

## Analysis of Fouling on RO VSEP (LFC 1) Membrane System used in Recovering Nutrients from Household Sewage Water of Skogaberg

*Master's Thesis in the International Master's Programme Applied Environmental  
Measurement Techniques*

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Cover page: Views of Analysis of Fouling on RO VSEP (LFC 1) Membrane System  
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## **ABSTRACT**

One of the major problems in concentrating nutrients in the VSEP RO system is fouling of the membrane surface by supersaturated inorganic metal salts or organic matter. Inorganic fouling can be delayed by acid or antiscalant dosing.

Three tests were performed on the VSEP with acid dose, antiscalant dose and no dose to check the concentration level up to which no fouling was observed. In each test membrane fouling was observed after concentration factors of 5-8 (approximately) irrespective of acid dose or antiscalant dose. Approximately 55 liters of black water was concentrated with the VSEP in each test. The unit was operated in the recirculation mode.

Samples, both filtrated and un-filtrated, were taken from the concentrate tank and permeate tank at the start, at 80% and at maximum volume reduction of the feed. The concentration of various chemical substances was measured on each sample taken.

The membrane taken out after the acid dose test was found to be coated with a thick layer of organic matter, whereas after antiscalant dose it was only moderately fouled.

A mass balance was performed on each chemical substance, at the end of each test. The efficiency of a membrane in each test could be determined by the extent of leakage of each chemical substance into the permeate.

For  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{PO}_4^{3-}$ ,  $\text{NH}_4^+$ , the difference between un-filtrated and filtrated samples started to increase at the 80% volume reduction point and was largest at maximum volume reduction. The increase in difference showed that a major precipitation of insoluble metal salts had started to occur after 80% feed concentration. The main salts that precipitated were probably  $\text{CaCO}_3(\text{s})$ ,  $\text{Ca}_3(\text{PO}_4)_3(\text{s})$  and  $\text{NH}_4\text{MgPO}_4(\text{s})$ . In each test the permeate flow started to drop after 80% volume reduction of feed, i.e. at a concentration factor of 5-8. Furthermore, the difference between un-filtrated and filtrated values of  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{PO}_4^{3-}$  and P-tot. was largest for the test with no dose, followed by the antiscalant and the acid dose tests. Hence, it can be predicted that most fouling should occur if neither acid nor antuscalant is dosed. Further investigations are, however, necessary before proper conclusions can be drawn.

The cleaning analysis of the membrane indicated that the fouling was mainly organic. The absence of inorganic fouling on the membrane surface could be due to formation of inorganic crystals on a pre-existing organic layer. Consequently it is possible that the inorganic salts were washed away during the cleaning long with the organic fouling.

The recovery of NH<sub>3</sub> and N-tot was higher (> 90%) for acid and antiscalant dose as compared to no dose test (~ 76%). Hence, by adding acid or antiscalant a better recovery of nutrients can be obtained.

It seems like a good strategy to stop the unit once a concentration factor of 5 has been attained and disposes of the permeate collected. Then the precipitation on the membrane is removed and the unit is restarted for further concentration. In this way, higher concentration factors could be reached.

**Key Words:** reverse osmosis, VSEP, concentrating nutrients, concentration factor, feed, concentrate, permeate, membrane cleaning analysis, fouling, inorganic metal salts, organic matter, acid dosing, antiscalant dosing, precipitation, percentage recoveries, percentage leakage, percentage loss

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# 1. INTRODUCTION

## 1.1 Background

Recycling of animal excreta back to the fields as fertilizers has been well known for ages. However there is a growing need of recycling crop nutrients from urban waste discharge (toilet flushing water and kitchen waste) back to farmlands and thereby making a sustainable agriculture. The City of Göteborg, Göteborg's Regional Sewage Works (GRYAAB), the Swedish Environmental Protection Agency and the Federation of Swedish Farmers (LRF), collectively aim to close the loop of ecocycle of plant nutrients, an essential step to build a sustainable society. In this context an extensive research is being carried out by combined contribution of City of Göteborg and Egnahembolaget, a local house construction company, to achieve this goal.

In Göteborg, as well as in many other towns in Sweden, there is only one wastewater treatment plant, Ryaverket, which collects wastewater from all sources i.e. from households, industries, landfills etc. It is a collection of nutrients along with heavy metals and pollutants. The wastewater is cleared from pollutants at the wastewater treatment plant. However, some pollutants are not degraded. The produced sludge is often accumulated with heavy metals and organic pollutants.

In the food industry there is a great fear of buying food products from farmlands where sewage sludge has been used as a fertilizer. There are also risks for infections from pharmaceuticals, hormones etc. As a result the Federation of Swedish Farmers (LRF) has no acceptance of sewage sludge fertilizers on there, resulting in no recycling of plant nutrients to Swedish farmlands. However LRF wishes to recycle nutrients from households on large scale provided they fulfill their requirements of safety and health is fulfilled.

The various sources of food nutrients like phosphorus and nitrogen include urine and faeces outlets from toilets, food leftovers from kitchen wastes, grocery and food shops and food industry. The sources of heavy metals and organic pollutants include industries, storm water and drainage. Recent studies have shown that the major contribution of heavy metals and pollutants is from grey water from households that includes water from bathing, dish washing and washing machine. In Göteborg, less than 7% of the heavy metals derive from industries <sup>[1]</sup>. Hence disconnecting the industry discharge into the sewage system will not make a significant contribution in collecting safe sewage sludge for farmlands. To make sustainable ecocycle of nutrients a modified household sewage system is needed and drinking water installations might be of other material than copper.

Thus in this context a pilot project is being carried out by the recycling office along with Egnahembolaget, the water and sewage works and GRYAAB, to make a separating household sewage collection system that facilitates safe recycling of nutrients to farmlands <sup>[1]</sup>.

## Separate sewage collection system



Egnahembolaget is a real estate company that has recently built 110 small houses in a place called Skogaberg, on Hisingen in Göteborg. The company wished to build a new sewage system along with the contribution from the recycling office that proves to collect cleaner and safer sewage from households, acceptable as fertilizers on agricultural lands. The conclusion was to make two separate household wastewater systems, one outlet collecting toilet wastewater plus food leftovers from kitchen as *black water* and the other collecting *grey water* from households

**Figure 1. A washbasin made for waste separation with two collection points: the left basin is used for collection of food leftovers and the right one is for rinsing.**

In ordinary kitchen basins in Sweden, there are two sections, the right one is for washing-up and the left one is for rinsing. At Skogaberg, food waste disposers are installed under the left section, which can be used for collecting food left over. This combined with the toilet flushing will be collected in one stream as black water. Very little amount of washing liquid present in black water coming from rinsing can be acceptable as sewage. Wastewater from the right washing-up basin along with bathing and laundry outlets can be collected as grey water. The grey water collection pipe discharges to Ryaverket sewage treatment plant while the black water will be transported in a different stream and treated for nutrient recycle in a pilot testing facility near the dwelling area of Skogaberg.

### 1.2 Aim of project

It is generally preferred to dispose off nutrient rich black water directly on field after little pretreatment. It is a cost effective and conventional way of recycling wastewater. However the Skogaberg residential area is located far from farmlands and it is unfeasible to transport household black water in bulk volume to the agricultural lands. The idea was to concentrate the black water to reduce its volume and recover useful nutrients like N, P and K in the form of concentrated slurry that is easy to transport to farmlands. The concentrated slurry can easily be digested and sprayed on farmlands. One key question is therefore how to concentrate nutrients in the best way.

A pre-study show that a pretreatment with a drum screen followed by a VSEP-reverse osmosis (RO) membrane should be suitable to concentrate the nutrients in the black water. As this application is new for the VSEP-RO system, a pilot testing plant has been built and test equipment installed in order to examine if the technology works as expected and to optimize the operation before a full scale plant is built.

The main aim of the project at the pilot testing facility is to:

- Getting a highly concentrated sludge from wastewater suitable for digestion as fertilizer.
- Reducing the volume of black water sludge as much as possible.
- Energy and chemical cost etc should be as low as possible and the final product that is produced should fit up to the environmental standards.
- The process should be easily manageable and should not give inconveniences for the surroundings <sup>[2]</sup>.

**Delimitation of the thesis**

The main aim of the thesis work was to investigate fouling potential on LFC1 reverse osmosis VSEP membrane system (L mode/single membrane unit) used for concentrating screened black water of Skogaberg. Other process parameters were not focused on in this study.

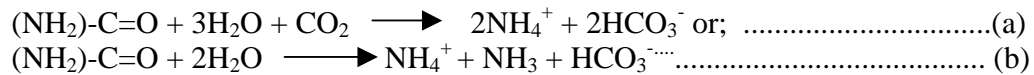


## 2. PROCESSES IN BLACK WATER

### 2.1 Biological Processes

#### 2.1.1 Ammonification of urea

Ammonification is a process in which urea from black water is transformed to ammonium<sup>[3]</sup>.



The above reaction increases the pH value to approximately 9. According to the stoichiometry, one mole of bicarbonate and one mole of ammonium are formed per mole of urea transformed. The main source of nitrogen in black water comes from urine/urea. The above reactions are mainly responsible for the values of pH and alkalinity in the black water. The transformation of urea to ammonium is a slow process that takes many days to get completed. However the reaction rate is accelerated/catalyzed by bacteria that emit an enzyme called *ureas* in black water. These types of bacteria are only present in faeces and not in urine. Hence, by mixing faeces with urine the reaction rate increases. The black water collected at Skogaberg is a mixture of urine and faeces. It is therefore expected to have high concentrations of alkalinity and ammonium. Furthermore, the rate of ammonification by enzymes is dependent on:

- 1) Temperature: The rate of reaction is directly proportional to the temperature of the black water and increases from 20 to 60 °C. When the temperature decreases from 20 to 10 °C the rate of reaction decreases to half its initial value. At 4 °C the rate of reaction is approximately zero<sup>[3]</sup>.
- 2) pH value: The rate of reaction is highest between pH range 7 to 8 (7 ≤ pH ≤ 8).

#### 2.1.2 Ammonification and acidification of organic substances

Hydrolysis of carbohydrates, fats and proteins present in black water converts them into fatty acids and sugars. These are further broken down into volatile organic fatty acids like acetic acid and propionic acid. Bacteria present in the black water carry out this transformation. This transformation into volatile acids lowers the bicarbonate alkalinity and pH value according to the following reaction<sup>[4]</sup>:

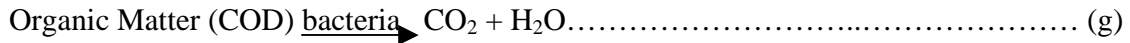


There are two different cases in which hydrolysis can occur:

- (i) Hydrolysis of organic particles (relatively slow).
- (ii) Hydrolysis of dissolved organics<sup>[3]</sup>

### 2.1.3 Aerobic degradation

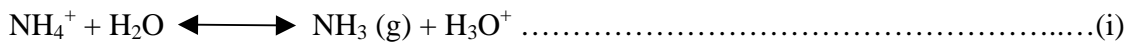
Aerobic degradations occur by heterotrophic bacteria, which oxidize the organic matter present in the black water to carbon dioxide (CO<sub>2</sub>), and water (H<sub>2</sub>O). The reaction is active at high organic loadings in wastewater. The reaction is as follows:



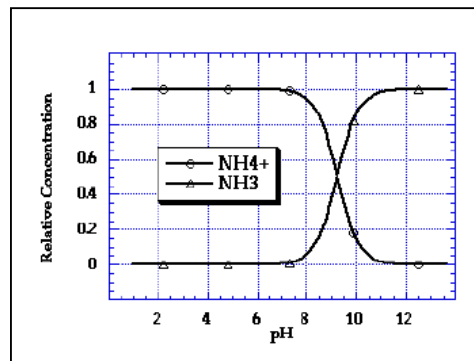
## 2.2 Chemical processes

### 2.2.1 Evaporation of ammonia

Ammonium and ammonia are produced during the ammonification process of urea. Ammonium and ammonia are in equilibrium with each other according to the following formula:



Ammonium and ammonia equilibrium is dependent on the pH and temperature. At a pH of around 9.0, 50% of (NH<sub>4</sub><sup>+</sup> + NH<sub>3</sub>) is NH<sub>4</sub><sup>+</sup> and 50% is NH<sub>3</sub>.



**Figure 2. Ammonium/ammonia concentration vs. pH of black water** <sup>[5]</sup>.

Ammonium is a positively charged ion and is highly soluble in water. Ammonia is a gas and has little solubility in water. For solutions above pH 8, ammonium concentrations quickly reduce, since the equilibrium between monovalent ammonium ions and neutral ammonia molecules begins to shift towards ammonia side. Since ammonia has little solubility in water, it diffuses out of the water

At a pH value of 9.2 half of ammonium is transformed into ammonia. If the pH value is 8, less than 10% of ammonium is ammonia. Vaporization of ammonia is significant at high pH values (8 or 9) and is minimum if pH is 7 or lower. If NH<sub>3</sub> vaporizes from the system, more NH<sub>3</sub> is produced by equilibrium, at the same time as the pH value and alkalinity decrease. In figure 2 ammonium and ammonia concentrations at different pH values of water are presented. At pH 6 almost all ammonium nitrogen is in ammonium form and at pH 11 all ammonium is converted into ammonia. If the aim of experiment is to retain the ammonium in the system, then pH must be kept less than or equal to 7. <sup>[5]</sup>

In the case of recovery of ammonia in VSEP reverse osmosis (RO) membranes, the membranes are charged to repel ions present in the feed water and this prevents them from passing through the membrane to permeate side. Ammonia gas molecule being neutral in nature easily permeates through the membrane. Ammonium on the other hand has a plus charge and permeates very little. During the experiments performed on digested hog manure in Canada, using LFC1 membrane, when pH is raised from 5 to 8.5 the ammonia rejection of an RO system dropped from 82% to 62 %. Therefore for maximum rejection/concentration of ammonium ions, the pH of the feed should be kept equal to or less than 7. The temperature change also affects the solubility of ammonia. The solubility decreases with increase in temperature<sup>[5]</sup>.

### 2.2.2 Solubility and precipitation

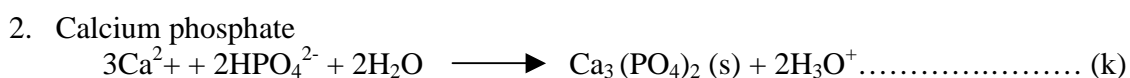
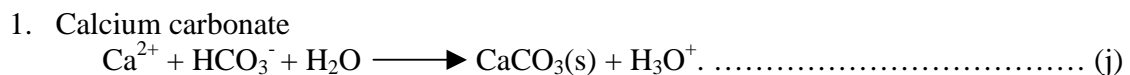
A solution containing anions and cations will tend to form ionic compounds depending upon the concentration or solubility limit of ions. In other words ionic compounds remain dissociated as cations and anions in solution depending upon their solubility limit. This is the maximum concentration of components (anions and cations) of an ionic compound that can remain dissociated in solution. This is measured in g/l and is termed as  $K_{sp}$  (solubility product constant).

If the concentration of components is below the amount that can be dissolved in solution, (below solubility limit), the solution is said to be dilute. In this case little or no precipitation of ionic compound occurs. As the concentration of reagents increases and reaches the solubility limit the solution is saturated. If the concentration exceeds the solubility limit, the solution becomes supersaturated. The level around the solubility limit, also known as metastable range, precipitation of ionic compound can occur if the conditions are favorable<sup>[6]</sup>.

The phase change from liquid to solid state occurs at liquid solid interface. These ions will look for nucleation sites (irregular solid surfaces) to get assistance in the initiation of the precipitation process. In a system with large numbers of liquid/solid interfaces or full of nucleation sites, precipitation can occur readily at the lower limit of the metastable range. For systems with low liquid/solid interface or nucleation sites, the solubility of the solution increases to super saturation state, as the access of ions to form precipitates at favorable nucleation site is limited. Precipitation of ions is also dependent on time, number of sites, temperature, pressure and many other variables<sup>[6]</sup>.

### 2.2.3 Precipitation of inorganic metal salts

The most important metal salts that may precipitate in black water are calcium carbonate, calcium phosphate, and magnesium ammonium phosphate. The reactions are as follows:



### 3. Magnesium ammonium phosphate



The precipitation of inorganic metal salts is highly dependent on pH and concentration of black water. The pH value and alkalinity of black water is lowered during by of precipitation<sup>[3]</sup>.

#### 2.2.4 Nucleation site

Chemical precipitation is a spontaneous process and takes place by itself. The free energy of the reaction for any spontaneous process decreases. However, for solutions full of saturated ions, precipitation/phase change from liquid to solid state does not take place without a presence of any pre-existing liquid/solid interfacial surface. This can be explained as follows:

The initiation of such a process in chemical solutions needs additional surface energy to take place spontaneously. At the start of any precipitation reaction, the phase change of ions from liquid to solid state results in a favorable energy decrease proportional to volume of solid precipitated or radius cubed, combined with an unfavorable energy increase proportional to new surface area created. The spontaneous precipitation reaction can occur by itself only when the overall free energy of reaction decreases. At the initiation point, the unfavorable surface energy increase is more than favorable volume energy decrease. This makes an increase in overall free energy of reaction, and stops the precipitation to occur by itself in absence of any pre-existing solid surface. In short, the precipitation of ions into solid state never occurs without presence of already present solid surfaces. Hence in the absence of solid surfaces/nucleation sites solution becomes oversaturated and no precipitation happens.

Hence in saturated solutions ions look for liquid/solid interface/nucleation sites for an initiation of precipitation. The surface energy proportional to the area of pre-existing solid surface becomes part of unfavorable energy increase of initial precipitation reaction. The overall energy change of initial precipitation reaction is only free energy decrease proportional to the volume of solid precipitated or radius cubed. Thus the nucleation site helps the precipitation reaction to occur at start by helping them to cross the initial energy barrier of new surface formation. If there are large numbers of nucleation sites, some are high on energy and some low. Precipitation will always occur on nucleation site high on energy to assist them to cross their energy hump<sup>[6]</sup>.



### 3. DIFFERENT ALTERNATIVES FOR NUTRIENT RECOVERY

In a recent study<sup>[2]</sup>, a comparison has been made on different technical alternatives for recycling of nutrients from black water collected at Skogaberg. They are as follows:

- Evaporation
- Ion exchange with zeolite/calcium silicate ( $\text{CaSiO}_3$ )
- Membrane techniques

#### 3.1 Evaporation

The black water collected at Skogaberg pilot station is still quite diluted and nutrient recovery through evaporation processes will end up in high-energy costs. The energy consumption is estimated to be 30-35 kWh/m<sup>3</sup>. The investment cost for an evaporation plant with a capacity of 1m<sup>3</sup>/h is estimated by VAPOTEC to be 2 MKr (Million SKr) (including other expenses like plumbing, construction and dosing). Thus due to high energy cost evaporation is not taken as a possible technique for black water filtration at Skogaberg<sup>[2]</sup>.

#### 3.2 Ion-exchange with zeolite

Ion Exchange is a process of removal of ions present in a solution using synthetic resins, which attract cations or anions, and release/exchange, other ions until the resin is completely saturated. Ion exchange resins can be regenerated or reused many times. The volume of water collected in treating the resin is small as compared to the amount of water treated by the resin. The concentrated slurry of nutrients/waste collected from resin-wash can be used for further treatment and other uses. In case of economical limitations the resins are not regenerated and disposed off after usage.

In the proposed black water treatment at Skogaberg by ion exchange, the water is first pretreated with precipitation and sedimentation to remove the suspended particles and phosphate present in it. In an ion exchange process the major ions like  $\text{K}^+$  and  $\text{NH}_4^+$  are exchanged with  $\text{Na}^+$  ions on filters containing resins like zeolite.

For a process with regeneration of zeolite, the filter has to be flushed with concentrated sodium chloride, which makes the handling too difficult at a small pilot station of Skogaberg.

In case of no regeneration large amount of zeolite is needed to absorb the major ions like ammonium present in black water. It is estimated to be 400kg zeolite/day. On annual basis it comes out to be 150mtons zeolite/yr. This becomes a too large figure regarding to resource efficiency.

Thus ion exchange process is not a good solution for nutrient recycling at Skogaberg [2].

### 3.3. Membrane Technique

The only technique that is appropriate for nutrient recovery at Skogaberg is membrane filtration. The energy costs are significantly less as compared to vacuum evaporation and ion exchange. The cost of dosing can be similar or lower. There are two alternatives for membrane-based separations.

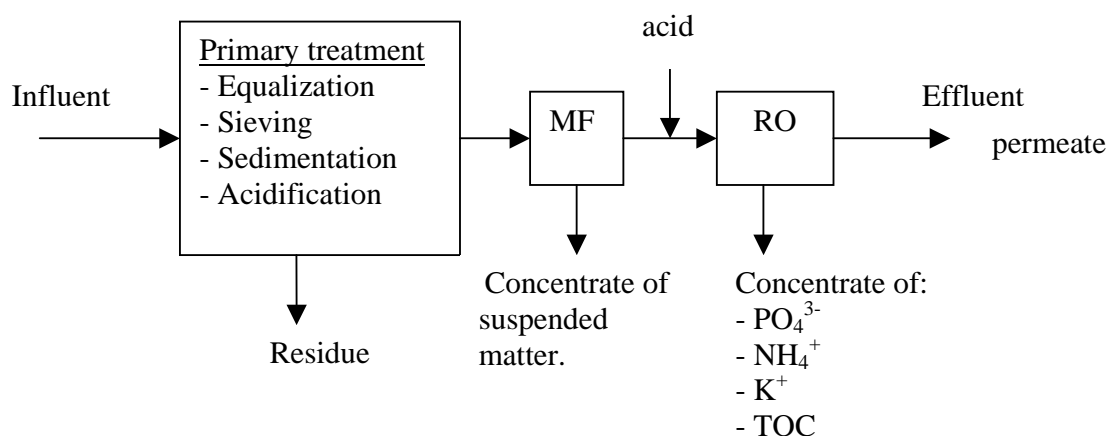
#### 3.3.1 Conventional membrane filtration

In this methodology black water is first filtrated through a Micro filtration (MF) system where major suspended solids are removed. The water is then passed to a RO reverse osmosis unit where nutrients are recycled. Acid is added after MF stage to stabilize the ammonium ions present in black water which need  $\text{pH} < 7$  to remain in solution as  $\text{NH}_4^+$  ions.

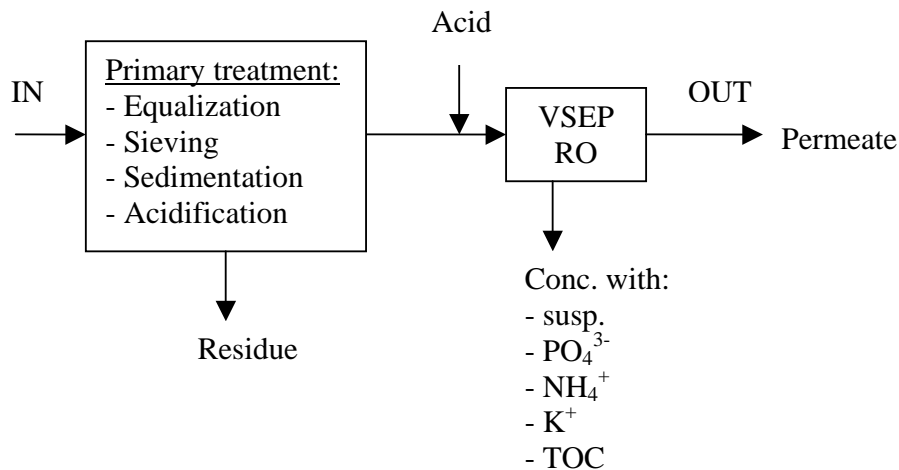
#### 3.3.2 VSEP membrane filtration

In VSEP membrane filtration the membrane is vibrated. This prevents it to get clogged by high concentration of suspended impurities. Laboratory tests have shown that it is possible to concentrate the black water directly after solid removal using RO membrane system.

In figure 3.1 and 3.2 both alternatives are presented and comparison is made in table 1. It has been found that energy consumption in the VSEP RO system is much smaller as compared to conventional membrane filtration and this brings VSEP RO treatment as the final alternative in nutrient recovery of black water at Skogaberg [2].



**Figure 3.1** Schematic description of micro filtration and reverse osmosis process.



**Figure 3.2 Schematic description of reverse osmosis with the VSEP technique.**

**Table 1. Comparison between conventional and VSEP membrane technique <sup>[2]</sup>**

System	Investment Mkr <sup>***)</sup>	Energy (KWh/m <sup>3</sup> )	Acid dosing mol H <sup>+</sup> / mol NH <sub>4</sub> <sup>+</sup>	Membrane cost in kkr/yr	Retention N/K/P	Ref.
MF – RO	1,5	5+8=13	< 1	ca 100	> 80 %	+/- <sup>*)</sup>
VSEP (RO)	2,5	3	< 1	ca 100	> 80 %	+/- <sup>**)   **)</sup>

<sup>\*)</sup> pig manure (Germany, The Netherlands), TRAAB (pilot-study, full-scale plant under construction)

<sup>\*\*) pig manure (Korea), several lab and pilot-experiments</sup>

<sup>\*\*\*)</sup> Coarse estimations, without extra costs due to construction etc.

### 3.4 Energy usage

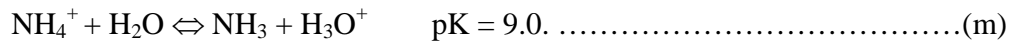
In cross flow membrane filtration system fluid is passed tangentially across the membrane surface and filtration takes place by water molecules permeating through the pores of membrane. By time these pores gets clogged due to suspended solids and deplete further filtration. To combat this, water is recirculated at a high velocity for preventing formation of cake layer on the membrane surface. Recirculation is needed to make filtration of clear permeate possible in the long run. Recirculation demands high-energy input.

In VSEP membrane filtration the recirculation is not strictly necessary. The vibration of membrane surface itself attains the filtration. This vibration of surface prevents any clogging of pores and lifts off suspended particles back into bulk solution that may set on it in static condition. Thus clogging is avoided.

The vibrating filter pack assembly needs much lower energy as compared to high energy consumption in recirculation of cross flow membranes. Hence energy consumption for VSEP is much less.

### 3.5 Dosing

Nutrient recovery of ions like nitrogen is an important objective of project. Nitrogen in black water is mainly in the form of ammonium ( $\text{NH}_4^+$ ) (50 – 70%). Ammonium is in equilibrium with ammonia gas in black water as:



By dosing acid to the black water the pH of the black water can be decreased below 7 ( $\text{pH} < 7$ ) and this shifts the equilibrium to the left making the loss of ammonium as ammonia limited. This highest relevant acid dosing is 1 mol  $\text{H}^+$ /mol  $\text{NH}_4^+$ . At this point all alkalinity of black water is removed and pH goes down to 4-5. Another reason for acid dosing is to inhibit precipitation of insoluble metal salts in the concentrate, which can clog the membrane surface. An alternative of acid dosing is antiscalant dosing<sup>[2]</sup>.

### 3.6 Choice of technique

After an analysis of various alternatives, VSEP RO membrane filtration is found to be the best alternative because a major part of the nutrients is recovered in the concentrate liquid. The energy consumption is low as compared to other technologies like Evaporation but investment costs are high. The technologies like evaporation or conventional membrane filtration either have high-energy consumption or are too intricate to be implemented at Skogaberg<sup>[2]</sup>.

## 4. REVERSE OSMOSIS

### 4.1 Donnan equilibrium

Donnan equilibrium is a state in which charge neutrality is maintained across the two sides of the membrane. Cations and anions present in concentrate and permeate pass randomly across the semi permeable membrane. The unionized molecules being small in size pass freely as no electrical balance is needed. The monovalent ions pass more easily as compared to multivalent ions being less in electric potential. However, the total electric potential of anions passing through membrane should be equal to that of cations. Donnan equilibrium is a condition in which the concentrate and permeate sides should always be electrically neutral <sup>[5]</sup>.

### 4.2 Concentration imbalance

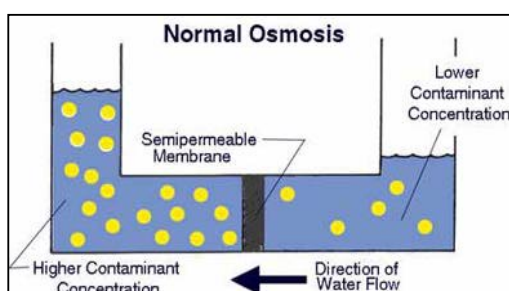


Figure 4. Osmosis process <sup>[7]</sup>.

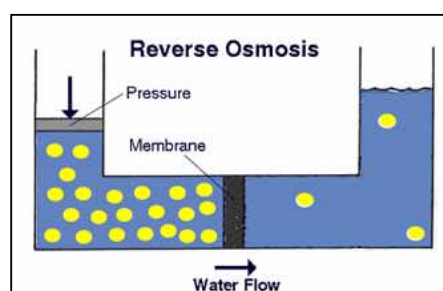
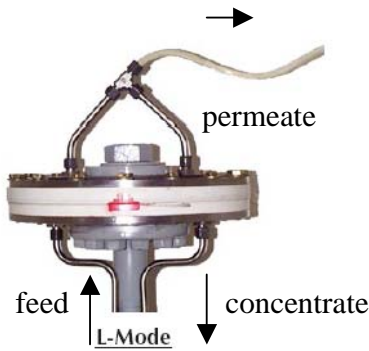


Figure 5. Reverse osmosis process <sup>[7]</sup>.

A concentration difference is established if the concentration of dissolved ions is higher on one side of the membrane than the other. The semi permeable membrane allows the small uncharged water molecules pass through it and prevents other ions. To maintain a concentration balance across the membrane water molecule will tend to pass across the membrane from lower ion concentration to higher ion concentration side. This process is called osmosis. Under atmospheric conditions a pressure is established on the higher concentration side to counter the diffusion of incoming water molecules. This pressure is known as osmotic pressure. As seen in figure 4 osmotic pressure is equal to the difference in water levels.

Reverse Osmosis is a process in which the natural osmosis process is reversed by pressurizing the water molecules from higher concentration side to pass to the lower concentration side across the semi permeable membrane (See figure 5). The semi

permeable membrane still permits passage of small water molecules and prevents ions and other bigger molecules pass to lower concentration side. Thus the term reverse osmosis denotes using a pressure higher than natural osmotic pressure of the system that pushes clean water molecules out of impure water and forces the water molecules in a reverse direction from where they would naturally migrate<sup>[5]</sup>.



**Figure 6. VSEP RO (1 mode) membrane side view<sup>[12]</sup>.**

In RO VSEP (1 mode) filtration, the feed side is concentrated with ions and water molecules and the permeate side is a clear liquid (see figure 6). Osmotic pressure is built around the membrane to counter the water molecules coming from permeate into the concentrate. To make a reverse osmosis process, a pressure higher than osmotic pressure is applied on the feed flow.

### 4.3 Charge Imbalance

A clear filtrate can be produced in the reverse osmosis process as the semi-permeable membrane prevents the passage of ions across it. However due to the donnan equilibrium, ions can pass through the semi permeable membrane and filtrate quality obtained is low. During the filtration process of a liquid containing a large quantity of monovalent anions and low number of monovalent cations, each monovalent anion that passes through the membrane will take a monovalent cation to maintain the charge neutrality. In a feed mixture of monovalent cations and multivalent cations, monovalent cations pass more freely across the membrane and a time will come when the feed side will be devoid of monovalent cations. The filtrate will have large concentrations of monovalent cations. This presents an inefficiency of RO membrane in retaining the monovalent cations in the feed.

In general the retention capability of RO membranes for monovalent ions like sodium and chloride is as high as 99%. However if the feed has weak acids like amino acids that have a small negative charge, they pass through semi permeable membrane easily. The ionic potential for these ions is much lower in comparison to chloride ion. To maintain a charge balance, each anion passing through the membrane, takes a cation like sodium or potassium along with itself to the filtrate side. In severe conditions monovalent cation concentration can be higher in permeate than in the feed. Thus, the membranes having 99% sodium chloride retention are found to have no rejection potential to sodium ions<sup>[5]</sup>.

#### **4.4 Saturation and precipitation of ions in RO filtration process**

In a reverse osmosis membrane filtration system, when pressure is applied and filtration occurs, permeate/clear water purges out through the membrane and consequently the concentration of solute to solvent changes. If the filtration process continues, the concentration of solute keeps on increasing until it reaches its solubility limit where precipitation is likely to occur. Once the precipitation has begun at appropriate nucleation sites, it increases more and more since the more water is removed more and more solid products are produced. During the process of precipitation, the solution will eventually try to keep its solute concentration around solubility limit due to precipitation. If the water is removed to an extent that concentration of solutes does not reach their solubility limit, precipitation will not occur. One common method to prevent precipitation, in conventional membrane systems is to concentrate the water to a limit where the solubility of the solutes is not reached. A second method is to add antiscalants that prevent the formation of precipitates. Antiscalants interfere with the formation of crystal growth by absorbing the nucleation site on which precipitation occurs. Another method is to add externally alternative sources of nucleation sites, also known as seeding, on which precipitation occur and fouling of membrane due to precipitation is minimized<sup>[6]</sup>.

#### **4.5 Kinetics of membrane scaling**

It has been thermodynamically proved that for an initiation of a precipitation process, nucleation sites are needed. During the process of membrane filtration there are simultaneously occurring processes, some of which are more prone to membrane fouling while some are not. As more and more water passes through the membrane, the solution gets more and more saturated. The degree of super saturation is maximum near the membrane and decreases with increases distance from it into the bulk solution. If there are nucleation sites in the form of solid particles present on the membrane surface these ions will precipitate on the membrane surface. The precipitation will occur at a high rate because the degree of super saturation is highest near the membrane surface. Hence the membrane gets fouled in this case. If there are no such particles present on the membrane surface, the ions will pass up into the bulk solution and will precipitate on the freely flowing colloidal particles serving as nucleation sites. These colloidal particles enter into the membrane system from the feed solution in the form of impurities. They can be any suspended solid, cake layer of organic matter or piece of metal etc. The precipitated colloidal particles will subsequently be drawn out with the reject stream and the membrane does not get fouled. It is important from fouling perspective whether the nucleation sites are present on the membrane surface or present in the bulk solution.

