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ABSTRACT

This study investigated the performance of Salsnes Filter (SF) as a primary treatment prior to hollow fiber membrane bioreactor (HFMB) for nitrogen removal. The objective was to determine whether influent particle size removed during primary treatment had any detrimental effect on downstream biological processes, especially nitrogen removal. The pilot scale testing was conducted at Nordre Follo Wastewater Treatment Plant, Oslo region, Norway.

This pilot system comprised of two anoxic tanks and one aerobic tank with hollow fiber membrane. Hollow fiber module type ZW-10 was provided by GE Water Technologies. One system was fed with influent wastewater that has been filtered with SF 2 mm and represents the degrittied wastewater (treatment Train C), while the other system was filtered specifically with SF 33 μm (treatment Train D). Two boundary conditions were used, the first investigated SF and MBR as the whole system, and the last reviewed the effect of different particle size on the performance of MBR system.

For both boundary conditions, it was found that both treatment trains have similar capability in reducing Total Suspended Solids (TSS), Total Chemical Oxygen Demand (TCOD), Total Biochemical Oxygen Demand (TBOD₅), and Total Phosphorus (TP) with average removal percentage of 99 %, 92 %, 99 %, and 79 % respectively, which met the discharge requirement criteria. However, Total Nitrogen (TN) effluent results showed that treatment Train C has better average removal efficiency of 73 % compared to treatment Train D's 68 % due to higher TCOD/TN ratio after SF treatment. Low TCOD/TN ratio in treatment Train D hampered the denitrification process, as confirmed by lower denitrification rate and higher NO₃-N concentration in the permeate than its counterpart. Nitrification and denitrification were proven to be the main factor of biological nitrogen removal compared to cell assimilation process.

The HFMB operated smoothly during the experiment, with no excessive fouling detected. Membrane in treatment Train C experienced more frequent rapid

transmembrane pressure (TMP) peaks due to abundance of organic and organic matters, making it more vulnerable of membrane fouling for long term operation.

Overall, both systems produced high quality effluent and free of TSS, even though treatment Train C was susceptible of membrane fouling and treatment Train D had slight problem in its nitrogen removal process. Further economic observation should be implemented to decide which system is more cost effective between the requirement of more frequent membrane maintenance cleaning for treatment Train C or external carbon source addition for treatment Train D.

Keywords : biological nitrogen removal, denitrification, membrane bioreactor, membrane fouling, nitrification, Salsnes Filter

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ACRONYMS

AUR	Ammonium Uptake Rate
BOD	Biochemical Oxygen Demand
CAS	Conventional Activated Sludge
CFU	Colony Forming Unit
COD	Chemical Oxygen Demand
C/N	Carbon/Nitrogen ratio
DM	Dry Matter
DO	Dissolved Oxygen
EBPR	Enhanced Biological Phosphorus Removal
EPS	Extracellular Polymeric Substances
F/M	Food/Microorganism ratio
HFMB	Hollow Fiber Membrane Bioreactor
HRT	Hydraulic Retention Time
MBR	Membrane Bioreactor
MF	Microfiltration
MLE	Modified Ludzak Ettinger
MLSS	Mixed Liquor Suspended Solids
MLVSS	Mixed Liquor Volatile Suspended Solids
N	Nitrogen
NH ₃ -N	Ammonia nitrogen
NH ₄ -N	Ammonium nitrogen
NO ₂ -N	Nitrite nitrogen
NO ₃ -N	Nitrate nitrogen
NTU	Nephelometric Turbidity Unit
NUR	Nitrogen Uptake Rate
OUR	Oxygen Uptake Rate

P	Phosphorus
PAO	Phosphorus Accumulating Organisms
pe	People Equivalent
PFU	Plaque Forming Unit
PO ₄ -P	Phosphate
rbCOD	Readily Biodegradable Chemical Oxygen Demand
sBOD	Soluble Biochemical Oxygen Demand
sCOD	Soluble Chemical Oxygen Demand
SDNR	Specific Denitrification Rate
SF	Salsnes Filter
SMP	Soluble Microbial Product
SNDN	Simultaneous Nitrification Denitrification
SNR	Specific Nitrification Rate
SOUR	Specific Oxygen Uptake Rate
SRT	Sludge Retention Time
SS	Suspended Solid
sTN	Soluble Total Nitrogen
sTP	Soluble Total Phosphorus
SVI	Sludge Volume Index
TCOD	Total Chemical Oxygen Demand
TBOD	Total Biochemical Oxygen Demand
TMP	Transmembrane Pressure
TN	Total Nitrogen
TP	Total Phosphorus
TSS	Total Suspended Solid
UF	Ultrafiltration
VSS	Volatile Suspended Solid
wwtp	Wastewater Treatment Plant

CHAPTER I

INTRODUCTION

1.1 Background

For the first half of the 20th century, pollution in the urban waterways often resulted in frequent occurrences of low dissolved oxygen, fish kills, algal blooms and bacterial contamination. Population growth, changes in industrial processes, technological developments, changes in land use, business innovations, and many other factors had increased the amount and complexity of the wastewater produced, thus increasing the need of advanced wastewater treatment process.

In Norway, there are approximately 2500 registered municipal wastewater treatment plants (wwtp) managing effluent from municipal to industrial wastewater (PRTR, 2012). Chemical treatment plants account for 36% of Norway's wastewater treatment capacity, chemical and biological plants for 28%, mechanical plants for 23%, and other unspecified type of plants treat the remaining 13% of total wastewater (PRTR, 2008). Biological treatment, however, has emerged as the leading process for many treatment plants. The obvious economic advantage, both in terms of capital investment and operating costs, of biological treatment over other treatment processes like chemical oxidation or thermal oxidation has cemented its place in any integrated wwtp, especially in the places where organic and nutrient removal are necessary.

Activated sludge process is the most widely used biological treatment process (Tchobanoglous et al., 2004). It refers to a mass of microorganisms cultivated in the treatment process to break down organic matter into carbon dioxide, water, and other inorganic compounds. Membrane bioreactor (MBR) is a technology which is operated similar to conventional activated sludge (CAS) process, only with the addition of microfiltration (MF) or ultrafiltration (UF) to separate the effluent from activated sludge (Melin et al., 2006). This leads to two big advantages of MBR compared with CAS, where MBR system does not need clarifier as secondary treatment or sand filtration as tertiary treatment. In the MBR system, suspended microbes in activated sludge consume organic matter in wastewater (quantified as Biochemical Oxygen

Demand (BOD) and Chemical Oxygen Demand (COD)), thus also providing efficient removal of BOD, COD, and nutrients for the effluent.

First generation of MBR dates back in the late 1990s, which focused mainly on microcontaminant removal and disinfection (Kraemer et al., 2012). Since then, MBR is advancing rapidly in research and commercial application. Nowadays, fifth generation of MBRs are used as biological secondary treatment in America, Europe, Asia, and Australia (Kraemer et al., 2012).

While there are several notable options for secondary treatment, the market for primary treatment is still dominated by primary sedimentation tanks. However, in Norway, the use of fine mesh sieves as primary treatment is undergoing intensive development because it decreases space requirements and investment costs in comparison with primary sedimentation (Rusten and Lundar, 2006). Salsnes Filter (SF) is a Norwegian company that produces fine mesh rotating belt sieves used for mechanical separation of particulate materials from wastewater. SF is widely used in Europe, North and South America, and currently implemented as primary treatment in various municipal and industrial wastewater applications. Feasibility studies and pilot tests suggested that effluent quality from SF has complied with European Union removal standard, with average Total Suspended Solids (TSS) removal ranged from 72 to 90 %, and average BOD-removal ranged from 39 to 80 % (Nussbaum et al., 2006). SF also has a potential for assisting biological nitrogen removal in the downstream treatment, as influent particle size is reported to affect nitrification rate in MBR system (Zhang et al., 1997).

At the moment, there are a lot of researches focusing on treatment system that have smaller footprint, simpler process, lower energy consumption, and lower costs, due to high initial and maintenance cost of wwtp. Salsnes Filter AS is looking to expand the use of their primary treatment technology to membrane bioreactor plants. Both SF and MBR are proven to be state-of-the-art technologies that offer smaller footprint compared to the other wastewater treatment process that exist today (Melin et al., 2006; Rusten and Lundar, 2006).

1.2 Objectives

The overall objective of this study is to test the performance of Salsnes Filter as a primary treatment to a membrane bioreactor for nitrogen removal and determine

whether influent particle size removed during primary treatment had any detrimental effect on downstream biological processes, especially nitrogen removal. Two types of water are used as feed as primary treatment: SF with 2 mm mesh size, and the other is SF with 33 μm mesh size. Samplings and tests were conducted in Nordre Follo wwtp in Oslo region. The specific objectives for this study are to :

- Compare and evaluate water effluent data from two train sets to determine which train offers the most advantage result for the treatment process : one with SF with 2 mm mesh size, and another is SF with 33 μm mesh size
- Assess the performance of membrane bioreactor as biological treatment for nitrogen removal, including the microfiltration membrane performance and membrane fouling characteristics during the experiment
- Evaluate nitrification and denitrification process in the MBR system, including the estimation of the nitrification and denitrification rate
- Determine the system capability from two boundary condition : first case investigates SF and MBR as a whole system, while the second case reviews the effect of particle size distribution from different mesh size of SF to the MBR system

1.3 Brief Outline of Thesis

There are five chapters in this thesis. Chapter 1 focuses on background and objectives of this research. Chapter 2 explains the scientific review behind the object of research, from SF as primary treatment and membrane bioreactor as secondary treatment. Chapter 3 illustrates methodology of the research, as well as materials and equipments used for the experiment. A discussion of results is presented in Chapter 4. Chapter 5 describes the conclusions from the experiment, in addition to recommendations for future research. Appendices are included in the last part to show the detailed methodology of certain experiments.

