	19	19	29	21	64	39	65	42	84	71		
PO_4-P (mg/L)	70	50	68	48	120	57	130	73	160	150		
Ntot (mg/L)	1000	810	870	800	4300	2000	3600	2100	2600	2300		
NH_4^+ (mg/L)	570	510	550	530	1000	1000	1100	1100	970	950		
K^+ (mg/L)	260	260	260	250	350	340	330	370	410	400		
Ca^{2+} (mg/L)	160	110	160	100	220	110	210	130	390	290		
Mg^{2+} (mg/L)	32	29	32	26	36	19	42	26	73	68		
Cl^- (mg/L)	420		420		620		640		2600			
Na^+ (mg/L)	370		380		590		570		730			
Sulfur (mg/L)	110	100	110	100	180	160	180	180	180	170		
SO_4^{2-} (mg/L)	210	190	180	180	320	340	350	370	290	330		

Appendix D2: Final concentrate composition

Parameter	Batch 4				Batch 5			
	TSED		CM		TSED		CM	
	un-filtrated	filtrated	un-filtrated	filtrated	un-filtrated	filtrated	un-filtrated	filtrated
pH	7.7	8.1	8	8.2	7.8	7.8	8.1	8.2
Alklinity (pH=??) (meq/L)	4800	4100	2100	4600	4200	1300	4200	3700
Conductivity (µs/cm)	1030	1020	1170	1150	850	56,9	930	910
Suspended solids (mg/L)	6500	620	4900	550	630	490	2000	600
COD(Cr) (mg/L)	15000	6600	10000	7000	9900	4200	5300	4700
COD(Mn) (mg/L)	2000	670	1700	30	460	45	760	55
TOC (mg/L)	3300	2300	3000	2400	2200	1300	1500	1500
Fatty acids (mg/L)	1600	210	800	220	1100	160	450	180
Ptot (mg/L)	52	47	31	40	20	20	28	17
PO_4-P (mg/L)	200	68	210	61	140	58	120	59
Ntot (mg/L)	2200	2000	2300	2100	4500	2200	4000	3900
NH_4^+ (mg/L)	660	1100	550	1100	990	970	1100	1100
K^+ (mg/L)	470	450	510	520	340	340	370	370
Ca^{2+} (mg/L)	460	170	360	180	280	130	220	140
Mg^{2+} (mg/L)	79	37	78	42	52	23	42	23
Cl^- (mg/L)	820		780		120		670	
Na^+ (mg/L)					530		560	
Sulfur (mg/L)	240	230	270	270	170	160	180	180
SO_4^{2-} (mg/L)	<2.0	<2.0	<2.0	170	48	340	350	420

Appendix D2: Final concentrate composition

Parameter	Batch 6				Batch 7			
	TSED		CM		TSED		CM	
	un-filtrated	filtrated	un-filtrated	filtrated	un-filtrated	filtrated	un-filtrated	filtrated
pH	5.8	5.9			5.6	5.8	5.8	
Alklinity (pH=??) (meq/L)	1800	1600			850	700	980	
Conductivity (µs/cm)	1860	1830			1320	1700		
Suspended solids (mg/L)	14000	2000			11000	620	3600	490
COD(Cr) (mg/L)		12000			22000	7200	16000	
COD(Mn) (mg/L)								
TOC (mg/L)	6400	4200			5000	2600	4900	
Fatty acids (mg/L)	3100	650			5400	490	1600	
Ptot (mg/L)	310	290			170	160	280	200
PO_4-P (mg/L)	390	310			230	220	350	290
Ntot (mg/L)	5300	4600			2400	2400	9100	
NH_4^+ (mg/L)	1700	1600			740	1300	1500	
K^+ (mg/L)	700	660			500	610	790	
Ca^{2+} (mg/L)	660	560			460	410	590	540
Mg^{2+} (mg/L)	130	120			71	79	110	110
Cl^- (mg/L)	4600				3500	4700	6300	
Na^+ (mg/L)	1100				880	1100	1400	
Sulfur (mg/L)	330	290			250	150	200	200
SO_4^{2-} (mg/L)	720	300			400	610	800	840

Appendix D3: Permeate composition

Batch 1

	pH	Conductivity	COD (Cr)	COD (Mn)	BOD7	TOC	Fatty acids	PO_4-P	P-tot	N-tot	NH_4	K	Ca	Mg	Cl	SO_4	S-tot	Na
		mS/m	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*
PA	7.6		<30	3		4.2	1.3	0.004	0.07	18	16	3.4	0.39	<0.1	14	<2.0	0.53	5.5
P0	6.6					4.1					3.6	<2.0			8.3			2.2
P50	7.3					4.8					11	2.1			11			4
PM	8.6		45	8		16	2.8	0.22	0.34	90	75	16	0.95	0.3	40	<2.0	1.6	23

Batch 2

	pH	Conductivity	COD (Cr)	COD (Mn)	BOD7	TOC	Fatty acids	PO_4-P	P-tot	N-tot	NH_4	K	Ca	Mg	Cl	SO_4	S-tot	Na
		mS/m	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*
PA-calc		*58.47*				*35.43*					*58.23*	*13.57*			*52.23*			*23.84*
P0	7.6	20.8				19					17	4.4			20		3.4	9.3
P50	7.9	34.4				24					35	7.3			37		2	13
P80	8.3	110	150	17		51	6.8			270	110	26			80			44
PM	8.5	200	260	23		96	3.9	3.1	3	560	220	56	4.2	1.4	170	14	8.5	90

Batch 3

	pH	Conductivity	COD (Cr)	COD (Mn)	BOD7	TOC	Fatty acids	PO_4-P	P-tot	N-tot	NH_4	K	Ca	Mg	Cl	SO_4	S-tot	Na
		mS/m	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*
PA	6.6	37.8	55	7		18	33	0.27	1.1	110	24	9.7	1.5	0.4	78	<2.0	1.2	17
P0	6	33				17				95	14	5.1			30		1.6	9.5
P50	6.2	26.1				18				62	18	6.7			50		0.9	12
P80	6.3	68.9	110	8		41	4			120	49	17			150		2.2	28

Appendix D3: Permeate composition

Batch 4

	pH	Conductivity	COD (Cr)	COD (Mn)	BOD7	TOC	Fatty acids	PO_4-P	P-tot	N-tot	NH_4	K	Ca	Mg	Cl	SO_4	S-tot	Na
		mS/m	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*
PA-calc		*34.36*				*20.44*				*54.2*	*31.45*	*9.45*			*28.53*		*2.11*	*15.68*
P0	6.8	9.1				10				12	6.5	2.3			7.1		1.5	4.8
P80	7.8	45.3	80	8		24	<1			52	39	11			33		2	18
PM	8.5	220	320	31		120	4	2.3	2.3	550	240	76	4.1	2.3	230	22	13	120