The nucleation sites can be any surface irregularity present on the liquid/solid interface. These surfaces can be any polymeric membrane, suspended solids or system components. Some nucleation sites are more preferred by precipitating ions than other, depending upon the amount of free energy available at each of them. Some solid surfaces have high free energy available while some do not depending upon the irregularity it has. The precipitation reactions always occur on nucleation sites highest on energy that assists

them to initiate spontaneously. In general, membrane surfaces are relatively low on free energy and are not the best destinations of crystal formation. Most precipitation will occur in the bulk solution where large numbers of solids with high irregular surfaces. Fouling occurs if the number of nucleation sites/irregular solid particles is more on the membrane surface than those away from the membrane surface. In other words the free energy of a nucleation site is directly proportional to irregularities present on it. The more the number of irregular particles the more the free energy it has and the more it is preferable to precipitate [6]

#### 4.6 Scaling problems in conventional membrane system

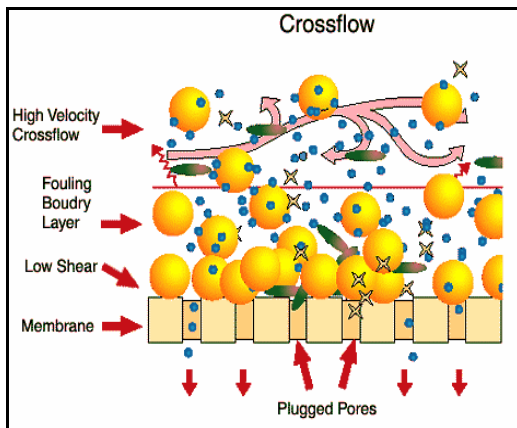
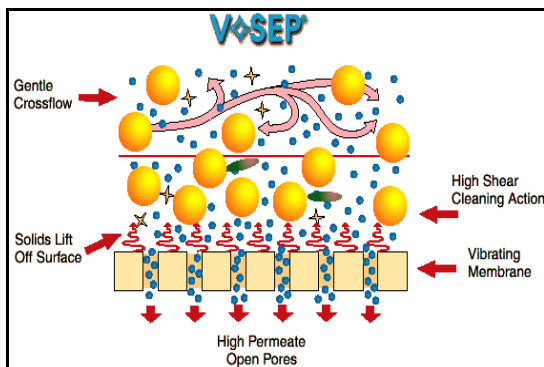


Figure 7. Low shear, high fouling in cross flow filtration system [8]

In conventional membrane filtration scaling problems occur because colloids of insoluble mineral salts are produced. These colloids float like any suspended solids. The main problem in conventional membrane filtrations is that these colloids become polarized on membrane pores and obstruct further filtration. The permeate flux gets reduced. To overcome this, concentration polarization cross flow of feed is used. However cross flow filtration cannot create shear force greater than 10-15 inverse per second and the polarized foulants are very little removed from the membrane surface. The membrane still gets fouled after some time. The conventional membrane systems have limits of total suspended solids TSS as fouling occurs if these levels are too high (see figure 7).

#### 4.7 Scaling formation in vibrating membranes



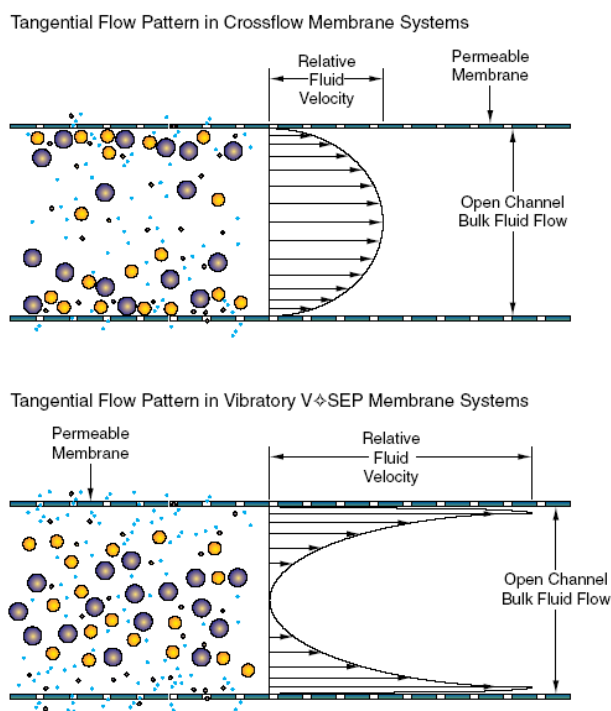


### **Figure 8. High shear, low fouling in VSEP system <sup>[8]</sup>**

In VSEP membrane systems the membrane surface is made to vibrate at a rate of 50 Hz, which prevents the diffusion polarization of suspended colloids. The sinusoidal shear waves created at the membrane surface pushes the incoming particles back into the bulk phase and leave the membrane surface clear for filtration. The result is that the repelled particles are kept as a parallel layer of suspended solids above the membrane surface that are removed by incoming tangential cross flow. This process of removal of colloids occurs at equilibrium. As suspended particles are removed more particles arrive. The removal and arrival rate occurs at equilibrium. The uplifted parallel layer also known as (diffusion/boundary layer) also acts as a nucleation site for precipitation of metal salts. The metal salts also behave like suspended colloids on the boundary layer. On increase in accumulation of precipitates, more are removed to maintain equilibrium. For high TSS concentrations in conventional membrane systems, more and more colloids settle on stationary membrane surface and the cake layer keeps on increasing. The filtration rate is completely reduced due to this. Unlike conventional membrane systems VSEP has no limitation of concentrations of TSS. This is because the diffusion boundary layer of suspended solids above the membrane surface is limited in size. As more particles arrive, more are removed to maintain equilibrium of the boundary layer. Thus VSEP has a capacity to filtrate almost any kind of liquid (See figure 8 above) <sup>[9]</sup>.

#### **4.8 The advantages of VSEP**

In general in VSEP, the precipitation of insoluble salts occurs on the suspended layer above the membrane surface instead of membrane surface itself. This is because the vibration makes the membrane surface low on free energy. Free energy is proportional to the amount of irregularities present in the form of peaks, valleys, and ridges present on any solid surface. With the movement of membrane surface back and forth at 50 Hz, the irregularities of the surface become more uniform or less prominent. The more uniform the membrane surface becomes the lower the free energy is available for crystallization. The solution becomes super saturated or the ions move into bulk solution and precipitate on the secondary layer acting as high energy nucleation site.



**Figure 9. Laminar flow patterns between conventional cross flow and VSEP [6].**

Crystallization of salts is also dependent on time factor. The vibrating membrane surface gives less time for crystal growth. The precipitation prefers to occur at other stationary targets of VSEP system. Similarly in conventional membrane systems, precipitation is more probable on membrane surface as it is stationary and gives enough time for crystallization (see figure 9 above).

Another advantage of VSEP is that filtration rate per  $m^2$  is much higher than conventional membrane systems due to suspension of colloids above the membrane surface. Studies have proved that flux increase of fifteen times is obtained with respect to conventional system. This makes the requirement of only one fifteenth of membrane area for same the same filtration rate as in conventional membranes. This makes an advantage of usage of large feed volumes in VSEP. Also since the filtration occurs quickly, the length of travel of feed water over membrane surface is reduced by as much as fifteen times. This makes less time for precipitation on membrane surface (see figure 8 above). [6]

Some other applications of VSEP are in calcium carbonate concentration, titanium dioxide dewatering, bentonite clay clarification, and other precipitated metal earth metal mineral slurries. [11]

#### 4.9 VSEP technical overview

Unlike flow-produced shear by conventional cross flow membranes; VSEP produces extremely high shear on the membrane surface because of high frequency oscillations. This is achieved due to combined resonance of high frequency torsion vibration of disk plate with mass spring system. The membrane is fixed to a plate that vibrates at amplitude of ½ inch to 1 inch peak to peak displacement. The frequency of vibration ranges from 50 to 55 Hz. The feed in the membrane remains almost stagnant producing a highly focused shear zone at the membrane surface. The accumulated solids are lifted off the membrane surface allowing for higher permeate rates. Feed pressure is provided by a feed-pump that continuously circulates new fluid to the membrane.

In general VSEP is simply two masses connected in between by a torsion spring, which is set to resonate at its natural frequency. The lighter of the two masses, i.e. a filter pack is set at the top of spring and the other mass which is seismic mass moves at smaller amplitude, proportional to the ratio of two masses. The use of two masses in this system allows the entire unit to resonate without attaching the device to a fixed mass.



**Figure 10. VSEP I mode unit** <sup>[10]</sup>.

An AC motor controlled by a solid-state speed controller of variable frequency provides the resonance to the two masses. The motor spins the eccentric weight coupled to the seismic mass. Since the eccentricity of the weight lies on one side, it produces a wobble in the system as the motor speed increases. This makes a similar wobble in the upper mass connected to the filter pack through the torsion spring, but with lag difference of 180degrees. With the increase in motor speed, the amplitude of the system reaches its maximum, and a greater speed only decreases amplitude. to reduce spring stress and infinite spring life VSEP is run below maximum amplitude <sup>[8]</sup>.

When the series l/p is configured for “L mode” operation (see figure 10 above), the filter pack consists of a single membrane enclosed in a “clamshell” assembly. The feed (process in) and concentrate (process out) piping connect at the bottom of the filter pack. The filtrate (permeate) piping connects at the top of the filter pack. The unit operates with continuous flow, limited by the “pumpability” of the feed slurry through the system.

When the series l/p is configured for “P mode” operation, the filter pack consists of a stack of membrane elements and spacers. Feed is pumped in at the top of the filter pack. The permeate exits at the top, and the concentrate exits at the bottom of the filter pack. Using a time cycled output valve can control the density of the concentrate. By adjusting the duty cycle of the valve the concentrate density can be optimized. The valve may also be controlled to maintain a constant vibration motor load or constant filter pack weight <sup>[12]</sup>.



## 5. MATERIAL AND METHODS

### 5.1 Experimental set-up

Black water was concentrated by using a LFC1 reverse osmosis VSEP membrane system. The operation was carried out with a single membrane stack (L mode) to check for the fouling potential on the membrane.

Black water treatment at Skogaberg is carried out in a series of step:

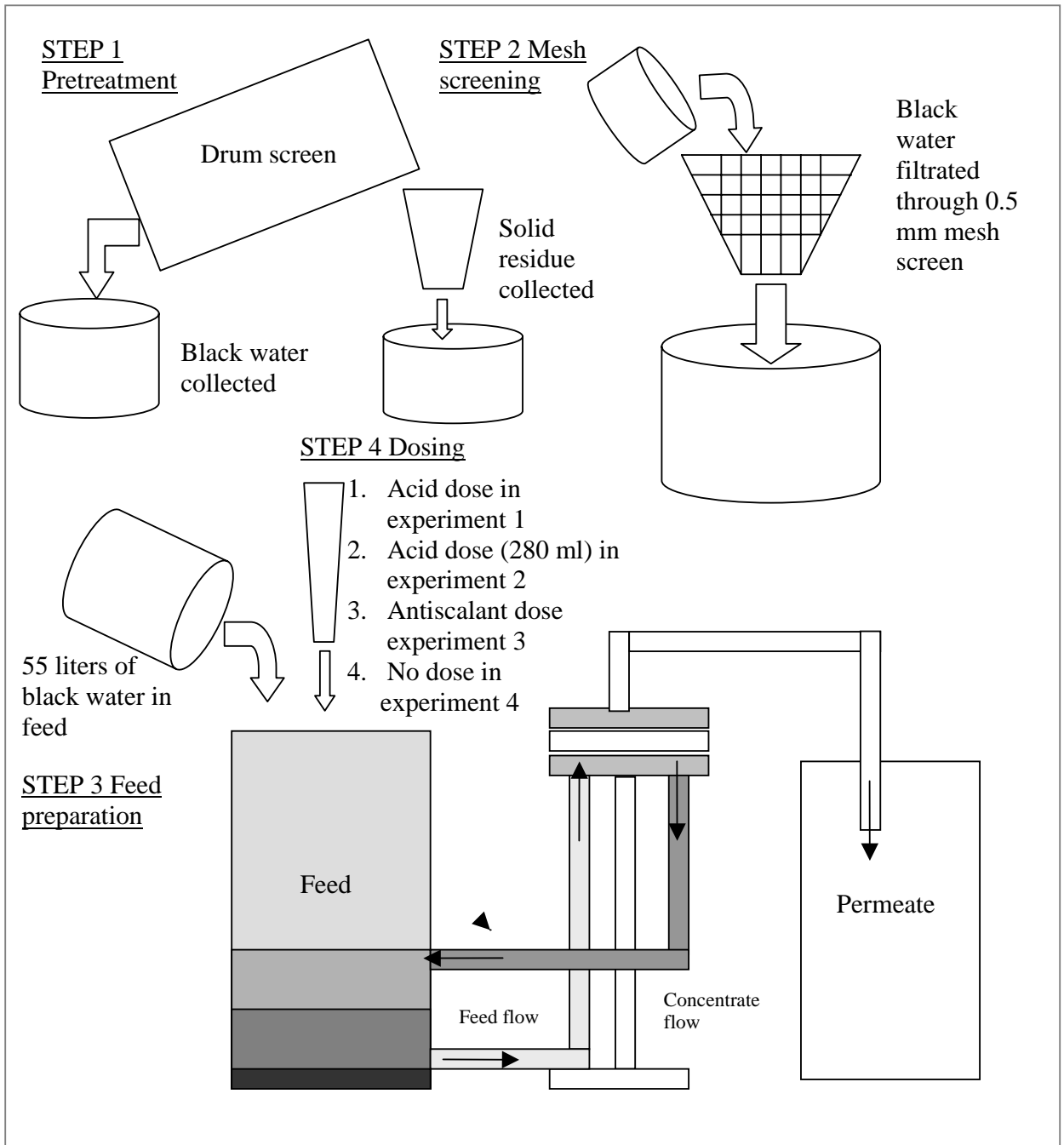
- First it is screened by a 0.6 mm drum screen, and then manually filtrated through 0.5 mm mesh screen to remove suspended particles.
- Then it is fed in a 60 liters batch container to make it ready for the VSEP system. It is predicted that when black water gets concentrated in the feed vessel some kind of fouling or precipitation in the tank will occur. The black water is a combined collection of kitchen leftovers and human faeces and urine. It has high concentrations of suspended solids, fatty acids and inorganic ions. It is predicted that when black water gets concentrated these metal salt will get oversaturated. The crystallized salts or highly concentrated organic impurities stick to the membrane surface and inhibits filtration across it. One common method is to add acids or antiscalants to the feed which prevents formation of crystals that can cause damage to the membrane and hamper the operation. Acid addition is also predicted to prevent the loss of ammonium ( $\text{NH}_4^+$ ) ions in the form of ammonia ( $\text{NH}_3$ ) gas. A loss can occur across the membrane surface if the pH of the black water exceeds 8. For this purpose three different VSEP batch tests were performed:

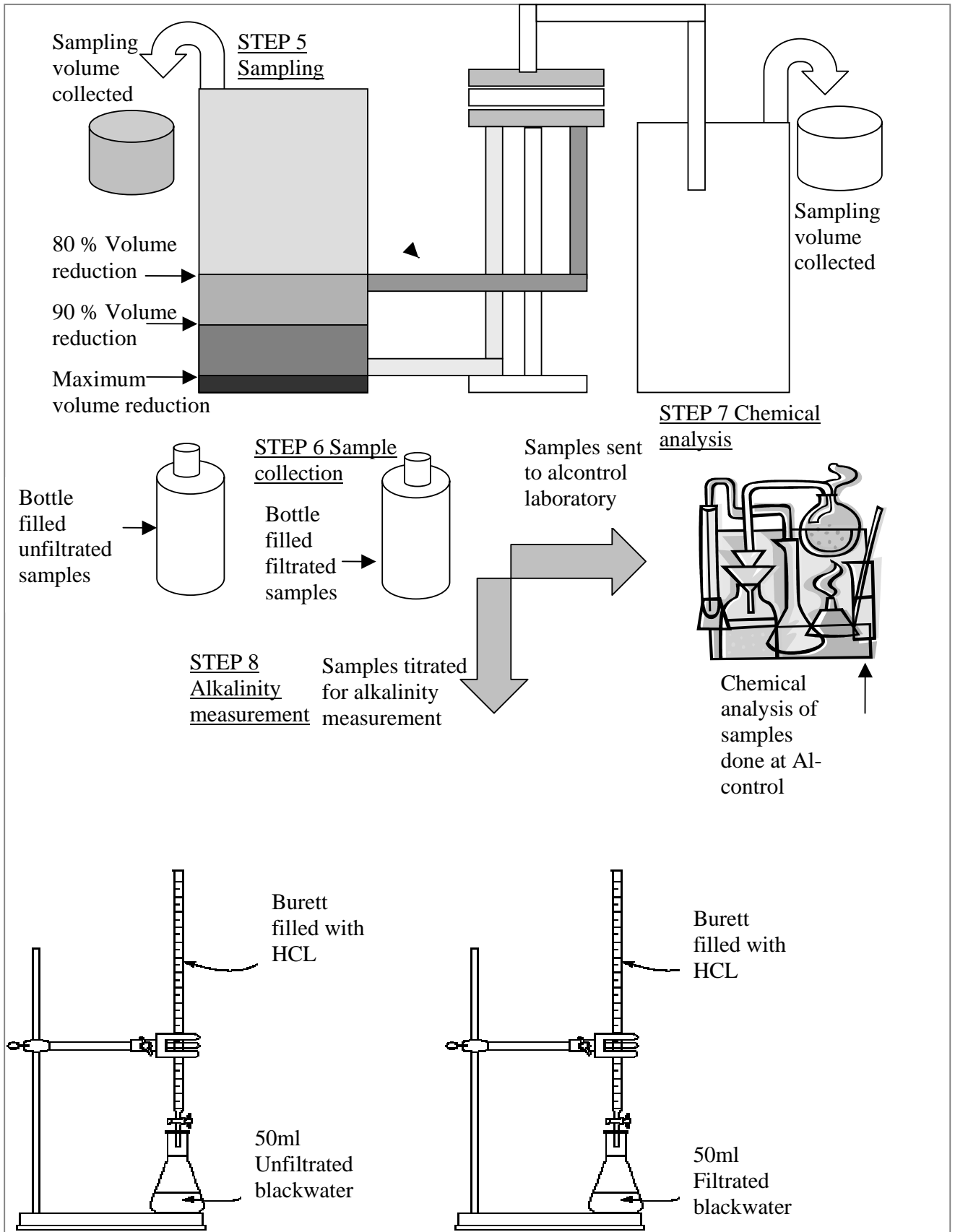
1. Acid dose (280 ml HCl)
2. Antiscalant dose
3. No dose

The first VSEP test with acid dose was not taken into consideration, as the concentration factor attained was too low to cause any significant fouling on the membrane.

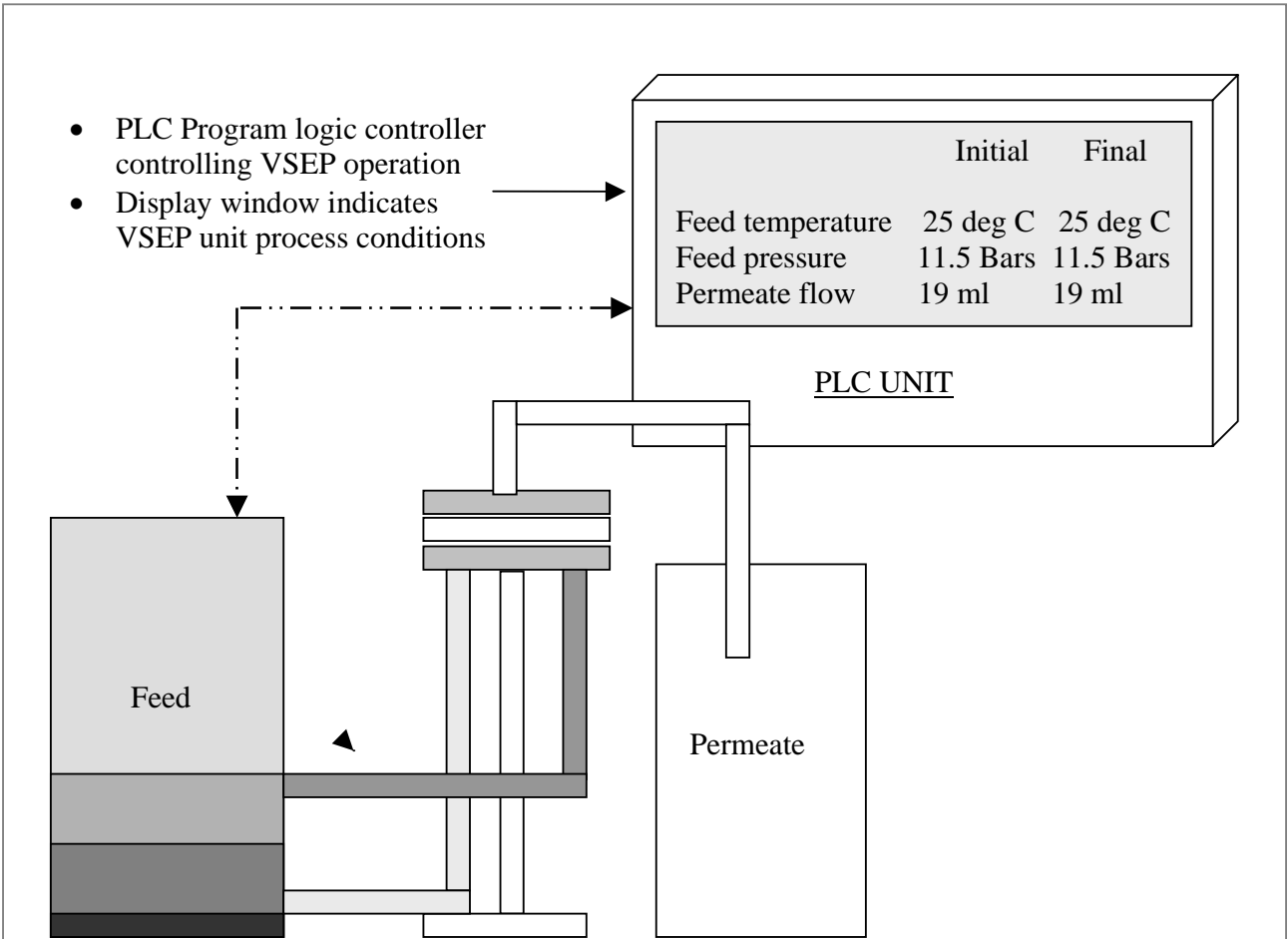
- During the course of experiment, samples were taken at different volume reduction. Samples were taken both on the concentrate and permeate. The samples were collected in two forms: one was filtrated and one un-filtrated. Chemical analysis and alkalinity measurements were carried out on both filtrated and un-filtrated samples. Chemical analysis was performed at Alcontrol laboratory and alkalinity titrations were carried out immediately at the pilot station. The difference in calculated values of filtrated samples and un-filtrated samples give an estimation of fouling potential on membrane or crystallization in tank.

- Membrane cleaning analysis was performed at the end of each VSEP test. The kind of fouling organic/inorganic was envisaged by cleaning the membrane with different kinds of detergents. The kind of fouling was assessed by the characteristic of a particular detergent on a certain kind of compound. For example detergent tornado was efficient in cleaning grease and aliphatic compounds. During the course of experimentation fouling was also predicted by the reduction in permeate flow across the VSEP membrane unit. The permeate flow reading was displayed by program logic control unit (PLC) that controls VSEP operation.
- A mass balance was carried out to estimate the loss or gain of the different chemical substances at the end of the VSEP tests under different dosing conditions.

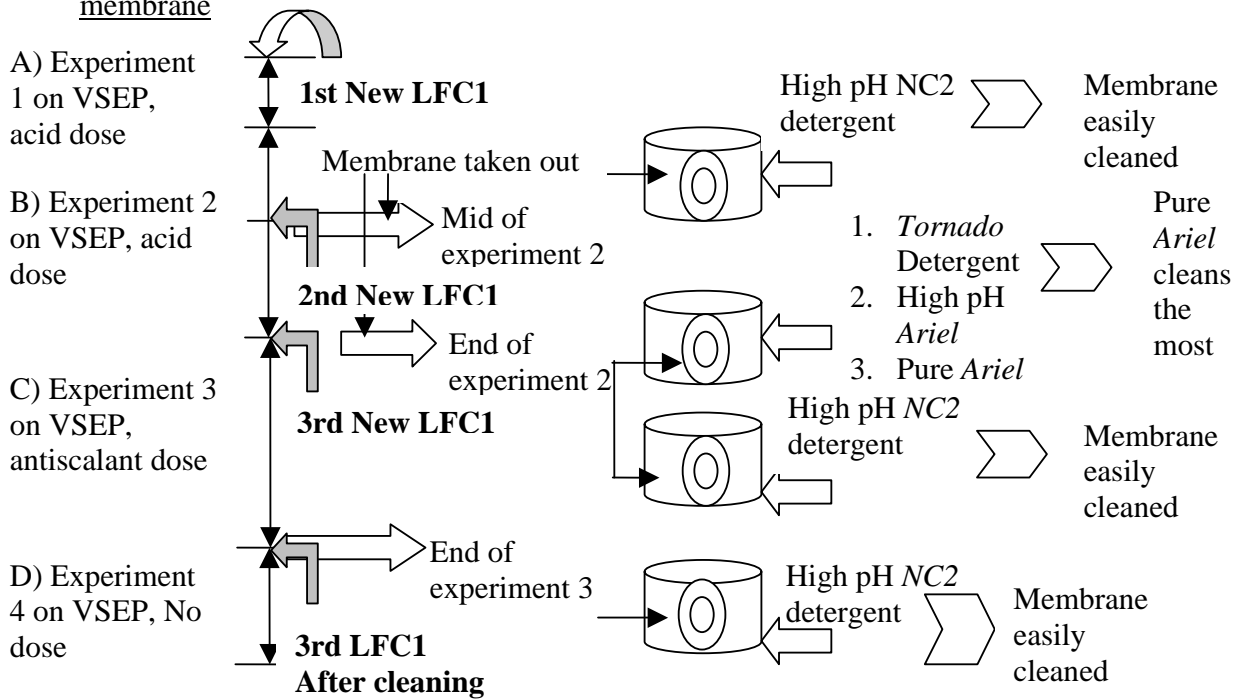


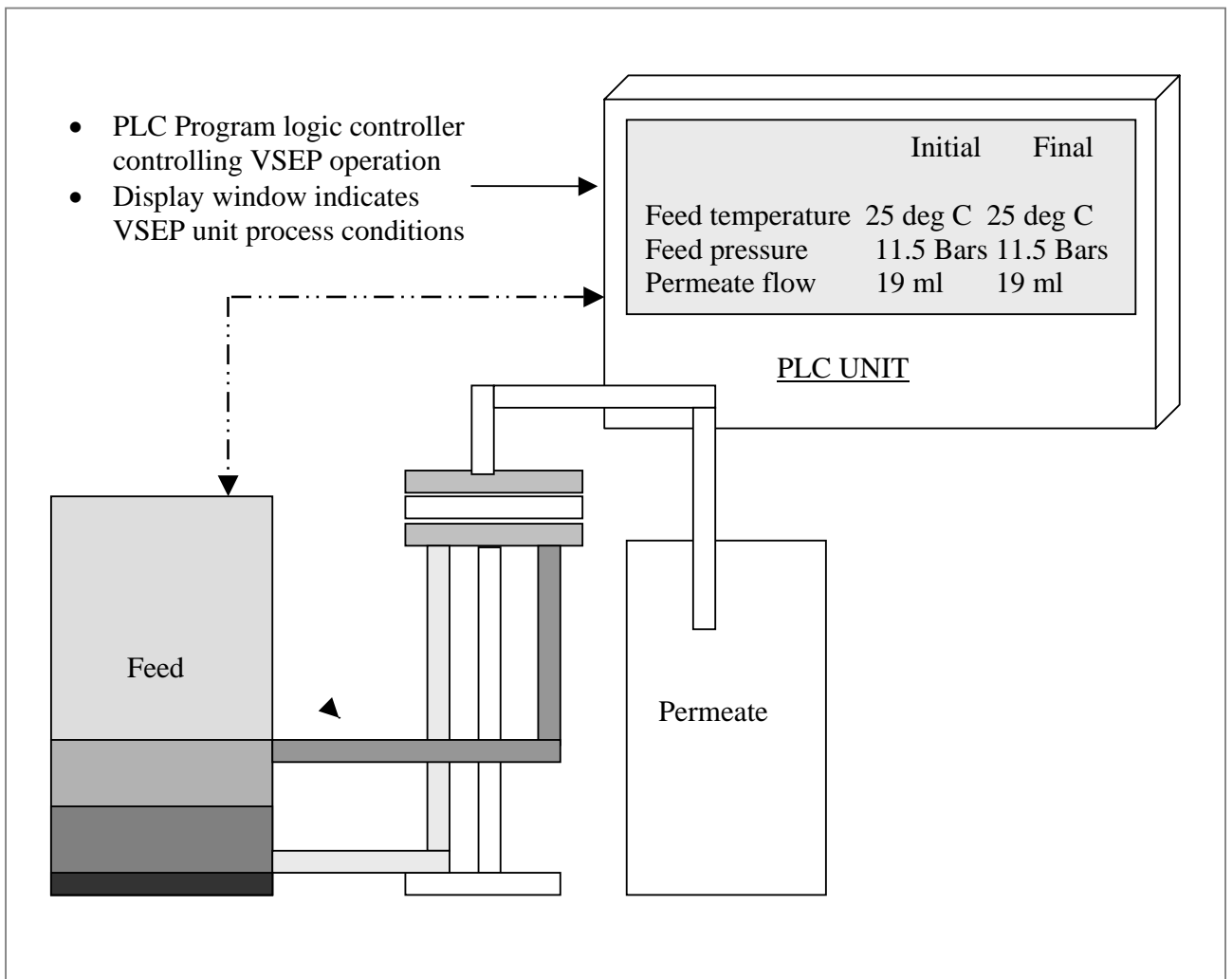






Step 8 Cleaning of membrane





**Figure 11. The step-by-step procedure of nutrient recovery from black water. The membrane cleaning procedure is also presented.**

## 5.2 Black Water Treatment at Skogaberg

### 5.2.1 Black water collection from drum screen

The drum screening process was carried out to remove big solid particles and impurities from the black water to be concentrated in the VSEP RO membrane system. (See step 1 in figure 11.) This makes the black water free from solid particles that can destroy the LFC1 membrane. The drum screen is a 0.5 mm mesh screen that filters the black water. It is an inclined cylindrical vessel in which sewage sludge enters from the right side (See appendix B: Pilot plant units). It has inner helical channels, which lift the solid sludge up to the right bottom outlet. The liquid black water pours down to the left bottom by gravity and drains out of the screen from the left bottom. Black water was collected in vessels from the left side of the bottom. Solid sludge was collected from the right bottom of the screen.

In all the three tests, black water was collected in the afternoon. It was assumed that the characteristics of the black water remained the same during a particular time of the day as the behavior of people is much related to the hour of a day.

### 5.2.2 Mesh Screening

The black water was again filtered through a 0.5 mm mesh screen to further eliminate any particles present in the black water. It was observed during mesh filtration that it got blocked with some “slimy” kind of substance, which appeared to be fat-like. The mesh was scraped and cleaned for further filtration. Care was taken that black water was not left for a long time, as then its characteristics would change. (See figure 11. step 2)

### 5.2.3 Dosing

Different dosing alternatives were tried to check their performance in controlling fouling/crystallization of metal salts on membrane.

In VSEP batch 2, 280 ml of HCl acid was dosed at the start of the experiment. In VSEP batch 3, antiscalant (falcon 260) was added and in the last experiment VSEP batch 4, no dosing is performed. (See figure 11, step 4)

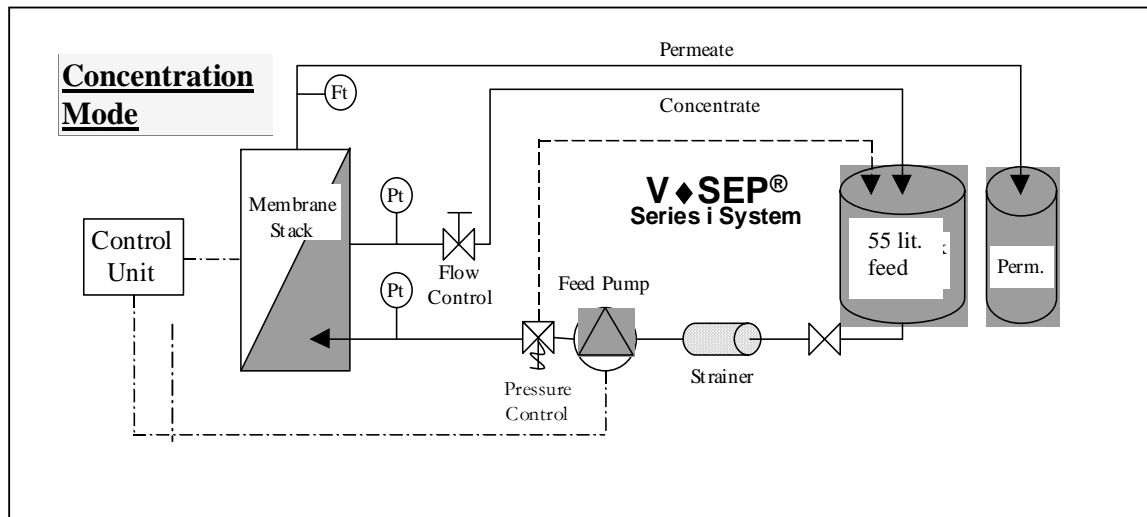
It was expected that acid dose will reduce precipitation of inorganic metal salts like  $\text{CaCO}_3$  and  $\text{CaPO}_4$  etc that can foul the membrane or crystallize in the tank. Antiscalant (falcon 260) is also expected to reduce fouling potential on membrane.

Falcon 260 controls  $\text{CaCO}_3$ ,  $\text{CaSO}_4$ , and  $\text{BaSO}_4$  scale formation. It is an aqueous solution of organic acids.

### 5.2.4 VSEP

The black water collected after pretreatment was filled in a 60 liter feed container. From this feed tank the black water was pumped into the VSEP system. The clear permeate was

collected from the top of the VSEP in a separate tank. The residual concentrate was pumped out of the VSEP system into a separate pipe and back into the feed vessel (concentration mode) to make the black water more and more concentrated. (See figure 12 VSEP batch test in concentration mode). The feed pressure was always fixed above the osmotic pressure build-up across the membrane. A Program Logic Controller PLC controlled the whole unit. PLC also displays the feed temperature, permeate flow and feed pressure in the system. During tests, conductivity and pH were manually measured. The conductivity is related to the concentration level in the tank and it should rise as the concentration increases.