CHAPTER II

LITERATURE REVIEW

A detailed scientific explanations about municipal wastewater, SF, MBR, and process treatment used in this study are explained in this section.

2.1 Municipal Wastewater Characteristics

Municipal wastewater mainly originates from domestic household sewage, with varying contribution from industrial sources. The wastewater contains organic and inorganic substances that are either suspended or dissolved in the water. The composition always varies depending on the inhabitants use of water, eating habits, quality of sewer system, weather conditions, etc. Table 2.1 shows typical municipal wastewater characteristics.

Table 2.1 : Typical composition of raw municipal wastewater with minor contributions of industrial wastewater [Adapted from Henze et al. (2008)]

Parameters	High (mg/L)	Medium (mg/L)	Low (mg/L)
COD total	1200	750	500
COD soluble	480	300	200
COD suspended	720	450	300
BOD ₅	560	350	230
Total nitrogen	100	60	30
Ammonia nitrogen	75	45	20
Total phosphorus	25	15	6
Orthophosphate	15	10	4
TSS	600	400	250
VSS	480	320	200

Large quantities of solids and organic compounds are unwanted in water bodies, as they can cause oxygen depletion and water turbidity. In addition, exceeded amount of nutrient in water bodies may cause a decline in water quality in the form of eutrophication or fish toxicity. Table 2.2 illustrates typical content of nutrients in raw municipal wastewater.

Table 2.2 : Typical content of nutrients in raw municipal wastewater with minor contributions of industrial wastewater [Adapted from Henze et al. (2008)]

Parameters	High (mg/L)	Medium (mg/L)	Low (mg/L)
Total nitrogen	100	60	30
Ammonia nitrogen	75	45	20
Nitrite and nitrate nitrogen	0.5	0.2	0.1
Organic nitrogen	25	10	15
Total Kjeldahl Nitrogen	100	60	30
Total phosphorus	25	15	6
Orthophosphate	15	10	4
Organic phosphorus	10	5	2

Suspended growth processes, i.e. activated sludge, and attached growth biological processes, i.e. trickling filters or rotating biological contactors, are some of the advanced technology adapted today in various wwtps for biological nitrogen removal. Activated sludge is the most widely used form of secondary biological treatment for nitrogen removal because of its efficiency and flexibility for modification (Tchobanoglous et al., 2004).

This research will study the combination of SF for primary treatment and membrane bioreactor for secondary treatment. The former serves as alternative to the typical primary sedimentation system, while the latter is a progressive modification of the conventional activated sludge system.

2.2 Salsnes Filter Fine Mesh Sieves

Salsnes Filter AS' history goes back to 1998, where its prototype systems were able to demonstrate that treated primary wastewater could meet European and Norwegian discharge requirement. Nowadays, SF has been installed in various municipal and industrial applications, including pulp and paper mills, food processing plants, breweries, fish hatcheries, and land-based fish farms. As shown in Table 2.3, there are four kinds of SF available in the market today for municipal wastewater treatment : SF 1000, SF 2000, SF 3000/4000, and SF 5000/6000.

Table 2.3 : Salsnes Filter capacity and dimensions

Parameter	SF1000	SF2000	SF 3000/4000	SF 5000/6000
Capacity	10-15 L/sec	20-40 L/sec	50-80 L/sec	100-140 L/sec
Length	1220 mm	1800 mm	2300 mm	2580 mm
Width	1050 mm	1350 mm	2150 mm	2720 mm
Height	1290 mm	950 mm	1300 mm	1630 mm
Weight	380 kg	475 kg	450/575 kg	580/725 kg

In municipal wastewater treatment plant, SFs are used as substitute for conventional primary water treatment such as primary sedimentation tanks, as SF only needs 1/10th of the land because sludge thickening and dewatering tools are already integrated into the system (SalsnesFilter, 2013a). In Riviera Wastewater Treatment Plant, Alabama (USA), SF is also proven to be able to remove 30-40% of BOD from its process, thus providing substantial savings in energy costs for aeration in the downstream process (SalsnesFilter, 2013b).

SF also demonstrates higher environmental benefits as it emits less carbon footprint than the conventional wwtp system. A study that compares the SF 6000 to a clarifier in a 2 MGD (315 m³/h) municipal wwtp in North America reveals that the SF system has a substantially lower environmental impact mainly because less concrete is required for installation, thus produced less CO₂ during construction and operation (SalsnesFilter, 2013a). The other substantial cost saving comes from reduction in sludge handling and sludge disposal cost, where the sludge volume is reduced to 20-25% than the usual operation with primary sedimentation.

2.2.1 Process Design of SF

SF operational design basically combines solid separation, sludge thickening and dewatering into one compact unit. Figure 2.1 shows the substantial operational tools in the treatment process. Wastewater enters from an inlet pipe, then filtered through a filter mesh. Solids above the filter mesh create a 'filter mat' of sludge, enhancing filtration performance. Particles build up on the mesh, creating progressively smaller holes that retain increasingly smaller particles. Filtered water flows out of the unit through an outlet pipe, while solids are transported on a rotating filter mesh. The filtered sludge goes into a sludge compartment by gravity and enabling

thickening process. First dewatering stage reduce the sludge thickness into 3– 8% dry matter (DM). The mesh is cleaned using compressed air blown to an air knife to remove any remaining sludge. A screw press further dewateres the sludge to 20–30% DM before it exits the unit. Hot water is regularly flushed for mesh cleaning and maintenance.

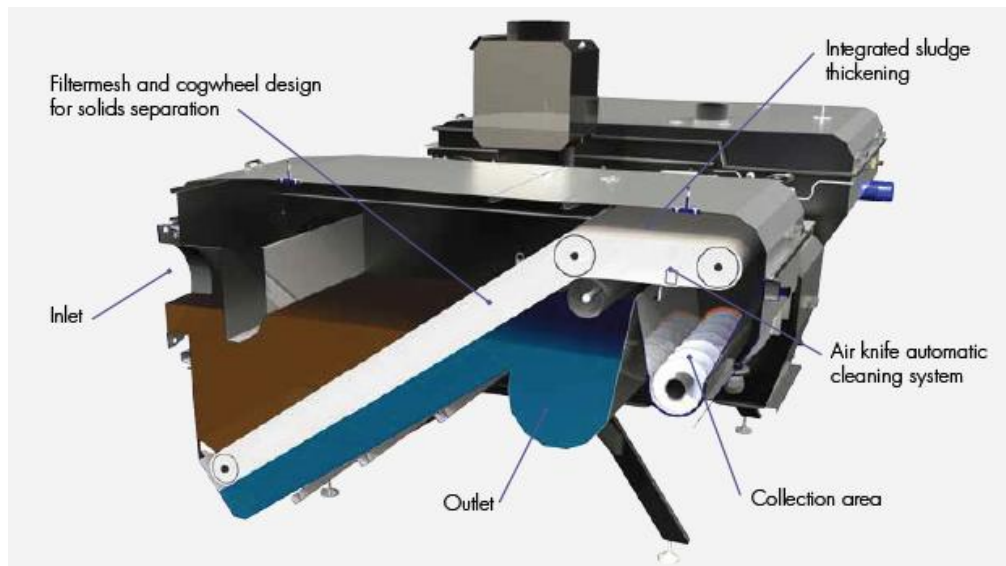


Figure 2.1 : Salsnes Filter [Adapted from SalsnesFilter (2013b)]

Several tests conclude that Salsnes filter's efficiency, if it is operated as single treatment without additional secondary treatment, depends on the development of filter mat, which is controlled by the sieve rate (Rusten and Lundar, 2006). The filter mat can affect removal efficiency because it rejects particulate matter and can act as an additional filtration barrier to the pollutant in the wastewater (Chu and Li, 2006). The filter mat typically forms in two stages: first, a pore wall deposition of sludge is formed followed by a simultaneous partial pore blocking and cake layer formation (Li et al., 2011). However, as the detention time goes higher, the filtration efficiency of filter mat will decrease because of the gradual accumulation of organic matters that can lead to irreversible fouling of filter mat. Hot water backwash can be implemented to remove the filter mat fouling.

2.3 Membrane Bioreactor

A membrane bioreactor is described as a combination of activated sludge process with a membrane separation process. The reactor is operated similar to a conventional activated sludge process but with an addition of low-pressure membrane

filtration, either microfiltration (MF) or ultrafiltration (UF), to separate effluent from activated sludge (Melin et al., 2006).

In 2011, the global MBR market was estimated at USD 838.2 million and is projected to grow at a average annual rate of 22.4 %, reaching a total market size of USD 3.44 billion in 2018 (Sartorius et al., 2013). Zenon (which is now part of GE Water Technologies) occupies the majority of the MBR market in North America, whereas Kubota and Mitsubishi-Rayon have a larger number of installations in other parts of the world due to Japan’s role as early adopter of MBR technology (Sartorius et al., 2013). Advantages and disadvantages of MBR are listed in Table 2.4 below.