Batch 5

	pH	Conductivity	COD (Cr)	COD (Mn)	BOD7	TOC	Fatty acids	PO_4-P	P-tot	N-tot	NH_4	K	Ca	Mg	Cl	SO_4	S-tot	Na
		mS/m	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*
PA-calc	6.42	30.86	52	10.95		11.95	2.21	0.31	0.43	43.8	31.68	7.16	0.52	0.16	23.98	5.01	1.25	12.03
P0	6.7	12.4	50	5		11	1.8	0.37	0.37	20	10	2.8	0.35	0.1	8.6	2.0	1.2	5.4
P75	7.4	32.1	35	20		3.8	3.1	0.02	0.24	49	32	6	0.32	0.1	25	2.0	0.64	11
P80	8	94.5	140	5		45	3	1.2	1.7	110	94	24	1.9	0.6	70	3.8	4.3	37
PM	8.7	150	210	16		70	1.8	1.8	1.8	200	190	45	2.9	0.9	130	71	5.5	67

Appendix D3: Permeate composition

Batch 6

	pH	Conductivity	COD (Cr)	COD (Mn)	BOD7	TOC	Fatty acids	PO_4-P	P-tot	N-tot	NH_4	K	Ca	Mg	Cl	SO_4	S-tot	Na
		mS/m	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*
PA	6.6	33.9			90	28	<1	0.51	1.2	31	25	9.2	1.5	0.3	68	<2.0	1.1	13
P0	6.4	20			70	20	5.2	0.53	0.72	18	14	5.6	1.1	0.2	35	<2.0	1.3	8,9
P50	6.3	24.7			60	22	2.2	0.9	0.86	22	18	6.7	0.92	0.2	48	<2.0	0.79	9.9
P80	6.3	70.4			160	57	1.3	2.7	2.5	61	53	19	3.2	0.7	130	2.3	2.1	25

Batch 7

	pH	Conductivity	COD (Cr)	COD (Mn)	BOD7	TOC	Fatty acids	PO_4-P	P-tot	N-tot	NH_4	K	Ca	Mg	Cl	SO_4	S-tot	Na
		mS/m	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*	*mg/l*
PA	6.6	22.8			50	14	4	0.42	0.42	19	16	6	0.58	0.1	48	<2.0	0.51	10
P0	6.2	12			30	10	16	0.13	0.26	10	8	3.1	0.32	<0.1	26	<2.0	0.3	5.9
P50	6.1	12.5			30	10	2.5	0.11	0.22	9.4	8.2	3.2	0.2	<0.1	23	<2.0	0.24	6
P80	6.3	36.7			80	27	3.9	0.42	0.67	30	27	10	0.89	0.2	83	<2.0	0.74	17
P90	6.3	81.1			150	54	5	1.4	1.5	66	60	21	2.1	0.4	160	<2.0	1.7	32
PM	6.4	130			230	83	11	2.6	2.7	110	100	36	4.8	0.9	280	3.9	3.5	53

Appendix E: Mass balances

Batch 1 - No acid dosing and higher cross flow

	Amount in feed (mg/l)	% in concentrate	% in permeate	% lost or gain by other meanings or inaccuacy
NH_4^+	120	80.14	11.08	8.77
COD(Cr)	1400	37.36	1.78	60.86
Fatty acids	130	35.04	0.83	64.13
PO_4-P	11	29.14	0.03	70.83
Ptot	20	59.05	0.29	40.66
Ca^{2+}	39	69.22	0.83	29.95
K^+	48	91.39	5.89	2.72
COD(Mn)	190	53.28	1.31	45.41
Cl^-	150	47.24	7.76	45.00
Ntot	250	67.49	5.99	26.53
Mg^{2+}	6,4	84.36	1.30	14.34
Na^+	67	93.18	6.82	0.00
SO_4^{2-}	32	110.73	5.20	-15.92
Suspended solids	610	44.26	0.00	55.74
Stot	22	84.36	2.00	13.64
TOC	250	52.64	1.40	45.96

Batch 2 - No acid dosing

	Amount in feed (mg/l)	% in concentrate	% in permeate	% lost or gain by other meanings or inaccuacy
NH_4^+	190	64.30		
COD(Cr)	1700	47.43		
Fatty acids	170	40.24		
PO_4-P	18	43.44		
Ptot	23	63.74		
Ca^{2+}	40	67.19		
K^+	55	77.74		
COD(Mn)	260	46.99		
Cl^-	110	68.86		
Ntot	200	262.65		
Mg^{2+}	6,9	63.74		
Na^+	93	77.50		
SO_4^{2-}	26	150.36		
Suspended solids	650	30.07		
Stot	19	115.73		

Appendix E: Mass balances

TOC	310	78.82		

Batch 3 - Acid dosing, constant pH - 6

	Amount in feed (mg/l)	% in concentrate	% in permeate	% lost or gain by other meanings or inaccuacy
NH_4^+	150	68.93	14.29	16.78
COD(Cr)	1600	86.61	3.07	10.32
Fatty acids	160	86.61	18.43	-5.03
PO_4-P	10	89.54	2.41	8.05
Ptot	21	81.21	4.68	14.11
Ca^{2+}	40	103.93	3.35	-7.28
K^+	50	87.41	17.33	-4.74
COD(Mn)	220	92.06	2.84	5.10
Cl^-	120	230.95	58.07	-189.02
Ntot	530	52.29	18.54	29.17
Mg^{2+}	8	97.27	4.47	-1.73
Na^+	93	83.67	16.33	0.00
SO_4^{2-}	13	237.78	13.74	-151.53
Suspended solids	750	100.91	0.00	-0.91
Stot	21	91.36	5.11	3.53
TOC	310	123.78	5.19	-28.97

Batch 4 - No acid dosing, higher amplitude (3/4 inch)

	Amount in feed (mg/l)	% in concentrate	% in permeate	% lost or gain by other meanings or inaccuacy
NH_4^+	97	43.34		
COD(Cr)	1300	73.50		
Fatty acids	150	67.95		
PO_4-P	10	33.12		
Ptot	18	70.78		
Ca^{2+}	39	75.13		
K^+	40	74.85		
COD(Mn)	230	55.39		
Cl^-	82	63.70		
Ntot	140	100.10		
Mg^{2+}	6,3	79.88		
Na^+	65	77.42		
SO_4^{2-}	15	0.85		
Suspended solids	650	63.70		
Stot	19	80.46		

Appendix E: Mass balances

TOC	210	100.10		

Batch 5 - No acid dosing, higher amplitude (1 inch)

	Amount in feed (mg/l)	% in concentrate	% in permeate	% lost or gain by other meanings or inaccuacy
NH_4^+	150	81.51	18.51	-0.02
COD(Cr)	1500	81.51	3.04	15.45
Fatty acids	140	97.04	1.38	1.58
PO_4-P	11	22.45	2.48	75.06
Ptot	23	75.17	1.66	23.17
Ca^{2+}	39	88.67	1.17	10.16
K^+	50	83.98	12.54	3.48
COD(Mn)	180	31.56	5.33	63.11
Cl^-	95	15.60	22.12	62.28
Ntot	460	120.82	8.35	-29.16
Mg^{2+}	7,3	87.97	1.92	10.11
Na^+	76	86.13	13.87	0.00
SO_4^{2-}	19	31.20	23.12	45.68
Suspended solids	630	12.35	0.00	87.65
Stot	24	87.48	4.58	7.94
TOC	270	100.63	3.88	-4.51