**Figure 12. VSEP Batch test in concentration mode**

### 5.2.5 Sampling

Samples of concentrate and permeate were collected at different volume reductions and chemical analyses of the samples were carried out. Samples were collected at the start of experiment, and then at 80%, 90% and maximum volume reduction (See figure 11 step 4 dosing). Sample were also taken from the permeate tank. At each sampling point two sets of samples (four 500ml samples) were collected. One 500ml sample was filtrated through 1  $\mu\text{m}$  filter paper and the other 500 ml sample was un-filtrated. Titrations were carried out immediately and the rest of the samples were sent to Alcontrol laboratory for chemical analysis (See figure 11 step 5).

The chemical substances that were analyzed are suspended solids (SS), total dry substances (TS), total organic carbon (TOC), sulphate ( $\text{SO}_4^{2-}$ ), magnesium ( $\text{Mg}^{2+}$ ), total nitrogen (N-Tot), chloride ( $\text{Cl}^-$ ), calcium ( $\text{Ca}^{2+}$ ), total phosphate (P-tot), phosphate ( $\text{PO}_4^{3-}$ ), fatty acids, chemical oxygen demand (COD), ammonium ( $\text{NH}_4^+$ ), potassium ( $\text{K}^+$ ) and sodium ( $\text{Na}^+$ ) (See Appendix C: Al-control laboratory results).

The concentrate sample taken at the start of the experiment was denoted: AAD = concentrated sample after acid/antiscalant/no-dosing; AADF = Filtrated sample after acid/antiscalant/no-dosing; C80 = concentrate at 80% volume reduction and C80f = filtrated concentrate at 80% volume reduction. PA is the permeate sample from the tank taken as an average of total permeates collected. B1, B2, B3 and B4 are VSEP batch 1

with acid dose, VSEP batch 2 with acid dose, VSEP batch 3 with antiscalant dose and VSEP batch 4 with no dose, respectively.

### 5.2.6 Titration

Titration was performed on concentrated and filtrated samples collected at different volume reductions of the feed tank. Titration was used to calculate the alkalinity of the black water at different concentration levels in the feed tank. (See figure 11.Step 8). Alkalinity is a measure of the capacity of the wastewater to neutralize acid addition. The alkalinity is defined as:  $\text{Alkalinity} = [\text{CO}_3^{2-}] + [\text{HCO}_3^-] + [\text{OH}^-] - [\text{H}^+]$ . However, at pH 7-9,  $\text{HCO}_3^-$  is the dominating compound.

50ml of black water was titrated with HCl acid filled in a burette to pH 2.5. The volume of acid added at different pH levels plotted was plotted as described in [Appendix D, E, F and G: Titration curves and readings for VSEP batch testing 1, 2, 3 and 4 (with acid dose, with acid dose, with antiscalant dose and with no dose)]. Different strengths of acid (0.05 M, 0.005 M or 0.0005M) were used to titrate with samples of different concentration levels. The alkalinity values were calculated for an acid dose to reach pH 4.5.

### 5.2.7 PLC display

The PLC program and logic controller unit controls the VSEP unit in the three batches. Once the feed pressure of VSEP is set at 11.5 bars (standard for starting a test), the PLC controller records the feed pressure, feed temperature and permeate flow of VSEP system during operation at every 6 minutes of counter (counter displayed on PLC as DL reading). (See figure 11). The record for these three tests is shown in figure 13, 14 and 15 (chapter 6). The conductivity and pH of feed were recorded manually using pH meter and conductivity meter. To maintain reverse osmosis, the feed pressure should always be greater than the osmotic pressure developing across the membrane. This becomes significant at high concentrations of feed water. The PLC is capable of auto-control of feed pressure, however, during the tests performed the feed pressure was controlled manually. It was increased by one unit every time the permeate flow dropped. The increase in feed pressure with decrease in permeate flow can be found in figure 13, 14 and 15. However, during the last part of experiment the permeate flow dropped even on feed pressure increase. This quick decrease proves that it is due to fouling of the membrane surface.



## 6. RESULTS AND DISCUSSION

### 6.1 Pressure and flow

The first batch on VSEP testing started with a new LFC1 membrane. It was acidified with HCl acid. The system was fed with 55 liters of black water was to be concentrated. The concentration factor attained at the end of experiment was around 2. This was due to the fact that a large part of the feed volume was taken out as samples that reduced the feed volume very much. The concentration factor attained was too low to make any conclusion on fouling or precipitation.

In VSEP batch 2, 280 ml of HCl acid was added to the 55 liter feed. The pH of the solution was set to 6.0. The feed pressure was set to 20 bars, permeate flow observed was 26 ml and feed temperature was 26 °C. In the middle of VSEP batch 2 the permeate flow dropped to 12 ml/min, feed pressure to 20 bars, feed temperature to 31.2 °C. The permeate flow did not recover and the membrane was taken out and checked for fouling and a cleaning analysis was performed.

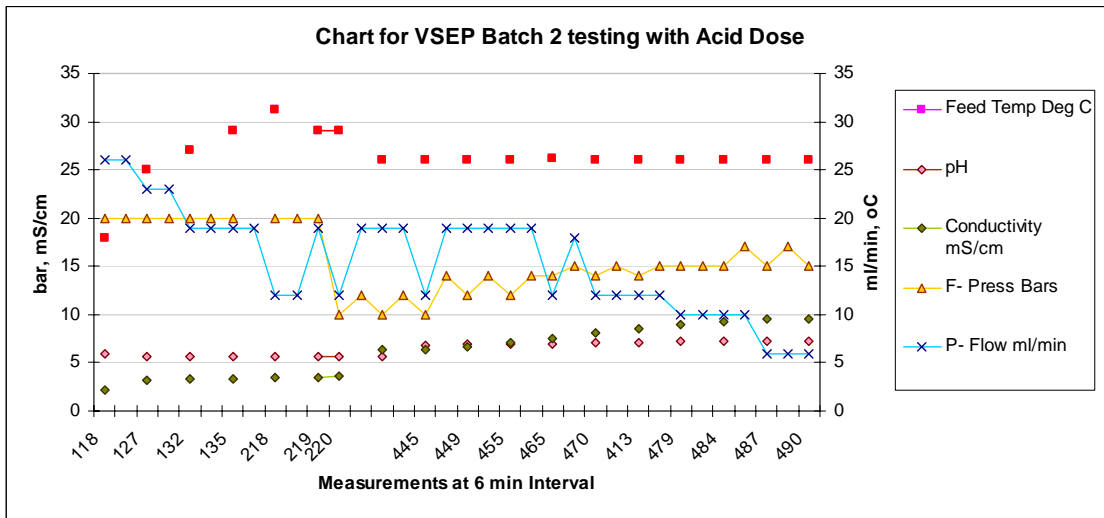
A new LFC1 membrane was installed for VSEP batch 2. The new feed pressure was 10 bars; feed temperature was 29 °C and permeates flow 12 ml/min. (See appendix 1: membrane cleaning). The black water got more and more concentrated until the permeate flow started to drop above 80% volume reduction of the feed. The feed pressure was increased to recover the permeate flow which brings up the permeate flow. Near maximal volume reduction the permeate flow was not recovered on feed pressure increase. The permeate flow finally dropped to 6 ml/min, feed temperature to 26 °C and feed pressure to 18 bars. The second LFC1 membrane was taken out and checked for fouling under cleaning of different detergents. The total duration of experiment was 49 hrs 40 min

For VSEP Batch 3, antiscalant *falcon 260* was added. A third LFC1 membrane was installed. The total duration of experiment was 47 hrs 50 min. the feed pressure, feed temperature and permeate flow at the start were 12 bars, 17 °C and 19 ml/min, respectively. As in previous test the feed pressure was increased on decrease in permeate flow level until the permeate flow did not recover at all. The final permeate flow attained was 10 ml/min. The feed pressure was increased to 20 bars and feed temperature to 32 °C. The increase in feed temperature makes the black water less viscous and increases the permeate flow across the membrane. In future experiments, it should be made constant in the feed by a heat exchanger to get proper condition for fouling investigation. The third LFC1 membrane was uninstalled from the VSEP unit and surveyed for fouling by cleaning in high pH detergent. The total duration of the experiment was 47 hrs 50 min.

After 4 days of cleaning the membrane was put into the VSEP system again for the fourth VSEP batch testing with no dosing. Initial feed conditions were feed temperature 21.2 °C, feed pressure 11.5 bars and permeate flow 23 ml/min. The permeate flow dropped in the

same way and completely ebbed out at 10 ml/min, feed temperature 30 °C and feed pressure 19 bars. The total duration of last experiment was 70 hrs 28 minutes.

The LFC1 membrane was not cleaning at the end of batch 4, no dose test. The PLC display observed during the working of three batch tests are presented in figure 13, 14 and 15. Figure 13 shows temperature, pH, and conductivity, feed pressure and permeate flow profile for VSEP batch test 2 with acid dose. The temperature of the feed and pH constantly rises and that can be the reason to the loss of  $\text{NH}_4^+$  and N-tot from the feed tank. The conductivity curve constantly rises, which corresponds to rising concentration in feed tank. When permeate flow decreases (see P-flow initial) the feed pressure is increased by one or two bars (F-pressure final). This makes the permeate flow back to normal value (P-flow final). The decrease in P-flow can be due to osmotic pressure developing across the across the membrane at higher concentration or due to membrane fouling. The F-pressure is increased to make it always higher than osmotic pressure (RO LFC1 membrane process). During the last part of experiment, the permeate flow dropped drastically even on increasing the feed pressure. This proves that drop in P-flow is due to fouling on membrane and not due to osmotic pressure across the membrane. The P-flow would drop smoothly if it is due to osmotic pressure.

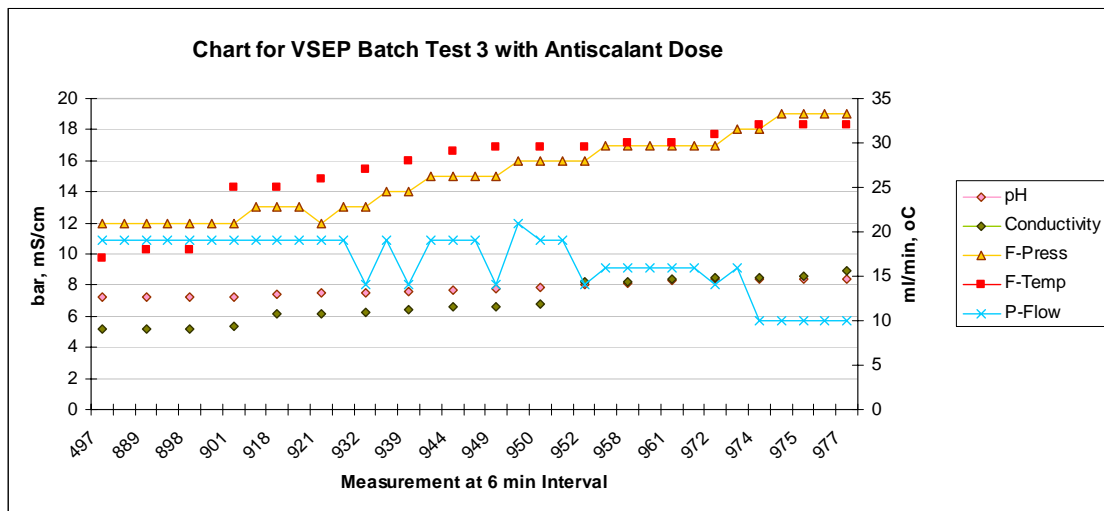


**Figure 13. Change in test parameters during VSEP batch 2 with acid dose.**

Figure 14 shows the temperature, pH, conductivity, feed pressure and permeate flow profile for VSEP batch test 3 with anti-scalant dose. The temperature of the feed and pH constantly rises and that can be the reason of loss of  $\text{NH}_4^+$  and N-tot from the feed tank. The conductivity curve constantly rises, proves rising concentration in feed tank. When permeate flow decreases (See P-flow initial) the feed pressure is increased by one or two bars (F-pressure final). This makes the permeate flow back to normal value ( P-flow final). The decrease in P-flow can be due to osmotic pressure developing across the across the membrane at higher concentration or due to membrane fouling. The F-pressure is



increased to make it always higher than osmotic pressure (RO LFC1 membrane process). During the last part of experiment, the permeate flow dropped drastically even on increasing the feed pressure. This proves that drop in P-flow is due to fouling on membrane and not due to osmotic pressure across the membrane. The P-flow would drop smoothly if it is due to osmotic pressure. The graph for antiscalant is almost same as for acid dose test (figure 13).



**Figure 14. Change in test parameters during VSEP batch 2 with antiscalant dose.**

Figure 15 shows temperature, pH, conductivity, feed pressure and permeate flow profile for VSEP batch testing 4 with no dose. The temperature of the feed and pH constantly rises and that can be the reason of loss of NH<sub>4</sub> and N-tot from the feed tank. The conductivity curve constantly rises, proves rising concentration in feed tank. When permeate flow decreases (See P-flow initial) the feed pressure is increased by one or two bars (F-press final). This makes the permeate flow back to normal value ( P-flow final). Decrease in P-flow can be due to osmotic pressure developing across the across the membrane at higher concentration or due to membrane fouling. The F-pressure is increased to make it always higher than osmotic pressure (RO LFC1 membrane process). During the last part of experiment, the permeate flow dropped drastically even on increasing the feed pressure. This proves that drop in P-flow is due to fouling on membrane and not due to osmotic pressure across the membrane. The P-flow would drop smoothly if it is due to osmotic pressure.

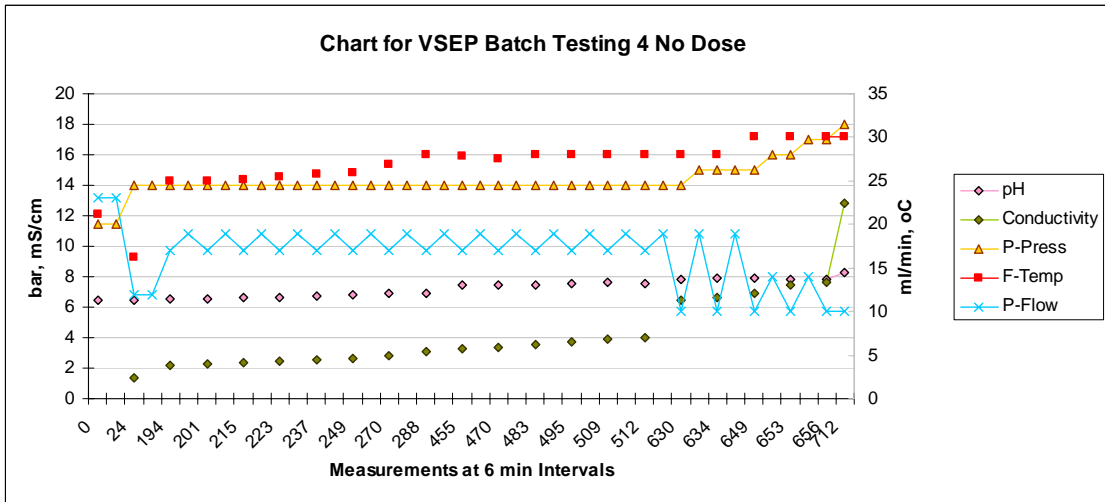


Figure 15. Change in test parameters during VSEP batch 3 with no dose.

## 6.2 Membrane Cleaning

Different membrane cleaning procedures were tested on fouled membranes taken out after each VSEP batch test. It was cleaned in different kinds of detergents to check what kind of fouling there was on the membrane. Different detergents have different characteristics towards a particular kind of impurity. The type of detergent that can clean it can assess the type of fouling on a membrane. For example the detergent called Tornado should be effective on fouling caused by grease/aliphatic compounds. Hence, if the fouling on the membrane could be removed by the detergent *Tornado*, the fouling most probably contained grease and/or aliphatic compounds (See figure 11 Step 8).

A new LFC1 membrane was installed at the start of VSEP batch testing 1. The concentration factor attained at the end of the experiment was around 2. The concentration factor was too low to bring any kind of fouling on the membrane. This was also observed by no reduction in permeate flow (as it should be in the case of membrane fouling) at the end of VSEP Batch test 1. This membrane was used again for VSEP batch test 2 after a fresh water test on VSEP to clean the membrane from any impurities clinging to its surface.

The permeate flow dropped abruptly in the middle of VSEP batch 2 and did not recover again. This was due to fouling on the LFC1 membrane. The membrane was taken out and underwent cleaning with different detergents (See appendix L: membrane cleaning analysis). It was found to contain little fouling on its surface. It was put in a solution of high pH NC<sub>2</sub> detergent for two days after which it was found to be perfectly clean (See appendix L: membrane cleaning analysis) as observed by visual analysis.

Another LFC1 membrane was installed in the middle of VSEP batch test 2. The test ran smoothly until the Permeate Flow started to drop. This was due to fouling on the membrane. The permeate flow dropped completely even when the feed pressure was increased which proved that the membrane was completely fouled. The membrane was taken out of the VSEP unit. It was highly fouled (See appendix L: membrane cleaning analysis). It was cut into small pieces. Each piece was cleaned in different kinds of detergent. One part was soaked in high pH NC<sub>2</sub> detergent for 2 days. It was found to be perfectly clean after only a little fresh water sprinkling over it. Three other pieces were put in high pH Ariel detergent, detergent Tornado and pure Ariel detergent for one day. The membrane part put in detergent Tornado was not cleaned after one day. The membrane part soaked in high pH Ariel detergent was also unaffected. However, the membrane part kept in pure Ariel detergent was completely clean after one day (See appendix L: membrane cleaning analysis). The detergent Tornado should be highly effective in cleaning impurities like grease/aliphatic compounds. Since the membrane was not cleaned after the treatment with Tornado detergent, it is most probable that there was some other kind of fouling on the second LFC1 membrane. The membrane section was completely cleaned in pure Ariel solution, which makes it highly probable, that the fouling was mainly organic.

A third LFC1 membrane was installed before VSEP batch test 3 with antiscalant dose. It was taken out at the end of the test when permeate flow dropped completely. It was moderately fouled. It was cleaned in high pH  $\text{NC}_2$  for 4 days. It was very much cleaned by this treatment, which indicates that the fouling was mainly organic. The conclusion is based on only visual observation in consent with membrane consultant.

The third LFC1 was installed back as it was into the VSEP unit for batch test 4, no dosing test. No further cleaning analysis were done on the membrane at the end of fourth test.

Table 2 summarizes the effectiveness of different cleaning detergents in cleaning the fouling of LFC1 membranes.

**Table 2. The table shows the cleaning performance of different detergents on LFC1 membrane surface.**

Detergent	Cleaning performance on membrane
Tornado	Poor
High pH $\text{NC}_2$	Good
Pure Ariel	Good
High pH Ariel	Poor

### 6.3 Alkalinity measurements

All alkalinity measurements and titration curves on feed; concentrate and permeate can be found in appendix D-I.

The alkalinity is here simply defined as the amount of acid, which is needed to lower the original pH of the sample to a pH of 4.5 (at which all bicarbonate is transformed to carbon dioxide).

The alkalinity difference between un-filtrated and filtrated samples for the different experiments is plotted in the Figure 16. A big alkalinity difference means that some compounds, which contributes to alkalinity, have been taken away by filtration. These compounds are probably precipitated salts like calcium carbonate and calcium phosphate.

As seen from the figure 16, the alkalinity difference is small up till 80 % volume reduction for all experiments. At CM though, the alkalinity difference is big, especially for B3 and B4 (antiscalant/no dosing). This is probably due to crystallization of supersaturated metal salts ( $\text{CaCO}_3$ ,  $\text{Ca}_3(\text{PO}_4)_2$  etc) in the feed tank during concentration. Further indications for this is that also a big part of the calcium and phosphate content can be taken away by filtration; see section 6.4.

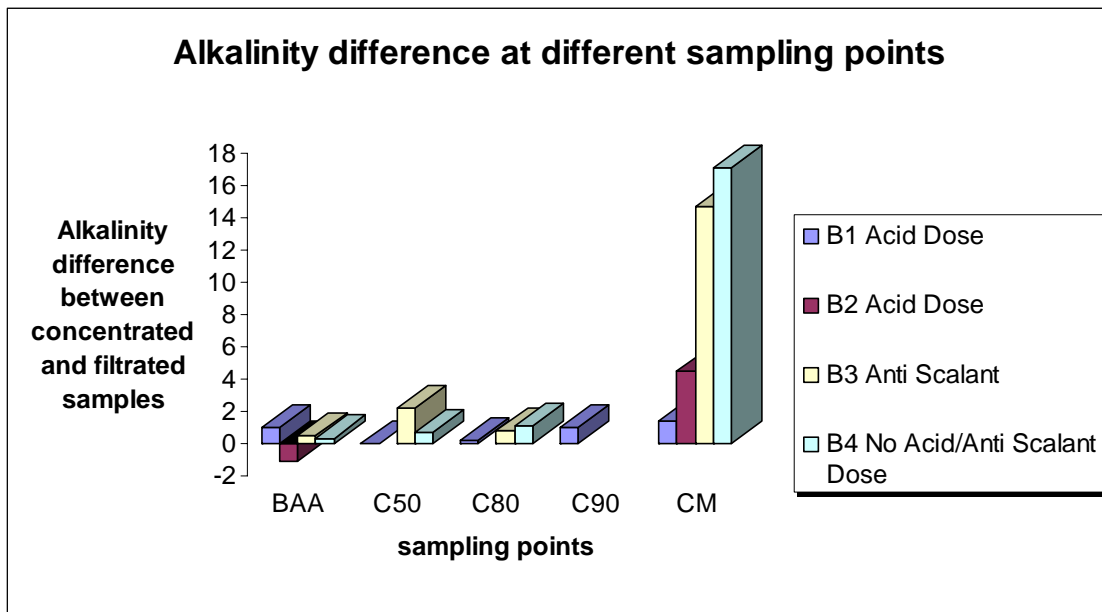


Figure 16. The graph shows values of difference of alkalinity (at pH 4.5) at each sampling points of three tests

#### 6.4 Chemical Analysis

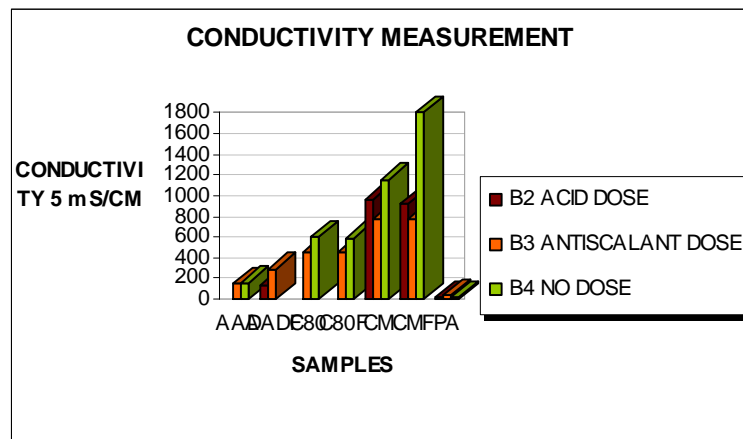
The 500 ml filtrated and concentrated black water samples taken at different concentration levels in the feed tank for the three VSEP batch tests, were analyzed for various chemical substances at the accredited AI-control laboratory. Suspended solids (SS), total dry substances (TS), total organic carbon (TOC), sulphate ( $\text{SO}_4^{2-}$ ), magnesium ( $\text{Mg}^{2+}$ ), total nitrogen (N-Tot), chloride (Cl<sup>-</sup>), calcium ( $\text{Ca}^{2+}$ ), total phosphate (P-tot), phosphate ( $\text{PO}_4^{3-}$ ), fatty acids, chemical oxygen demand (COD), ammonium ( $\text{NH}_4^+$ ), potassium ( $\text{K}^+$ ) and sodium ( $\text{Na}^+$ ) (See Table 6, 7 for AI-control results, SEE APP C).

Chemical analyses were done for both filtrated and un-filtrated samples at different concentration levels. Samples from the permeate tank, known as permeate average (PA) sample were also analyzed. The black water feed got more and more concentrated in the course of experiment as more and more water purges out as permeate across the membrane. The black water contains a series of metal salts and organic impurities. It was expected that at high concentrations of black water the metal salts got oversaturated and could crystallize. This could cause fouling on the membrane surface and reduce its performance. Black water also has organic impurities that can cling to the membrane surface at higher concentrations.

The concentrations of the chemical substances in the permeate gives information of the retention of substances by the membrane. The concentrations at the start of the experiment (P-Start) make a good basis of comparison with the concentration in other samples taken later on.

#### 6.4.1 Concentration factor difference between three tests

The concentration factor attained for VSEP batch test 4 with no dosing is 11.36, for VSEP batch test 3 with antiscalant dosing is 6.8 and VSEP batch 2 with acid dose is 9.0. This can be seen in figure 17, where the conductivity at point CM is highest (1200 mS/cm) for B4, then (1000 ms/cm) for B2 and minimum (800 ms/cm) for B3 antiscalant dose test. However, it has to be pointed out that it should have been 55 liters for all three tests, but error in preparing the initial batch made a difference in final concentration level.



**Figure 17.** The graph above shows conductivity values of black water samples taken at different sampling points for three batch tests.

The conductivity values were lowest at the start AAD and increased with further volume reduction. It was highest at CM. This proves that that concentration of black water is increasing with volume reduction. (See figure 17)

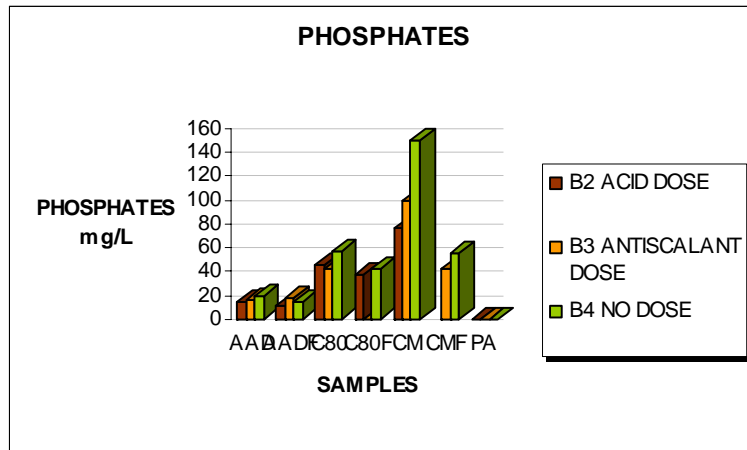
The conductivity value was very low for permeate average samples (PA) which shows that they are much less concentrated or clear.

#### 6.4.2 Difference in $PO_4^{3-}$ , $P_{-tot}$ , $Mg^{2+}$ and $Ca^{2+}$ concentrations

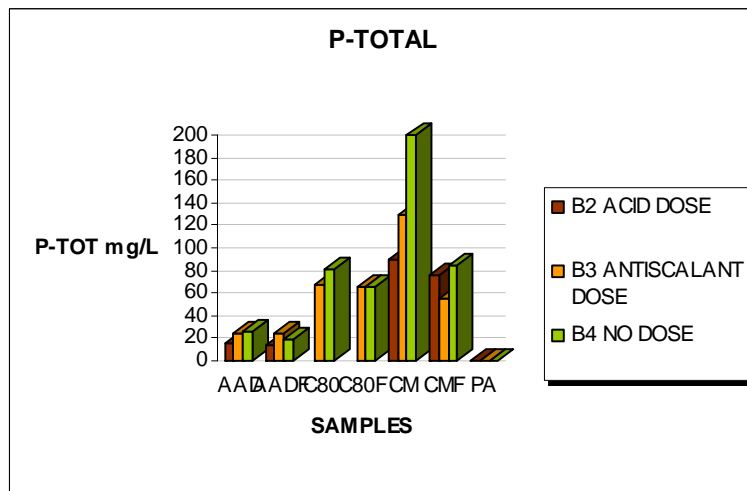
High difference is observed in phosphate, P-tot, magnesium and calcium values of samples taken at maximum concentration (See figure 18, 19, 20 and 21) in all three tests. The high difference indicates precipitation of metal salts in black water or on the membrane surface. Precipitation of inorganic salts is estimated in all the three tests after 80% concentration. Loss of  $Ca^{2+}$ ,  $Mg^{2+}$  and  $PO_4^{3-}$  is observed in all three tests which shows that acid dose/antiscalants are ineffective in preventing precipitation of insoluble metal salts after 80% volume reduction. The main insoluble salts that can produce are probably  $Ca_3(PO_4)_2(s)$ ,  $CaCO_3(s)$ ,  $MgNH_4PO_4(s)$ .

Cleaning analysis of membranes taken out after batch 2, 3 and 4 indicated fouling to be organic and not by inorganic salts. The precipitation may have taken place on the boundary layer above the vibrating membrane surface. The feed water had lower numbers of particles but a cake layer of organic matter and suspended solids. These can become

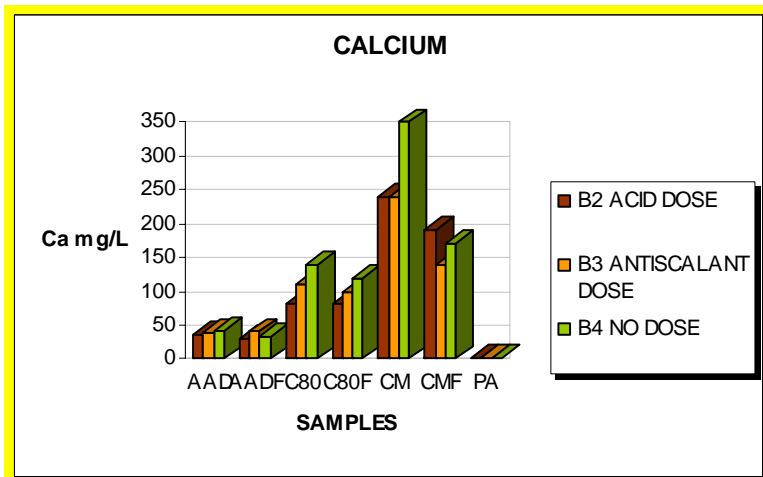
the part of boundary layer on which precipitation may take place. It is also possible that inorganic crystals are absorbed into the organic molecules that stick on the membrane surface. Since the main particles are organic they are easily removed in the cleaning. The inorganic crystals may remain inside the organics. This condition is beneficial for the process. High level of organics in black water can prevent inorganic fouling on LFC1 membrane.



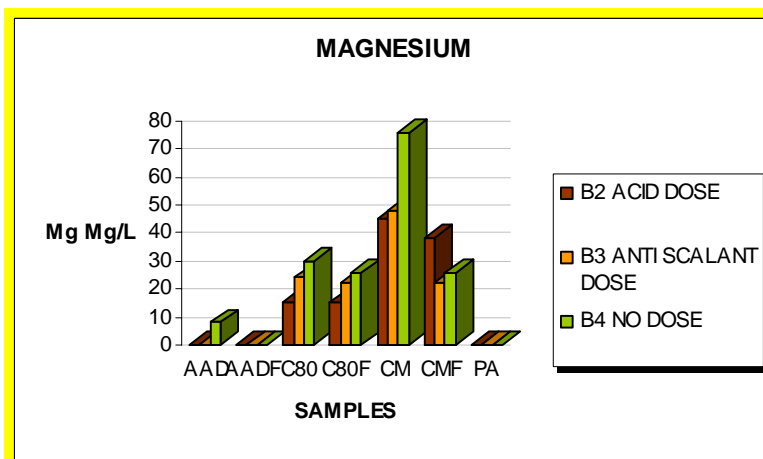
**Figure 18. Difference in phosphate values of filtrated and un-filtrated samples.**



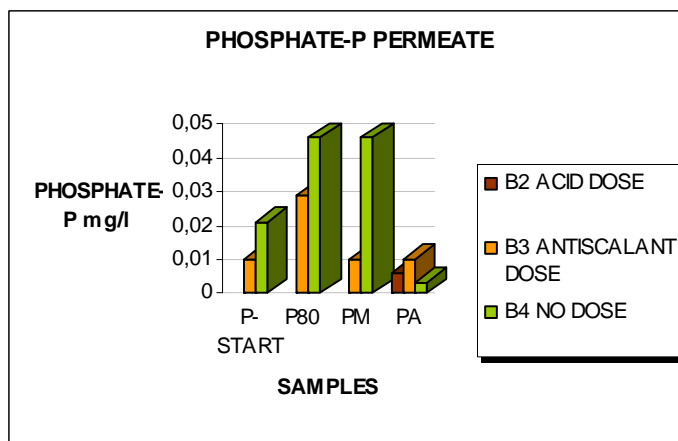
**Figure 19. Difference in P-tot values of filtrated and un-filtrated samples.**



**Figure 20. Difference in calcium values of filtrated and concentrates samples**

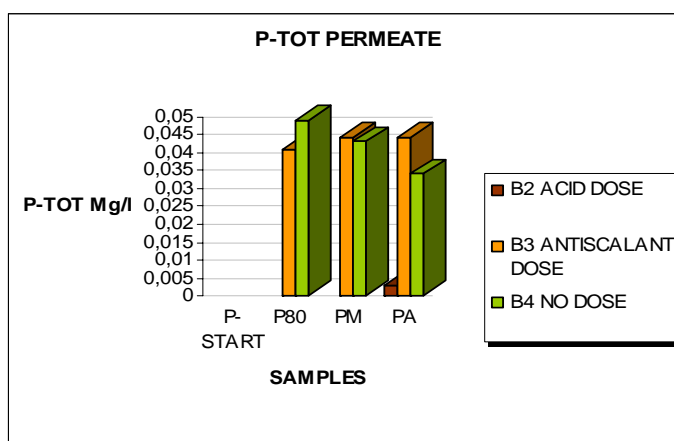


**Figure 21. Difference in magnesium values of filtrated and concentrates samples**

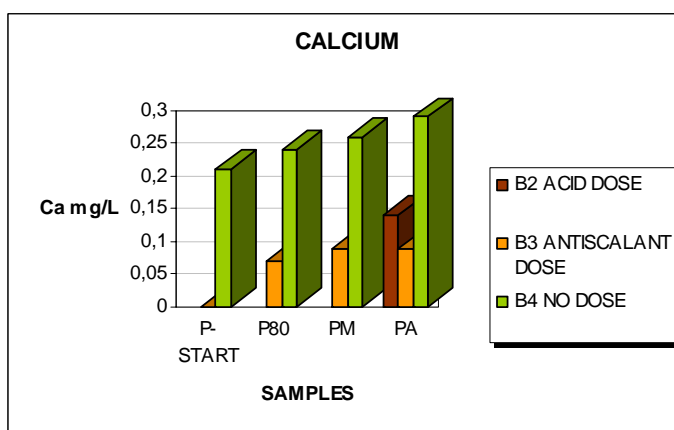


**Figure 22. Phosphate values of permeate samples**





**Figure 23. P-tot values of permeate samples**

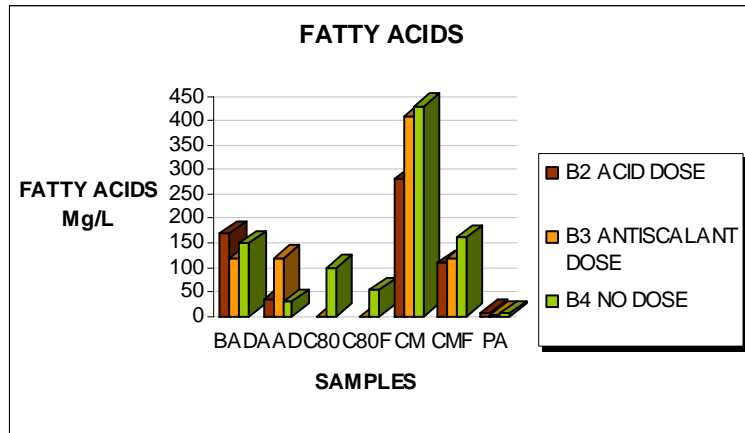


**Figure 24. Calcium values of permeate samples**

It can be seen in figure 18, 19, 20 and 21 that the difference in un-filtrated and filtrated value of P-tot,  $\text{Ca}^{2+}$ ,  $\text{PO}_4^{3-}$ ,  $\text{Mg}^{2+}$  at maximum volume reduction point, is highest for batch 4 no dose, followed by batch 3 antiscalant dose, and least for batch 2 acid dose. It can therefore be predicted the maximum precipitation of inorganic metal salts will occur in the no dose test, followed by antiscalant dose test and acid dose test.