Table 2.4 : Advantages and disadvantages of MBR [Adapted from Kraemer et al. (2012); Tchobanoglous et al. (2004); Gómez et al. (2012); Melin et al. (2006)]

Advantages	Disadvantages
Smaller footprint and smaller reactor volume as a consequence of higher MLSS concentration and loading rate	Higher operating cost (higher energy and chemical consumption)
Shorter reactor hydraulic retention times	Greater mechanical complexity and new technology for many owners and operators
Less sludge production	Membrane fouling
High quality effluent in terms of low turbidity, bacteria, TSS, BOD	Limitations imposed by pressure, temperature, and pH requirements to meet membrane tolerances
Lower sensitivity to contaminant peaks	Poor peak flow performance
High level of automation can be achieved	

MBRs are mostly utilized as secondary treatment, downstream to primary treatment, to remove dissolved and particulate carbonaceous BOD, stabilize the organic matters, and eliminate nutrients. Table 2.5 shows typical contaminant removal efficiencies and effluent quality achieved by MBR.

Table 2.5 : Submerged MBR removal efficiencies and effluent quality [Adapted from Melin et al. (2006)]

Parameters	Removal efficiency (%)
TSS, mg/L	>99
Turbidity, NTU	98.8–100
COD, mg/L	89–98
BOD, mg/L	>97
Ammonia nitrogen, mg/L	80 – 90

Parameters	Removal efficiency (%)
Total nitrogen, mg/L	36–80
Total phosphorus, mg/L	62–97
Total coliforms, CFU/100 mL	5–8 log
Faecal coliforms, CFU/100 mL	-
Bacteriophages, PFU/100 mL	>3.8 log

2.3.1 Process Design of MBR

Essential elements of the design of MBR process are divided into three parts : the design of pre-treatment, the biological process, and the membrane separation process. Pre-treatment is important to protect membrane integrity and prevent physical damage to the membrane fibers for full scale treatment plants (Stefanski et al., 2011). A pre-filtration with grid distance of maximum 3 mm is advised to ensure long-term membrane operation (Melin et al., 2006).

The second aspect, biological process, is determined by the quantity and quality of the Mixed Liquor Suspended Solids (MLSS). MLSS concentrations of 8000-10000 mg/L appear to be the most cost-effective. Even though MBR can operate at much higher MLSS, up to 15000-25000 mg/L, at higher concentrations it can cause operational problems like clogging of the membrane, decreased oxygen transfer efficiency and cake formation (Kraemer et al., 2012; Melin et al., 2006).

Finally, membrane separation process is one significant aspect that differs the MBR system from CAS. With a membrane functioning as selective barrier for activated sludge, the MBR can operate without the need for secondary clarification and tertiary steps like sand filtration. The membrane permit passage of certain components as permeate (i.e. water and the treated organic and inorganic matters of certain quantity and size) and retain certain other components of a mixture as retentate (i.e. the activated sludge).

The major membrane separation processes are microfiltration, ultrafiltration, nanofiltration, and reverse osmosis (Cheryan, 1998). Figure 2.2 depicts the primary separation process, their various range of particle cutting size, and the specific particle cutting size of of membrane and SF used in this study.

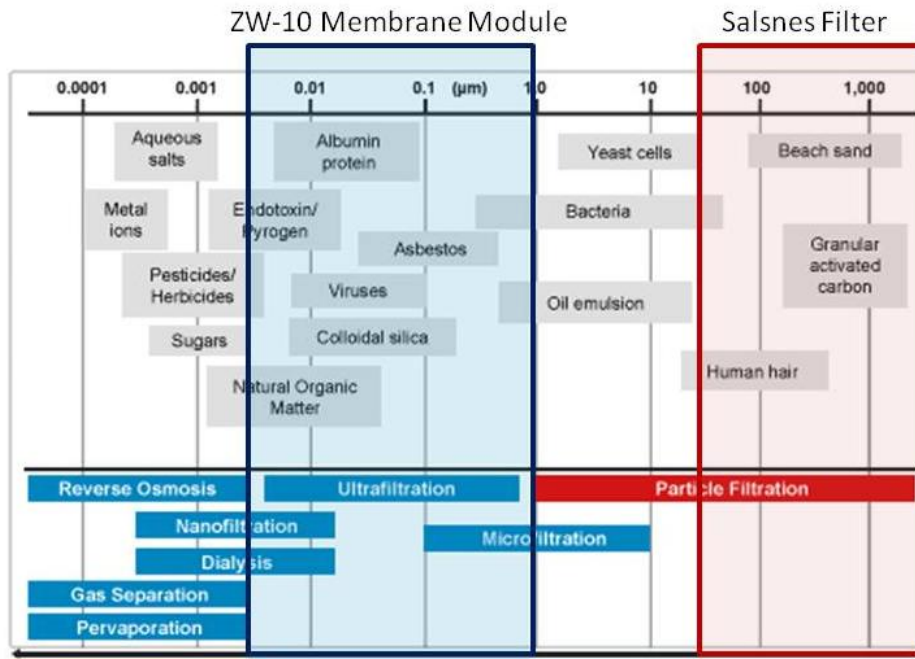


Figure 2.2 : Various range separation process and their particle cutting size [Figure adapted from Hyflux (2008)]

2.3.2 Membrane Configuration and Module

The two main membrane configurations are integrated MBR with a submerged membrane module and MBR with external circulation/sidestream membrane separation unit, as shown in Figure 2.3(a) and Figure 2.3(b), respectively. In a submerged MBR, the membrane is submerged into the aeration tank and separation occurs within the bioreactor. Compressed air is introduced to the membrane module in order to maintain MLSS within bioreactor, minimize solid deposition, and provide oxygen to maintain aerobic conditions. Submerged MBR is used in municipal wwtps worldwide, as it requires lower energy consumption and space requirement than external configuration (Melin et al., 2006).

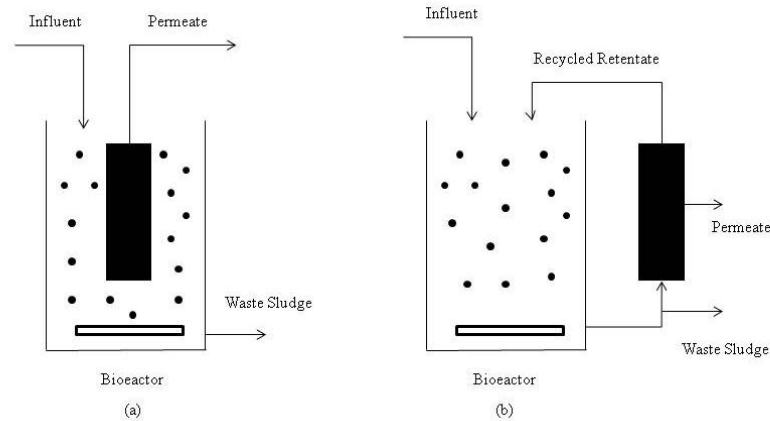


Figure 2.3 : (a) Integrated/submerged MBR, (b) External/sidestream MBR [Adapted from Melin et al. (2006)]

For the second configuration, the membrane is located outside the bioreactor. Feed enters the bioreactor where it undergoes biological treatment, then the water is pumped in a recirculation loop that contains a membrane units where the permeate is extracted and the retentate on the feeding side returns to the aeration tank. External MBR has smaller worldwide application than submerged MBR because it has higher costs in fabrication and more difficult maintenance than submerged MBR (Chen et al., 2010).

Furthermore, membrane module applied in the configurations can vary depends on the need of influent flow capacity and particle size. Tubular, hollow fiber, and spiral wound are three module types mostly used by industrial users of membrane technology, as seen in Table 2.6.

Table 2.6 : Advantages and disadvantages of various membrane modules [Adapted from Cheryan (1998)]

Membrane module	Advantages	Disadvantages
Tubular	<ol style="list-style-type: none"> 1) Tolerate to large particles from feed water 2) Can handle higher viscosity products 3) Easy to clean when heavily fouled 4) High internal volume 	<ol style="list-style-type: none"> 1) Lowest membrane area to volume ratio 2) Highest energy use 3) Large space requirement 4) Need expensive investment
Hollow fiber	<ol style="list-style-type: none"> 1) Highest surface area to volume ratio 2) Backflushing capability 	<ol style="list-style-type: none"> 1) Intolerant to large pressure changes 2) Low pressure ratings

Membrane module	Advantages	Disadvantages
	3) Self-supporting fibers 4) Low energy operating costs and capital costs	3) Susceptible to plugging, need pre-treatment for SS control
Spiral wound	1) High packing density 2) Easy replacement 3) Pressure tolerant 4) Lowest initial and operating costs and capital costs 5) Energy efficient	1) Easily clogged by particles, need pre-treatment for SS control 2) Difficult to clean when heavily plugged 3) Intolerant to large pressure differences

This study used a submerged MBR configuration with a hollow fiber module. Hollow fiber is chosen because of its backflushing capability (which prevents buildup of cake formation in membrane surface) and its geometry (which provides a greater filtration surface area). Self supporting nature of hollow fiber also provides a more practical operation, it does not need to be assembled with spacers or porous supports like any other type of modules (Cheryan, 1998). Figure 2.4 shows the detailed flow movement in the hollow fiber module.