Batch 6 - Acid dosing, constant pH = 6

NH_4^+	140	71.49	16.81	11.70
COD(Cr)	2400			
Fatty acids	330	55.31	0.29	44.41
PO_4-P	21	86.91	2.29	10.80
Ptot	30	76.54	3.76	19.69
Ca^{2+}	50	77.72	2.82	19.46
K^+	51	80.81	16.98	2.21
COD(Mn)	380			
Cl^-	95	285.09	67.36	-252.46
Ntot	350	89.16	8.34	2.51
Mg^{2+}	8,5	90.05	3.32	6.63
Na^+	77	84.11	15.89	0.00
SO_4^{2-}	18	235.51	10.46	-145.97
Suspended solids	1200	68.69	0.00	31.31
Stot	24	80.96	4.31	14.73

Appendix E: Mass balances

	4.62	29.27

Batch 7 Acid dosing, constant pH = 6 and higher ampliude (3/4 inch)

	Amount in feed (mg/l)	% in concentrate	% in permeate	% lost or gain by other meanings or inaccuacy
NH_4^+	120	53.87	12.17	33.96
COD(Cr)	1500	128.12	3.04	-31.16
Fatty acids	120	393.10	3.04	-296.15
PO_4-P	14	106.08	2.74	-8.81
Ptot	19	105.75	2.02	-7.76
Ca^{2+}	36	111.62	1.47	-13.09
K^+	49	89.14	11.18	-0.31
COD(Mn)				
Cl^-	210	145.59	20.86	-66.45
Ntot	260	80.64	6.67	12.69
Mg^{2+}	6,1	101.68	1.50	-3.17
Na^+	86	89.39	10.61	0.00
SO_4^{2-}	40	87.36	4.56	8.08
Suspended solids	750	128.12	0.00	-28.12
Stot	25	87.36	1.86	10.78
TOC	370	118.05	3.45	-21.50

Appendix F: Concentration and concentration difference in filtrated and un-filtrated

Ca^{+}	Initial feed				50% volume reduction				80% volume reduction				Maximum volume reduction			
Batch	Un-filtr [mg/l]	Filtr [mg/l]	diff [mg/l]	diff [mmole/l]	Un-filtr [mg/l]	Filtr [mg/l]	diff [mg/l]	diff [mmole/l]	Un-filtr [mg/l]	Filtr [mg/l]	diff [mg/l]	diff [mmole/l]	Un-filtr [mg/l]	Filtr [mg/l]	diff [mg/l]	diff [mmole/l]
1	39	28	11	0.274	70	58	12	0.299	-	-			160	100	60	1.497
2	40	30	10	0.250	84	73	11	0.274	180	120	60	1.497	210	130	80	1.996
3	40	27	13	0.324	88	74	14	0.349	220	190	30	0.749	-	-		
4	39	22	17	0.424	-	-			220	190	30	0.749	360	180	180	4.491
5	39	28	11	0.274	-	-			160	130	30	0.749	220	140	80	1.996
6	50	30	20	0.499	110	89	21	0.524	300	260	40	0.998	-	-		
7	36	26	10	0.250	83	78	5	0.125	270	270	0	0.000	590	540	50	1.248

Mg^{2+}	Initial feed				50% volume reduction				80% volume reduction				Maximum volume reduction			
Batch	Un-filtr [mg/l]	Filtr [mg/l]	diff [mg/l]	diff [mmole/l]	Un-filtr [mg/l]	Filtr [mg/l]	diff [mg/l]	diff [mmole/l]	Un-filtr [mg/l]	Filtr [mg/l]	diff [mg/l]	diff [mmole/l]	Un-filtr [mg/l]	Filtr [mg/l]	diff [mg/l]	diff [mmole/l]
1	6.4	5.5	0.9	0.037	-	-			-	-			32	26	6	0.247
2	6.9	5.7	1.2	0.049	16	15	1	0.041	34	29	5	0.206	42	26	16	0.658
3	8	6.9	1.1	0.045	-	-			45	43	2	0.082	-	-		
4	6.3	5	1.3	0.053	-	-			45	43	2	0.082	78	42	36	1.481
5	7.3	6.1	1.2	0.049	-	-			33	29	4	0.165	42	23	19	0.782
6	8.5	7.2	1.3	0.053	20	18	2	0.082	57	55	2	0.082	-	-		
7	6.1	5.2	0.9	0.037	14	14	0	0.000	34	34	0	0.000	110	110	0	0.000

NH_4^{+}	Initial feed				50% volume reduction				80% volume reduction				Maximum volume reduction			
Batch	Un-filtr [mg/l]	Filtr [mg/l]	diff [mg/l]	diff [mmole/l]	Un-filtr [mg/l]	Filtr [mg/l]	diff [mg/l]	diff [mmole/l]	Un-filtr [mg/l]	Filtr [mg/l]	diff [mg/l]	diff [mmole/l]	Un-filtr [mg/l]	Filtr [mg/l]	diff [mg/l]	diff [mmole/l]
1	120	130	-10	-0.555	410	450	-40	-2.218	-	-			550	530	20	1.109
2	190	170	20	1.109	-	-			820	850	-30	-1.664	1100	1100	0	0.000
3	150	130	20	1.109	270	-			670	640	30	1.664	-	-		
4	97	93	4	0.222	-	-			520	500	20	1.109	550	1100	-550	-30.501
5	150	150	0	0.000	-	-			780	750	30	1.664	1100	1100	0	0.000
6	140	120	20	1.109	310	310	0	0.000	860	990	-130	-7.209	-	-		
7	120	-			250	270	-20	-1.109	370	300	70	3ee882	1500	-		

Appendix F: Concentration and concentration difference in filtrated and un-filtrated

SO_4^-	Initial feed				50% volume reduction				80% volume reduction				Maximum volume reduction			
Batch	Un-filtr [mg/l]	Filtr [mg/l]	diff [mg/l]	diff [mmole/l]	Un-filtr [mg/l]	Filtr [mg/l]	diff [mg/l]	diff [mmole/l]	Un-filtr [mg/l]	Filtr [mg/l]	diff [mg/l]	diff [mmole/l]	Un-filtr [mg/l]	Filtr [mg/l]	diff [mg/l]	diff [mmole/l]
1	32	41	-9	-0.094	-	-			-	-			180	180	0	0.000
2	26	41	-15	-0.156	89	98	-9	-0.094	280	270	10	0.104	350	370	-20	-0.208
3	13	34	-21	-0.219	-	-			-	240			-	-		
4	15	26	-11	-0.114	-	-			160	160	0	0.000	2	170	-168	-1.749
5	19	37	-18	-0.187	-	-			210	280	-70	-0.729	350	420	-70	-0.729
6	18	26	-8	-0.083	88	84	4	0.042	280	300	-20	-0.208	-	-		
7	40	43	-3	-0.031	61	68	-7	-0.073	250	320	-70	-0.729	800	840	-40	-0.416