The concentrations of phosphate and total phosphorus in the permeate were low which shows that the membrane is efficient in retaining the molecules in the concentrate (figure 22, 23, 24). The concentration of magnesium was extremely low in the permeate.

#### 6.4.3 Difference in fatty acids values



**Figure 25. High difference in fatty acids values of filtrated and concentrated samples**

High difference was observed in fatty acids values in all tests (See figure 25). The high difference gives indication of organic fouling on the membrane surface. Cleaning analysis of membranes taken out in the middle of batch 2, end of batch 2 and end of batch 3 showed some kind of organic fouling. Organic fouling on membrane is possibly by fatty acids present in feed water. But it should be kept in mind that the LFC1 membrane taken out after end of batch 2 did not become clean when washed in detergent tornado. Tornado detergent should be highly effective in cleaning impurities like grease and alifates.

It can also be seen from figure 25, that the largest difference in un-filtrated and filtrated value of fatty acid at CM sampling point was found for batch 4 (no-dose), followed by antiscalant dose and acid dose. It can be assessed by this that most organic fouling can be predicted in batch 4.

6.4.4 Difference in TOC and COD values

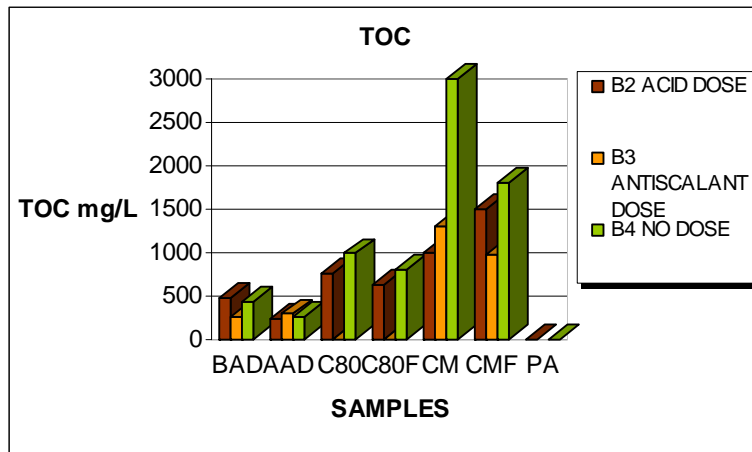


Figure 26. High difference in TOC values of filtrated and un-filtrated samples.

COD and TOC are measures to calculate the concentration of organic matter present in the black water. All COD values are higher than the TOC values (figure 26 and 27).

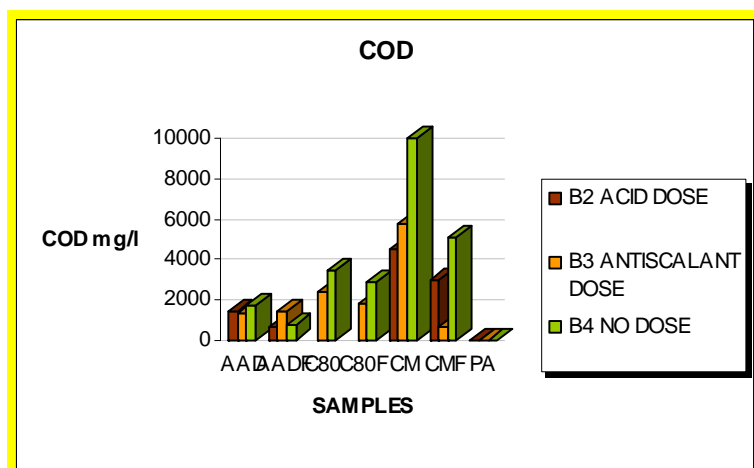
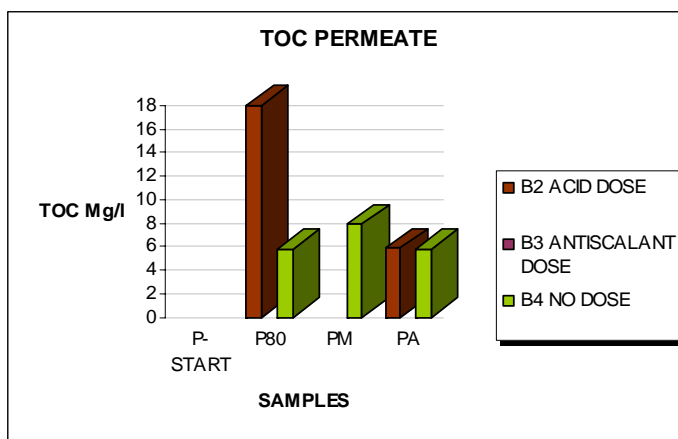


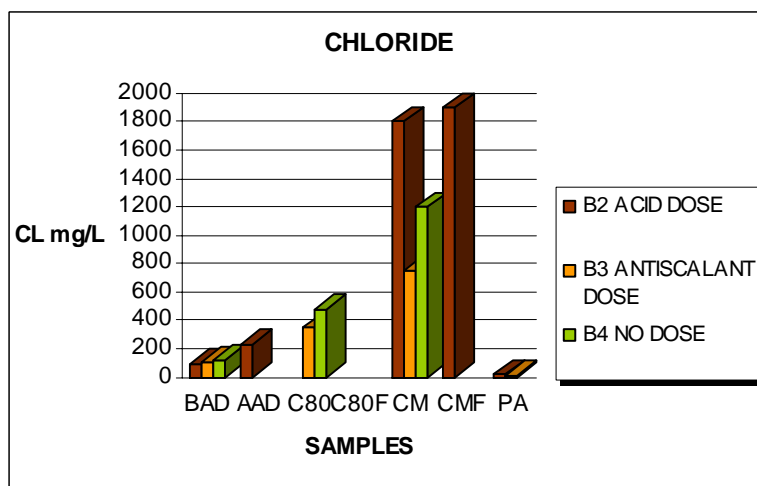
Figure 27. High difference in COD values of filtrated and un-filtrated samples.

The concentrations of TOC in the permeate were low but highest when acid was dosed and at the 80% concentration point (figure 28).



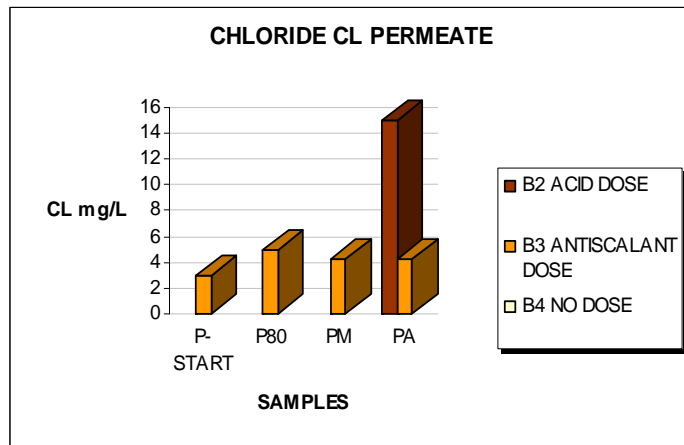
**Figure 28. TOC values of permeate samples**

*6.4.5 Difference in Cl<sup>-</sup> concentration*



**Figure 29. High chloride values in batch 2 with acid dose.**

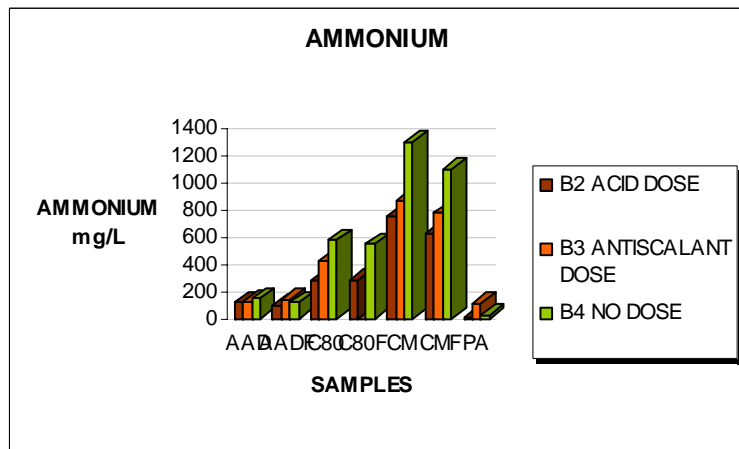
High concentrations of chloride were observed for the batch 2 (acid dose) samples at the maximum concentration point. This is due to HCl dosage. (See figure 29). Some chloride also passed the membrane to the permeate (figure 30).



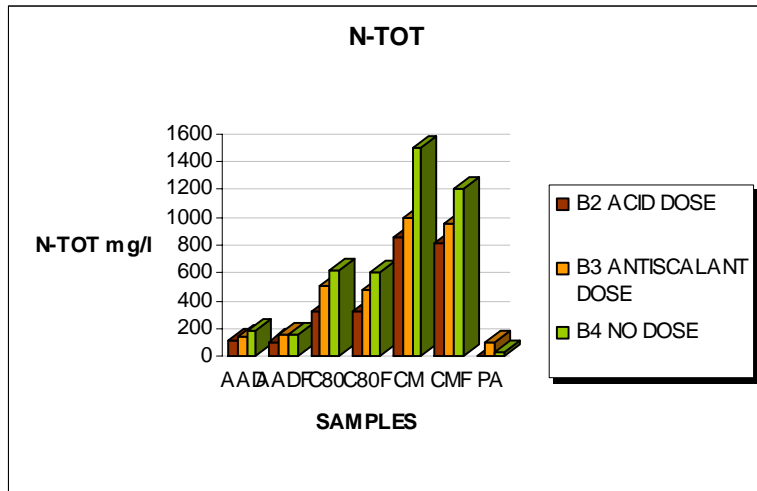
**Figure 30. Chloride values of permeate samples**

**6.4.6 Difference in concentration of  $NH_4^+$ , N-tot,  $K^+$  and  $Na^+$**

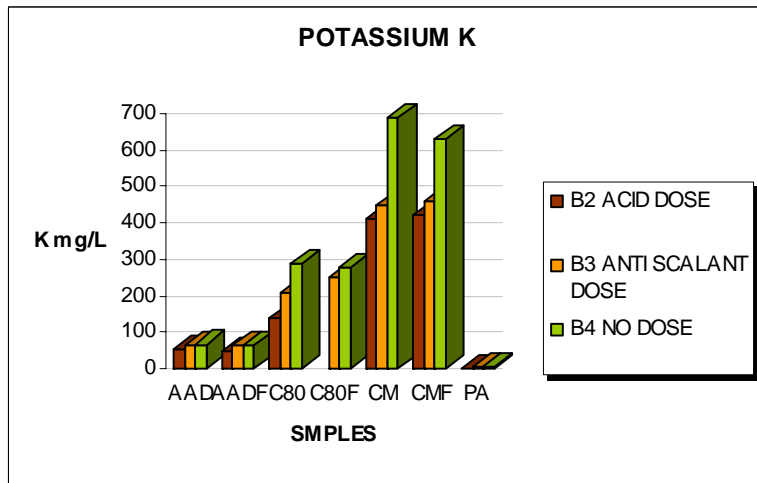
The values of  $NH_4^+$ , N-tot,  $K^+$  and  $Cl^-$  in the filtrated and un-filtrated samples are showed in figure 31, 32, 33 and 34. Monovalent ions like  $NH_4^+$ ,  $K^+$  and  $Cl^-$  have a low charge potential and are small in size. They pass across (LFC1) semi-permeable membrane easily. High concentrations of  $NH_4^+$  and  $K^+$  were observed in the permeate average (See figure 35-39).



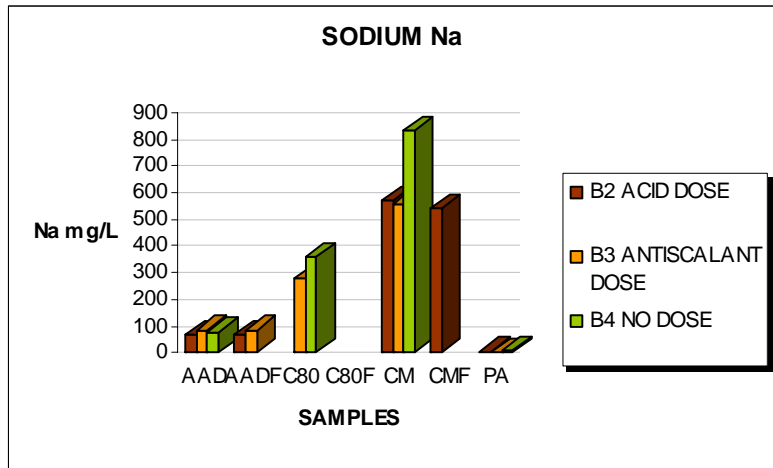
**Figure 31. Difference in ammonium values of filtrated and un-filtrated samples.**



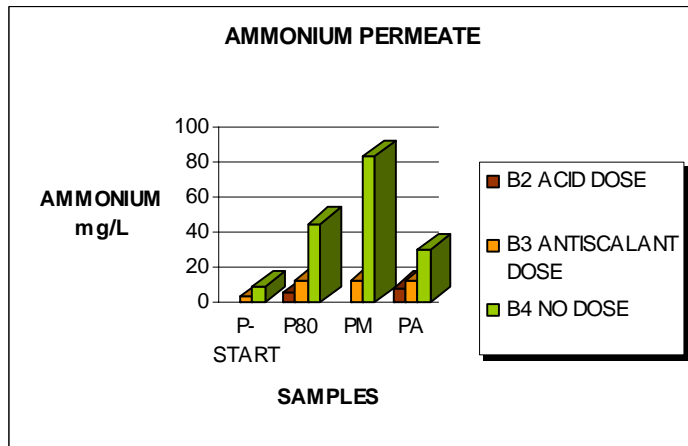
**Figure 32. Difference in N-tot values of filtrated and un-filtrated samples.**



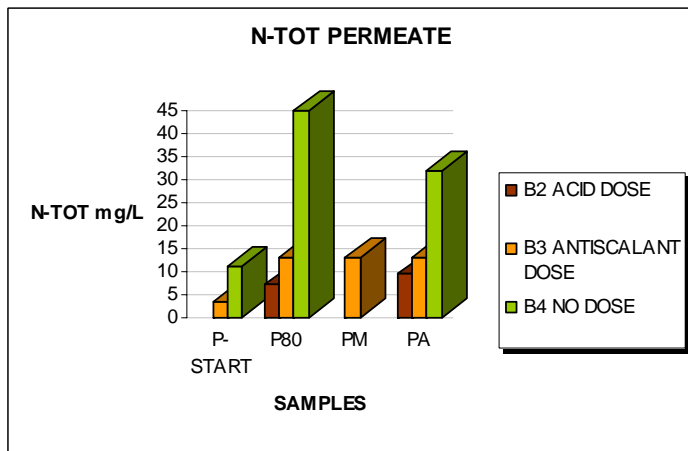
**Figure 33. Difference in potassium values of filtrated and un-filtrated samples.**



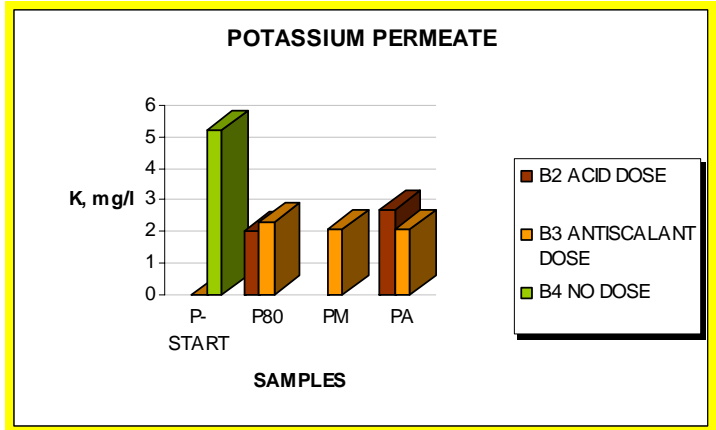
**Figure 34. Difference in sodium values of filtrated and un-filtrated samples.**



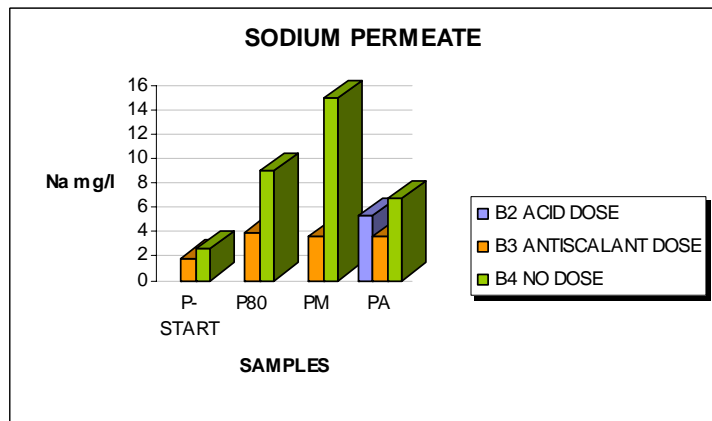
**Figure 35. Large Ammonium concentration in Permeate average samples**



**Figure 36. N-tot values of permeate samples**



**Figure 37. Large  $K^+$  concentration in permeate average samples**

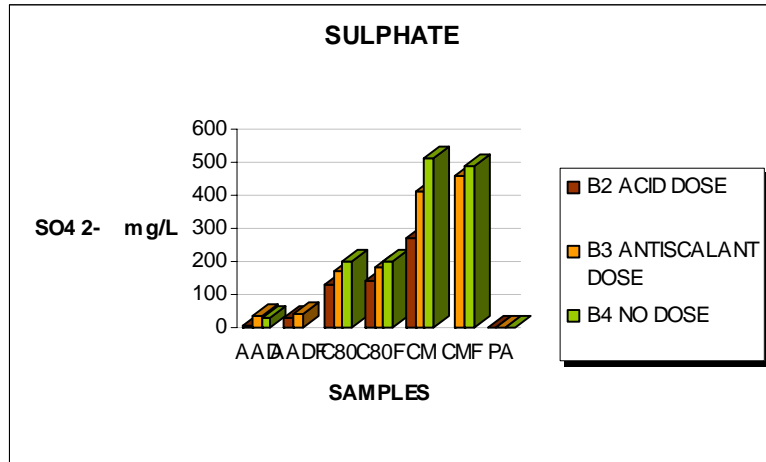


**Figure 38. The graph shows concentration of sodium in permeates at different sampling points and permeate average sample for three batch tests**



#### 6.4.7 Difference in $SO_4^{2-}$ concentration

The difference in sulphate concentration between un-filtrated and filtrated samples is shown in figure 39.



**Figure 39. Difference in sulphate values of filtrated and un-filtrated samples.**

Negligible concentrations of sulphate ions were found in permeate samples in all tests. Divalent ions have higher charge potential (+2) than monovalent ions. They are bigger in size due to this. Big size and high charge potential prevent them from leaking across the membrane into the permeate tank unlike monovalent ions.

## 6.5 PLC curves analysis

### 6.5.1 Permeate flow drops to the maximum in batch 2 Acid dose

The permeate flow dropped most (6 ml/min) at the end of Batch 2 as compared to the Batch 3 and Batch 4 tests, (See figure 13, 14 and 15). This proves that LFC1 membrane taken out at the end of batch 2 fouled to the most as compared to those taken out at the end of batch 3 and 4. This was also found on visual analysis of membrane pieces when taken out at the end of each experiment.

### 6.5.2 Permeate flow drop at almost same point in all tests

There is not much difference in permeate flow profiles for all three tests. This permeate flow reduction is unaffected by the kind of dosing each test had. In each experiment permeate flow dropped after 80% volume reduction (approx) (See figure 13, 14 and 15). This shows that membrane starts to foul after 80% volume reduction in feed. The fouling potential is unaffected by the kind of dosing feed has. Permeate flow drops to 10 ml/min in antiscalant dose test and 10ml/min in no dose (See figure 13, 14 and 15). The role of acid dose or antiscalant is negligible in inhibiting fouling on membrane surface.

The analysis can be better if the feed temperature is kept constant.

## 6.6 Mass Balance

Mass balances were performed on the different chemical compounds. At first a mass balance was performed on sodium on the assumption that the amount of  $\text{Na}^+$  in the feed is the sum of the amount of  $\text{Na}^+$  in the concentrate and in the permeate, hence no sodium disappears from the system by precipitation/sedimentation or evaporation to the air. This assumption is correct for a “tracer component” like sodium.

Then with the aid of this mass balance, you can derive that the following equation is valid:

$$\text{CF} = \text{CM} \times f + (1-f) \times \text{PA}$$

Or;

$$f = (\text{CF} - \text{PA}) / (\text{CM} - \text{PA})$$

Where  $f$  is the fraction of the original volume which is left after concentration (volume of concentrate/volume of feed). Hence,  $1/f$  is the “concentration factor” (volume of feed/volume of concentrate, the maximal factor that a component can be concentrated with if no leakage to permeate occurs).

This method for calculating the factor  $f$  by sodium content is more accurate than simply taking the measured rest volume of the concentrate divided by the feed volume. This is

because the rest volume cannot be measured accurately due to an unknown volume which is left in the piping system and membrane package.

*Note that no attention is paid to the sample volumes which have been taken out during the experiment. This is not completely right and it should be checked that the effect of sampling on mass balances is negligible.*

For batch 4,  $f = 0,088$ , hence the concentration factor is  $1/f = 1/0.088 = 11.36$  for batch 4. By carrying out the same calculations for each batch, the concentration factors could be estimated to be 6.8 for batch 3 and 9.0 for batch 2.

Once you know the factor  $f$ , it is easy to make a mass balance for each component. The results of these mass balance calculations are shown in table 3 to 8 and figure 40. The loss/inaccuracy post is introduced to make the balance correct:

The amounts of a component in the feed are equal to the amount of that component in the concentrate and permeate plus the amount which is lost due to for example evaporation or precipitation/sedimentation. A negative loss is a gain; somehow this component has been produced in the system.

**Table 3. Percentage recovery of chemical substances in concentrate and leakage into permeate and total percentage loss for batch 4 no dose test.**

Component	Batch 4 No Dose		
	In Concentrate %	In Permeate %	Loss/Inaccuracy
Na <sup>+</sup>	92.4	7.7	0.0
NH <sub>4</sub> <sup>+</sup>	76.3	18.2	5.5
N-tot.	69.5	15.3	15.2
K <sup>+</sup>	90.62	7.07	2.31
PO <sub>4</sub> <sup>3-</sup>	66	0.01	33.99
P-Tot	70.4	0.12ta	29.48
COD	51.76	0	48.23
Fatty Acids	25.22	3.52	71.26
Ca <sup>2+</sup>	73.33	0.62	26.05
Cl <sup>-</sup>	88	10.63	1.37
Mg <sup>2+</sup>	83.5	0	16.5
SO <sub>4</sub> <sup>2-</sup>	136	0	-36
TOC	61.39	1.23	37.38
Torr Subs.	86.24	0	13.76
Susp.Solids			

**Table 4. Percentage recovery of chemical substances in concentrate and leakage into permeate and total percentage loss for batch 3 antiscalant dose test.**

Component	Batch 3 Antiscalant dose		
	In Concentrate %	In Permeate %	Loss/Inaccuracy
Na <sup>+</sup>	96,2	3,60)α	
NH <sub>4</sub> <sup>+</sup>	97,7	8,54	-6,24
N-tot.	104,3	7,93	-12,23
K <sup>+</sup>	102,65	2,8	-5,45
PO <sub>4</sub> <sup>3-</sup>	85,9	0,05	14,05
P-Tot	82,6	0,16	17,24
COD	60,48	0	39,52
Fatty Acids	49,88	2,41	47,71
Ca <sup>2+</sup>	89,84	0,19	9,97
Cl <sup>-</sup>	99,54	3,34	-2,88
Mg <sup>2+</sup>	97,2	0	2,8
SO <sub>4</sub> <sup>2-</sup>	166,27	0	-66,27
TOC	73	0	27
Torr Subs.	84,94	0	15
Susp.Solids	71,72	NA	28,28

**Table 5. Percentage recovery of chemical substances in concentrate and leakage into permeate and total percentage loss for batch 2 acid dose test.**

Component	Batch 2 Acid dose		
	In Concentrate %	In Permeate %	Loss/Inaccuracy
Na <sup>+</sup>	93,06	6,910)α	
NH <sub>4</sub> <sup>+</sup>	87,17	7,95	4,18
N-tot.	78,95	6,95	14,1
K <sup>+</sup>	85,86	4,52	9,62
PO <sub>4</sub> <sup>3-</sup>	52,26	0,03	47,71
P-Tot	65,86	0,2	33,94
COD	33,3	0	66,7
Fatty Acids	18,28	4,02	77,7
Ca <sup>2+</sup>	76,11	0,34	23,55
Cl <sup>-</sup>	205,97	13,74	-119,71
Mg <sup>2+</sup>	84,57	0	-15,42
SO <sub>4</sub> <sup>2-</sup>	370	0	-270
TOC	44,92	0	-55,07
Torr Subs.	23,61	1,11	75,28
Susp.Solids			

**Table 6. Percentage loss/gain of chemical substances in three batches**

Component	% Loss/Inaccuracy in batch 4, B4 No dose Membrane 3, LFC1	% Loss/Inaccuracy in batch 3, B3, Antiscalant dose Membrane 2, LFC1	% Loss/Inaccuracy in batch 2 Acid dose Membrane 1, LFC1
<b>Na<sup>+</sup></b>	0)α	0)α	0)α
<b>NH<sub>4</sub><sup>+</sup></b>	5,5	-6,24	4,18
<b>N-Tot</b>	15,18	-12,23	14,1
<b>K<sup>+</sup></b>	2,31	-5,45	9,62
<b>PO<sub>4</sub><sup>3-</sup></b>	33,99	14,05	47,71
<b>P-Tot</b>	29,48	17,24	33,94
<b>COD</b>	48,23	39,52	66,7
<b>Fatty Acids</b>	71,26	47,71	77,7
<b>Ca<sup>2+</sup></b>	26,05	9,97	23,55
<b>Cl<sup>-</sup></b>	1,37	-2,88	-119,71
<b>Mg<sup>2+</sup></b>	16,5	2,8	-15,42
<b>SO<sub>4</sub><sup>2-</sup></b>	-36	-66,27	-270
<b>TOC</b>	37,38	27	-55,07
<b>Torr Subs.</b>	13,76	15	75,28
<b>Susp.Solids</b>		28,28	

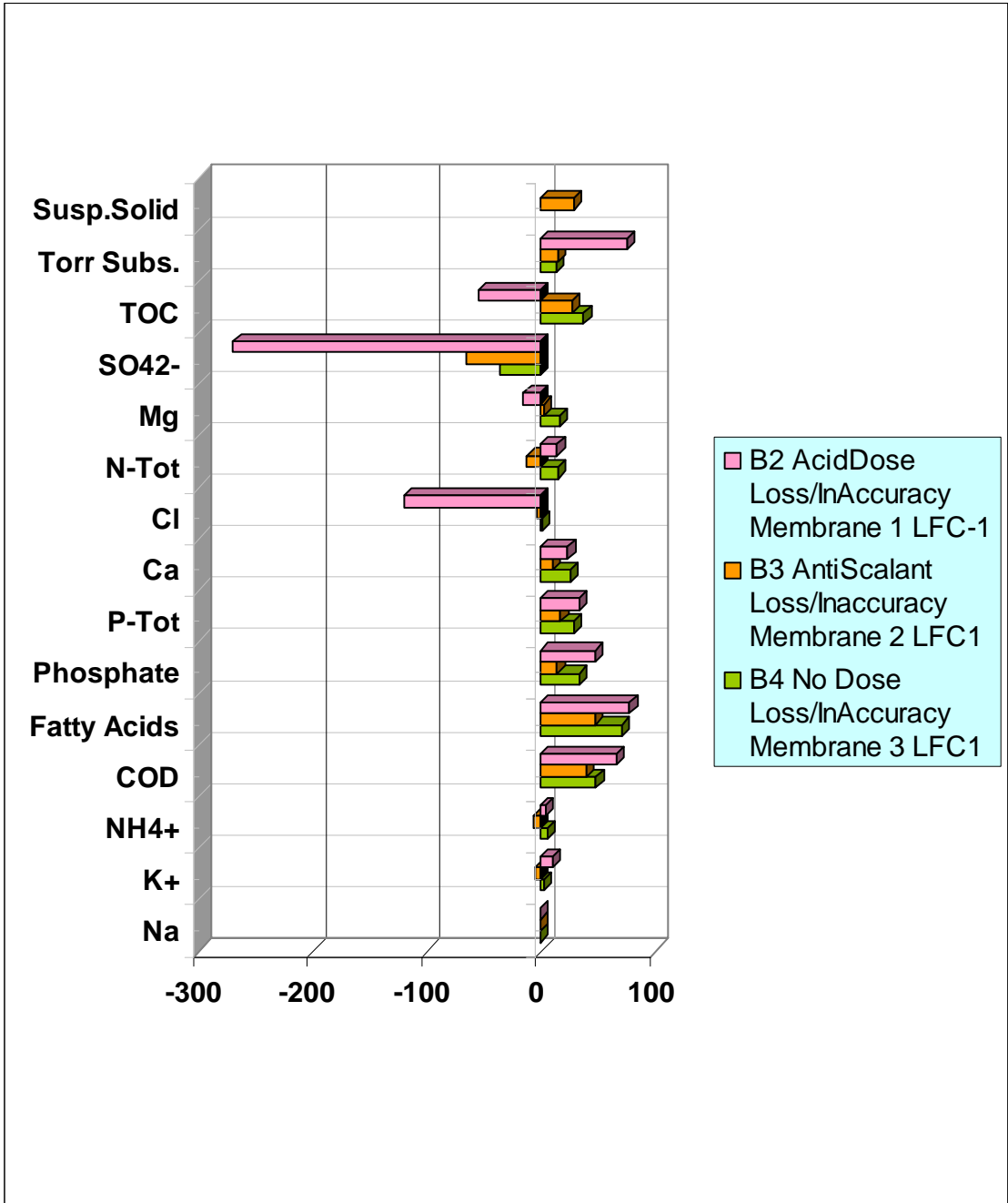


Figure 40. The graph shows the percentage disappearance or production in concentration values of chemical substances at the end of each batch test.

**Table 7. Percentage values of chemical substance that get recovered in the concentrate**

	<b>Batch 4 No Dose Membrane 3 LFC1 After wash</b>	<b>Batch 3 Antiscalant dose Membrane 3 LFC1</b>	<b>Batch 2 Acid dose Membrane 2 LFC1</b>
<b>Component</b>	<b>% in Concentrate</b>	<b>% in Concentrate</b>	<b>% in Concentrate</b>
<b>Na</b>	92,4	96,2	93,06
<b>NH<sup>4+</sup></b>	76,26	97,7	87,17
<b>N-tot.</b>	69,47	104,3	78,95
<b>K<sup>+</sup></b>	90,62	102,65	85,86
<b>Phosphate</b>	66	85,9	52,26
<b>P-Tot</b>	70,4	82,6	65,86
<b>COD</b>	51,76	60,48	33,3
<b>Fatty Acids</b>	25,22	49,88	18,28
<b>Ca<sup>2+</sup></b>	73,33	89,84	76,11
<b>Cl<sup>-</sup></b>	83,5	99,54	205,97
<b>Mg<sup>2+</sup></b>	88	97,2	84,57
<b>SO<sub>4</sub><sup>2-</sup></b>	136	166,27	370
<b>TOC</b>	61,39	73	44,92
<b>Torr Subs.</b>	86,24	84,94	23,61
<b>Susp.Solids</b>		71,72	

**Table 8. Percentage values of chemical substance that leak into permeate**

<b>Component</b>	<b>In Permeate % batch 4</b>	<b>In Permeate % batch 3</b>	<b>In Permeate % batch 2</b>
<b>Na</b>	7.7	3,6	6,91
<b>NH<sup>4+</sup></b>	18.2	8,54	7,95
<b>N-tot.</b>	15.3	7,93	6,95
<b>K<sup>+</sup></b>	7.07	2,8	4,52
<b>Phosphate</b>	0.01	0,05	0,03
<b>P-Tot</b>	0.12	0,16	0,2
<b>COD</b>	0	0	0
<b>Fatty Acids</b>	3.52	2,41	4,02
<b>Ca<sup>2+</sup></b>	0.62	0,19	0,34
<b>Cl<sup>-</sup></b>	10.63	3,34	13,74
<b>Mg<sup>2+</sup></b>	0	0	0
<b>SO<sub>4</sub><sup>2-</sup></b>	0	0	0
<b>TOC</b>	1.23	0	0
<b>Torr Subs.</b>	0	0	1,11
<b>Susp.Solids</b>		NA	

The analysis of the various results from the mass balance is described in section 6.5.