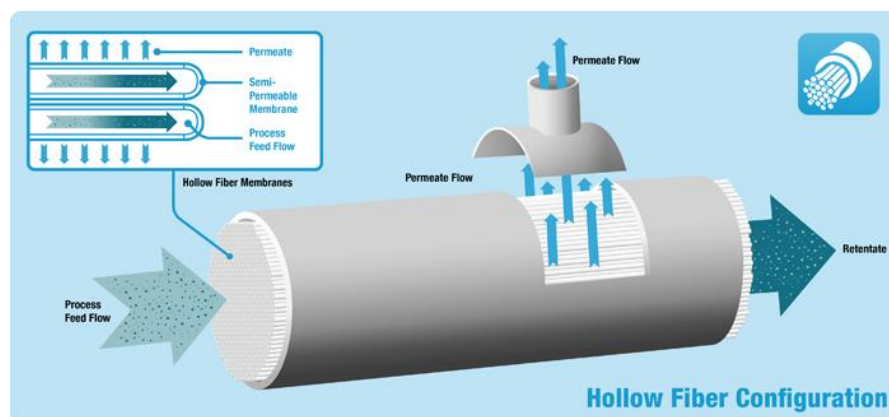


Figure 2.4 : Hollow fiber module [Adapted from Koch (2014)]

2.3.3 Membrane Fouling

Membrane fouling is a major problem that limits the performance of a membrane. It results from the deposition of soluble and particulate materials onto and into the membrane, causing permeate flux to decline over time and an increase of

transmembrane pressure (TMP) (Cosenza et al., 2013). In long term operation, membrane fouling results in reduced productivity, shorter membrane lifespan and increased operation costs. The extent of fouling can vary due to several factors such as (Cheryan, 1998; Le-Clech et al., 2006; Zhang et al., 2006):

- membrane characteristics (hydrophilicity, surface topography, membrane charge, pore size)
- feed-biomass characteristics (proteins, salts, pH, amount of filamentous bacteria, extracellular polymeric substances (EPS) and soluble microbial products (SMP))
- operating conditions (temperature, flow rate, turbulence, pressure)

As controlling membrane fouling is the key issue in the operation of an MBR, many methods have been proposed to reduce the chance of membrane fouling. Corrective methods to control fouling include periodic backwash, backflush, and chemical maintenance cleaning with sodium hypochlorite (for organic foulants) and citric acid (for inorganics). Severe membrane fouling occurs above a critical permeate flux or at too low aeration rate.

In a submerged MBR, shear forces can be utilized for the purpose of avoiding membrane fouling. Shear forces by air scouring creates turbulence of uprising air and liquid inside the membrane modules, resulting in removal of cake layer deposited on membrane surfaces before the cake layer becomes compacted.

Other strategies to limit fouling include improving the anti-fouling properties of the membrane, adjusting hydrodynamics and flux, and pre-treating the biomass suspension to limit its fouling propensity with coagulant/flocculant (Le-Clech et al., 2006).

2.3.4 MBR for Organic Degradation and Removal

Organic matters are normally composed of combination of carbon, hydrogen, oxygen, and nitrogen, forming compounds such as proteins, carbohydrates, oils, and fats. MBR has a proven track record in removing organic matters and even recalcitrant compound, as extended contact time of sludge and substrate allows development of specialized, slow-growing microorganism that are able to remove recalcitrant compounds (Melin et al., 2006). Organic matter removal can be achieved by a suspended growth biomass in the bioreactor, where the microorganisms use molecular/free oxygen to assimilate organic impurities as main substrate and convert them into carbon dioxide, water and biomass as described in equation below.



Heterotrophic microorganisms are the ones responsible for this process, as they use organic carbon for cell growth and formation of new biomass. They perform electron transfer with organic matter as electron donor to oxygen as electron acceptor. Heterotrophs bound to have higher cell yields and growth rates because they do not have to synthesize inorganic carbon to cellular carbon compounds. *Pseudomonas sp* has the highest degradation potential among the common heterotrophs found in activated sludge (Tchobanoglous et al., 2004).

In MBR system, it is important to maintain microorganisms in low growth rate where they mainly utilise available substrates for maintenance purposes. In an excess substrate condition, microorganisms tend to produce additional biomass, thus creating excess sludge that must be disposed of at costs. Contact time is provided in the MBR for mixing and aerating influent wastewater with the microbial suspension, generally referred as mixed liquor suspended solids (MLSS) or mixed liquor volatile suspended solids (MLVSS). COD and BOD₅ removal are found to increase with MLSS concentration, but eventually the reaction rate between substrate and MLSS could be hampered by less oxygen transfer rate if the MLSS concentration is higher than 20,000 mg/L (Kraemer et al., 2012). Biomass in an MBR process have less tendency to be washed out like often encountered in conventional activated sludge, which also one factor attributing to high organic removal rate in MBR process. Growth rate of the biomass in the system can be calculated as observed yield with below formula.

$$Y_{\text{obs}} = \frac{Q_w \times \text{MLSS}}{Q_i \times (\text{TCOD}_{\text{in}} - \text{TCOD}_{\text{out}})} \quad (\text{Equation 1})$$

where Q_w is waste sludging rate, MLSS is MLSS concentration in the system, Q_i is influent rate, TCOD_{in} is influent TCOD concentration, and TCOD_{out} is effluent TCOD concentration.

In addition, Sludge Retention Time (SRT) and the amount of readily biodegradable (soluble) COD affects the performance rate of the removal process. A high loading rate generally able to enhance heterotrophic growth, although it does not always generate the optimum organic percentage removal. On a MBR full scale treatment plant, optimal SRT time should be balanced between 15 and 40 days to

achieve optimum biodegradation removal and lowest fouling rate (Grelier et al., 2006).

Readily biodegradable COD (rbCOD) has direct effect on the biological kinetics and process performance because this portion is quickly assimilated by biomass, thus increasing the organic reduction rate. In a wastewater characterization process, rbCOD can be determined by the biological response method called the oxygen uptake rate (OUR). An ideal OUR curve for municipal wastewater is shown in Figure 2.5 below.

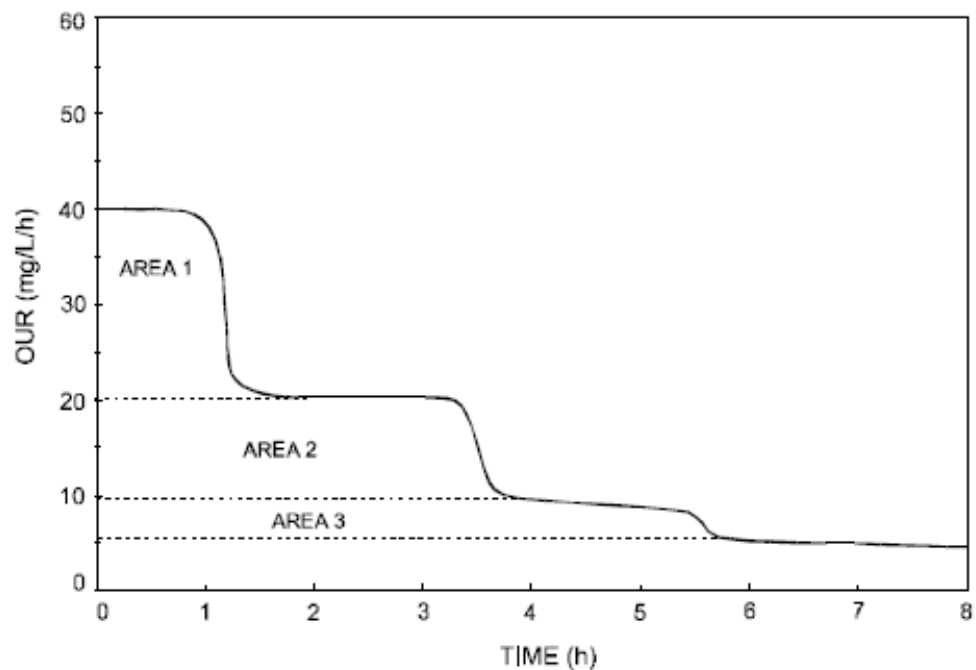


Figure 2.5 : Typical OUR curve [Adapted from Razafimanantsoa (2014)]

The OUR is obtained from the slope of the linear section DO response – that is, the decrease in DO over a measured time interval. The area under the OUR curve is divided into four sections: area 1 indicates the mass of oxygen utilized for the oxidation of rbCOD, area 2 indicates the mass utilized for nitrification, and area 3 denotes the mass consumed for oxidation of slowly biodegradable COD. The remaining area under the OUR curve indicates the oxygen associated with endogenous respiration (Razafimanantsoa, 2014).

2.3.5 MBR for Biological Nitrogen Removal

Biological nitrogen removal in wastewater treatment is used to protect water quality against negative effect of discharged nitrogen such as eutrophication or fish toxicity in water bodies. Nitrogen removal by microorganism can be further divided

into microbial cell assimilation and/or conversion to gaseous nitrogen by nitrification-denitrification process (Tan and Ng, 2008). The first method, nitrogen removal by cell assimilation, is achieved by wasting sludge from the MBR. Nitrogen assimilated into bacteria cells and higher organisms (protozoa and worms) will be removed from the MBR, thus decreasing total nitrogen in the system (Tan and Ng, 2008). Estimation of nitrogen removal through assimilation can be calculated using formula below :

$$DN_{waste} = i_{xvss} \times X_{vss} \times Q_w \quad (\text{Equation 2})$$

where i_{xvss} is nitrogen content by weight of MLVSS concentration, X_{vss} is the waste sludge MLVSS, and Q_w is the sludge wasting rate.

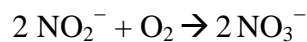
However, the most common practiced of biological nitrogen removal is nitrification-denitrification process. Nitrification is a term used to describe the two-step biological process in which ammonia (NH_4^-) is oxidized to nitrite (NO_2^-) and nitrite is oxidized to nitrate (NO_3^-), while denitrification describes the reduction of nitrate to nitric oxide, nitrous oxide, and nitrogen gas. Together, these two processes are widely used in wwtp to remove nitrogen content from the wastewater.