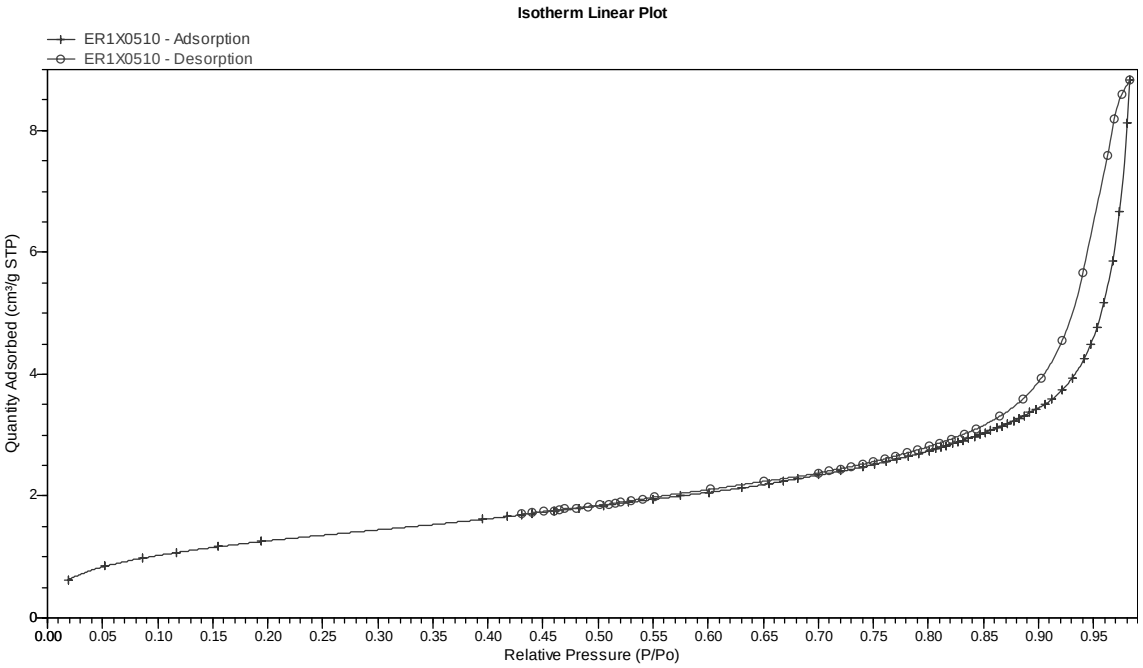
PO_4-P	Initial feed				50% volume reduction				80% volume reduction				Maximum volume reduction			
Batch	Un-filtr [mg/l]	Filtr [mg/l]	diff [mg/l]	diff [mmole/l]	Un-filtr [mg/l]	Filtr [mg/l]	diff [mg/l]	diff [mmole/l]	Un-filtr [mg/l]	Filtr [mg/l]	diff [mg/l]	diff [mmole/l]	Un-filtr [mg/l]	Filtr [mg/l]	diff [mg/l]	diff [mmole/l]
1	11	12	-1	-0.011	19	14	5	0.053	-	-			29	21	8	0.084
2	18	15	3	0.032	36	39	-3	-0.032	67	56	11	0.116	65	42	23	0.242
3	10	11	-1	-0.011	-	-			67	57	10	0.105	-	-		
4	18	12	6	0.063	-	-			82	64	18	0.190	210	61	149	1.569
5	23	15	8	0.084	-	-			83	52	31	0.326	120	59	61	0.642
6	30	21	9	0.095	57	56	1	0.011	160	150	10	0.105	-	-		
7	19	13	6	0.063	37	37	0	0.000	110	95	15	0.158	350	290	60	0.632

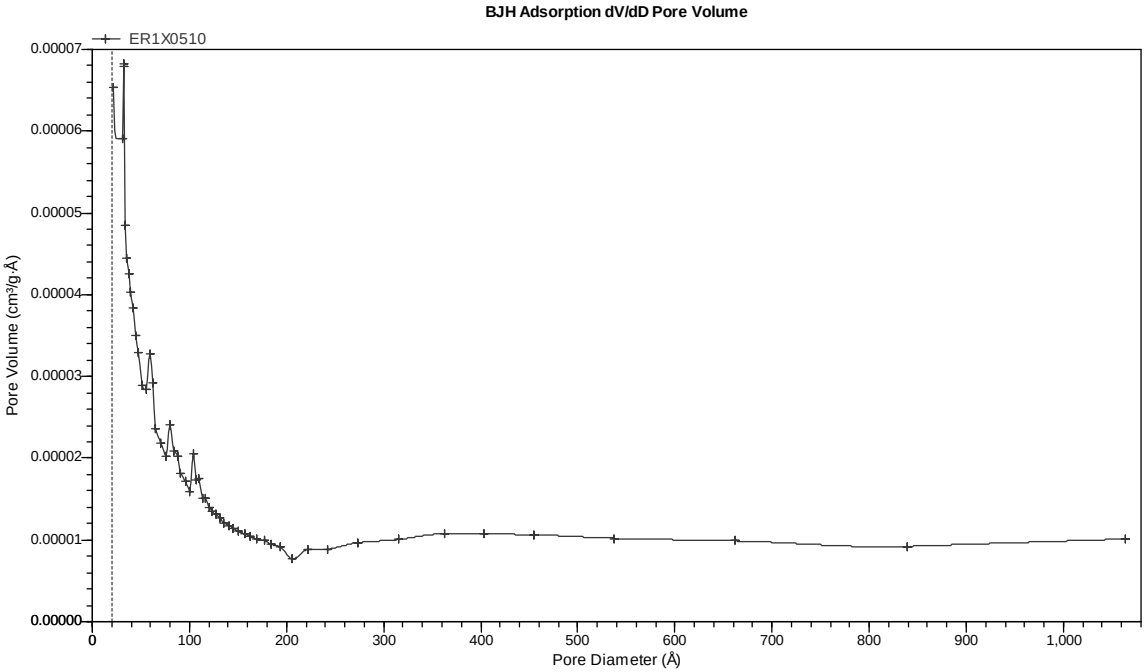
Appendix G: Ratio between permeate and concentrate concentrations