## **6.5 Percentage recovery, leakage and loss of chemical substances**

### *6.5.1 Recovery of $NH_4^+$ & N-tot*

The amount of chemical substances recovered in concentrate at the end of each test is compared in table 7. A very high recovery of  $NH_4^+$  and N-tot is observed in the acid dose and antiscalant experiments (% recovery > 90%) as compared to no dose batch (% recovery ~ 76%). This proves that due to some reason  $NH_4^+$  and N-tot are more retained in acid or antiscalant dosing as compared to no dose experiment.

### *6.5.2 Loss of COD*

COD loss is observed in all tests (See figure 59).

The major loss in COD is estimated due to sedimentation into tank.

The loss can also be due to degradation of organic matter into  $CH_4$  and  $CO_2$  by heterotrophic bacteria present in black water. The DO dissolve oxygen level in black water feed is expected to be as low as 0-0.5 mg/l. It is possible that anaerobic conditions prevail in black water that stimulates the heterotrophic bacteria present in it to convert the organic matter into  $CO_2$  and  $CH_4$ . The feed temperature for all three tests is as high as 33 °C. The activity of heterotrophic bacteria doubles by every 10 °C increase in temperature and is highest at 35 °C. High level of bacterial activity is expected at such high temperatures.

It is also possible that VFAs and acetates are produced in the sewer system before water enters the VSEP system. Once water is in feed these can be consumed by anaerobic bacteria (if no nitrate is present). If we assume that VFA is mainly acetate and propionate, their fraction of dissolved COD is about 25 and 36% for B2 and less in B3 and B4. The amount formed is dependent on the retention time in pipes etc. Once in the feed tank the VFA and easily degradable COD are reduced easily by the bacteria present. Acidic conditions, (pH 6) or antiscalant dose do not seem to affect degradation.

### *6.5.3 Gain in $SO_4^{2-}$*

A percentage gain is observed for sulphate at the end of all three VSEP test (See figure 59). It is possible that anaerobic condition can prevail in black water before entering the feed vessel as it contains a lot of organics.  $H_2S$  is produced. On entering the feed vessel if oxygen is present,  $H_2S$  can be oxidized to  $SO_4^{2-}$  and a gain is observed.

### *6.5.4 Gain in Cl*

A percentage gain is observed for antiscalant or acid dose test (See figure 59). The gain in batch test 2 can be due to HCl acid dose. Cl<sup>-</sup> at high concentrations might have disturbed the analysis of other compounds. This can be checked in future tests.



#### *6.5.5 Determination of permeate to concentrate ratio*

To find a comparison between relative leakages of chemical substances across the membrane surface for three batch test, permeate to concentrate ratio should be found out at the sampling points like 50%, 80% and maximum volume reduction. The ratio will be P50/C50, P80/C80 and PM/CM.



## 7. CONCLUSION

While concentrating the black water to a concentration of approximately 10, with acid/antiscalant/no dosing, some kind of fouling was observed. After the membrane cleaning analysis it was found that fouling was mainly organic. The major precipitation took place in the concentrate tank at concentration factors of 5 to 10. The main salts that crystallized were probably  $\text{Ca}_3(\text{PO}_4)_2$ ,  $\text{CaCO}_3$ , and  $\text{NH}_4\text{MgPO}_4$ .

There was not much difference observed between the permeate flow profile for all three tests of acid dose/antiscalant/no dose. The permeate flow dropped after 80% volume reduction in all three cases. (See figure 13, 14 and 15). Fouling was observed on each membrane taken out in the middle of batch 2 (acid dose), end of batch 2 (acid dose) and end of batch 3 (antiscalant). The membrane at the end of batch 4 was not analysed.

After the cleaning analysis of the membrane some kind of organic fouling was observed. The fouling probably does not mainly consist of grease or aliphatic compounds as the membrane was not affected by tornado detergent wash. On cleaning of membrane with *NC2* and *Ariel*, it was found to become perfectly clean and the permeate flow could be totally regained (checked after batch 3). This indicates that the fouling was mainly organic.

Loss of  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$  and  $\text{PO}_4^{3-}$  was observed in all three tests and this shows that acid dose/antiscalants are ineffective in preventing precipitation of insoluble metal salts after 80% volume reduction. The difference between un-filtrated and filtrated values of  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{PO}_4^{3-}$ , P-tot,  $\text{NH}_4^+$ , N-tot and fatty acids, taken at different concentration degrees, started to increase after 80% volume reduction point and was largest at the maximal volume reduction point (CM). This shows that precipitation of insoluble metal salts or organic fouling took place after 80% volume reduction in black water.

The difference between un-filtrated and filtrated values of  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{PO}_4^{3-}$ , P-tot,  $\text{NH}_4^+$ , N-tot and fatty acids was largest for no-dose, then for antiscalant dose and then for acid dose. From this it can be predicted that maximum fouling on membrane or precipitation of insoluble salts will occur when nothing is dosed followed by antiscalant dose and least by acid dose. However definite conclusions cannot be drawn until further analysis has been done.

High levels of precipitations should increase the probability for inorganic fouling on membrane surfaces but this was not found after the membrane cleaning analysis. One reason can be that inorganic fouling occurred on a pre-existing organic fouling layer on the membrane surface. The inorganic precipitations could have been trapped in an organic layer and thus become ineffective in causing any fouling. On cleaning they were washed along with organic fouling.



## **8. RECOMMENDATIONS**

The amplitude of the VSEP membrane can be raised to make the surface of the membrane smoother and lower on free energy. This would reduce the formation of precipitates on the membrane surface. Higher shear energy on the membrane surface would lift up any organic particle trying to settle on the membrane surface and eventually prevent fouling of membrane.

It could be a good idea to perform VSEP experiments in two steps. First concentrating the feed approximately 5 times, so a clear permeate is collected which can be safely disposed off. Then as crystallization takes place in the concentrate, the unit can be brought to a standstill for a day or two till all the crystallization goes away. Then the experiment can be continued until a desired concentration level has been obtained. The need for antiscalant/acid dosing is discarded by this methodology.

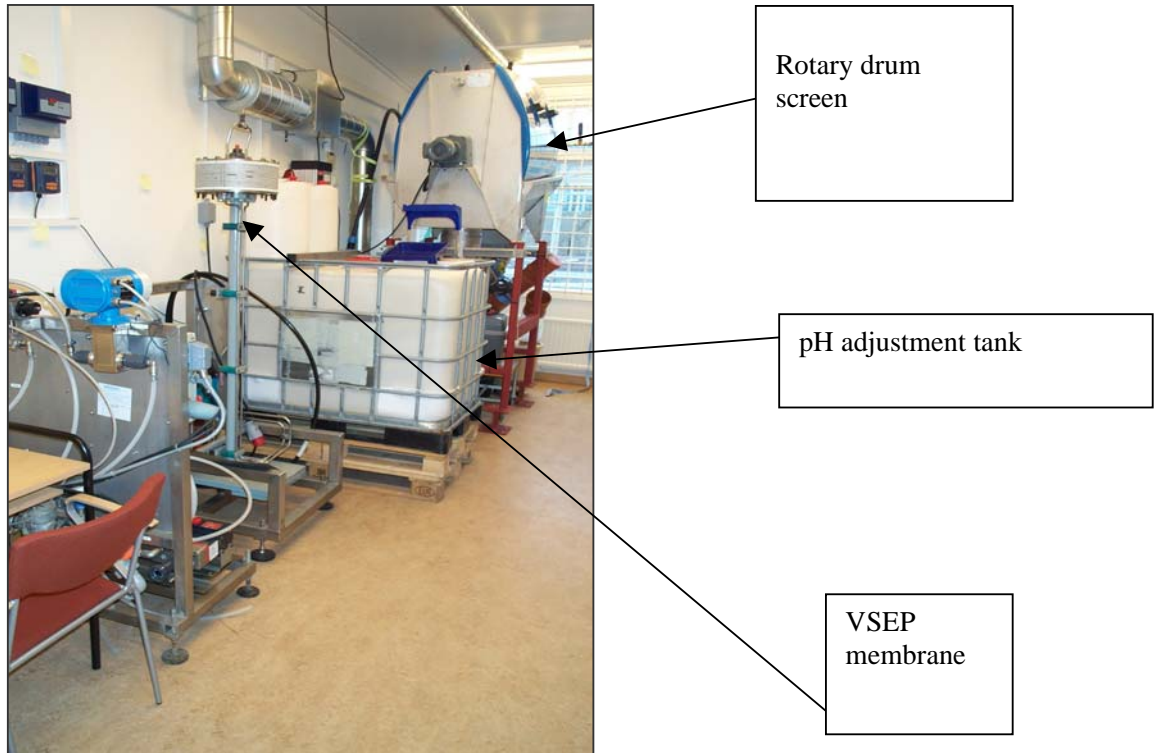
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## Appendix A: Pilot plant set-up



**Figure 9. View of pilot plant, for nutrient-recycling from black water sewage, at Skogaberg**



## Appendix B Pilot plant units



**Figure 10. Rotary drum screen operated to separate solids from the black water.**



**Figure 11. Inlet for black water sewage into the drum screen**



**Figure 12. Solid sludge collected from the right Side bottom of the drum screen**



**Figure15. The VSEP membrane filtration unit**



**Figure 13. Screened black water, from Roto-Sieve collection point from the left bottom side of the drum screen.**



**Figure 14. A PLC (Program Logic Controller) is controlling the whole process.**

## Appendix C: AI-control laboratory results

- B1 Experiment 1 black water filtration on VSEP membrane with *acid dose*  
 B2 Experiment 2 black water filtration on VSEP membrane with *acid dose*  
 B3 Experiment 3 black water filtration on VSEP membrane with *anti-scalant dose*  
 B4 Experiment 4 black water filtration on VSEP membrane without *acid/antiscalant dose*  
 AAD Concentrated sample after acid/antiscalant/no-dosing at the start of experiment.  
 AADF Filtrated sample after acid/antiscalant/no-dosing at the start of experiment.  
 C50 Concentrate sample at 50% volume reduction of feed tank  
 C50f Filtrated concentrate sample at 50% volume reduction of feed tank  
 C80 Concentrate sample at 80% volume reduction of feed tank  
 C80f Filtrated concentrate sample at 80% volume reduction of feed tank  
 C90 Concentrate sample at 90% volume reduction of feed tank  
 C90f Filtrated concentrate sample at 90% volume reduction of feed tank  
 CM Concentrate sample at maximal volume reduction of feed tank  
 CMF Filtrated concentrate sample at maximal volume reduction of feed tank

Parameters	AAD	AADF	C80	C80f	CM	CMF	PA
Conductivity, S, B2		140			970	920	11.9
Conductivity, S, B3	150	285	460	460	780	780	14.6
Conductivity, S, B4	160		600	590	1150	1800	20.9
Parameters	AAD	AADF	C80	C80f	CM	CMF	PA
Ammonium, B2	96	96	280	280	760	630	8.6
Ammonium, B3	130	140	430	430	870	780	13
Ammonium, B4	150	120	580	550	1300	1100	30
Parameters	AAD	AADF	C80	C80f	CM	CMF	PA
COD(cr), B2	1500	710			4500	3000	<30
COD(cr), B3	1400	1500	2400	1800	5800	700	<30
COD(cr), B4	1700	790	3500	2900	10000	5100	<30
Parameters	AAD	AADF	C80	C80f	CM	CMF	PA
Fatty acids, tot, B2	170	35			280	110	7.7
Fatty acids, tot, B3	120	120	430	430	410	120	3.4
Fatty acids, tot, B4	150	29	100	53	430	160	5.8
Parameters	AAD	AADF	C80	C80f	CM	CMF	PA
Phosphate-P, B2	15	12	46	38	76		0.006
Phosphate-P, B3	17	18	43	43	100	42	0.010
Phosphate-P, B4	20	15	57	43	150	55	0.003
Parameters	AAD	AADF	C80	C80f	CM	CMF	PA
P-tot, B2	15	13			89	76	0.033
P-tot, B3	23	23	68	65	130	55	0.044
P-tot, B4	25	18	81	66	200	85	0.034
Parameters	AAD	AADF	C80	C80f	CM	CMF	PA
Calcium, Ca, mg/l, B2	35	31	82	81	240	190	0.14
Calcium, Ca, mg/l, B3	39	40	110	98	240	140	0.09

Calcium, Ca, mg/l, B4	42	33	140	120	350	170	0.29
Parameters	AAD	AADF	C80	C80F	CM	CMF	PA
Potassium, K, mg/l, B2	53	48	140		410	420	2.7
Potassium, K, mg/l, B3	64	64	210	250	450	460	2.1
Potassium, K, mg/l, B4	67	65	290	280	690	630	5.2
Parameters	AAD	AADF	C80	C80F	CM	CMF	PA
Chloride, Cl, mg/l, B2	97	230			1800	1900	15
Chloride, Cl, mg/l, B3	110		350		750		4.3
Chloride, Cl, mg/l, B4	120		480		1200		
Parameters	AAD	AADF	C80	C80F	CM	CMF	PA
N-tot, mg/l, B2	120	110	330	320	850	810	9.4
N-tot, mg/l, B3	140	160	510	480	1000	950	13
N-tot, mg/l, B4	190	160	620	600	1500	1200	32
Parameters	AAD	AADF	C80	C80F	CM	CMF	PA
Magnesium, Mg, mg/l, B2	5.9	5.1	15	15	45	38	<.1
Magnesium, Mg, mg/l, B3	7.2	7.2	24	22	48	22	<.1
Magnesium, Mg, mg/l, B4	8	7.3	30	26	76	26	<.1
Parameters	AAD	AADF	C80	C80F	CM	CMF	PA
Sodium, Na, mg/l, B2	68	70			570	540	5.3
Sodium, Na, mg/l, B3	85	85	280		560		3.6
Sodium, Na, mg/l, B4	79		360		830		6.7
Parameters	AAD	AADF	C80	C80F	CM	CMF	PA
Sulphate, S, mg/l, B2	8.1	33	130	140	270		<2
Sulphate, S, mg/l, B3	36	39	170	180	410	460	<2
Sulphate, S, mg/l, B4	33		200	200	510	490	<2
Parameters	AAD	AADF	C80	C80F	CM	CMF	PA
Suspended solids, mg/l, B2	420	52	430		1700	230	
Suspended solids, mg/l, B3	570	650	810		2800		
Suspended solids, mg/l, B4	540	94	1200				
Parameters	AAD	AADF	C80	C80F	CM	CMF	PA
TOC, mg/l, B2	470	240	760	630	1000	1500	5.9
TOC, mg/l, B3	260	290	xxx	xxx	1300	980	
TOC, mg/l, B4	430	260	990	810	3000	1800	5.8
Parameters	AAD	AADF	C80	C80F	CM	CMF	PA
Torrsubstans, mg/l, B2	860	590			6800	5500	<5
Torrsubstans, mg/l, B3	1100	1100	2600	2500	6400	4800	xxx
Torrsubstans, mg/l, B4	1000	720	3500	3600	9800	6800	<5

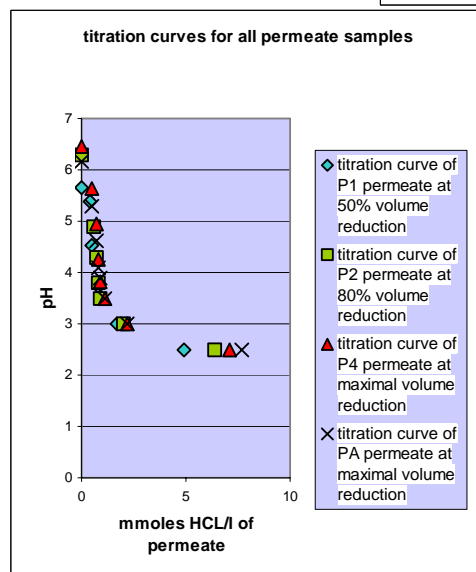
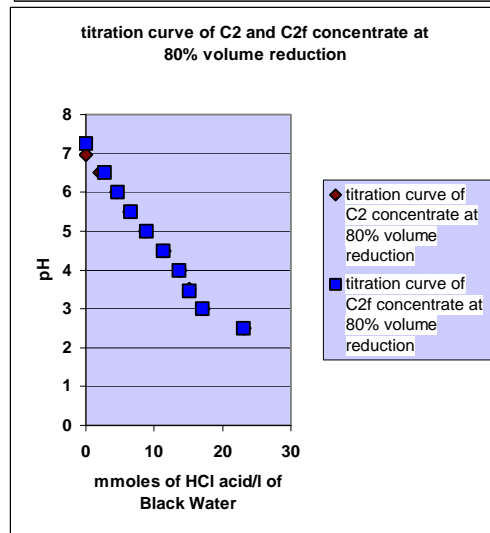
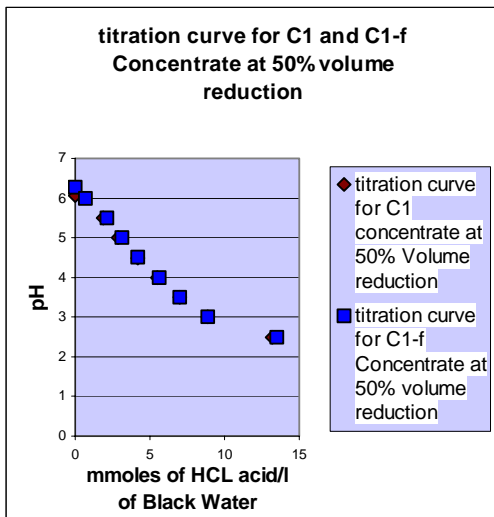
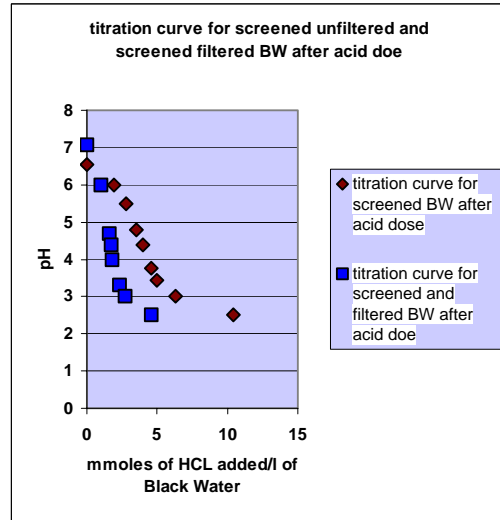
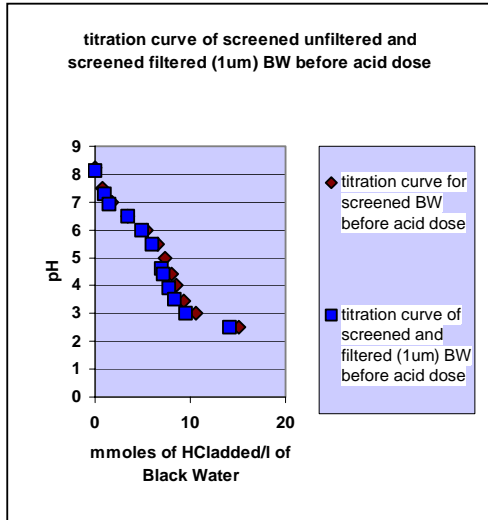
P-Start Permeate sample taken at the start of experiment  
P50 Permeate sample taken at 50% volume reduction of feed tank  
P80 Permeate sample taken at 80% volume reduction of feed tank  
P90 Permeate sample taken at 90% volume reduction of feed tank  
PM Permeate sample taken at maximal volume reduction of feed tank  
PA Permeate sample taken from permeate collection container also called average permeate

Parameters	P-START	P80	PM	PA
Conductivity, S, B2				11.9
Conductivity, S, B3	5.7	14.7	14.6	14.6
Conductivity, S, B4	7.3	24.9	38.8	20.9
Parameters	P-START	P80	PM	PA
Ammonium, B2		6.2		8.6
Ammonium, B3	3.3	13	110	13
Ammonium, B4	9.4	45	83	30
Parameters	P-START	P80	PM	PA
COD(Cr), B2				<30
COD(Cr), B3	<30	<30	<30	<30
COD(Cr), B4	<30	<30	<30	<30
Parameters	P-START	P80	PM	PA
Fatty Acids, tot, B2				7.7
Fatty Acids, tot, B3	4.7	2	3.4	3.5
Fatty Acids, tot, B4	6.2	3.8	6.9	5.8
Parameters	P-START	P80	PM	PA
Phosphate-P, B2				0.006
Phosphate-P, B3	0.01	0.029	0.01	0.01
Phosphate-P, B4	0.021	0.046	0.046	0.003
Parameters	P-START	P80	PM	PA
P-tot, mg/l, B2				0.033
P-tot, mg/l, B3	0.015	0.041	0.044	0.044
P-tot, mg/l, B4	0.025	0.049	0.043	0.034
Parameters	P-START	P80	PM	PA
Calcium, Ca, mg/l, B2				0.14
Calcium, Ca, mg/l, B3	<.05	0.07	0.09	0.09
Calcium, Ca, mg/l, B4	0.21	0.24	0.26	0.29
Parameters	P-START	P80	PM	PA
Potassium, K, mg/l, B2		2		2.7
Potassium, K, mg/l, B3	<2	2.3	2.1	2.1
Potassium, K, mg/l, B4	<2.5	7.2	12	5.2
Parameters	P-START	P80	PM	PA
Chloride, Cl, mg/l, B2				15
Chloride, Cl, mg/l, B3	3	4.9	4.3	4.3
Chloride, Cl, mg/l, B4				
Parameters	P-START	P80	PM	PA
N-tot, mg/l, B2		7.1		9.4

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N-tot, mg/l, B3	3.4	13	13	13
N-tot, mg/l, B4	11	45		32
Parameters	P-START	P80	PM	PA
Magnesium, Mg, mg/l,				<.1
Magnesium, Mg, mg/l,	<.1	<.1	<.1	<.1
Magnesium, Mg, mg/l,	<.1	<.1	<.1	<.1
Parameters	P-START	P80	PM	PA
Sodium, Na, mg/l, B2				5.3
Sodium, Na, mg/l, B3	1.8	3.9	3.6	3.6
Sodium, Na, mg/l, B4	2.6	9	15	6.7
Parameters	P-START	P80	PM	PA
Sulphate, S, mg/l, B2				<2
Sulphate, S, mg/l, B3	<2	<2	<2	<2
Sulphate, S, mg/l, B4				<2
Parameters	P-START	P80	PM	PA
Suspended solids, mg/l,		<5		
Suspended solids, mg/l,				
Suspended solids, mg/l,				
Parameters	P-START	P80	PM	PA
TOC, mg/l, B2		18		5.9
TOC, mg/l, B3				
TOC, mg/l, B4		5.8	7.9	5.8
Parameters	P-START	P80	PM	PA
Torrsubstans, mg/l, B2		2100		<5
Torrsubstans, mg/l, B3	∞∞∞	∞∞∞	∞∞∞	∞∞∞
Torrsubstans, mg/l, B4	<5			<5

**Appendix D: Titration curves and readings for VSEP batch testing 1. With acid dose, date of experiment 190405**



1) Alkalinity for screened black water before acid dose

mmoles of HCl acid/l of black water	pH
0	8
0.8	8
1.8	7
3.4	7
5.4	6
6.6	6
7.4	5
8	4
8.5	4
9.3	3
10.6	3
15.1	3

3). Titration curve for screened black water after acid dose

mmoles of HCl acid/l of black water	pH
0	6.6
1.9	6
2.8	5.5
3.5	4.8
4	4.4
4.6	3.8
5	3.4
6.3	3
10.4	2.5

2) Titration curve for screened and filtered (1um) black water before acid dose

mmoles of HCl acid/l of black water	pH
0	8
1	7
1.5	7
3.4	7
4.9	6
6	6
7	5
7.2	4
7.7	4
8.3	4
9.5	3
14.1	3

4). Titration curve for screened and filtered black water after acid dose

mmoles of HCl acid/l of black water	pH
0	7.1
1	6
1.6	4.7
1.7	4.4
1.8	4
2.3	3.3
2.7	3
4.6	2.5



1. Alkalinity measurements for C1 concentrate at 50% volume reduction

mmoles of HCl acid/l of black water	pH
0	6.07
1.9	5.5
2.9	5
4.2	4.48
5.5	4
7	3.5
8.9	3
13.2	2.5

2. Alkalinity measurements for C1-f concentrate at 50% volume reduction

mmoles of HCl acid/l of black water	pH
0	6.27
0.7	6
2.1	5.5
3.1	5
4.2	4.5
5.6	4
7	3.5
8.9	3
13.5	2.5

4. Alkalinity measurements for C2 concentrate at 80% volume reduction

mmoles of HCl acid/l of black water	pH
0	6.96
2.1	6.5
4.5	6
6.4	5.5
8.7	5
11.5	4.5
13.8	4
15.2	3.5
17.2	3
23.2	2.5

5. Alkalinity measurements for C2-f concentrate at 80% volume reduction

mmoles of HCl acid/l of black water	pH
0	7.25
2.7	6.5
4.7	6
6.6	5.5
8.8	5
11.3	4.5
13.6	4
15.2	3.45
17	3
23.1	2.5

7. Alkalinity measurements for C3 concentrate at 90% volume reduction

mmoles of HCl acid/l of black water	pH
0	7.03
3.4	6.5
6.5	6
9.1	5.5
12.9	5
16.8	4.5
19.8	4
22	3.5
24.5	3
32.2	2.5

8. Alkalinity measurements for C3-f Concentrate at 90% volume reduction.

mmoles of HCl acid/l of black water	pH
0	7.2
3.3	6.5
6.4	6
9.2	5.5
12	5
15.8	4.5
19	4
21	3.5
23.4	3
29.8	2.5

10. Alkalinity measurements for C4 concentrate at maximal volume reduction.

mmoles of HCl acid/l of black water	pH
0	7.13
3.9	6.5
7.3	6
10.1	5.5
13.6	5
17.7	4.5
20.8	4
22.8	3.5
25.6	3
33.4	2.5

11. Alkalinity measurements for C4-f concentrate at maximal volume reduction.

mmoles of HCl acid/l of black water	pH
0	7.34
1.4	7
3.9	6.5
6.9	6
9.3	5.5
12.4	5
16.3	4.5
19.6	4
21.5	3.5
24	3
31.2	2.5

3. Alkalinity measurements for P1 permeate at 50% volume reduction

mmoles of HCl acid/l of permeate	pH
0	5.66
0.4	5.38
0.5	4.52
0.8	3.73
0.9	3.52
1.7	3
4.9	2.5

6. Alkalinity measurements for P2 permeate at 80% volume reduction

mmoles of HCl acid/l of permeate	pH
0	6.29
0.6	4.9
0.7	4.3
0.8	3.8
0.9	3.5
2	3
6.4	2.5

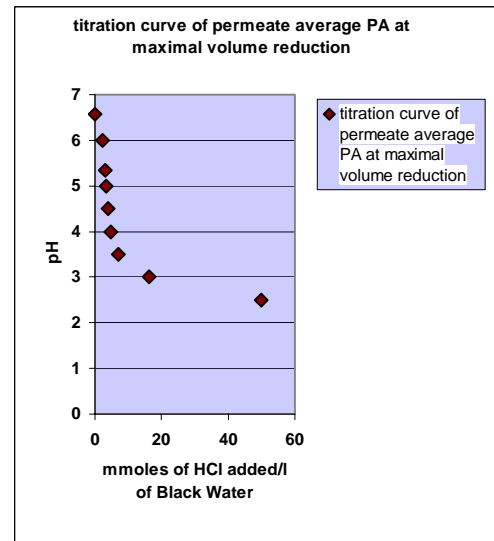
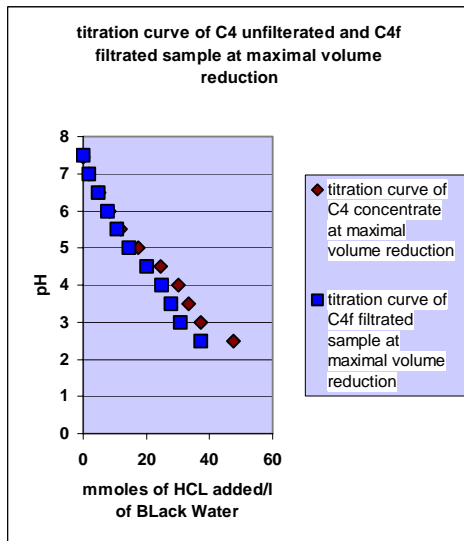
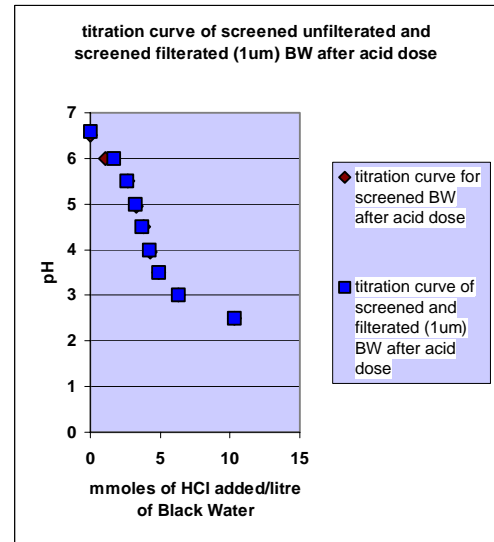
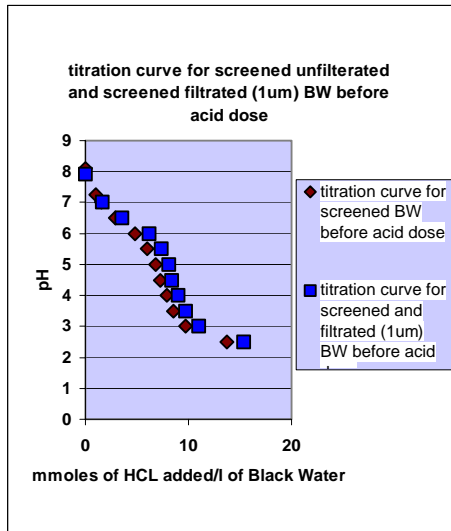
12. Alkalinity measurements for P4 permeate at maximal volume reduction.

mmoles of HCl acid/l of permeate	pH
0	6.45
0.5	5.63
0.7	4.95
0.8	4.26
0.9	3.82
1.1	3.5
2.2	3
7.1	2.5

13. Alkalinity measurements for PA permeate at maximal volume reduction.

mmoles of HCl acid/l of permeate	pH
0	6.16
0.5	5.3
0.7	4.62
0.8	4.1
0.9	3.9
1.1	3.5
2.2	3
7.7	2.5

**Appendix E: Titration curves and readings for VSEP batch testing 2 with acid dosing  
date of experiment 020505**



1. Titration curve for screened unfiltered black water before acid dose

mmoles of HCl acid/l of black water	pH
0	8.09
1	7.25
1.5	7
2.9	6.5
4.8	6
6	5.5
6.8	5
7.3	4.5
7.9	4
8.5	3.5
9.7	3
13.7	2.5

2. Titration curve for screened and filtrated (1um) black water before acid dose

mmoles of HCl acid/l of black water	pH
0	7.91
1.6	7
3.5	6.5
6.2	6
7.4	5.5
8.1	5
8.4	4.5
9	4
9.7	3.5
11	3
15.4	2.5

3. Titration curve for screened unfiltered black water after acid dose

mmoles of HCl acid/l of black water	pH
0	6.5
1.1	6
2.7	5.5
3.3	4.95
3.8	4.5
4.3	3.96
4.9	3.5
6.3	3
10.3	2.5

4. Titration curve for screened and filtrated (1um) black water after acid dose

mmoles of HCl acid/l of black water	pH
0	6.6
1.7	6
2.6	5.5
3.2	5
3.7	4.5
4.2	4
4.9	3.5
6.3	3
10.3	2.5

1. Alkalinity measurements for C4 concentrate at maximal volume reduction

mmoles of HCl acid/l of black water	pH
0	7.45
1.8	7
4.9	6.5
8.3	6
11.9	5.5
17.5	5
24.5	4.5
30	4
33.3	3.5
37.2	3
47.6	2.5

2. Alkalinity measurements for C4f concentrate at maximal volume reduction

mmoles of HCl acid/l of black water	pH
0	7.5
1.8	7
4.6	6.5
7.8	6
10.5	5.5
14.5	5
20	4.5
24.8	4
27.7	3.5
30.6	3
37.1	2.5

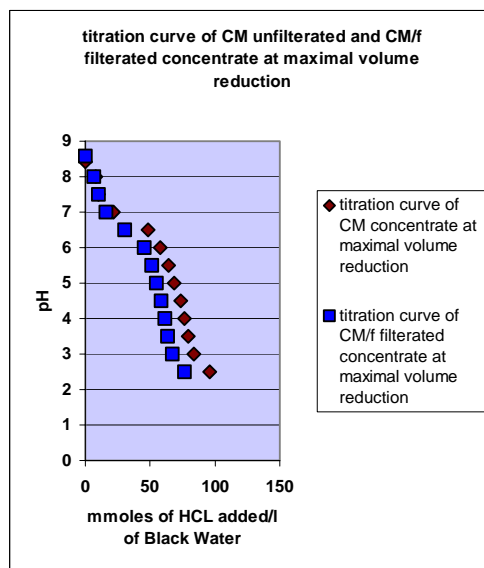
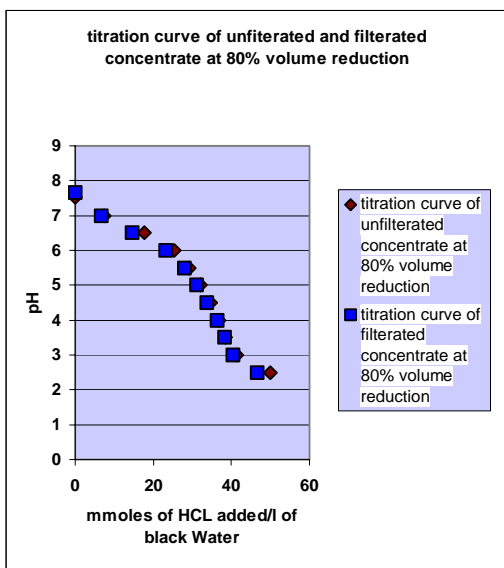
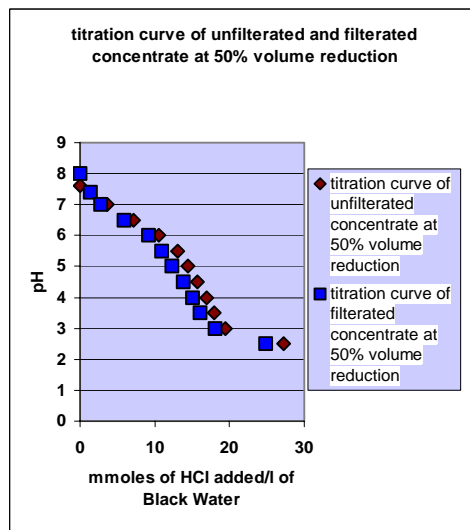
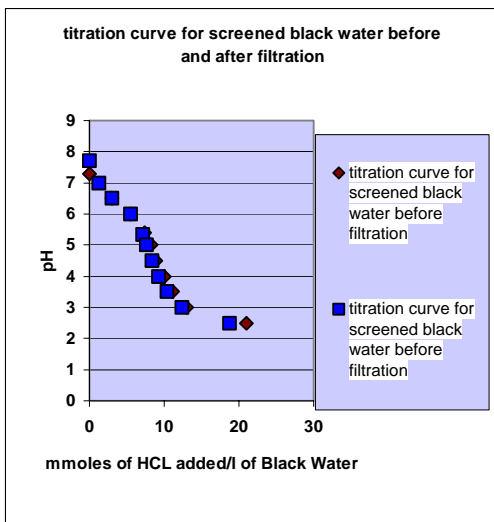
3. Alkalinity measurements for permeate average (PA) at maximal volume reduction

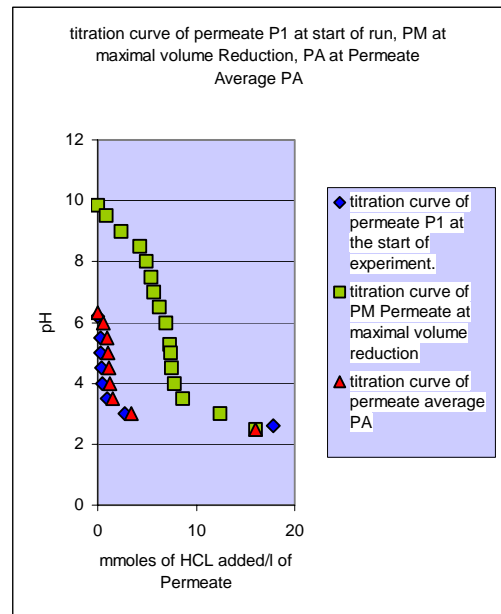
mmoles of HCl acid/l of black water	pH
0	6.56
2.2	6
3	5.34
3.4	5
3.9	4.5
4.7	4
6.9	3.5
16.2	3
50	2.5

RESULT ANALYSIS

The big difference in titration curves of filtrated and un-filtrated samples clearly shows that an inorganic fouling is quite well expected. The fouling can be from calcium carbonate and calcium phosphate.