2.3.5.1 Biological Nitrification

Nitrification process occurs optimally in the aerobic zone with two main steps. In the first step, bacteria such as *Nitrosomonas* (and other genera with prefix *Nitroso-*) responsible for oxidizing ammonia to nitrite (Tchobanoglous et al., 2004) :



In the second step, *Nitrobacter* (and other genera with prefix *Nitro-*) oxidize nitrite to nitrate:



Both genera are aerobic autotrophic bacteria and they demonstrate more sensitive behavior towards their surrounding environment, such as pH, toxic compounds, metals, and un-ionized ammonia than heterotrophic bacteria. They also have low growth and yield rate, thus system designed for nitrification generally requires long hydraulic and solids retention times and adequate level of nitrifiers at all times to complete the process.

Nitrification rate would be halted to half of the maximum rate if the DO fall within the range of 0.3 - 1.3 mg/l , thus DO value below 1 mg/l could possibly reduce the nitrification rate (Charley et al., 1980). Barnes and Bliss (1993) reported that optimum pH for nitrification lies in the range of 7.5 – 8.5, while optimum temperature is range of 25 – 30 °C.

Tchobanoglous et al. (2004) states that maximum specific nitrification rate of the activated sludge in MBR is affected by the fraction of nitrifying organisms present in the mixed liquor, and this fraction is reasonably related with C/N ratio in the system. They also highly dependent on NH_4^- loading from the influent to survive, as ammonia serves as electron donor for this process, as well as oxygen as electron acceptor. A minimum sludge age of 5 days is necessary in order to ensure complete nitrification. For municipal wastewater, the maximum specific nitrification rates reported are between 0.91-1.12 mgNO_x-N/(gMLVSS.h) (Harremoës and Sinkjær, 1995). Zhang et al. (1997) also reported that nitrification rate is affected by floc size in the activated sludge.

Nitrification rate is measured by Ammonia Utilization Rate (AUR) test. During the AUR test, activated sludge is exposed to excessive NH_4 -N and aerobic condition. Continuous or frequent measurement of the decrease in NH_4 -N concentration over time allows the determination of the specific nitrification rate (SNR). SNR can be calculated using the formula below.

$$SNR = 60000 \times \frac{dN}{dt} / X \quad (\text{Equation 3})$$

where SNR is specific nitrification rate (mgNO_x-N/gMLVSS.h), dN/dt is the initial slope of the NH_4 -N versus time curve (mg NO_x-N /L.min), and X is MLVSS concentration during the test (mg MLVSS/L).

2.3.5.2 Biological Denitrification

Most of the bacteria carrying out denitrification are facultative aerobic organisms with ability to use oxygen as well as nitrate or nitrite as electron acceptor, such as *Pseudomonas* sp (Tchobanoglous et al., 2004). Typically the environment for denitrification is set to be anoxic, so in the absence or limited concentration of DO, these bacteria choose to use nitrate or nitrite as the electron acceptor instead of oxygen. The electron donor itself comes from internal organic source, such as BOD and COD in the wastewater, or external source like the addition of methanol and

acetate. Therefore, it is necessary to provide a sufficient amount of BOD or COD to induce a proper nitrate removal. Barth et al. (1968) found that approximately 4 gram of BOD is needed per gram of NO₃ reduced.

However, the actual value will depend on the operating conditions of the system and the type of electron donors used for denitrification, such as the presence of easily biodegradable substrate. Kraume et al. (2005) concluded from his study that higher soluble carbon concentration in influent can lead to higher nitrogen removal, and this removal rate can be further increased to meet effluent values less than 3 mg/L through addition of external carbon source. Synthetic wastewater with more easily biodegradable substrate (e.g. acetate) leads to higher denitrification rates (up to 20 mgNO₃-N/(gVSSh) than a substrate like raw water that is harder to degrade (1-6 mgNO₃-N/(gVSSh) (Kraume et al., 2005).

Nitrate utilization rate (NUR) test is a bioassay commonly used for the determination of denitrification rate. This test is commonly divided into two types, low food/microorganism (F/M) ratio NUR test (if the F/M ratio is between 0.02-0.05 mgCOD/mgVSS) and high F/M ratio NUR test (if the F/M ratio is between 2-3 mgCOD/mgVSS). Procedure of NUR test is almost similar with AUR, with only differences in electron donor and oxygen availability. NUR test is implemented under excessive nitrate and anoxic condition. Continuous or frequent measurement of the decrease in nitrate as electron acceptor concentration over time allows the determination of the specific denitrification rate (SDNR). SDNR can be calculated using the formula below.

$$SDNR = 60000 \times \frac{dN}{dt} / X \quad (\text{Equation 4})$$

where SDNR is specific denitrification rate associated with rbCOD consumption (mg NO_x-N/gVSS.h), dN/dt is the initial slope of the nitrate versus time curve (mg NO_x-N /L.min), and X is MLVSS concentration during the test (mg MLVSS/L).

2.3.5.3 Process Design of Nitrogen Removal in MBR System

In Modified Ludzak Ettinger (MLE) or preanoxic denitrification process, the anoxic tank precedes the aeration tank where nitrification occurs (Figure 2.6(a)). Nitrate produced in aeration tank is recycled back to the anoxic tank. Organic substrate in the influent wastewater provides the electron donor for oxidation reduction reactions using nitrate. Preanoxic denitrification is the most common

biological nitrogen removal process used in municipal wwtp. Biodegradable organic matter that is available in the anoxic zone via influent, improves denitrification rates, hence cutting out the need of external carbon source. Secondly, the oxidation capacity of nitrate degrades part of the organic matter, hence reducing oxygen demand and achieving savings in aeration requirement (Chen et al., 2010).

Figure 2.6(b) is termed postanoxic denitrification as denitrification occurs after nitrification, hence BOD removal has occurred first and is not available to drive the nitrate reduction reaction. Electron donor source comes from endogenous decay or external carbon source such as methanol or acetate. This process is more costly compared to preanoxic denitrification process.

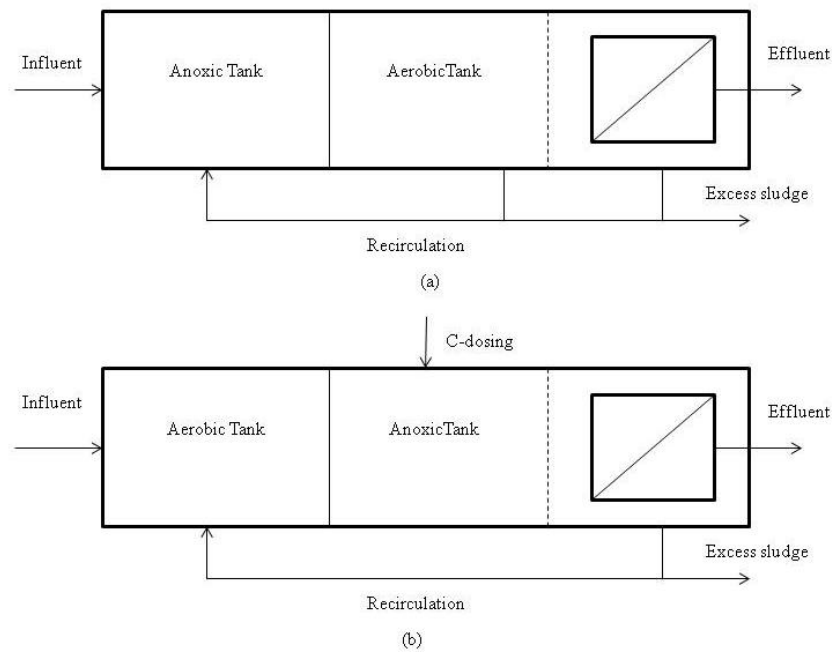


Figure 2.6: (a) Pre-denitrification process, (b) Post-denitrification process [Adapted from Chen et al. (2010)]

CHAPTER III

MATERIALS AND METHODS

This chapter contains an explanation about field description, experimental set up, and analytical methods used during this study.

3.1 Field Description

For this research, field activity was divided between Nordre Follo wwtp and Bekkelaget wwtp, although most of the experiment was implemented at Nordre Follo wwtp in Oslo, Norway.

3.1.1 Feedwater Source

Influent for the MBR was obtained from Nordre Follo wwtp, where the field experiment was also implemented. The wwtp is located 40 km southeast of Oslo and served 40000 people equivalent (pe) from Ås, Ski, and Oppegård Kommune. Water treatment methods consists of a combination of primary sedimentation, chemical treatment, MBBR process, and flotation. Effluent quality must meet with Norwegian Pollution Control Authorities standard of 90% of phosphorus removal or equivalent with 1 mg/L TP, 70% of nitrogen removal or equivalent with 10 mg/L TN, and 70% of organic removal or equivalent with 125 mg/L COD. Sample of wastewater was taken after grit removal. It was pumped into SF 1000 before continued to feed tank.

3.1.2 Activated Sludge Source

Activated sludge for MBR was obtained from Bekkelaget wwtp, which serves 35-40% of all wastewater from Oslo, or approximately 280000 pe. Bekkelaget uses primary clarifier, activated sludge, and sand filter for its water treatment process, with additional biogas production. Activated sludge was taken at March 10, 2014 at 10.15. TSS concentration is measured directly from the activated sludge and the concentration is approximately 6 mg/L.

3.2 Design of Experiments

The whole experiment started from January to June 2014, with data collection was implemented between 18 March-21 June 2014.