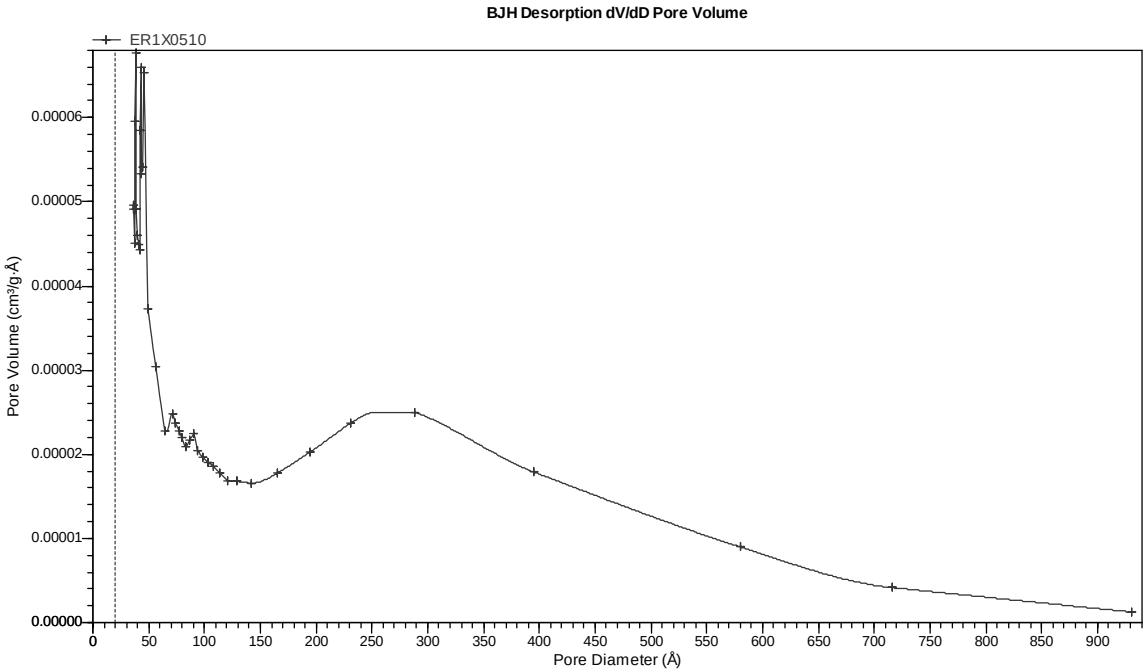
		COD(Cr)	PO4-P	Ptot	Ca	K	Na	TOC	Ntot	NH4-N
Batch 1	P0/F0	0.000	0.000	0.000	0.000	0.042	0.000	0.016	0.000	0.030
	P50/C50	-	-	-	-	-	-	-	-	-
	PM/CM	0.013	0.008	0.005	0.006	0.062	0.061	0.019	0.103	0.136
Batch 2	P0/F0	-	-	-	-	0.080	-	0.061	0.000	0.089
	P50/C50	-	-	-	-	0.066	0.065	0.043	0	0.085
	P80/C80	0.032	-	-	-	0.096	0.096	0.039	0.129	0.134
	PM/CM	0.045	0.048	0.023	0.020	0.170	0.158	0.051	0.156	0.200
Batch 3	P0/F0	0.000	0.000	0.000	0.000	0.102	-	0.055	0.000	0.093
	P50/C50	-	-	-	-	0.056	0.057	0.029	0.083	-
	P80/C80	0.020	-	-	-	0.061	0.055	0.023	0.063	0.073
Batch 4	P0/F0	0.000	0.000	0.000	0.000	0.058	-	0.048	0.000	0.067
	P80/C80	0.024	-	-	-	0.050	0.049	0.022	0.085	0.075
	PM/CM	0.032	0.074	0.011	0.011	0.149	0.140	0.040	0.239	0.436
Batch 5	P0/F0	0.033	0.034	0.016	0.009	0.056	0.071	0.041	-	0.0667
	P80/C80	0.033	0.043	0.020	0.011	0.092	0.090	0.041	0.038	0.121
	PM/CM	0.040	0.064	0.015	0.013	0.122	0.120	0.047	0.050	0.173
Batch 6	P0/F0	0.029	0.025	0.024	0.022	0.110	0.116	0.035	0.024	0.100
	P50/C50	0.016	0.016	0.016	0.008	0.056	0.055	0.020	0.033	0.058
	P80/C80	0.017	0.019	0.016	0.011	0.058	0.048	0.020	0.032	0.064
Batch 7	P0/F0	0.020	0.009	0.014	0.009	0.063	0.069	0.027	-	0.067
	P50/C50	0.012	0.003	0.005	0.002	0.029	0.030	0.017	0.014	0.033
	P80/C80	0.017	0.004	0.006	0.005	0.037	0.035	0.018	0.027	0.047
	PM/CM	0.014	0.009	0.008	0.008	0.046	0.038	0.017	0.012	0.067