**Appendix F: Titration curves and readings for VSEP batch testing 3 with anti scalant, date of experiment: 110505**







1. Screened black water before filtration.

mmoles of HCl acid/l of black water	pH
0	7.28
3	6.5
5.5	6
7.4	5.4
8.3	5
8.9	4.5
10	4
11.1	3.5
13	3
21	2.5

2. Screened black water after filtration

mmoles of HCl acid/l of black water	pH
0	7.71
1.3	7
3	6.5
5.5	6
7.1	5.34
7.6	5
8.4	4.5
9.3	4
10.4	3.5
12.4	3
18.7	2.5

3. Alkalinity measurements of C50 unfiltered concentrate at 50% volume reduction.

mmoles of HCl acid/l of black water	pH
0	7.6
3.6	7
7.2	6.5
10.5	6
13.1	5.5
14.4	5
15.7	4.5
16.9	4
17.9	3.5
19.4	3
27.2	2.5

4. Alkalinity measurements of C50/f filtered concentrate at 50% volume reduction.

mmoles of HCl acid/l of black water	pH
0	8.01
1.4	7.4
2.7	7
5.9	6.5
9.2	6
10.9	5.5
12.3	5
13.8	4.5
15.1	4
16.1	3.5
18.1	3
24.9	2.5

5. Alkalinity measurements of C80 unfiltered concentrate at 80% volume reduction.

mmoles of HCl acid/l of black water	pH
0	7.52
7.5	7
17.8	6.5
25.4	6
29.1	5.5
32	5
34.6	4.5
36.8	4
38.6	3.5
41.3	3
50	2.5

6. Alkalinity measurements of C80/f filtrated concentrate at 80% volume reduction.

mmoles of HCl acid/l of black water	pH
0	7.65
6.6	7
14.7	6.5
23.3	6
27.9	5.5
31	5
33.8	4.5
36.4	4
38.3	3.5
40.5	3
46.7	2.5

7. Alkalinity measurements of CM unfiltered concentrate at maximal volume reduction.

mmoles of HCl acid/l of black water	pH
0	8.41
7.7	8
10.3	7.5
21.8	7
48.2	6.5
57.6	6
63.9	5.5
68.7	5
73.2	4.5
76.6	4
79.2	3.5
83.7	3
95.6	2.5

8. Alkalinity measurements of CM/f filtrated concentrate at maximal volume reduction.

mmoles of HCl acid/l of black water	pH
0	8.57
6.8	8
9.9	7.5
15.8	7
30	6.5
45.7	6
51.4	5.5
55	5
58.5	4.5
61.3	4
63.6	3.5
66.9	3
76.6	2.5

9. Alkalinity measurements of P1 permeate at the start of experiment.

mmoles of HCl acid/l of permeate	pH
0	6.19
0.24	5.5
0.29	5
0.35	4.5
0.48	4
0.91	3.5
2.73	3
17.85	2.6

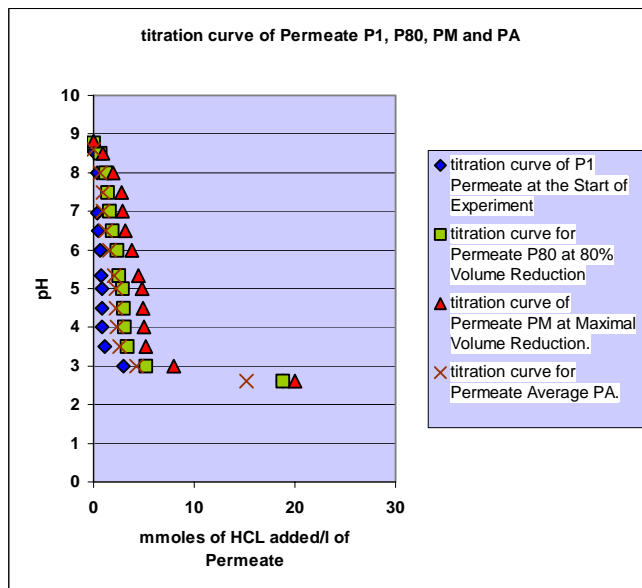
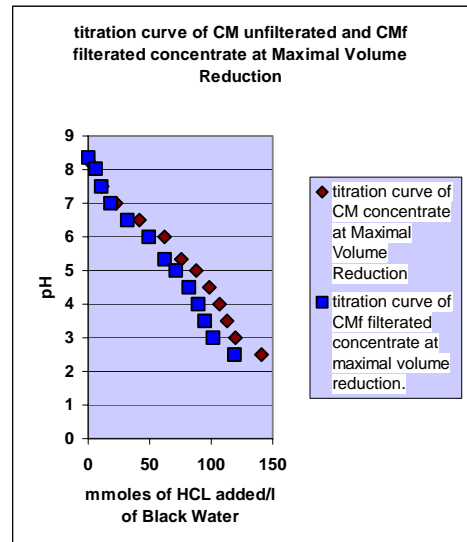
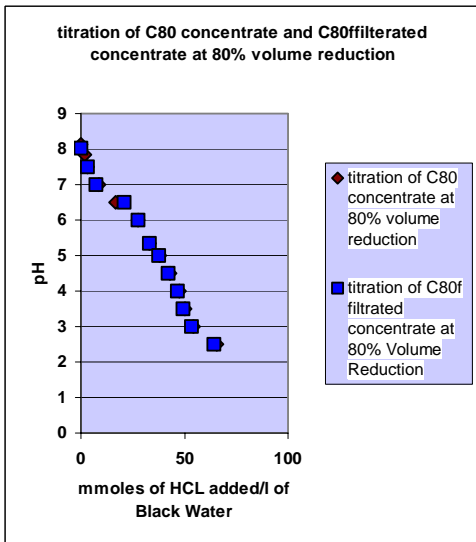
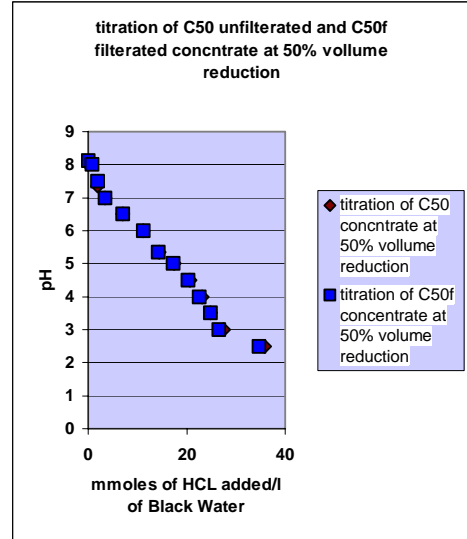
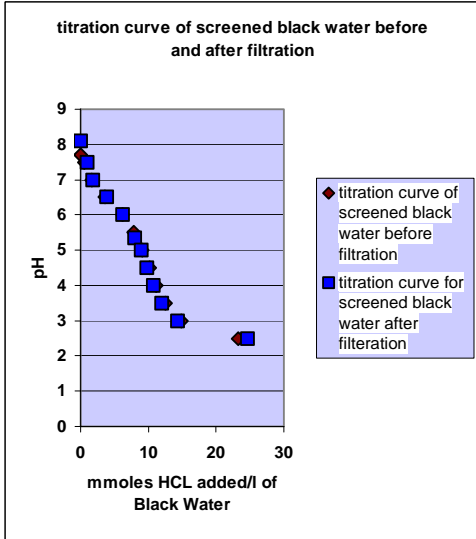
11. Alkalinity measurements of PM permeate at maximal volume reduction.

mmoles of HCl acid/l of permeate	pH
0	9.86
0.84	9.5
2.37	9
4.24	8.5
4.95	8
5.42	7.5
5.72	7
6.27	6.5
6.88	6
7.33	5.29
7.41	5
7.53	4.5
7.77	4
8.59	3.5
12.4	3
16	2.5

12. Alkalinity measurements of PA permeate average of the whole collected permeate.

mmoles of HCl acid/l of permeate	pH
0	6.32
0.54	6
0.97	5.5
1.09	5
1.15	4.5
1.2	4
1.48	3.5
3.45	3
16	2.5

**Appendix G: Titration curves and readings for VSEP batch testing 4 (without dosing)  
date of experiment: 180505**



1. Screened black water before filtration.

mmoles of HCl acid/l of black water	pH
0	7.69
0.55	7.5
1.6	7
3.6	6.5
6.15	6
7.9	5.5
9.1	5
10.15	4.5
11.15	4
12.55	3.5
14.9	3
23.2	2.5

2. Screened black water after filtration

mmoles of HCl acid/l of black water	pH
0	8.09
1	7.5
1.85	7
3.85	6.5
6.25	6
7.95	5.34
8.95	5
9.8	4.5
10.75	4
11.95	3.5
14.25	3
24.65	2.5

3. Alkalinity measurements of C50 un-filtrated concentrate at 50% volume reduction

mmoles of HCl acid/l of black water	pH
0	8.02
1.9	7.35
3.4	7
7.1	6.5
11.2	6
14.4	5.34
17.5	5
20.6	4.5
23.1	4
24.8	3.5
27.4	3
35.8	2.5

4. Alkalinity measurements of C50/f filtrated concentrate at 50% volume reduction

mmoles of HCl acid/l of black water	pH
0	8.13
0.8	8
1.9	7.5
3.4	7
7	6.5
11.1	6
14.2	5.34
17.2	5
20.3	4.5
22.6	4
24.9	3.5
26.6	3
34.7	2.5

5. Alkalinity measurements of C80 unfiltered concentrate at 80% volume reduction.

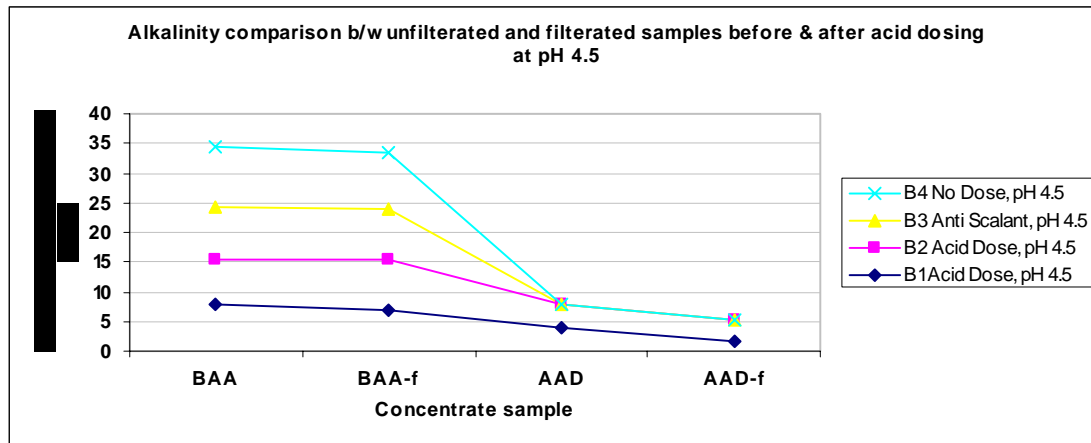
mmoles of HCl acid/l of black water	pH
0	8.13
1.6	7.84
3.2	7.5
8.4	7
16.6	6.5
27.8	6
33.2	5.34
38	5
43.2	4.5
47.3	4
50	3.5
54.3	3
65.5	2.5

6. Alkalinity measurements of C80/filtrated concentrate at 80% volume reduction.

mmoles of HCl acid/l of black water	pH
0	8.02
3.1	7.5
7.4	7
21	6.5
27.8	6
33	5.34
37.5	5
42.1	4.5
46.7	4
49.3	3.5
53.2	3
64.4	2.5

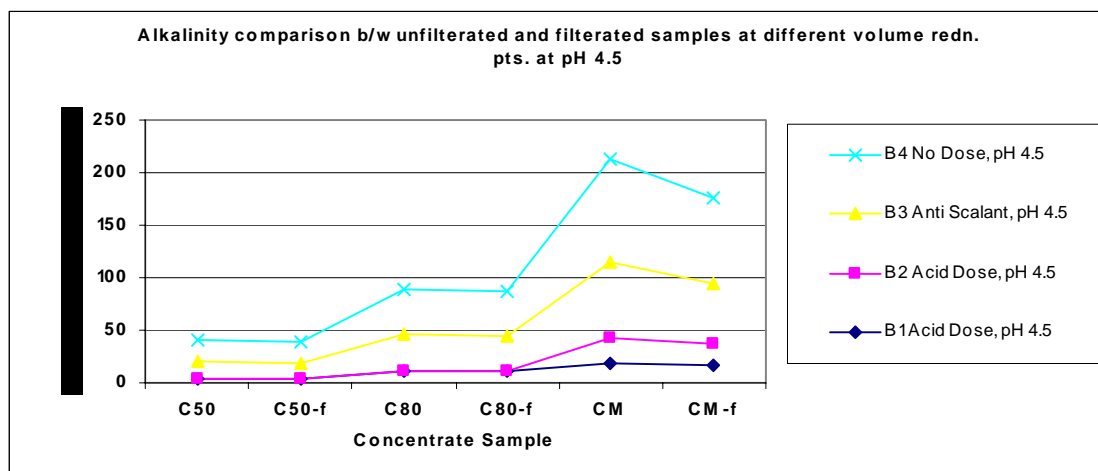
**Appendix H: Tables for alkalinity (pH 4.5) values for filtrated and un-filtrated samples at different sampling points of VSEP testing**

	BAA	BAA-f	AAD	AAD-f
	mmole HCl/l Black-Water	mmole HCl/l Black-Water	mmole HCl/l Black-Water	mmole HCl/l Black-Water
B1 Acid Dose, pH 4.5	8	7	4	1.7
B2 Acid Dose, pH 4.5	7.3	8.4	3.8	3.7
B3 Anti Scalant, pH 4.5	8.9	8.4		
B4 No Dose, pH 4.5	10.15	9.8		



**Graph 1. Alkalinity values of concentrated and filtrated samples before and after acid dosing at pH 4.5**

	C50	C50-f	C80	C80-f	CM	CM-f
	mmole HCl/l Black- Water	mmole HCl/l Black- Water	mmole HCl/l Black- Water	mmole HCl/l Black- Water	mmole HCl/l Black- Water	mmole HCl/l Black- Water
B1 Acid Dose, pH 4.5	4.2	4.2	11.5	11.3	17.7	16.3
B2 Acid Dose, pH 4.5					24.5	20
B3 Anti Scalant, pH 4.5	15.7	13.8	34.6	33.8	73.2	58.5
B4 No Dose, pH 4.5	20.6	20.3	43.2	42.1	98.4	81.5



**Graph 2. Alkalinity values of concentrated and filtrated samples at different sampling points, at pH 4.5**



**Appendix I: Table of alkalinity difference values (pH 4.5) at different sampling points of VSEP testing**

	BAA-BAAf	AAD-AADf	C50-C50f	C80-C80f	CM-CMf
B1 Acid Dose pH 4.5	1	2.3	0	0.2	1.4
B2 Acid Dose pH 4.5	-1.1	0.1			4.5
B3 Anti Scalant pH 4.5	0.5		2.2	0.8	14.7
B4 No Dose pH 4.5	0.35		0.7	1.1	17.1

**Appendix J: Graphs on al-control results of concentrate samples**

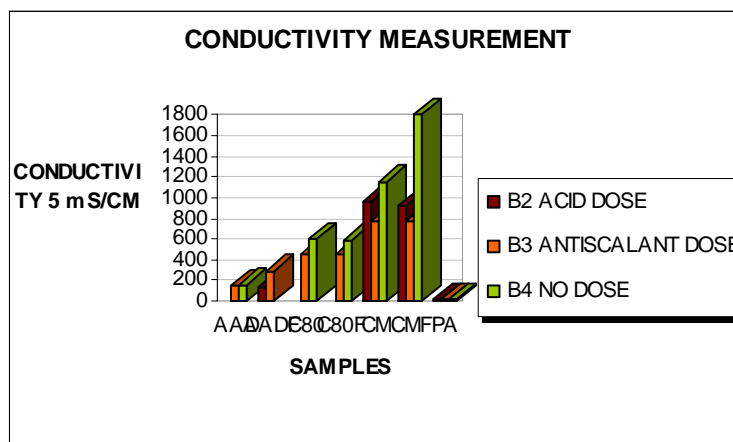
The concentration factor for B2 is  $C_f = 9.0$

The concentration factor for B3 is  $C_f = 7.4$

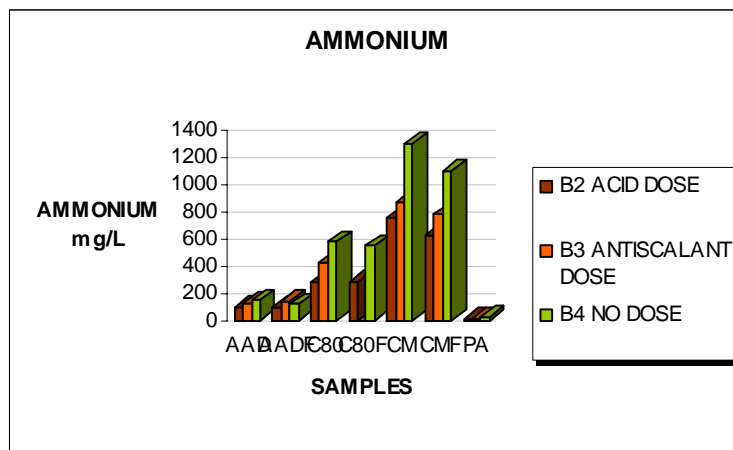
The concentration factor for B4 is  $C_f = 11.36$

In general it is assumed that maximum difference in un-filtrated and filtrated values could be found in B4 samples, then in B2 and then in B3.

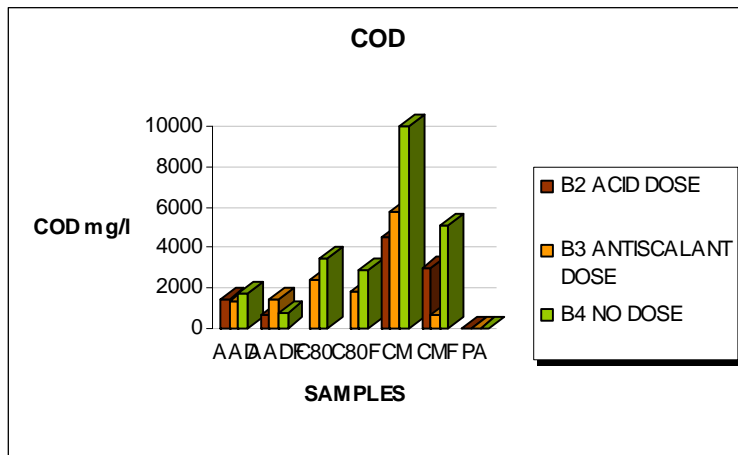
Difference in un-filtrated & filtrated values:  $B4 > B2 > B3$  (in general)



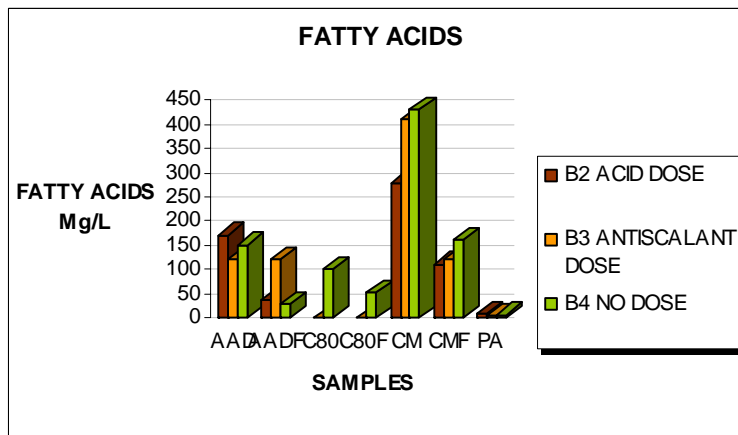
**Graph 4** The graph above shows conductivity values of black water samples taken at different sampling points for three batch tests.



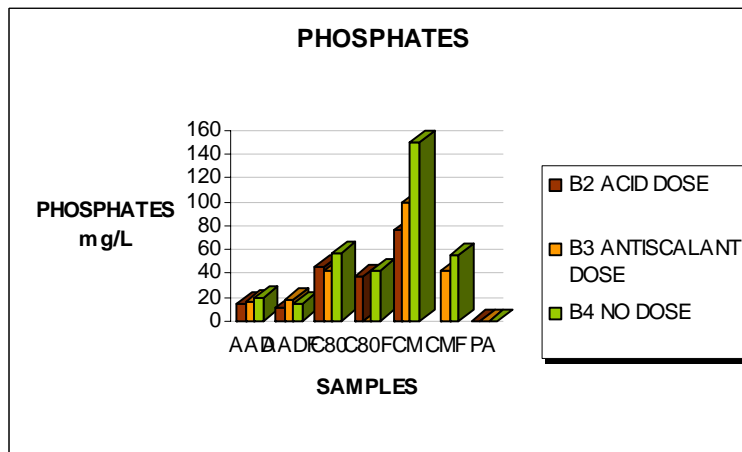
**Graph 5.** Difference in ammonium values of filtrated and concentrates samples



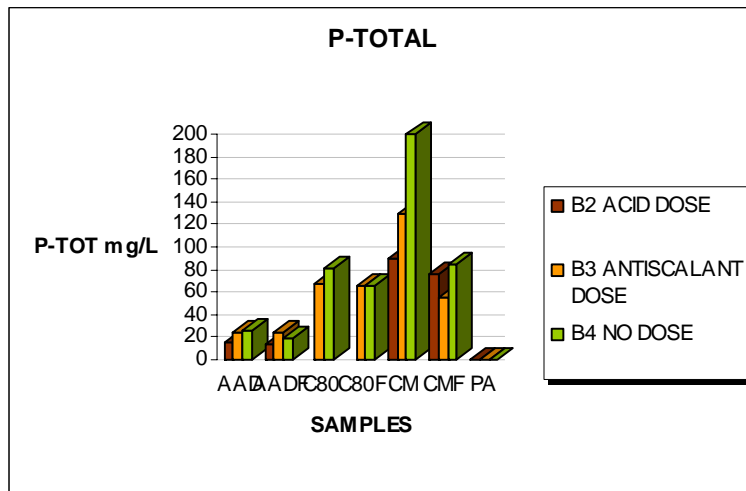
**Graph 6. High difference in COD values of filtrated and concentrate sample**



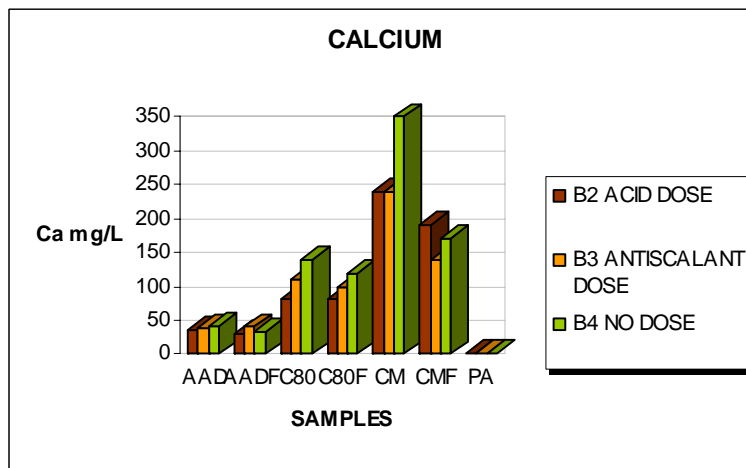
**Graph 7. High difference in fatty acids values of filtrated and concentrates samples**



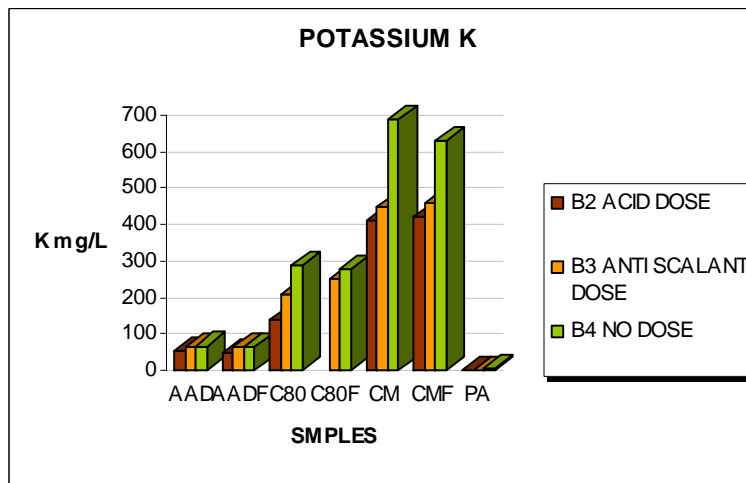
**Graph 8. Difference in phosphate values of filtrated and concentrates samples**



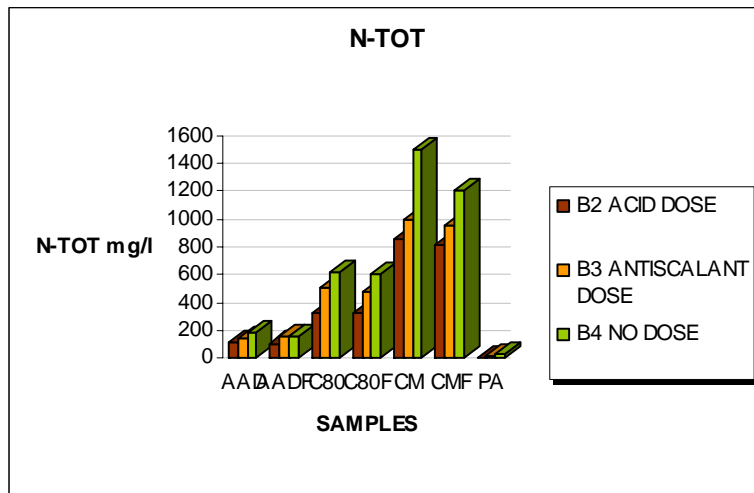
**Graph 9. Difference in P-tot values of filtrated and concentrated samples**



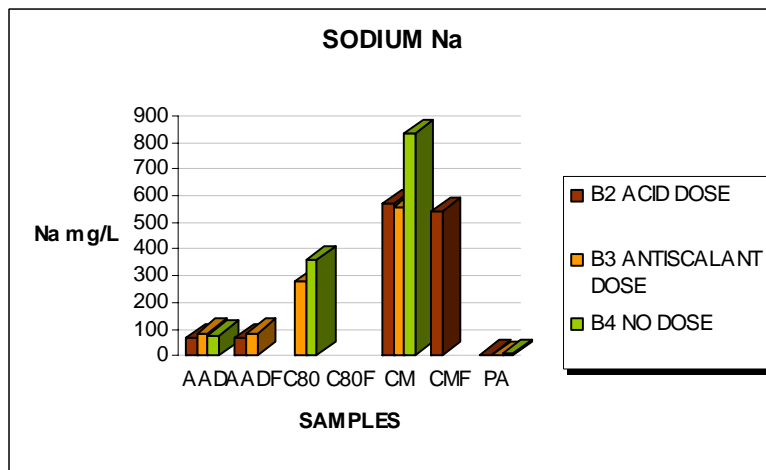
**Graph 10. Difference in calcium values of filtrated and concentrates samples**



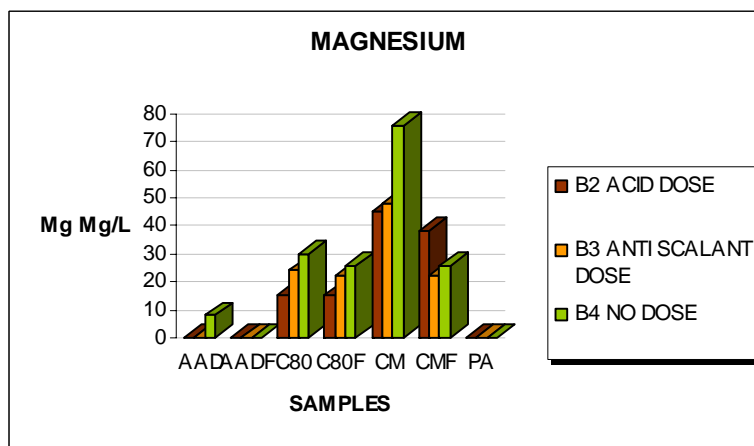
**Graph 11. Difference in potassium values of filtrated and concentrates samples**



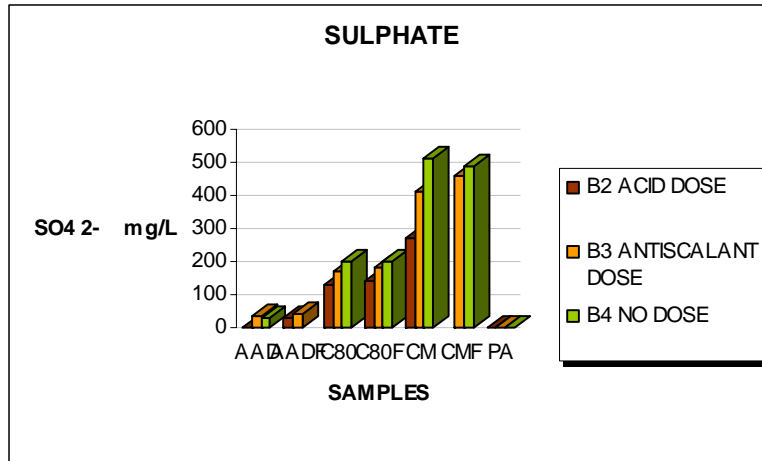
**Graph 12. Difference in N-tot values of filtrated and concentrates samples**



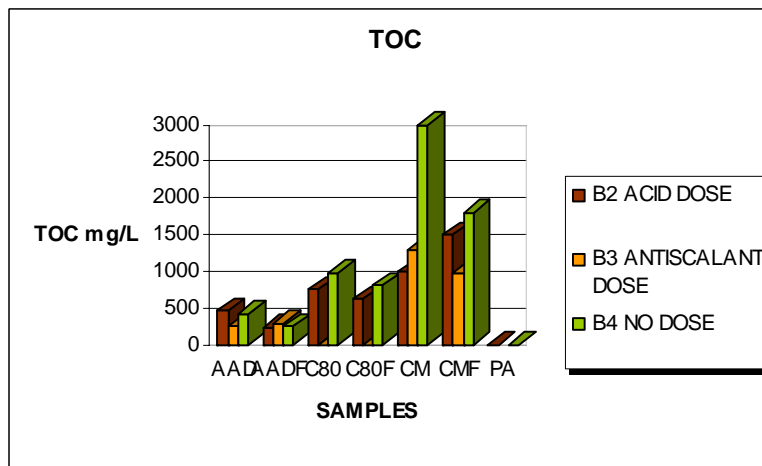
**Graph 13. Difference in sodium values of filtrated and concentrates samples**



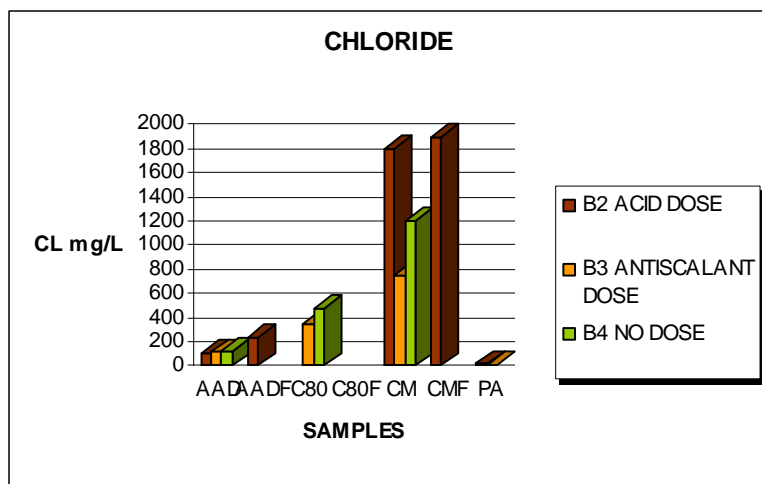
**raph 14. Difference in magnesium values of filtrated and concentrates samples**