3.2.1 Reactor Setup

Two sets of MBR plants were operated in a parallel. The first one will treat municipal wastewater filtered with SF 1000 with 2 mm mesh size, which represents de gritted wastewater. Meanwhile, the other tank will receive wastewater that filtered with SF 1000 with 33 μm mesh size. The first set will further reference as treatment Train C, while the latter will be called treatment Train D. For both treatment trains, SF 1000 was operated without filter mat to prevent the formation of additional filtration barrier. The water from each sources were stored in a 500 L feed tank to ensure continuous operation. Both feed water were collected at the same time to ensure identical composition. Feed tanks were equipped with propeller mixer to avoid the settlement of particle matters.

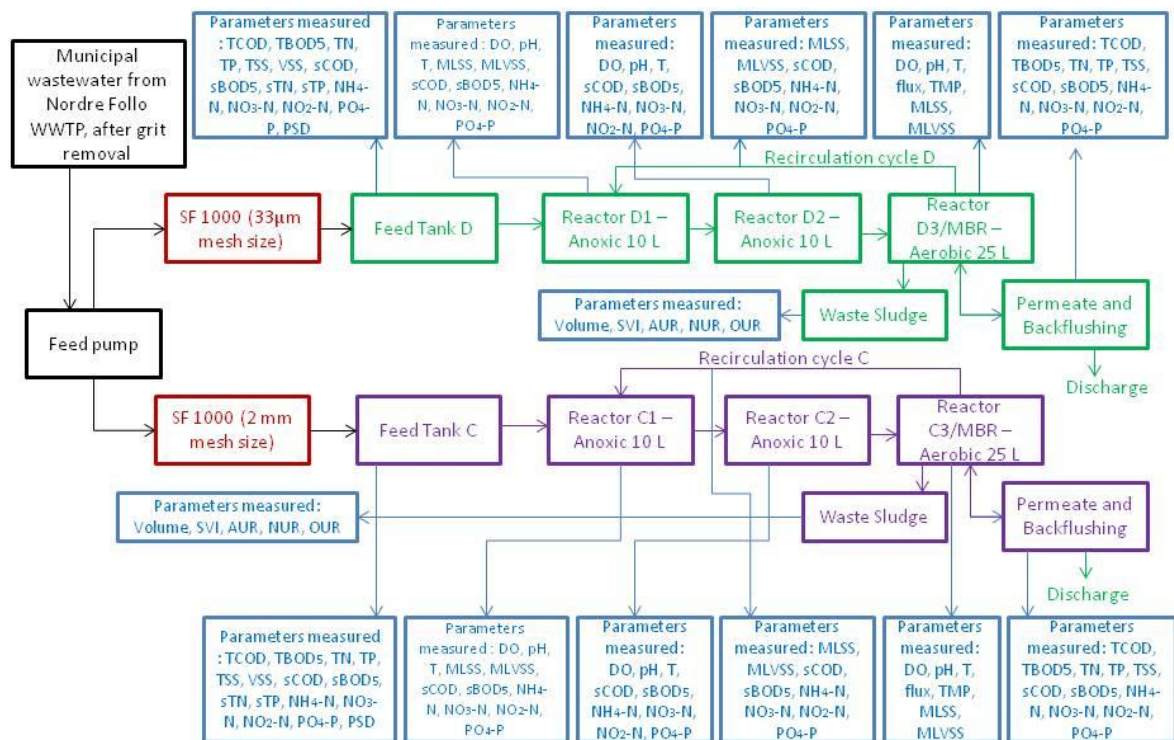


Figure 3.1: Schematic flowsheet of the MBR experiment

Figure 3.1 shows schematic flowsheet of MBR system. Water from feed tanks are pumped with inflow rate 5 L/h to both reactor tanks : tanks in treatment Train C received water from SF1000 with 2 mm mesh size, while tanks in treatment Train D accepted filtered water from SF 1000 with 33 μm mesh size. Each treatment train consists of two anoxic tanks for denitrification (tank C1, C2, D1, and D2) and one aerobic nitrification tanks with membrane module (tank C3 and D3). Anoxic tanks

have volume of 10 L and they were both equipped by propeller mixer. The aerobic tank have continuous aeration from the bottom of the reactor to supply oxygen required for the microorganism. Aerobic tank has volume of 25 L and it is equipped by pressure indicator, level sensor, thermometer, and DOmeter to ensure optimum membrane operation. These parameters, including flowrate for influent, permeate, and backflushing were controlled from a Programmable Logic Controller (PLC) to guarantee automatic operation.

The PLC was set to measure flowrate, DO, temperature, level, and pressure every five minutes. Programmable logic was also applied for influent, permeate, and backflushing flowrate. If water level in MBR reached a certain high level, influent rate was automatically stopped in order to restore the condition into normal water level. On the contrary, when water level in MBR dropped into a certain level, permeate flowrate was automatically stopped to prevent a further reduction of water level in the tank. Depending on DO concentration in the MBR, PLC was also able to turning on and off the emergency air supply on the tank.

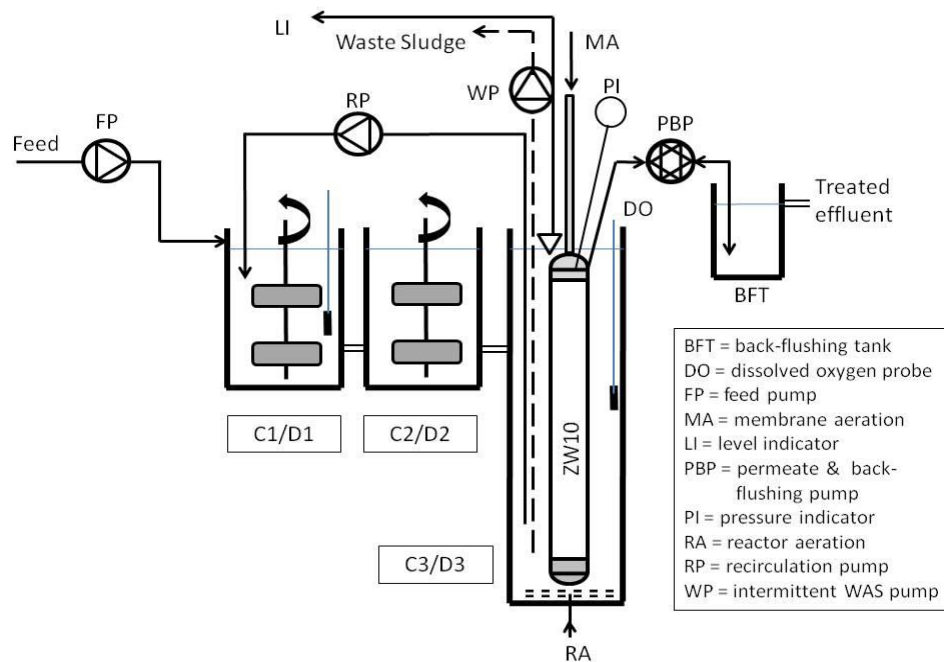


Figure 3.2: Schematic configuration of MBR system in each train

The retentate containing biomass and particles was recycled to the the first anoxic tank. The permeate from membrane was discharged. Excess biomass was withdrawn from the tanks C3 and D3 on a continuous basis to keep MLSS

concentration in the system revolved around 4000 mg/L in anoxic tank and 6000 mg/L in aerobic tank. A schematic configuration of treatment Train C and D system in the experiment is shown in Figure 3.2, while Figure 3.3 depicts the actual MBR system in the laboratory.

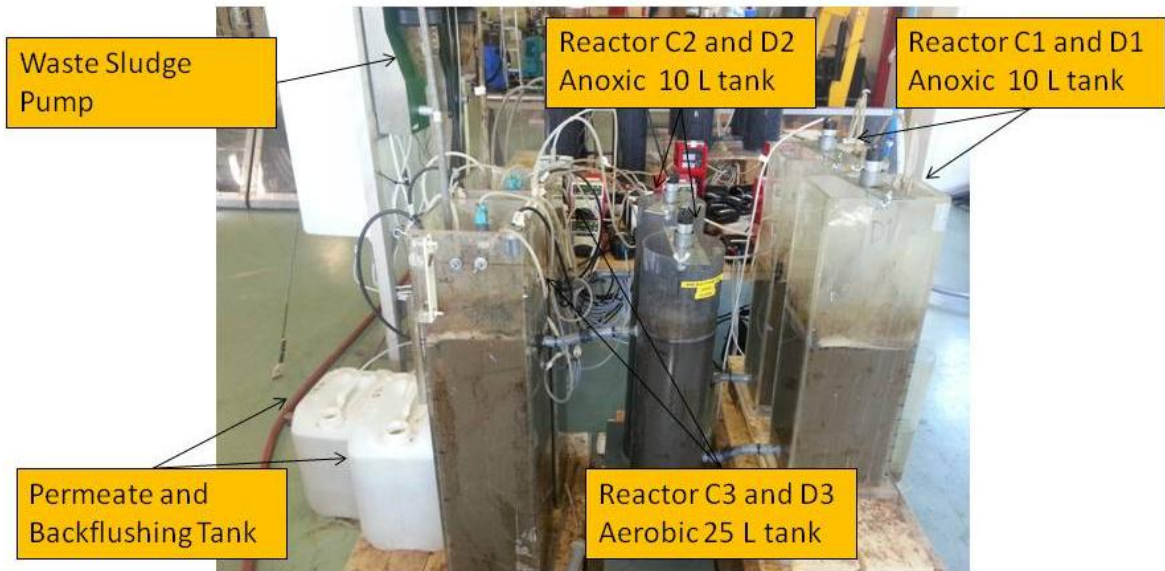


Figure 3.3: MBR system during the experiment

A ZeeWeed-10 Bench Test Unit by GE Water Technologies was used as submerged membrane module in the aerobic reactor. Air scouring was implemented to keep the hollow fibers moving and create a flow along the membrane surface, thus also decreasing the chance of membrane fouling. Membrane cleaning has to be done if TMP reach 300 mbar. For maintenance cleaning, sodium hypochlorite was added to the backpulse tank to a concentration of 500-1000 ppm. Soak cleaning was also performed if regular maintenance cleaning could not prevent membrane fouling. Membrane module must be soaked in 200 ppm NaOCl (for inorganic fouling) or HCl (for organic fouling) for a minimum of 5 hours. Membrane specification can be seen in Table 3.1 below.