Appendix H: Results from BET analyse

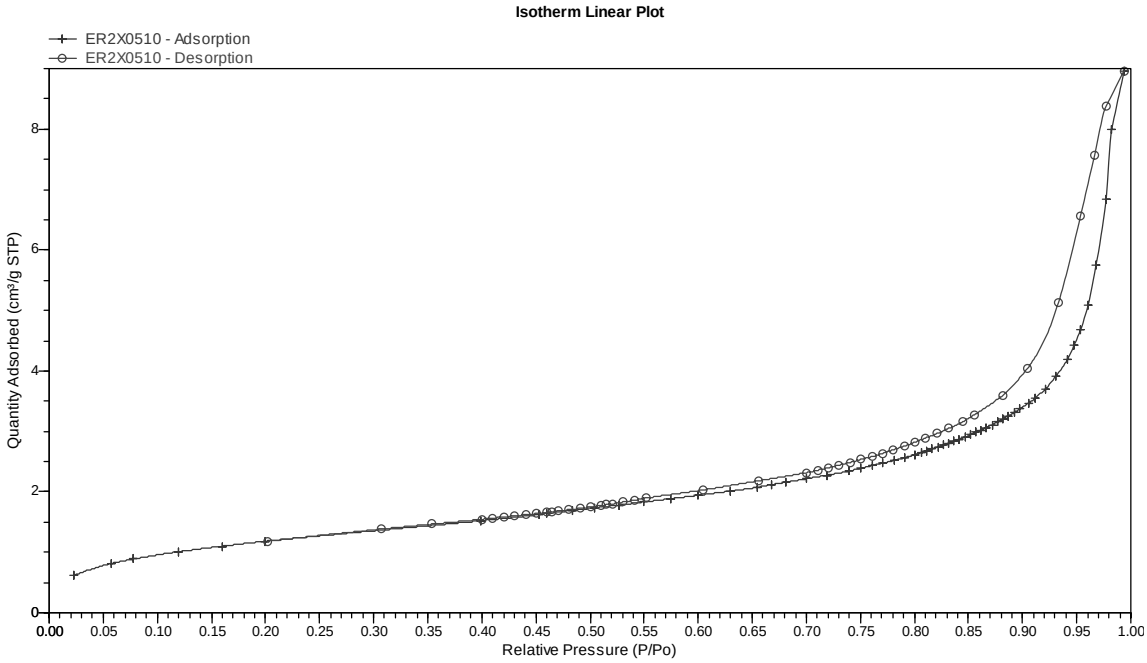
New LFC3 membrane

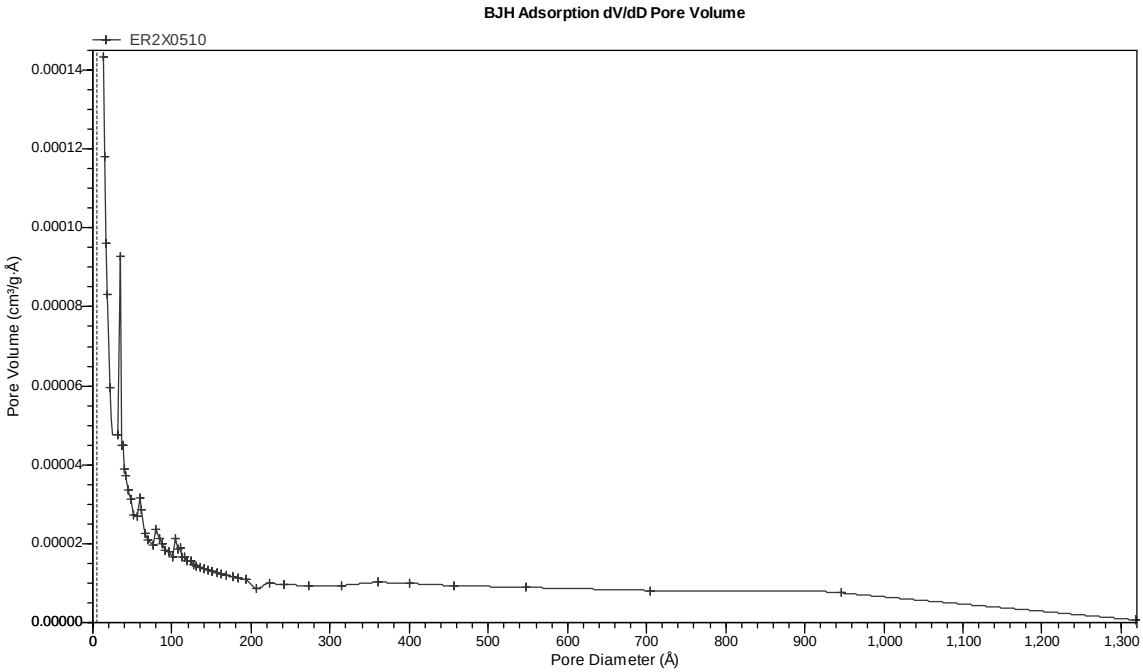


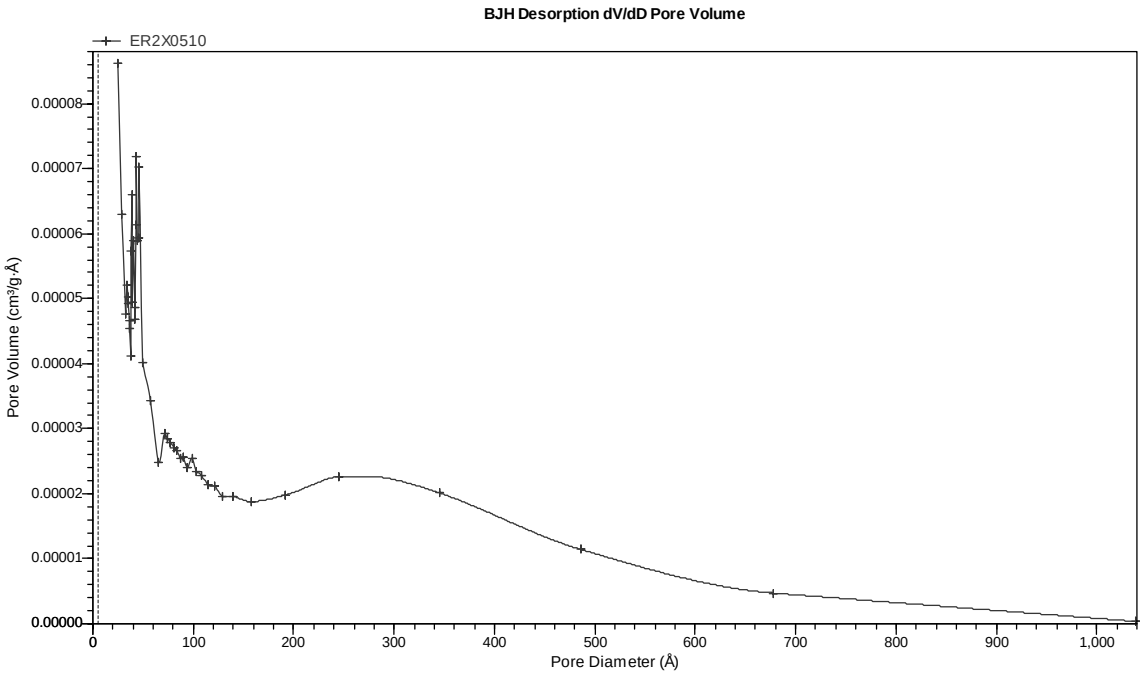




Used LCF1 membrane




BJH Adsorption dV/dD Pore Volume
ER2X0510
Pore Volume (cm³/g·Å)
0.00014
0.00012
0.00010
0.00008
0.00006
0.00004
0.00002
0.00000
0
100
200
300
400
500
600
700
800
900
1,000
1,100
1,200
1,300
Pore Diameter (Å)


BJH Desorption dV/dD Pore Volume
ER2X0510
Pore Volume (cm³/g·Å)
0.00008
0.00007
0.00006
0.00005
0.00004
0.00003
0.00002
0.00001
0.00000
0
100
200
300
400
500
600
700
800
900
1,000
Pore Diameter (Å)

Appendix I: All results from ALcontrol

Batch 0	Ca^{2+}
	mg/l
F0	25
F80	130
F80-f	130
PM	3.3
C0	31
C80	170
C80-f	140
CM	750
CM-f	660

Batch 1	pH	Conductivity	Suspended solids	TOC	COD(Cr)	COD(Mn)	Alkalinity	Fatty acids
		mS/m	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l
F0	7.5	150	610	250	1400	190	540	130
F0-f			72	150	490	95		15
P0	6.6			4.1				
P50	7.3			4.8				
PA	7.6			4.2	<30	3		1.3
PM	8.6			16	45	8		2.8
C50	7.7		530					
C50-f	7.8							
CM	8		1600	830	3400	580		240
CM-f	8.2		220	740	2400	410		66
TSED	8		1600	780	3100	600		270
TSED-f	8.2		420	700	2500	430		72

	Ptot	PO_4-P	NH_4^+	Ntot	K^+	Ca^{2+}	Mg^{2+}	Na^+	Cl^-	SO_4^{2-}	Sulfur
	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l
F0	20	11	120	250	48	39	6.4	67	150	32	22
F0-f	13	12	130	250		28	5.5			41	20
P0			3.6		<2.0			2.2	8.3		
P50			11		2.1			4	11		
PA	0.07	0.004	16	18	3.4	0.39	<0.1	5.5	14	<2.0	0.53
PM	0.34	0.22	75	90	16	0.95	0.3	23	40	<2.0	1.6
C50		19				70					
C50-f		14				58					
CM	68	29	550	870	260	160	32	380	420	180	110
CM-f	48	21	530	800	250	100	26			180	100
TSED	70	19	570	1000	260	160	32	370	420	210	110
TSED-f	50	19	510	810	260	110	29			190	100

Appendix I: All results from ALcontrol

Batch 2	pH	Conductivity	Suspended solids	TOC	COD(Cr)	COD(Mn)	Alkalinity	Fatty acids
		mS/m	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l
F0	7.7	190	650	310	1700	260	770	170
F0-f			30	230	680	110		21
P0	7.6	20.8		19				
P50	7.9	34.4		24				
P80	8.3	110		51	150	17		6.8
PM	8.5	200		96	260	23		3.9
C50	7.8	410	580	560	2300	280		270
C50-f	7.9	420		450	1600	240		62
C80	7.8	820	1500	1300	4700	610		420
C80-f	8	820		1100	3400	400		110
CM	8	1000	2000	1900	5800	860		450
CM-f	8.1	990	250	1600	4300	740		160
TSED	8	970	1600	2000	6600	1000		560
TSED-f	8.1	920	130	1500	3700	600		120