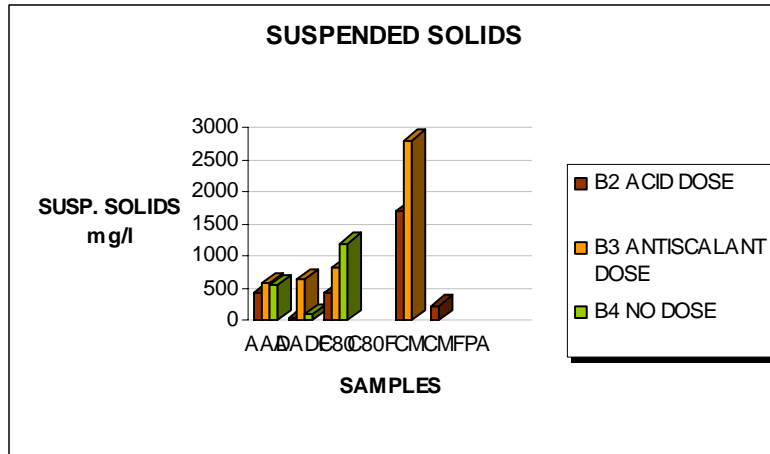
**Graph 15. Difference in sulphate values of filtrated and concentrates samples**



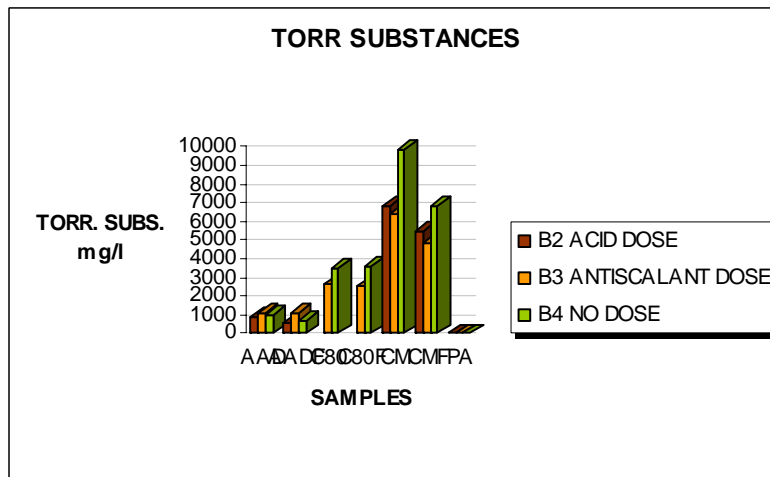
**Graph 16. Difference in TOC values of filtrated and concentrates samples**



**Graph 17. Difference in chloride values of filtrated and concentrates samples**

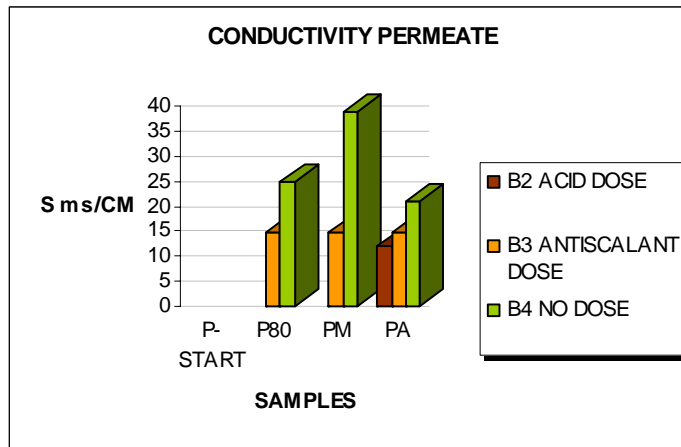


**Graph 18. Difference in suspended solids values of filtrated and concentrates samples**

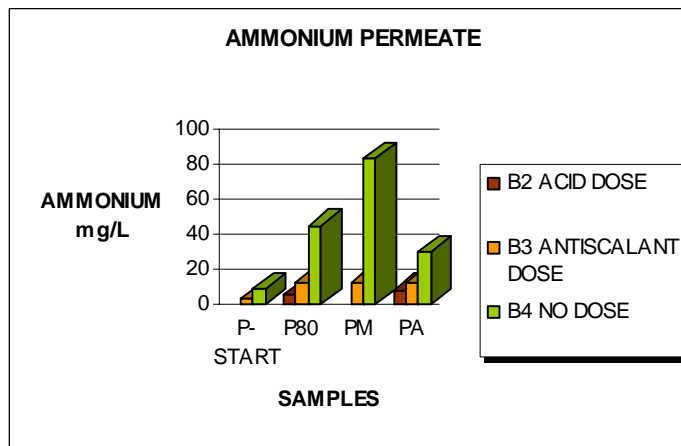


**Graph 19. Difference in torr substance values of filtrated and concentrates samples**

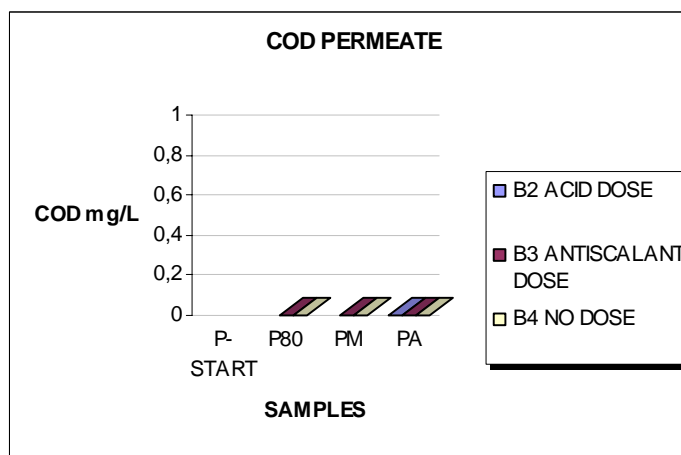
**Appendix K: Graphs on al-control results of permeate samples**



**Graph 20. Conductivity values of permeate samples**

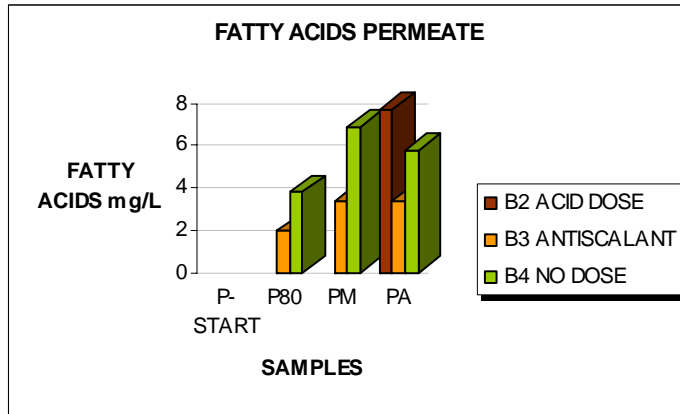


**Graph 21. Ammonium values of permeate samples**

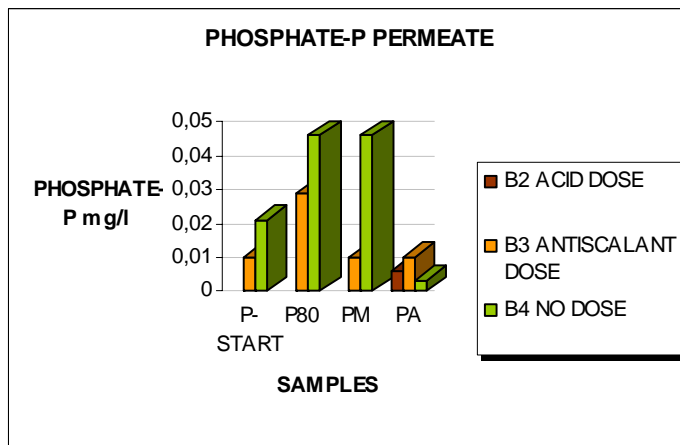


**Graph 22. COD values of permeate samples**

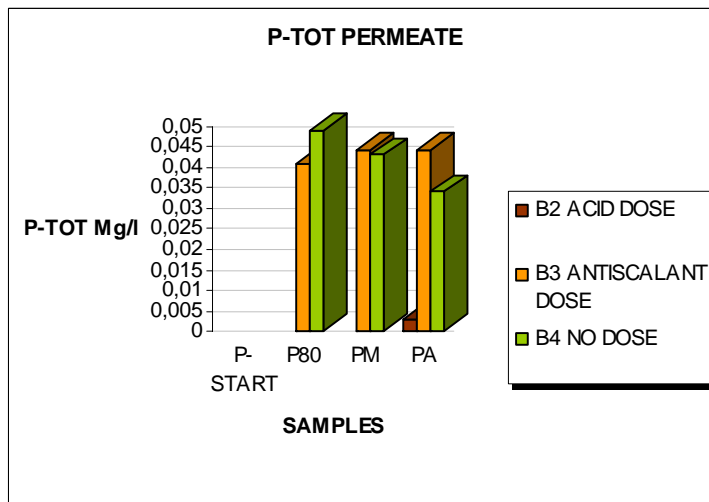




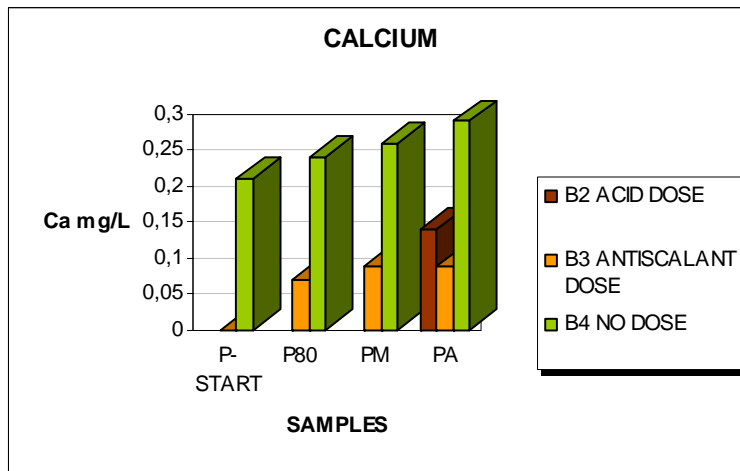
**Graph 23. Fatty acids values of permeate samples**



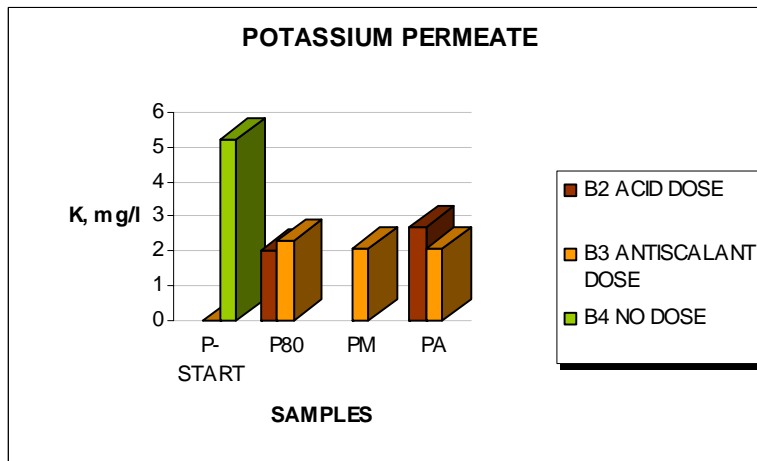
**Graph 24. Phosphate values of permeate samples**



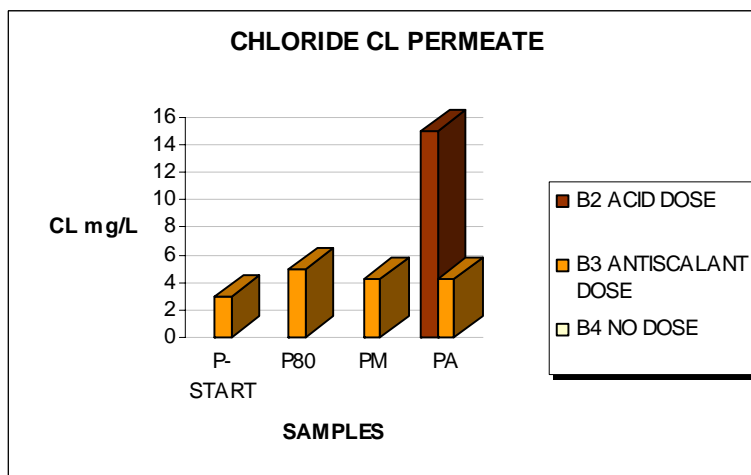
**Graph 25. P-tot values of permeate samples**



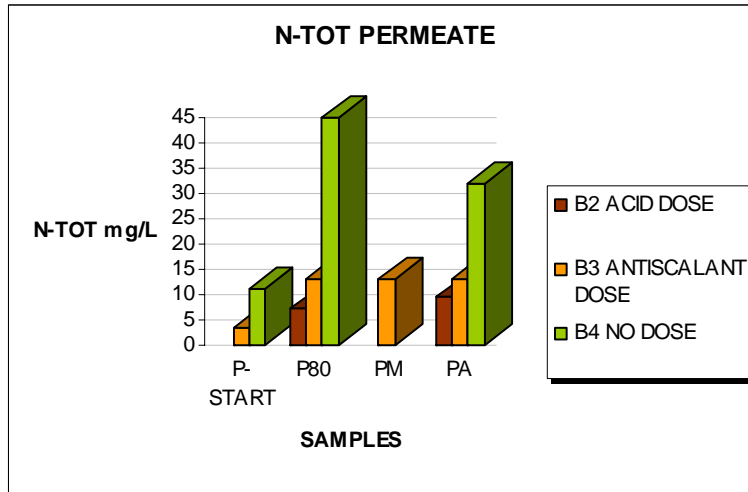
**Graph 26 Calcium values of permeate samples**



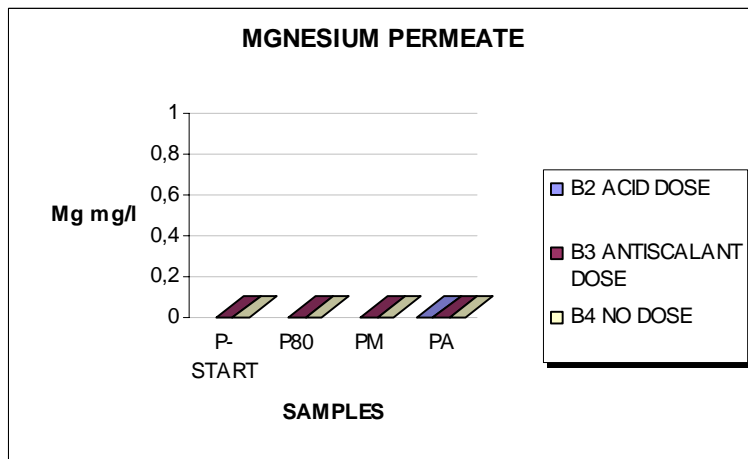
**Graph 27. Potassium values of permeate samples**



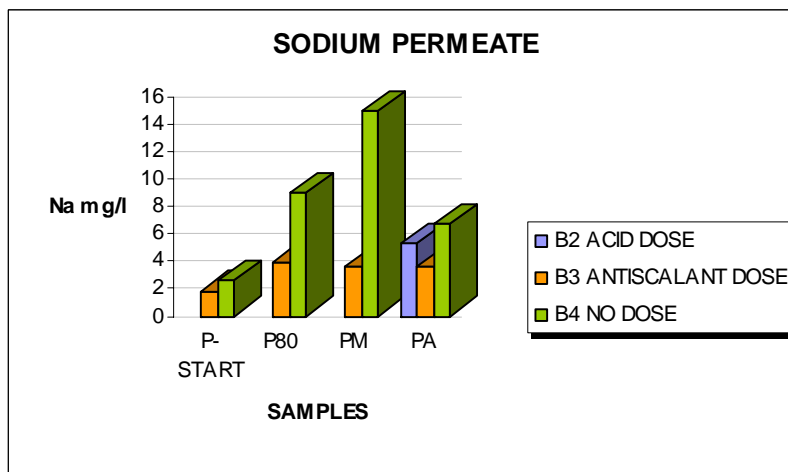
**Graph 28. Chloride values of permeate samples**



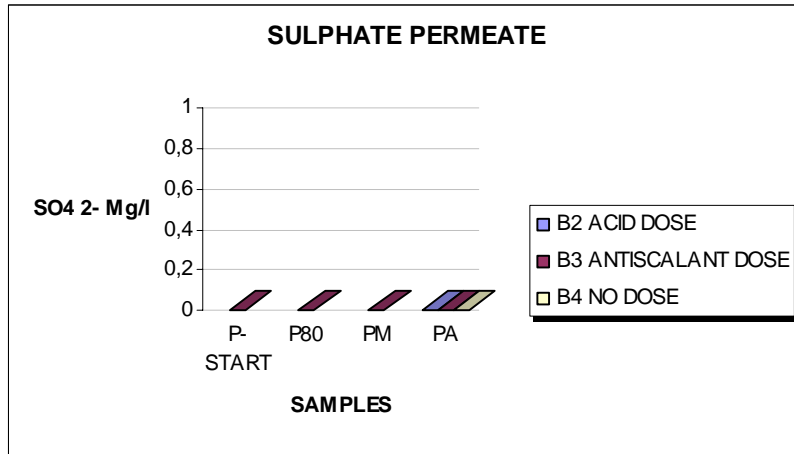
**Graph 29. N-tot values of permeate samples**



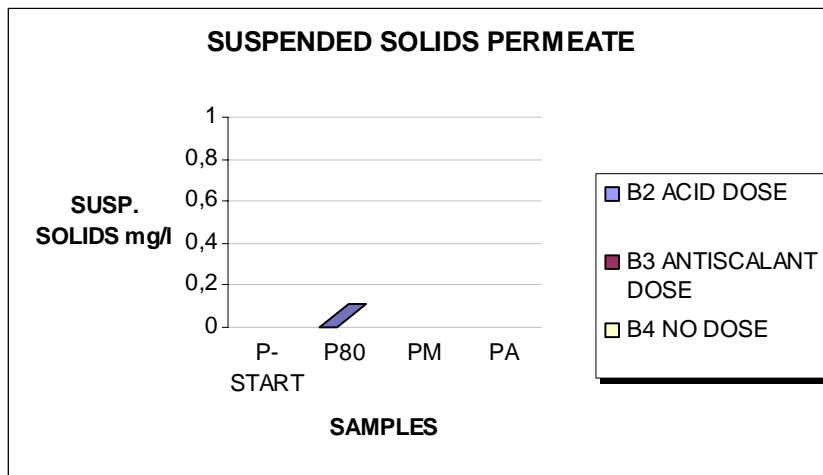
**Graph 30. Magnesium values of permeate samples**



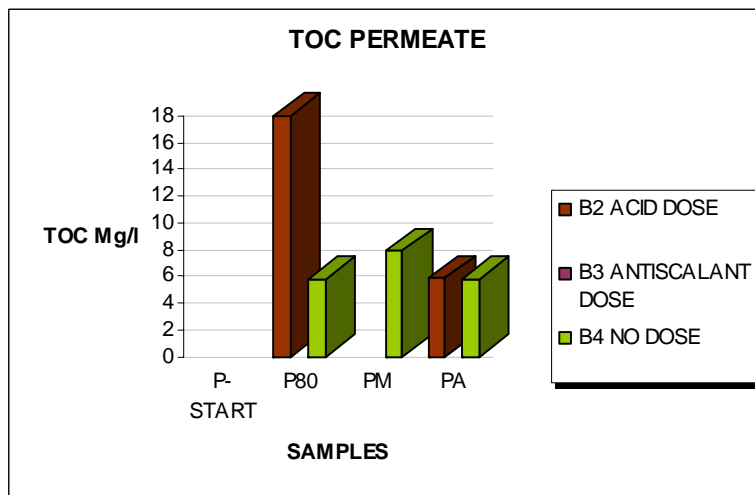
**Graph 31. Sodium values of permeate samples**



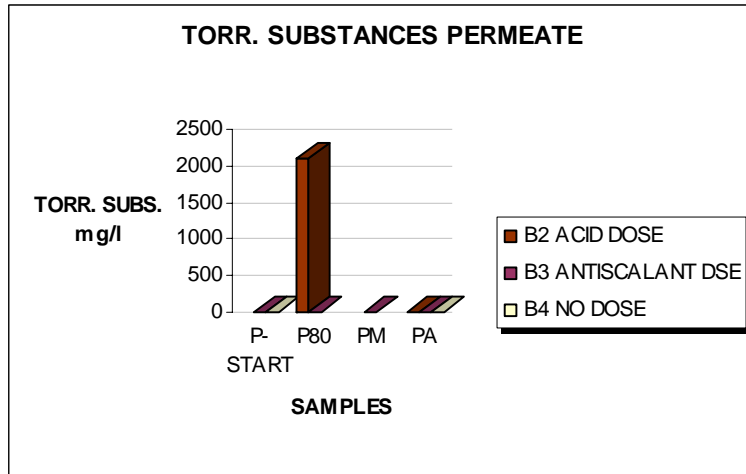
**Graph 32. Sulphate values of permeate samples**



**Graph 33. Suspended solids values of permeate samples**



**Graph 34. TOC values of permeate samples**

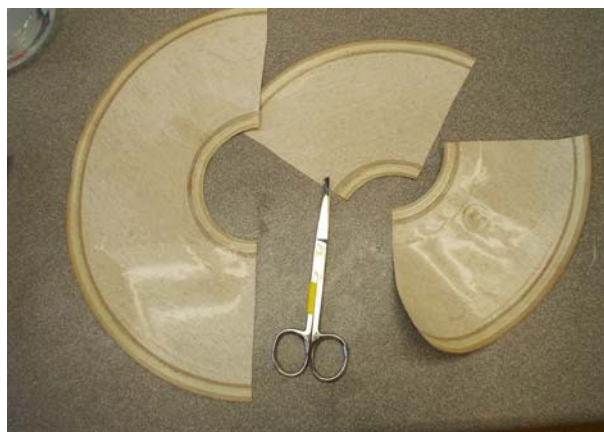


**Graph 35. Torr. substance values of permeate samples**

## Appendix L: Membrane cleaning analysis



**Figure 1.** Membrane picture above is used on 1<sup>st</sup> VSEP batch test. Used from VSEP test 1 to the middle of VSEP test 2.



**Figure 2.** Membrane picture above is on 1<sup>st</sup> VSEP batch test. Used from VSEP test 1 to the middle of VSEP test 2. One quarter part is cleaned and rubbed in 150 ml distilled water on 10.05.05. Another quarter part is soaked in (50 ml NC2 + 1 lit water + NaOH) pH 11 solution for 2days on 9.05.05.



**Figure 3. Membrane Part used on 2<sup>nd</sup> Run. Used from middle of VSEP Batch Test 2 to its end. It was uninstalled on 09.05.05.**



**Figure 4. Membrane part used on 2<sup>nd</sup> run. Used from middle of VSEP batch test 2 to its end. It was uninstalled on 09.05.05. One quarter part is cleaned and rubbed in 150 ml distilled water on 10.05.05. Another quarter part is soaked in ( 50 ml NC2 + 1 lit water + NaOH ) pH 11 solution for 2 days on 9.05.05**



**Figure 5.** The two membrane pieces above are from membrane used on 2<sup>nd</sup> VSEP testing. One is in pure *Ariel* solution and another in (*Ariel* + *NaOH*) pH 11 solution.

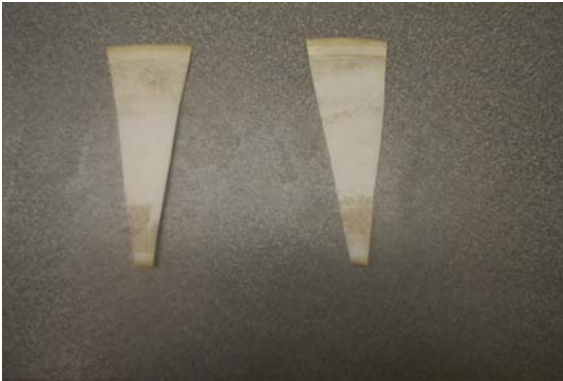


**Figure 6.** The same picture above but taken from sides of cup.





**Figure 7.** Membrane piece above is from membrane used on 2<sup>nd</sup> VSEP run. When soaked in detergent called *Tornado* for 1 day and cleaned in fresh water, no effect on foulants was observed.



**Figure 8.** The two pieces are also from membrane used on 2<sup>nd</sup> VSEP run. Left one was soaked in one cup solution of detergent (*Ariel* + *NaOH*) pH 11 for 1 day. Right one was kept in one-cup solution of pure *Ariel* detergent. The left piece after one day was found to be much cleaner than right one.



**Figure 9. Membrane picture above is that used on VSEP batch 3 (with anti scalant). It is fouled. This was put uncut into a solution of (NC2 2450 ml + NaOH) ph 11 solution for 4 days.**



**Figure 10. The same membrane part used on VSEP batch testing 3(with anti-scalant). It is cleaned in fresh water after being kept in detergent solution for 4 days. It is found to be perfectly clean however some color fading is observed, may be due to high detergent. This membrane is again installed in VSEP for batch testing 4 (without acid/antiscalant).**

## Appendix M: Methodology of membrane cleaning

The cleaning analysis on the two fouled membranes was performed on 10.05.05. The procedure is as follows.

1. 1<sup>st</sup> and 2<sup>nd</sup> LFC1 membranes are cut into two quarters and one half.
2. One quarter of each is put into a solution of (25ml NC2 + 1 lit Water + NaOH) pH 11 for one day.
3. Another quarter of each is put into distilled water 150 ml and rubbed after some time.
4. One quarter of 2<sup>nd</sup> LFC1 is cut in 3 equal pieces 2c, 2d, 2e.
5. These pieces from 2<sup>nd</sup> membrane are put into detergents Tornado, Pure *Ariel* and (*Ariel* + *NaOH*) pH 11 solutions.
6. The third membrane taken from VSEP Run 3 (With Anti-Scalant) is put in solution of (50 ml NC2 + 1400 ml Water + NaOH) pH 11 for 4 days (12.05.05 to 16.05.05).

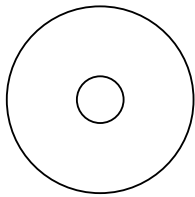


Figure 1. Membrane on VSEP 1<sup>st</sup> Run.

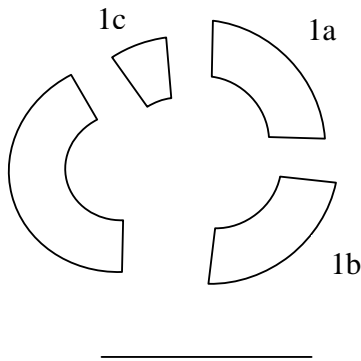


Figure 3. Part 1a put into a solution of (NC2 + water + NaOH), pH 11.

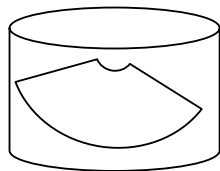


Figure 5. Part 1b put into distilled water and rubbed after some time.

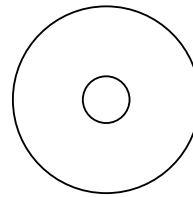


Figure 2. Membrane on VSEP 2<sup>nd</sup> Run.

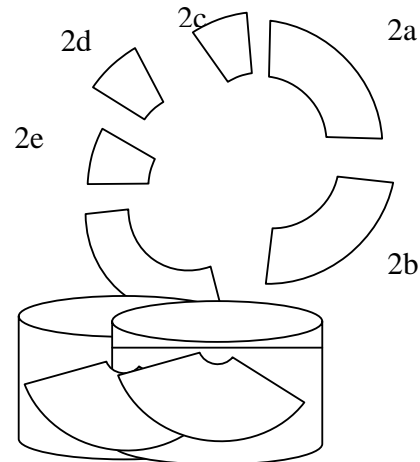


Figure 4. Part 2a put into a solution of (NC2 + water + NaOH), pH 11.

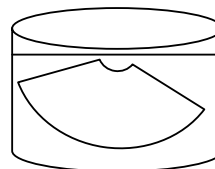


Figure 6. Part 2b put into distilled water and rubbed after some time.

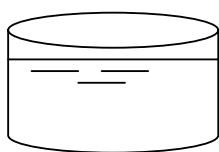


Figure 7. Chemical Analysis of remaining water from Part 1b cleaning is done

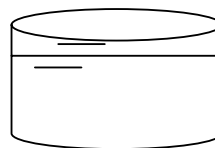


Figure 8. Chemical Analysis of remaining water from Part 2b cleaning is done

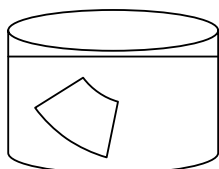


Figure 11. Part 2e put in *Ariel* + *NaOH* pH 11 detergents.

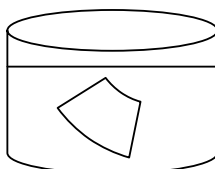


Figure 9. Part 2c put Pure *Tornado* detergent.

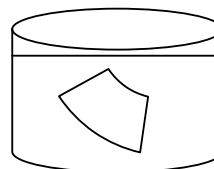


Figure 10. Part 2d put *Pure Ariel* detergent.

## CONCLUSIONS

1. Part 1a and 2a put in NC2 solution is clean of Organics. It is taken out and dried and compared with part 1b and 2b cleaned in fresh water. (*No Pictures are taken but dry membrane parts are kept in transparent folders at Testing Station Skogaberg*).
2. Part 2c(*figure 9 above and figure 7 in Appendix*) is put in pure detergent called *Tornado* had not been cleaned. This shows that *Tornado* had no effect on membrane fouling. Generally *Tornado* cleans grease and aliphatic compounds very well. Its failure in cleaning the membrane part proves that fouling is something other than grease or aliphatic compounds.
3. Part 2d (*figure 10 above and figure 8 in Appendix*) put in Pure Ariel detergent solution is cleaned to the maximum.
4. Part 2e (*Figure 11 above and figure 8 in Appendix*) put in Ariel with NaOH pH 11 Solution is very little cleaned. This shows that Ariel with NaOH has little effect on Membrane Fouling.
5. The membrane taken out after end of VSEP Run 3 when cleaned in NC2 pH 11 solution is found to be very clean after 4 days,(*See figure 9 and 10 in Appendix*). This membrane is again used for VSEP Run 4 (*No Dosing*).

**Appendix N: Mass balance calculation results**

B4	Na	B3	85	B2	Na
Cf FEED	79	Cf FEED	81.76	Cf FEED	68
CM predicted	73.04	CM predicted	3.07	CM predicted	63.287
PA predicted	6.11	PA predicted	84.83	PA predicted	4.7
TOT.	79.15	TOT.	6.8	TOT.	67.987
Cf Conc.fac.	11.36	Cf Conc.fac.	<b>K+</b>	Cf Conc.fac.	9
	<b>K+</b>		6.8		<b>K+</b>
Cf Conc.fac.	11.36	Cf Conc.fac.	64	Cf Conc.fac.	9
Cf FEED	67	Cf FEED	65.7	Cf FEED	53
CM predicted	60.72	CM predicted	1.79	CM predicted	45.51
PA predicted	4.742	PA predicted	67.5	PA predicted	2.4003
TOT.	65.4	TOT.	<b>NH4+</b>	TOT.	47.9103
	<b>NH4+</b>		6.8		<b>NH4+</b>
Cf Conc.fac.	11.36	Cf Conc.fac.	130	Cf Conc.fac.	9
Cf FEED	150	Cf FEED	127.02	Cf FEED	96
CM predicted	114.4	CM predicted	11.1	CM predicted	84.36
PA predicted	27.36	PA predicted	138.12	PA predicted	7.64
TOT.	141.76	TOT.	<b>COD</b>	TOT.	92.0
	<b>COD</b>		6.8		<b>COD</b>
Cf Conc.fac.	11.36	Cf Conc.fac.	1400	Cf Conc.fac.	9
Cf FEED	1700	Cf FEED	846.8	Cf FEED	1500
CM predicted	880	CM predicted	0	CM predicted	499.5
PA predicted	0	PA predicted	846.8	PA predicted	0
TOT.	880	TOT.	85	TOT.	499.5
	Fatty Acids		Fatty Acids		Fatty Acids
Cf Conc.fac.	11.36	Cf Conc.fac.	6.8	Cf Conc.fac.	9
Cf FEED	150	Cf FEED	120	Cf FEED	170
CM predicted	37.84	CM predicted	59.86	CM predicted	31.08
PA predicted	5.289	PA predicted	2.9	PA predicted	6.8453

predicted					
TOT.	43.129	TOT.	58.37	TOT.	37.9253
	Phosphate		Phosphate		Phosphate
Cf Conc.fac.	11.36	Cf Conc.fac.	6.8	Cf Conc.fac.	9
Cf FEED	20	Cf FEED	17	Cf FEED	15
CM predicted	13.2	CM predicted	14.6	CM predicted	8.436
PA predicted	0.0027	PA predicted	0.0085	PA predicted	0.005
TOT.	13.2027	TOT.	14.608	TOT.	8.441

	P-TOT		P-TOT		P-TOT
Cf Conc.fac.	11.36	Cf Conc.fac.	6.8	Cf Conc.fac.	9
Cf FEED	25	Cf FEED	23	Cf FEED	15
CM predicted	17.6	CM predicted	19	CM predicted	9.879
PA predicted	0.031	PA predicted	0.037	PA predicted	0.029
TOT.	53.991	TOT.	19.03	TOT.	9.908
	Ca		Ca		Ca
Cf Conc.fac.	11.36	Cf Conc.fac.	6.8	Cf Conc.fac.	9
Cf FEED	42	Cf FEED	39	Cf FEED	35
CM predicted	30.8	CM predicted	35.04	CM predicted	26.64
PA predicted	0.2644	PA predicted	0.077	PA predicted	0.124
TOT.	31.0644	TOT.	35.18	TOT.	26.764
	Cl		Cl		Cl
Cf Conc.fac.	11.36	Cf Conc.fac.	6.8	Cf Conc.fac.	9
Cf FEED	120	Cf FEED	110	Cf FEED	97
CM predicted	105.6	CM predicted	109.5	CM predicted	199.8
PA predicted	12.768	PA predicted	3.67	PA predicted	13.335
TOT.	118.368	TOT.	113.2	TOT.	213.135
	N tot		N tot		N tot
Cf Conc.fac.	11.36	Cf Conc.fac.	6.8	Cf Conc.fac.	9
Cf FEED	190	Cf FEED	140	Cf FEED	120
CM predicted	132	CM predicted	146	CM predicted	94.35
PA predicted	29.184	PA predicted	11.1	PA predicted	8.3566

predicted					
TOT.	161.184	TOT.	157.6	TOT.	102.7066
	Mg		Mg		Mg
Cf Conc.fac.	11.36	Cf Conc.fac.	6.8	Cf Conc.fac.	9
Cf FEED	8	Cf FEED	7,2	Cf FEED	5.9
CM predicted	6.688	CM predicted	7	CM predicted	4.995
PA predicted	0	PA predicted	0	PA predicted	0
TOT.	6.688	TOT.	7	TOT.	4.995
	SO4 2-		SO4 2-		SO4 2-
Cf Conc.fac.	11.36	Cf Conc.fac.	6.8	Cf Conc.fac.	9
Cf FEED	33	Cf FEED	36	Cf FEED	8.1
CM predicted	44.88	CM predicted	59.86	CM predicted	29.97
PA predicted	0	PA predicted	0	PA predicted	0
TOT.	44.88	TOT.	59.86	TOT.	29.97
	TOC		TOC		TOC
Cf Conc.fac.	11.36	Cf Conc.fac.	6.8	Cf Conc.fac.	9
Cf FEED	430	Cf FEED	260	Cf FEED	420
CM predicted	264	CM predicted	189.8	CM predicted	111
PA predicted	5.2896	PA predicted	na	PA predicted	5.24
TOT.	269.2896	TOT.	189.8	TOT.	116.24
	Torr Subs.		Torr Subs.		Torr Subs.
Cf Conc.fac.	11.36	Cf Conc.fac.	6.8	Cf Conc.fac.	9
Cf FEED	1000	Cf FEED	1100	Cf FEED	470
CM predicted	862.4	CM predicted	934.4	CM predicted	111
PA predicted	0	PA predicted	0	PA predicted	5.245
TOT.	862.4	TOT.	934.4	TOT.	116.245
			<b>Sus. Solids.</b>		
		Cf Conc.fac.	6.8		
		Cf FEED	570		
		CM predicted	408.8		
		PA predicted	na		
		TOT.	408.8		



Mass balance calculations

*For Batch 4*

The calculations are based on sodium na as it neither precipitated in feed tank as salt nor it is lost from permeate tank as gas.