Table 3.1 : Specifications of ZW-10 membrane

Parameter	Value/Type
Model	ZW-10, submersible module
Configuration	Submerged hollow fiber
Nominal Membrane Surface Area	0.93 m ²
Pore size	0.4 μm ²
Weight of Module (Drained)	1.9 kg
Weight of Module (Wet)	2.1 kg
Dimensions (length x width)	692.15 x 109.54 mm

Parameter	Value/Type
Permeate (Fiber Side) Hold-up Volume	0.13 liters
Typical Operating Transmembrane Pressure	1-7 psi @ 40°C
Operating pH range	5-9
Cleaning pH Range	2-10.5

In addition, specifications for SF1000 used as primary treatment for feed water is shown on Table 3.2, while Figure 3.4 shows SF1000 machine used in this study. SF1000 was operated with average flowrate of 1-2 L/s, belt speed of 30 Hz, and without filter mat. Water cleaning was always implemented after SF operation to prevent buildup of filter mat or other pollutants.

Table 3.2 : Specifications of SF1000

Specification	SF1000
Treated flow	0.2 MGD (31 m ³ /hr)
TSS removal efficiency	40 - 80% (design dependent)
BOD removal efficiency	20 - 35% (design dependent)
Sludge dry matter after thickening	3 – 8%
Maximum head loss	-
Dimensions (length x width x height)	1.4 x 1.3 x 1.4 m
Weight (dry)	415 kg



Figure 3.4: SF1000 machine

3.2.2 Operational Condition

Operating condition for all the tanks are described in Table 3.3.

Table 3.3 : Operating condition for the study

Parameter	Value
Feed flow rate	5 L/h
Recycle flow rate	10 L/h
Permeate flow rate	6.0 L/h
Back-flush flow rate	10 L/h
ZW10 operating cycle	9.5 minutes permeate 0.5 minutes back-flush
Recycle ratio	2
Operating TMP	< 300 mbar
Back-flush TMP	< 200 mbar
MLSS in anoxic tank (C1, C2, D1, D2)	~ 4000 mg/L
MLSS in aerobic tank (C3, D3)	~ 6000 mg/L
DO in anoxic tank (C1, C2, D1, D2)	~ 0 mg O ₂ /L
DO in aerobic tank (C3, D3)	> 2 mg O ₂ /L
Temperature	15-20 °C
Hydraulic Retention Time (HRT)	8 hours
Sludge Retention Time (SRT)	14 days (Train C), 17 days (Train D)

Several preparations that had to be done to achieve the desired operational conditions were equipment calibrations, PLC programming, activated sludge mixing, and membrane permeability test. Feed water pump, recycle pump, permeate and backflush pump, multimeter, DOmeter, and weighing scale were all calibrated before the experiment began.

To achieve MLSS concentration of 4000 mg/L in anoxic tank, 3 L of water was mixed with 7 L of activated sludge from Bekkelaget wwtp, which has MLSS concentration of 6000 mg/L. The desired MLSS concentration in tank C3 and D3 was achieved by putting 25 L of activated sludge into those aerobic tanks. SRT for each treatment train was not similar due to the difference of organic matter concentration after SF. Treatment Train C would experience faster microbial growth, thus a shorter SRT was needed to keep MLSS concentrations between 4000-6000 mg/L.

A membrane permeability test with clean water also had been implemented before the membrane was put in the process water. Membrane was also soaked and rinsed with NaOCl before use.

3.2.3 Experimental Program

Daily tasks during the entire period of experiments are shown in Table 3.4.

Table 3.4 : Daily tasks (Day 0-96)

	Week XX						
	Mon	Tue	Wed	Thu	Fri	Sat	Sun
Storage tank Fill	X		X		X	X	
Particle size distribution	X	X		X			
Influent analysis	X	X		X		X	
Reactor analysis		X		X			
Effluents analysis	X	X		X		X	
OUR Test*			X				
Low F/M ratio NUR Test*		X					
AUR Test*			X				
High F/M ratio NUR Test*				X			

* : tests are implemented every two weeks

3.2.3.1 Major Change During Experiment

Table 3.5 lists major changes that were applied to both of MBR systems during the time of experiment.

Table 3.5 : Major changes (Day 0-96)

Day	Date	Change	Train
37	23 April 2014	Waste sludge uptake	C (SF 2 mm) and D (SF 33 μ m)
41	27 April 2014		
43	29 April 2014		
66	22 May 2014		
81	6 June 2014		

3.2.4 Sampling Point

List of measured parameters in each of sampling points are described in Table 3.5. Sampling from the reactor was done by using a syringe from the middle of tank.

Table 3.6 : Sampling points

Sampling point	Location	Type of sample	Analytical parameters
Influent	Feed storage tank C and D	Unfiltered	TSS, VSS, PSD, TBOD ₅ , TCOD, TN, TP
		Filtered	sCOD, sBOD ₅ , NO ₃ -N, NO ₂ -N, NH ₄ -

Sampling point	Location	Type of sample	Analytical parameters
			N, PO ₄ -P, sTN, sTP
Effluent	The end of permeate tube	Unfiltered	TSS, TBOD ₅ , TCOD, TN, TP
		Filtered	sCOD, sBOD ₅ , NO ₃ -N, NO ₂ -N, NH ₄ -N, PO ₄ -P
Reactor C1, D1	Reactor	Unfiltered	DO, pH, T°C
	Effluent	Unfiltered	MLSS, MLVSS, SVI
		Filtered	sCOD, sBOD ₅ , NO ₃ -N, NO ₂ -N, NH ₄ -N, PO ₄ -P
Reactor C2, D2	Reactor	Unfiltered	DO, pH, T°C
	Effluent	Filtered	sCOD, sBOD ₅ , NO ₃ -N, NO ₂ -N, NH ₄ -N, PO ₄ -P
Reactor C3, D3 and Recirculation line C/D	Reactor	Unfiltered	DO, pH, T°C,
	Effluent	Unfiltered	MLSS, MLVSS
		Filtered	sCOD, sBOD ₅ , NO ₃ -N, NO ₂ -N, NH ₄ -N, PO ₄ -P
Membrane			TMP, Flux
Waste sludge	Waste sludge tank	Unfiltered	SVI

3.3 Analytical Methods

Unless specified otherwise, all Dr. Lange Cuvette kits for photometric analysis are produced by Hach Lange GmbH (Figure 3.5). Hach Lange Thermostat LT 200 was part of the kits and it was compatible for digestion of all sample and reagents at the cuvette kits. Also included in the package was Spectrophotometer Hach Lange DR 5000, which has wavelength range of 190-1100 nm and provides digital readouts in direct concentration units, absorbance or percent transmittance.



(a)



(b)



(c)

Figure 3.5: Hach Lange kits, (a) Hach Lange Thermostat LT 200, (b) Spectrophotometer Hach Lange DR 5000, (c) One of Hach Lange cuvette kits

3.3.1 Total Chemical Oxygen Demand (TCOD) and Soluble Chemical Oxygen Demand (sCOD)

Measurement of TCOD and sCOD concentration was done by Dr. Lange cuvette test LCK 114 (150-1000 mg/L O₂) or LCK 314 (15-150 mg/L O₂), and followed by two hours of digestion in 200°C temperature. After the digestion, sample scan was done by spectrophotometer DR 5000. sCOD sample was filtered through 1.2 µm Whatman glass before it was inserted into Dr. Lange cuvette kit.

3.3.2 Total Biochemical Oxygen Demand (BOD₅) and Soluble Total Biochemical Oxygen Demand (sBOD₅)

BOD₅ is used to measure dissolved oxygen in wastewater that is used by microorganisms in the biochemical oxidation of organic matters. Sample was tested with Dr. Lange cuvette test LCK 555 (4-1650 mg/L BOD₅), stored in 20°C dark place within five days, and followed by a sample scan by spectrophotometer DR 5000. sBOD₅ sample had to be filtered through 1.2 µm Whatman glass first before it was inserted into Dr. Lange cuvette kit.

3.3.3 Total Nitrogen (TN) and Soluble Total Nitrogen (sTN)

Total nitrogen consists of organic nitrogen, ammonia, nitrite and nitrate. To measure total nitrogen concentration, Dr. Lange cuvette test LCK 238 (5-40 mg/L TN) was used. Sample then digested for one hour in 100°C, then scanning was implemented by spectrophotometer DR 5000. sTN sample had to be filtered through 1.2 µm Whatman glass before it was inserted into Dr. Lange cuvette kit.

3.3.4 Nitrite Nitrogen (NO₂-N)

Nitrite nitrogen is toxic to most aquatic species, but it is easily oxidized to nitrate form. To measure nitrite nitrogen, sample was filtered through 1.2 µm Whatman glass before it was analyzed by Dr. Lange Cuvette test LCK 341 (0.015-0.06 mg/L NO₂-N), followed by spectrophotometer DR 5000 for sample scan.