	Ptot	PO_4-P	NH_4^+	Ntot	K^+	Ca^{2+}	Mg^{2+}	Na^+	Cl^-	SO_4^{2-}	Sulfur
	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l
F0	23	18	190	200	55	40	6.9	93	110	26	19
F0-f	19	15	170	270		30	5.7			41	24
P0			17		4.4			9.3	20		3.4
P50			35		7.3			13	37		2
P80			110	270	26			44	80		
PM	3	3.1	220	560	56	4.2	1.4	90	170	14	8.5
C50	49	36	410	1100	110	84	16	200	230	89	62
C50-f	45	39	450	450		73	15			98	62
C80	89	67	820	2100	270	180	34	460	530	280	140
C80-f	72	56	850	1800		120	29			270	140
CM	130	65	1100	3600	330	210	42	570	640	350	180
CM-f	73	42	1100	2100	370	130	26			370	180
TSED	120	64	1000	4300	350	220	36	590	620	320	180
TSED-f	57	39	1000	2000	340	110	19			340	160

Appendix I: All results from ALcontrol

Batch 3	**pH**	**Conductivity**	**Suspended solids**	**TOC**	**COD(Cr)**	**COD(Mn)**	**Alkalinity**	**Fatty acids**
		mS/m	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l
F	7.4	160	750	310	1600	220	620	160
F0	6.6	170	640	410	1500	220	330	170
F0-f				210	660	100		18
F-f			13	170	590	100		24
P0	6	33		17				
P50	6.2	26.1		18				
P80	6.3	68.9		41	110	8		4
PA	6.6	37.8		18	55	7		33
C50	6.5	400	690	620				
C50-f			40	510				
C80	6.2	870	1900	1800	5400	760		540
C80-f	6.3	860		1200	3300	550		150
TSED	6.1	1180	7100	3600	13000	1900	1000	1300
TSED-f	6.1	1190	150	2100	5400	890	750	230

	Ptot	**PO_4-P**	**NH_4^+**	**Ntot**	**K^+**	**Ca^{2+}**	**Mg^{2+}**	**Na^+**	**Cl^-**	**SO_4^{2-}**	**Sulfur**
	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l
F	21	10	150	530	50	40	8	93	120	13	
F0	19	9.5		450		43					20
F0-f	15	12		390						34	20
F-f	14	11	130	410		27	6.9	90	130	34	21
P0			14	95	5.1			9.5	30		1.6
P50			18	62	6.7			12	50		0.9
P80			49	120	17			28	150		2.2
PA	1.1	0.27	24	110	9.7	1.5	0.4	17	78	<2.0	1.2
C50			270	750	120	88		210	710		49
C50-f				810		74					45
C80	100	67	670	1900	280	220	45	510	1800	<2.0	120
C80-f	94	57	640	1600		190	43			240	120
TSED	160	84	970	2600	410	390	73	730	2600	290	180
TSED-f	150	71	950	2300	400	290	68			330	170

Appendix I: All results from ALcontrol

Batch 4	**pH**	**Conductivity**	**Suspended solids**	**TOC**	**COD(Cr)**	**COD(Mn)**	**Alkalinity**	**Fatty acids**
		mS/m	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l
F0	7.4	120	650	210	1300	230	450	150
F0-f	7.7		41	170	490	70	490	15
P0	6.8	9.1		10			45	
P80	7.8	45.3		24	80	8	170	<1
PM	8.5	220		120	320	31	790	4
C80	7.9	580	1200	1100	3400	490	2200	330
C80-f	8	560		1800	2700	420	2300	79
CM	8	1170	4900	3000	10000	1700	2100	800
CM-f	8.2	1150	550	2400	7000	30	4600	220
TSED	7.7	1030	6500	3300	15000	2000	4800	1600
TSED-f	8.1	1020	620	2300	6600	670	4100	210

	Ptot	**PO_4-P**	**NH_4^+**	**Ntot**	**K^+**	**Ca^{2+}**	**Mg^{2+}**	**Na^+**	**Cl^-**	**SO_4^{2-}**	**Sulfur**
	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l
F0	18	10	97	140	40	39	6.3	65	82	15	19
F0-f	12	9.8	93	100		22	5			26	18
P0			6.5	12	2.3			4.8	7.1		1.5
P80			39	52	11			18	33		2
PM	2.3	2.3	240	550	76	4.1	2.3	120	230	22	13
C80	82	36	520	610	220	160	33	370	450	160	110
C80-f	64	38	500	550		130	29			160	100
CM	210	31	550	2300	510	360	78	860	780	<2.0	270
CM-f	61	40	1100	2100	520	180	42			170	270
TSED	200	52	660	2200	470	460	79	790	820	<2.0	240
TSED-f	68	47	1100	2000	450	170	37			<2.0	230

Appendix I: All results from ALcontrol

Batch 5	**pH**	**Conductivity**	**Suspended solids**	**Dry Solid**	**TOC**	**COD(Cr)**	**COD(Mn)**	**Alkalinity**	**Fatty acids**
		mS/m	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l
F0	7.2	160	630		270	1500	180	630	140
F0-f	7.6		64		200	640	15	620	23
P0	6.7	12.4			11	50	5	36	1.8
P75	7.4	32.1			3.8	35	20	140	3.1
P80	8	94.5			45	140	5	330	3
PM	8.7	150	<5		70	210	16	530	1.8
C80	7.8	730	1400		1100	4200	530	3100	360
C80-f	8	700	300		1000	3200	70	2900	130
CM	8.1	930	2000		1500	5300	760	4200	450
CM-f	8.2	910	600		1500	4700	55	3700	180
TSED	7.8	850	630		2200	9900	460	4200	1100
TSED-f	7.8	56.9	490		1300	4200	45	1300	160

	Ptot	**PO_4-P**	**NH_4^+**	**Ntot**	**K^+**	**Ca^{2+}**	**Mg^{2+}**	**Na^+**	**Cl^-**	**SO_4^{2-}**	**Sulfur**
	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l
F0	23	11	150	460	50	39	7.3	76	95	19	24
F0-f	15	8.1	150	390		28	6.1			37	23
P0	0.37	0.37	10	20	2.8	0.35	<0.1	5.4	8.6	<2.0	1.2
P75	0.24	0.02	32	49	6	0.32	<0.1	11	25	<2.0	0.64
P80	1.7	1.2	94	110	24	1.9	0.6	37	70	3.8	4.3
PM	1.8	1.8	190	200	45	2.9	0.9	67	130	71	5.5
C80	83	28	780	2900	260	170	37	410	490	210	120
C80-f	52	17	750	3100		120	21			280	120
CM	120	28	1100	4000	370	220	42	560	670	350	180
CM-f	59	17	1100	3900	370	140	23			420	180
TSED	140	20	990	4500	340	280	52	530	120	48	170
TSED-f	58	20	970	2200	340	130	23			340	160

	Nitrate + Nitrite	**Nitrite nitrogen**
	mg/l	mg/l
F0	0.19	0.089
F0-f		
P0	0.007	<0.001
P75	0.009	0.007
P80	<0.005	0.009
PM	0.009	0.004
C80	1.3	0.21
C80-f		
CM	1.6	0.2
CM-f		
TSED	1.8	0.26
TSED-f		