Sodium Na

Concentration value of feed cf: 79 mg/l

Concentration value of concentrate at maximum volume reduction cm: 830 mg/l

Average permeate concentration pa: 6.7 mg/l

Taking mass balance:

Mass of a component in feed = Mass in concentrate + Mass in permeate

$$79 = f \times 830 + (1-f) \times 6.7$$

$$= 830f - 6.7f + 6.7$$

$$72.3 = 823.3 f$$

$$F = 0.088$$

$$1-f = 0.912$$

$$\text{Concentration factor } C_f = 1/0.088 = 11.36$$

$$\text{CM predicted} = 830 \times 0.088 = 73.04$$

$$\text{PA predicted} = 6.7 \times 0.912 = 6.11$$

Potassium K+

Concentration value of feed Cf: 67 mg/l

Concentration value of concentrate at maximum volume reduction cm: 690 mg/l

Average permeate concentration pa: 5.2 mg/l

$$\text{CM Predicted} = 0.088 \times 690 = 60.72$$

$$\text{PA Predicted} = 0.912 \times 5.2 = 4.742$$

$$\text{CM Predicted} + \text{PA Predicted} = 60.72 + 4.742 = 65.4$$

In percentage values:

$$\text{In concentrate} = (100 \times 60.72) / 67.0 = 90.62\%$$

$$\text{In permeate} = (100 \times 4.742) / 67.0 = 7.07\%$$

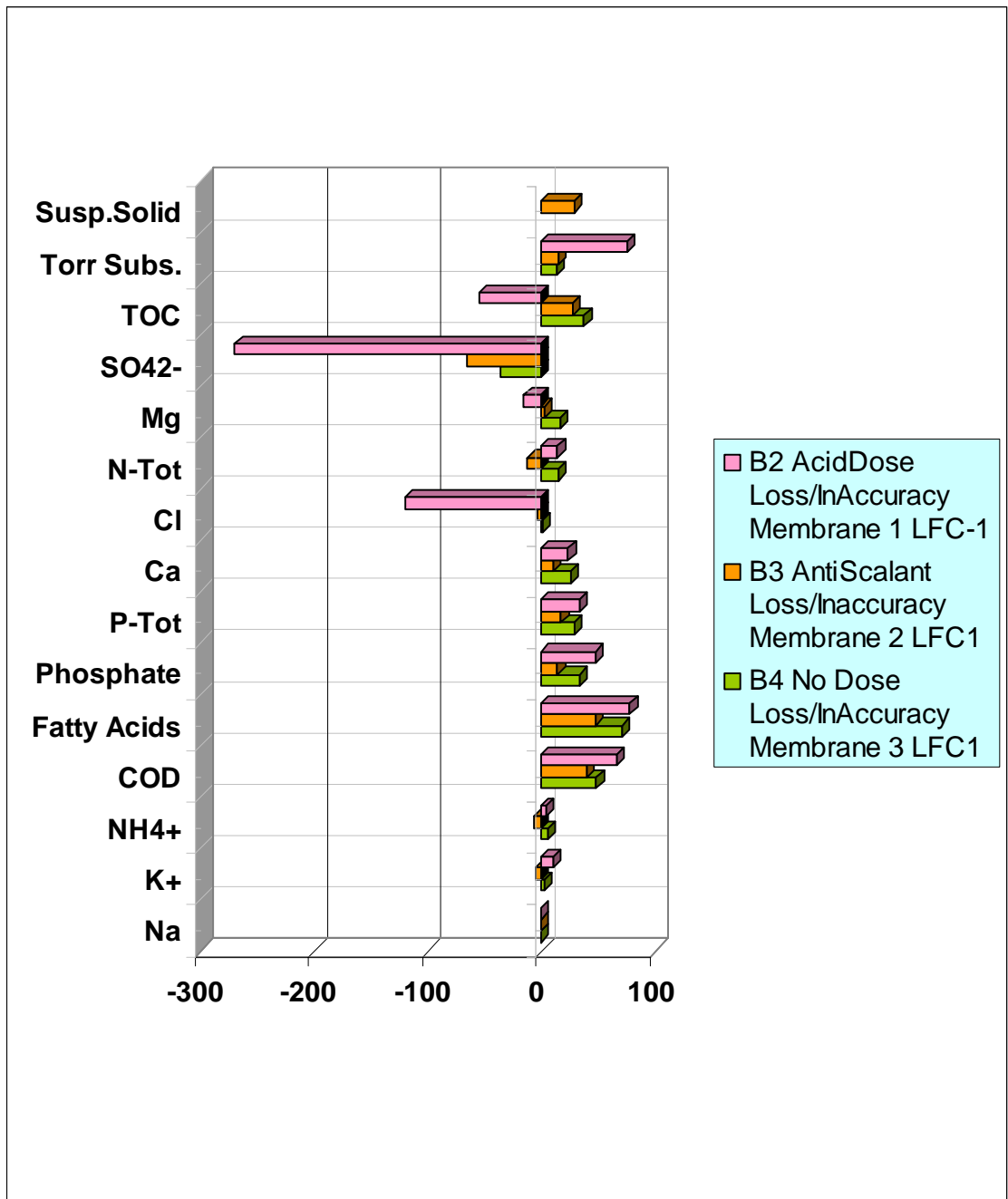
$$\text{Loss/inaccuracy} = 100 - 90.62 - 7.07 = 2.31\%$$

Batch 4 No Dose			
Component	In Concentrate %	In Permeate %	Loss/Inaccuracy
Na <sup>+</sup>	92.4	7.7	0 <sup>α</sup>
NH <sub>4</sub> <sup>+</sup>	76.3	18.2	5.5
N-tot.	69.5	15.3	15.2
K <sup>+</sup>	90.62	7.07	2.31
PO <sub>4</sub> <sup>3-</sup>	66	0.01	33.99
P-Tot	70.4	0.12 <sup>ta</sup>	29.48
COD	51.76	0	48.23
Fatty Acids	25.22	3.52	71.26
Ca <sup>2+</sup>	73.33	0.62	26.05
Cl <sup>-</sup>	88	10.63	1.37
Mg <sup>2+</sup>	83.5	0	16.5
SO <sub>4</sub> <sup>2-</sup>	136	0	-36
TOC	61.39	1.23	37.38
Torr Subs.	86.24	0	13.76
Susp.Solids			

Batch 3 Antiscalant dose			
Component	In Concentrate %	In Permeate %	Loss/Inaccuracy
Na <sup>+</sup>	96,2	3,60 <sup>α</sup>	
NH <sub>4</sub> <sup>+</sup>	97,7	8,54	-6,24
N-tot.	104,3	7,93	-12,23
K <sup>+</sup>	102,65	2,8	-5,45
PO <sub>4</sub> <sup>3-</sup>	85,9	0,05	14,05
P-Tot	82,6	0,16	17,24
COD	60,48	0	39,52
Fatty Acids	49,88	2,41	47,71
Ca <sup>2+</sup>	89,84	0,19	9,97
Cl <sup>-</sup>	99,54	3,34	-2,88
Mg <sup>2+</sup>	97,2	0	2,8
SO <sub>4</sub> <sup>2-</sup>	166,27	0	-66,27
TOC	73	0	27
Torr Subs.	84,94	0	15
Susp.Solids	71,72	NA	28,28

Batch 2 Acid dose			
Component	In Concentrate %	In Permeate %	Loss/Inaccuracy
Na <sup>+</sup>	93,06	6,91	0)α
NH <sub>4</sub> <sup>+</sup>	87,17	7,95	4,18
N-tot.	78,95	6,95	14,1
K <sup>+</sup>	85,86	4,52	9,62
PO <sub>4</sub> <sup>3-</sup>	52,26	0,03	47,71
P-Tot	65,86	0,2	33,94
COD	33,3	0	66,7
Fatty Acids	18,28	4,02	77,7
Ca <sup>2+</sup>	76,11	0,34	23,55
Cl <sup>-</sup>	205,97	13,74	-119,71
Mg <sup>2+</sup>	84,57	0	-15,42
SO <sub>4</sub> <sup>2-</sup>	370	0	-270
TOC	44,92	0	-55,07
Torr Subs.	23,61	1,11	75,28
Susp.Solids			

Component	% Loss/Inaccuracy in batch 4, B4 No dose Membrane 3, LFC1	% Loss/Inaccuracy in batch 3, B3, Antiscalant dose Membrane 2, LFC1	% Loss/Inaccuracy in batch 2 Acid dose Membrane 1, LFC1
Na <sup>+</sup>	0)α	0)α	0)α
NH <sub>4</sub> <sup>+</sup>	5,5	-6,24	4,18
N-Tot	15,18	-12,23	14,1
K <sup>+</sup>	2,31	-5,45	9,62
PO <sub>4</sub> <sup>3-</sup>	33,99	14,05	47,71
P-Tot	29,48	17,24	33,94
COD	48,23	39,52	66,7
Fatty Acids	71,26	47,71	77,7
Ca <sup>2+</sup>	26,05	9,97	23,55
Cl <sup>-</sup>	1,37	-2,88	-119,71
Mg <sup>2+</sup>	16,5	2,8	-15,42
SO <sub>4</sub> <sup>2-</sup>	-36	-66,27	-270
TOC	37,38	27	-55,07
Torr Subs.	13,76	15	75,28
Susp.Solids		28,28	



**Graph 36.** The graph shows the percentage disappearance or production in concentration values of chemical substances at the end of each batch test.

- **It is observed from the above graph that maximum loss/inaccuracy of components is observed in the first lfc1 membrane, used in the first test with acid dose b2. This also denotes that it leaked the most as compared to other membranes.**
- **The above graph presents losses/gains in various chemical substances measured from black water samples taken from VSEP batch testing under different dosing conditions**

### Appendix O: PLC data and their variables for four VSEP testing

STATUS	DATE	TIME	TOT. TIME	DL	pH	S (mS/cm) IN FEED	F-press Initial	F-press Final	FT (DEG C)	P- Flow Initial ml/min	P-Flow Final ml/min	REMARKS
START OF VSEP BATCH 2 ACID DOSE (LFC1 MEMBRANE USED AFTER VSEP BATCH TEST 1), VOL OF FEED= 55 LT., AMT.ACID= 280 ML	02.05.05	1754 PM	0	118	6	2.11	20	20	18	26	26	NO FOULING ON LFC1 MEMBRANE AFTER BATCH 1 NO PERMEATE FLUX DECREASE AT THE END OF THE TEST. MEMBRANE IS USED FOR VSEP BATCH TEST 2.
	03.05.05	1120 AM	17' 25"	127	5.65	3.2	20	20	25	23	23	
	03.05.05	1150 AM	17' 55"	132	5.6	3.3	20	20	27	19	19	
	03.05.05	1220 PM	18' 25"	135	5.62	3.3	20	20	29	19	19	
DECREASE IN PF	03.05.05	1545 PM	21' 50"	218	5.62	3.5	20	20	31.2	12	12	SYSTEM IS CLOSED DOWN AND A NEW LFC-1 MEMBRANE IS INSTALLED
START OF VSEP BATCH 2 ACID DOSE USING NEW (2ND) LFC-1 MEMBRANE	03.05.05	1600 PM	21' 50"	219	5.62	3.5	10	10	29	19	19	THE PHOTOGRAPH OF 1ST LFC1 MEMBRANE IS TAKEN AND SOME INORGANIC FOULING HAS BEEN OBSERVED
DECREASE IN PF	03.05.05	1606 PM	21' 56"	220	5.63	3.55	10	12	29	12	19	PERMEATE FLOW DROPPED
	04.05.05	1411 PM	44' 59"		5.63	6.3	12	12	26	19	19	
DECREASE IN PF	04.05.05	1528 PM	45' 16"	445	6.83	6.37	12	14	26	12	19-20	FEED PRESSURE IS INCREASED BY 2 BARS
	04.05.05	1556 PM	45' 44"	449	6.98	6.69	14	14	26	19	19	
	04.05.05	1632 PM	46' 21"	455	6.99	7.04	14	14	26	19	19	
DECREASE IN PF	04.05.05	1745 PM	47' 34"	465	7	7.5	14	15	26.2	12	16-19	PRESSURE IS INCREASED
	04.05.05	1758 PM	47' 47"	470	7.11	8.17	15	15	26	12	12	
	04.05.05	1816 PM	48' 5"	413	7.08	8.57	15	15	26	12	12	
	04.05.05	1854 PM	48' 43"	479	7.3	8.93	15	15	26	10	10	
DECREASE IN PF	04.05.05	1923 PM	49' 13"	484	7.3	9.25	15	17	26	10	10	PRESSURE IS INCREASED
	04.05.05	1943 PM	49' 33"	487	7.3	9.48	17	17	26	6	6	

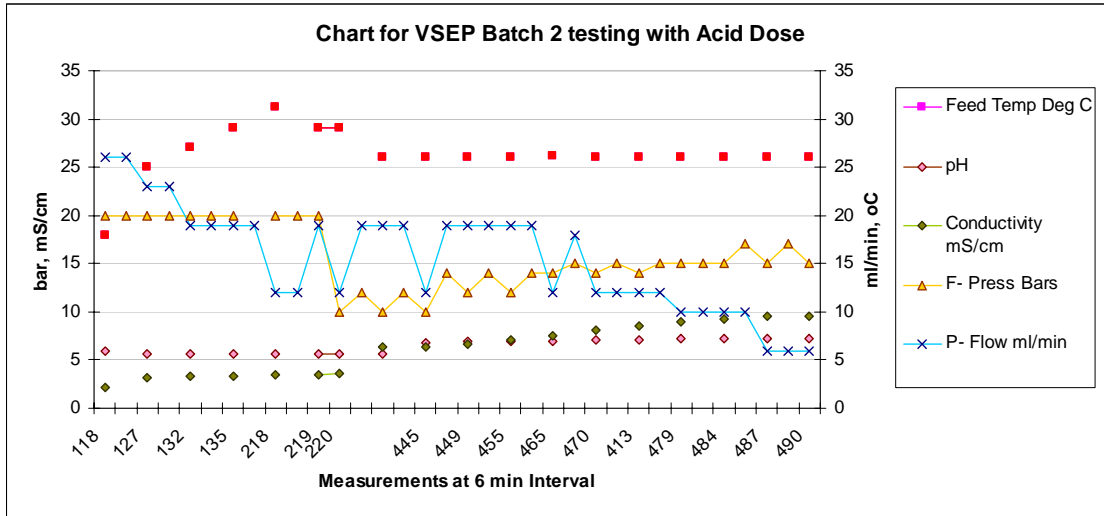
DECREASE IN PF. END OF VSEP BATCH TEST 2	04.05.05	1950 PM	49' 40"	490	7.3	9.48	17	18	26	6	6	PERMEATE FLOW DROPPED PROBABLY DUE TO FOULING. EXPERIMENT IS ENDED. CONCENTRATE C AND CF ARE TAKEN. THERE IS APPROX. 2- 2.5 LITERS LEFT IN FEED TANK. PHOTOGRAPH OF 2ND LFC1 MEMBRANE IS TAKEN
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STATUS	DATE	TIME	TOT. TIME	DL	pH	S (mS/cm) IN FEED	F-press Initial	F-press Final	FT (DEG C)	P- Flow Initial ml/min	P-Flow Final ml/min	REMARKS
START OF VSEP BATCH 3 <i>ANTI-SCALANT</i> NEW LFC1 MEMBRANE, VOL OF FEED= 55 LT.	09.05.05	1930 PM	0'	497	7.2	5.22	12	12	17	19	19	NEW LFC1 MEMBRANE IS INSTALLED FOR BATCH 3 TESTING
80 % VOLUME REDUCTION.	11.05.05			889	7.2	5.22	12	12	18	19	19	AT THIS POINT WE TAKE 1 LIT OF C80, C80f AND 1 L PERMEATE
	11.05.05	1102 AM	39' 30"	898	7.2	5.22	12	12	18	19	19	
DECREASE IN PF	11.05.05	1123 AM	38' 53"	901	7.27	5.32	12	13	25	19	19	PERMEATE FLOW DROPS TO 9-16ML, SO PR IS INCREASED
	11.05.05	1304 PM	40' 34"	918	7.44	6.17	13	13	25	19	19	
EMERGENCY STOP				921	7.48	6.2	12	13	26	19	19	
90% VOL. REDN. DECREASE IN PF	11.05.05	1459 PM	42' 29"	932	7.53	6.29	13	14	27	14	19	AT APPROX. 90% VOLUME REDUCTION, FEED PRESSURE IS INCREASED TO 14 BARS
DECREASE IN PF				939	7.6	6.4	14	15	28	14	19	PERMEATE FLOW DROPS TO 9-16ML, SO PR IS INCREASED TO 15 BARS
	11.05.05	1610 PM	43' 40"	944	7.7	6.58	15	15	29	19	19	
DECREASE IN PF	11.05.05	1640 PM	44' 10"	949	7.8	6.6	15	16	29.6	14	21	PERMEATE FLOW DROPS TO 9-16ML, SO PR IS INCREASED TO 16 BARS
	11.05.05	1642 PM	44' 12"	950	7.9	6.77	16	16	29.6	19	19	
DECREASE IN PF	11.05.05			952	8	8.2	16	17	29.6	14	16	PERMEATE FLOW AGAIN DROPS TO 9-16ML BUT DOESN'T RECOVER ON INCREASING PRESSURE. APPROX. 2 LIT OF CONCENTRATE IS LEFT IN FEED TANK
	11.05.05	1732 PM	45' 02"	958	8.15	8.25	17	17	30	16	16	
	11.05.05	1748 PM	45' 18"	961	8.34	8.36	17	17	30	16	16	
DECREASE IN PF	11.05.05			972	8.35	8.44	17	18	31	14	16	PERMEATE FLOW DOESN'T RECOVER ON INCREASING PRESSURE,

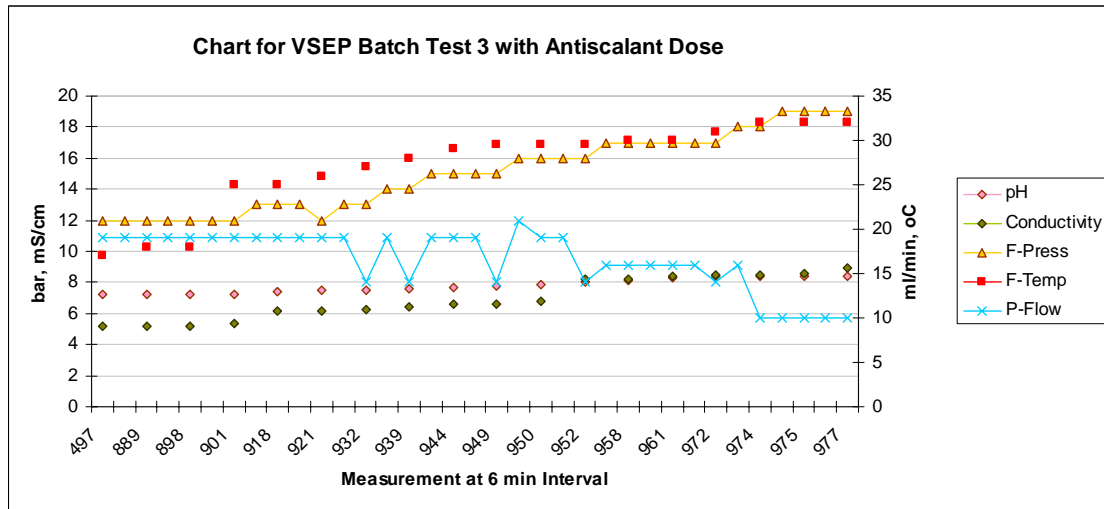


												APPROX. 1.5 L LEFT IN TANK.
DECREASE IN PF	11.05.05	1904 PM	47' 34"	974	8.36	8.5	18	19	32	10	10	PERMEATE FLOW DOESNT RECOVER ON INCREASE IN PRESSURE, CONSTANT AT 9-12 ML
	11.05.05	1911 PM	47' 41"	975	8.38	8.6	19	19	32	10	10	
MAXIMAL VOL. REDN. DECREASE IN PF. END OF VSEP BATCH 3 (ANTISCALANT)	11.05.05	1920 PM	47' 50"	977	8.41	8.9	19	20	32	10	10	PERMEATE FLOW DOESNT RECOVER ON PRESSURE INCREASE, SAMPLES CM, CMF ARE TAKEN. MEMBRANE IS TAKEN OUT, EASILY WASHED OFF FOULING BY DETERGENT, ORGANIC FOULING IS MOST PROBABLE.

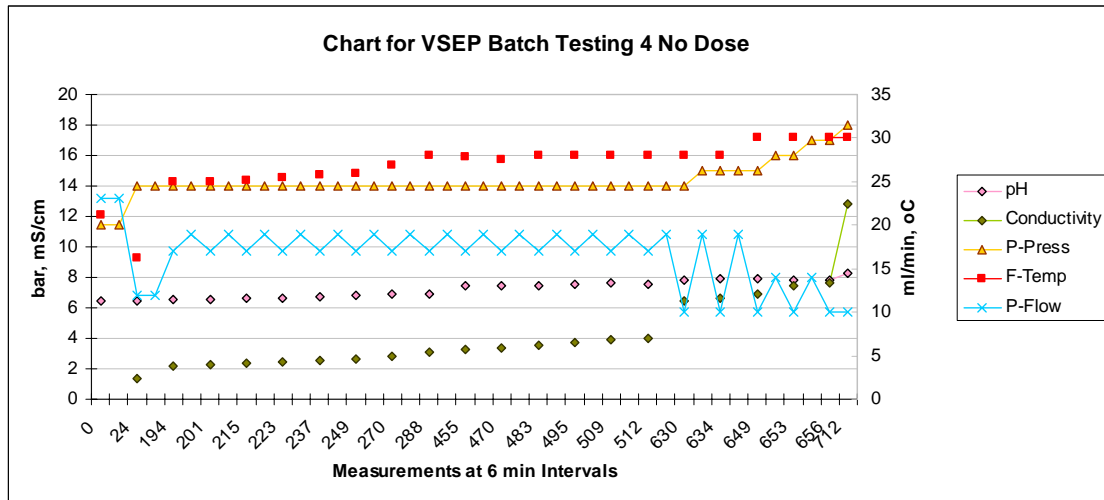
STATUS	DATE	TIME	TOT. TIME	DL	pH	S (mS/cm) IN FEED	F-press Initial	F-press Final	FT (DEG C)	P- Flow Initial ml/min	P-Flow Final ml/min	REMARKS
BEFORE VSEP BATCH 4 , FRESH WATER TEST	16.05.05			0	6.45		11.5	11.5	21.2	23	23	SAME MEMBRANE WHEN INSTALLED GIVES SAME PERMEATE FLOW AS PREVIOUSLY UNDER SAME PRESSURE TEMPERATURE CONDITIONS, PROVES THAT NO INORGANIC FOULING ON MEMBRANE.
VSEP BATCH 4, NO DOSE, VOL FEED TANK= 55 LT.	17.05.05	1800 PM	0'	24	6.45	1.4	14	14	16.2	12	12	
	18.05.05	1204 PM	17' 04"	194	6.56	2.2	14	14	24.9	17	19	
	18.05.05	1304 PM	19' 04"	201	6.58	2.31	14	14	25	17	19	
	18.05.05	1404 PM	20' 04"	215	6.63	2.38	14	14	25.2	17	19	
	18.05.05	1504 PM	21' 04"	223	6.66	2.47	14	14	25.4	17	19	
	18.05.05	1634 PM	22' 34"	237	6.73	2.56	14	14	25.7	17	19	
	18.05.05	1748 PM	23' 48"	249	6.82	2.68	14	14	25.9	17	19	
	18.05.05	2004 PM	26' 04"	270	6.9	2.86	14	14	26.86	17	19	
	18.05.05	2143 PM	27' 43"	288	6.9	3.08	14	14	28	17	19	
	19.05.05	1427 PM	44' 32"	455	7.41	3.24	14	14	27.79	17	19	
	19.05.05	1553 PM	45' 58"	470	7.43	3.36	14	14	27.6	17	19	
	19.05.05	1714 PM	47' 19"	483	7.49	3.55	14	14	28	17	19	
	19.05.05	1830 PM	48' 35"	495	7.54	3.74	14	14	28	17	19	
	19.05.05	1948 PM	49' 53"	509	7.63	3.9	14	14	28	17	19	
	19.05.05	2008 PM	50' 13"	512	7.57	3.96	14	14	28	17	19	
	20.05.05	800 AM	73' 13"	630	7.84	6.5	14	15	28	10	19	
	20.05.05	909 AM	74' 22'	634	7.9	6.64	15	15	28	10	19	
	20.05.05	954 AM	75' 07"	649	7.9	6.94	15	16	30	10	14	
	20.05.05	1015 AM	75' 28"	653	7.84	7.41	16	17	30	10	14	
	20.05.05	1032 AM	75' 45"	656	7.83	7.61	17	18	30	10	14	
END OF EXPERIMENT VSEP B4 NO DOSE	20.05.05	1615 PM	70' 28"	712	8.23	12.86	18	19	30	10	10	NO INCREASE IN PERMEATE FLOW



**Graph 37. The above graph represents temperature, ph, conductivity, feed pressure and permeate flow profile for VSEP batch testing 2 with acid dose. The temperature of the feed and pH constantly rises and that can be the reason of loss of NH<sub>4</sub> and n-tot from the feed tank. The conductivity curve constantly rises, proves rising concentration in feed tank. When permeate flow decreases (see P-flow initial) the feed pressure is increased by one or two bars (F-pressure final). This makes the permeate flow back to normal value ( P-flow final) .decrease in P-flow can be due to osmotic pressure developing across the across the membrane at higher concentration or due to membrane fouling. F-pressure is increased to make it always higher than osmotic pressure (RO LFC1 membrane process). During the last part of experiment, the permeate flow dropped drastically even on increasing the feed pressure. This proves that drop in P-flow is due to fouling on membrane and not due to osmotic pressure across the membrane. The P-flow would drop smoothly if it is due to osmotic pressure.**

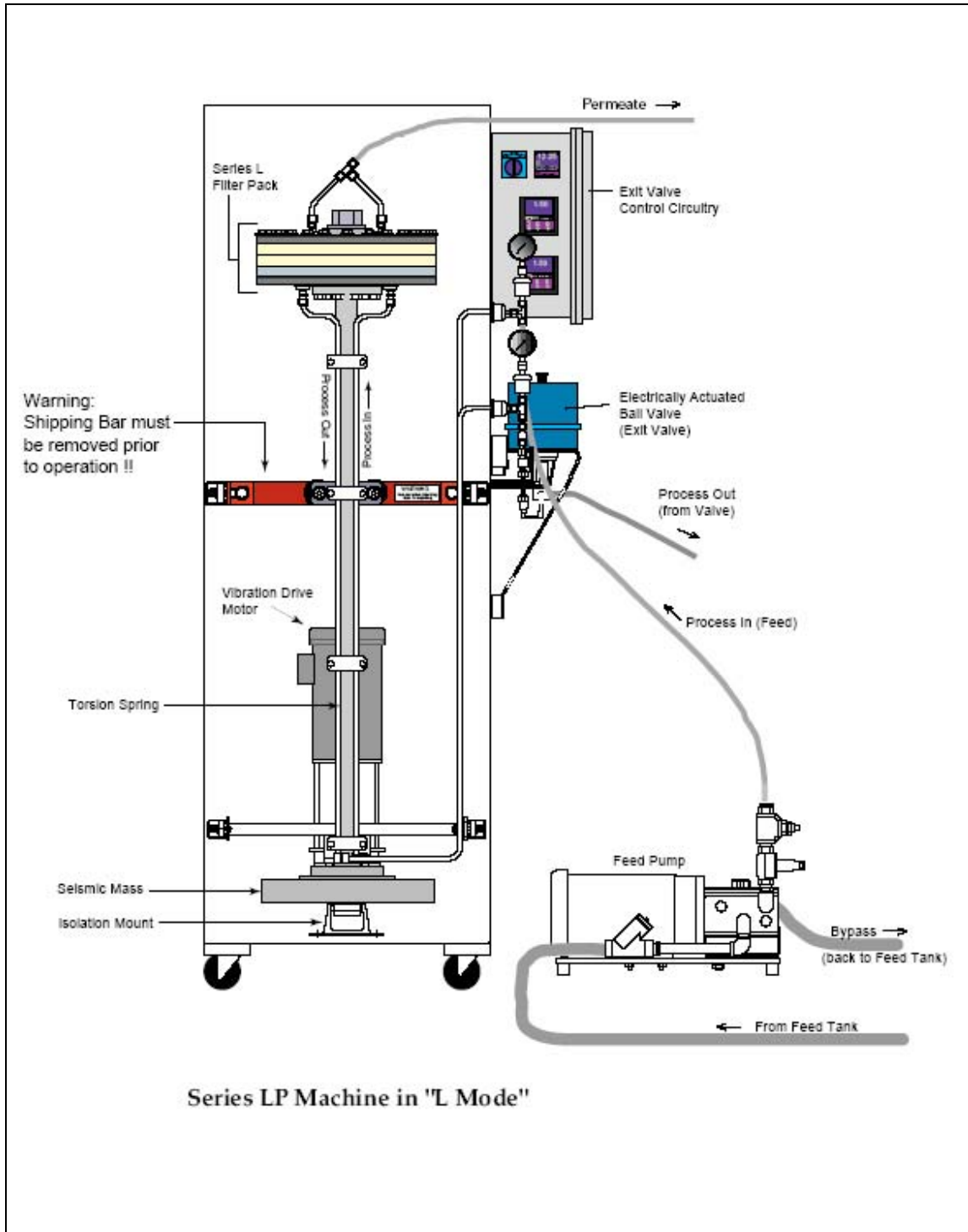


**Graph 38.** The above graph represents temperature, ph, conductivity, feed pressure and permeate flow profile for VSEP batch testing 3 with anti-scalant dose. The temperature of the feed and ph constantly rises and that can be the reason of loss of NH4 and N-tot from the feed tank. The conductivity curve constantly rises, proves rising concentration in feed tank. When permeate flow decreases (see P-flow initial) the feed pressure is increased by one or two bars (F-press final). This makes the permeate flow back to normal value ( P-flow final ).decrease in p-flow can be due to osmotic pressure developing across the across the membrane at higher concentration or due to membrane fouling. F-pressure is increased to make it always higher than osmotic pressure (RO LFC1 membrane process). During the last part of experiment, the permeate flow dropped drastically even on increasing the feed pressure. This proves that drop in P-flow is due to fouling on membrane and not due to osmotic pressure across the membrane. The P-flow would drop smoothly if it is due to osmotic pressure. The graph for antiscalant is almost same as for acid dose test (figure 1).



**Graph 39.** The above graph represents temperature, ph, conductivity, feed pressure and permeate flow profile for VSEP batch testing 4 with no dose. The temperature of the feed and ph constantly rises and that can be the reason of loss of NH<sub>4</sub> and N-tot from the feed tank. The conductivity curve constantly rises, proves rising concentration in feed tank. When permeate flow decreases (see P-flow initial) the feed pressure is increased by one or two bars (F-press final). This makes the permeate flow back to normal value ( P-flow final) .decrease in p-flow can be due to osmotic pressure developing across the across the membrane at higher concentration or due to membrane fouling. F-pressure is increased to make it always higher than osmotic pressure (ro lfc1 membrane process). During the last part of experiment, the permeate flow dropped drastically even on increasing the feed pressure. This proves that drop in P-flow is due to fouling on membrane and not due to osmotic pressure across the membrane. The P-flow would drop smoothly if it is due to osmotic pressure.

## Appendix P: The VSEP membrane unit in "P" mode



**Appendix Q: The VSEP membrane unit in “p” mode**