Appendix I: All results from ALcontrol

Batch 6	pH	Conductivity	Suspended solids	Dry Solid	TOC	COD(Cr)	COD(Mn)	Alkalinity	Fatty acids
		mS/m	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l
F	7.4	160	1200		570	2400	380	560	330
F0	6.6	170	1100		560	2400		250	330
F0-f	7	170	100		300	950		430	35
F-f	7.6	160	100		270	1200		610	34
P0	6.4	20			20	70		49	5.2
P50	6.3	24.7			22	60		26	2.2
P80	6.3	70.4			57	160		52	1.3
PA	6.6	33.9			28	90		43	<1
C50	6.2	410	1500		1100	3800	560	310	470
C50-f	6.4	410	340		730	2000		310	110
C80	6	990	2700		2800	9600	1200	860	1100
C80-f	6.1	1040	800		1700	5500		990	310
TSED	5.8	1860	14000	27100	6400			1800	3100
TSED-f	5.9	1830	2000		4200	12000		1600	650

	Ptot	PO_4-P	NH_4^+	Ntot	K^+	Ca^{2+}	Mg^{2+}	Na^+	Cl^-	SO_4^{2-}	Sulfur
	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l
F	30	21	140	350	51	50	8.5	77	95	18	24
F0	32	23		380	51	52	8.5	77	260	<2.0	22
F0-f	22	21		300	50	37	7.7			12	21
F-f	21	20	120	280	48	30	7.2			26	20
P0	0.72	0.53	14	18	5.6	1.1	0.2	8.9	35	<2.0	1.3
P50	0.86	0.9	18	22	6.7	0.92	0.2	9.9	48	<2.0	0.79
P80	2.5	2.7	53	61	19	3.2	0.7	25	130	2.3	2.1
PA	1.2	0.51	25	31	9.2	1.5	0.3	13	68	<2.0	1.1
C50	54	57	310	660	120	110	20	180	810	88	52
C50-f	52	56	310	690	110	89	18			84	49
C80	160	140	830	1900	330	300	57	520	2000	280	150
C80-f	150	140	820	1600	320	260	55			300	140
TSED	390	310	1700	5300	700	660	130	1100	4600	720	330
TSED-f	310	290	1600	4600	660	560	120			300	290

Batch 6	Red heated loss	Red heated loss	Red heated rest	Red heated rest of susp
	% av TS	% av susp	% av TS	% av susp
TSED		83		17

Batch 7	Red heated loss	Red heated loss	Red heated rest	Red heated rest of susp
	% av TS	% av susp	% av TS	% av susp
F	57.6	>95	42.4	<5
F0	59.8	>95	40.2	<5
C50		>95		<5
C80	61.3	>95	38.7	<5
C90	65.2	93	34.8	7
TSED	76.1	>95	23.9	<5

Appendix I: All results from ALcontrol

Batch 7	pH	Conductivity	Suspended solids	Dry Solid	TOC	COD(Cr)	Alkalinity	Fatty acids
		mS/m	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l
F	7.3	150	750	1110	370	1500	590	120
F0	6.3	160	420	1120	300	1200	220	160
F0-f	7		37		180	580	230	30
F-f	7.8		42		170	580	550	29
P0	6.2	12			10	30	16	16
P50	6.1	12.5			10	30	12	2.5
P80	6.3	36.7			27	80	23	3.9
P90	6.3	81.1			54	150	42	5
PA	6.6	22.8			14	50	23	4
PM	6.4	130			83	230	54	11
C50	6.3	350	680		600	2500	200	290
C50-f	6.4		52		490	1600	200	130
C80	5.9	850	1100	440	1500	4600	370	500
C80-f	6		170	5980	1100	3400	300	190
C90	5.8	1520	2500	14600	3100	9400	750	1000
C90-f	5.9		520	10000	2400	5900	810	480
CM	5.8		3600		4900	16000	980	1600
CM-f			490					
TSED	5.6	1320	11000	20000	5000	22000	850	5400
TSED-f	5.8	1700	620		2600	7200	700	490

	Ptot	PO_4-P	NH_4^+	Ntot	K^+	Ca^{2+}	Mg^{2+}	Na^+	Cl^-	SO_4^{2-}	Sulfur
	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l
F	19	14	120	260	49	36	6.1	86	210	40	25
F0	18	15	110	240	48	34	5.9	86	330	37	23
F0-f	16	13		270		30	5.3			43	22
F-f	13	12		300		26	5.2			43	22
P0	0.26	0.13	8	10	3.1	0.32	<0.1	5.9	26	<2.0	0.3
P50	0.22	0.11	8.2	9.4	3.2	0.2	<0.1	6	23	<2.0	0.24
P80	0.67	0.42	27	30	10	0.89	0.2	17	83	<2.0	0.74
P90	1.5	1.4	60	66	21	2.1	0.4	32	160	<2.0	1.7
PA	0.42	0.42	16	19	6	0.58	0.1	10	48	<2.0	0.51
PM	2.7	2.6	100	110	36	4.8	0.9	53	280	3.9	3.5
C50	41	37	250	660	110	83	14	200	870	61	28
C50-f	39	37	270	600	120	78	14	210	920	68	57
C80	110	94	570	1100	270	190	34	490	1900	250	130
C80-f	95	82	650	1100	270	180	34	490	1900	320	140
C90	210	180	1100	2100	550	410	70	990	4100	490	260
C90-f	190	180	1000	2000	560	380	72	1000	4400	540	130
CM	350	280	1500	9100	790	590	110	1400	6300	800	200
CM-f	290	200				540	110			840	200
TSED	230	170	740	2400	500	460	71	880	3500	400	250
TSED-f	220	160	1300	2400	610	410	79	1100	4700	610	150

Appendix I: All results from ALcontrol

	pH	Conductivity	Suspended solids	Dry Solid	TOC	COD(Cr)	BOD7	Alkalinity	Fatty acids
		mS/m	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l
Dissolved foul.in HCl					<1.0				
Filtrated last 19			120	0.14	68	430			13
Un filtr. last 19			1100	0.05	250	1400			110
Filtrated first 19			58	0.02	41	240			6.2
Un filtrated first19			530	0.07	130	830			72
NC2 without fouling	12	150	7		150	1900	<3	640	380
NC4 without fouling	<3.0	280	16		110	670	<3	<1	62
NC4 with fouling	<3.0	190	22		110	590	<3	<1	36
NC2 with fouling	12	150	43		180	2200	58	680	380
fouling.filtrated			77	0.047	88	630	200		20
fouling.un-filtrated			2300	0.18	410	2400	1300		140

	Ptot	PO_4-P	NH_4^+	Ntot	K^+	Ca^{2+}	Mg^{2+}	SO_4^{2-}	Sulfur
	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l
Dissolved foul.in HCl	1.6	1.1	<0.05	0.54		4	0.2	<2.0	0.14
Filtrated last 19	27	19	3.9	32	<2.5	49	4.1	<2.0	3
Un filtr. last 19	66	28	9.8	73	3.9	140	9	<2.0	5.7
Filtrated first 19	13	11	2.2	15	<2.5	25	2	<2.0	1.8
Un filtrated first19	31	19	2.7	41	<2.5	65	4.3	<2.0	3.1
NC2 without fouling	0.8	0.91	0.07	40	42	24	2.3	25	79
NC4 without fouling	450	450	0.32	13	15	22	2.5	<10	43
NC4 with fouling	430	430	0.33	120	16	30	3.1	<10	39
NC2 with fouling	1.6	1.2	0.2	97	43	28	2.4	27	89
fouling.filtrated	10	3.8	0.7	38	<2.5	42	1.6	2.5	4.5
fouling.un-filtrated	45	6.4	6.6	100	4.2	220	4	<2.0	12