3.3.5 Nitrate Nitrogen (NO₃-N)

Nitrate nitrogen is the most oxidized form of nitrogen found in wastewaters. To measure nitrate nitrogen, sample was filtered through 1.2 µm Whatman glass before it is examined by Dr. Lange cuvette test LCK 339 (0.23-13.5 mg/L NO₃-N). Spectrophotometer DR 5000 was then used for sample scan.

3.3.6 Ammonia Nitrogen (NH₄-N)

Ammonia nitrogen exists in aqueous solution as either ammonium ion (NH₄⁺) or dissolved ammonia (NH₃) depending on the pH of the solution. To measure ammonia nitrogen concentration, sample was filtered through 1.2 µm Whatman glass before it is analyzed by Dr. Lange Cuvette test LCK 303 (2-47 mg/L NH₄-N), LCK 304 (0.015-2 mg/L NH₄-N), or LCK 305 (1-12 mg/L NH₄-N), and followed with sample scan by spectrophotometer DR 5000.

3.3.7 Orthophosphate (PO₄-P)

Orthophosphate is the most abundant type of phosphorus, it is available for biological metabolism without further breakdown. Sample was filtered through 1.2 µm Whatman glass before it was examined by Dr. Lange Cuvette test LCK 348 (0.5-5 mg/L PO₄-P) or LCK 349 (0.05-1.5 mg/L PO₄-P). Spectrophotometer DR 5000 was then utilized for sample scan.

3.3.8 Total Phosphorus and Soluble Total Phosphorus (sTP)

Total phosphorus consists of orthophosphate, polyphosphate, and organic phosphorus. Sample was examined using Dr. Lange cuvette test LCK 348 (0.5-5 mg/L PO₄-P) or LCK 349 (0.05-1.5 mg/L PO₄-P) before it was digested for one hour at 100⁰C. Spectrophotometer DR 5000 is then utilized for sample scan. sTP sample had to be filtered through 1.2 µm Whatman glass before it was inserted into Dr. Lange cuvette kit.

3.3.9 pH, Temperature, and DO

Measurement of pH, temperature, and dissolved oxygen are important for the design and operation of biological process in the experiment. A multiparameter WTW 3420 was used to manually monitor pH, temperature, and dissolved oxygen rate in the reactors. In addition, a PLC was also installed to automatically record temperature and DO per five minutes.

3.3.10 Total Suspended Solids (TSS)

TSS is portion of total solids retained on a 1.2 μm Whatman glass fiber filter, measured after being dried at 105°C. Procedure used in the experiment is according to APHA Method no 2540D : Total Suspended Solids Dried at 103-105 °C in the Standard Methods for the Examination of Water and Wastewater (AWWA, 1999), as seen in Appendix A.1. Filters must be pre-cleaned first before they were used in the experiment. Pre-cleaned process includes addition of distilled water to new filters and then they were burnt in 550 °C for two hours. Pre-cleaned filters then weighted and placed in a petri dish for use.

3.3.11 Volatile Suspended Solids (VSS)

VSS is solids that can be volatilized and burned off when TSS are ignited in 550°C. Procedure used in the experiment is according to method no 2540 E : Fixed and Volatile Solids Ignited at 550 °C in the Standard Methods for the Examination of Water and Wastewater (AWWA, 1999), as seen in Appendix A.2.

3.3.12 Transmembrane Pressure (TMP)

Transmembrane pressure was measured automatically by PLC that is connected to MBR tank in both trains.

3.3.13 Particle Size Distribution (PSD)

In this study, a Mastersizer 3000 was used to characterize wastewater particles (Figure 3.6). Mastersizer 3000 uses laser diffraction method to analyze particle size in the sample. The instrument uses a 633 nm red laser and a 470 nm blue laser for measurements which allows it to cover the particle size distribution (PSD) from 10 nm to 3.5 mm (3500 μm).



Figure 3.6 : Malvern Mastersizer 3000

3.3.14 Membrane Flux

Membrane flux was determined manually by collecting permeate in graduated cylinder and time was measured by stopwatch.

3.3.15 Oxygen Uptake Rate (OUR)

To determine OUR, 1500 ml of waste sludge from Train C and D are mixed with 500 ml of feed wastewater from feed tank C and D in a batch reactor. The batch was aerated to have an initial concentration of around 8 mg O₂/L. Oxygen utilisation was measured by introducing an oxygen probe into the flask. Aeration was then terminated and the ensuing decrease in DO concentration with time was recorded until the DO had reduced to approximately 2 mg/L O₂. The OUR was calculated from the slope of the resulting oxygen utilisation curve.

3.3.16 Nitrate Utilization Rate (NUR)

NUR is used to determine denitrification rate. This experiment was divided into two parts, low F/M ratio and high F/M ratio NUR test. In the low F/M NUR test, 1500 ml of waste sludge from Train C and D were mixed with 500 ml of feed wastewater from feed tank C and D in a batch reactor, while in high F/M NUR test, 200 ml of sludge and 1800 ml of feed wastewater were used. Nitrate was added to reach a concentration of 30 mg/L. Samples of 3 ml was drawn off every 10 minute for the first 30 minute, and then every 30 minute for the rest of the experiment (total duration: 3 hours). MLSS, MLVSS, TCOD, sCOD, nitrate plus nitrite nitrogen were determined from the samples. The NUR was calculated from the slope of the resulting nitrate plus nitrite utilisation curve.

3.3.17 Ammonium Utilization Rate (AUR)

AUR is used to determine nitrification rate. To determine the AUR, 1500 ml of waste sludge from Train C and D were mixed with 500 ml of feed wastewater from feed tank C and D in a batch reactor. The liquor was kept in suspension by aeration through diffusers, which also provided the sludge with oxygen at a concentration of 6–8 mg/L O₂. Samples of 3 ml of mixed liquor were drawn off at 30-min intervals for 3 hours. Part of these samples were immediately measured for TCOD, MLSS, and MLVSS, while the other part was filtered and analysed for ammonia nitrogen and sCOD. The AUR was calculated from the slope of the resulting ammonium consumption curve.

3.3.18 Sludge Volume Index (SVI)

SVI is defined as the volume of sludge in milliliters occupied by 1 gram of activated sludge. SVI measurement is based on Norsk Standard NS-EN 14702-1 (Standard Norge, 2006), as seen in Appendix A.3. SVI was obtained by pouring a mixed liquor sample in a graduated cylinder, measuring the settled volume after 30 minutes and the corresponding sample MLSS concentration, and calculating the index using below formula :

$$SVI = \frac{30 \text{ min settling volume}}{MLSS} \times 1000 \quad (\text{Equation 5})$$

3.3.19 Bacterial Microscopy

Microscopic analysis was done to prove the abundance existence of filamentous bacteria in the activated sludge. Sample was taken from tank C1 and D1, where bulking sludge condition were the worst. Nikon Eclipse 50i with supporting tools such as Nikon DS-V11 digital camera and Digital Sight DS-02 was used to examine the sample. Magnification of 4x, 10x, and 40x were implemented to get the best result, with phase contrast Ph1, Ph2, Ph3, A, and C.

CHAPTER IV

RESULTS AND DISCUSSIONS

This chapter describes the result of the experiment and its analyses, including influent characteristics, removal percentage from every parameter, and membrane performance.

4.1 Influent Wastewater Characteristics

Characteristics of feed water that passed through grit removal of Nordre Follo wwtp during 18 March–21 June 2014 are summarized in Table 4.1. This categorization is based on municipal wastewater characterization by Henze et al. (2008), as already explained in sub-chapter 2.1. The high, medium, and low category displayed in the following table are compared to the typical concentration of pollutant in European municipal wastewater.

Table 4.1 : Average concentration of pollutant in influent wastewater (Day 0-96)

Parameters	Average influent after grit removal (mg/L)	Category [Adapted from Henze et al. (2008)]
TSS	269 ± 156	Low
VSS	226 ± 101	Low
TCOD	513 ± 166	Medium
sCOD	166 ± 44	Low
TBOD ₅	122 ± 62	Low
sBOD ₅	34 ± 17	Low
TN	43 ± 12	Medium
sTN	30 ± 10	Medium
NH ₄ -N	31 ± 10	Medium
NO ₃ -N	0.39 ± 0.2	High
NO ₂ -N	0.03 ± 0.02	Medium
TP	4.62 ± 1.79	Low
sTP	1.94 ± 0.81	Low
PO ₄ -P	1.85 ± 0.75	Low

In 96 days of research study, results indicate that influent could be categorized as medium to low concentrated wastewater, with only $\text{NO}_3\text{-N}$ could be considered as high concentration. Low concentrated wastewater represents high water consumption, infiltration in the sewage system, and/or stormwater effect (Henze et al., 2008). During March-June 2014, the months of the experiment, average precipitation reached 56.65 mm, slightly higher than the usual precipitation of 47 mm during these months (NRK, 2014). High precipitation volume is directly proportional with stormwater effect, which could be one of the reason why influent concentration is quite low. Medium percentage of incoming total nitrogen emphasizes the importance of nitrogen removal process for this wastewater treatment.

4.2 Boundary Conditions

Removal efficiency discussion is divided into two different boundary conditions : first case explains SF and MBR as one system (Figure 4.1(a)), while the second case discuss the effect of SF's different mesh size to MBR system (Figure 4.1(b)). For the first case, only influent from SF 2 mm used as reference as it represents the degrittied wastewater. Meanwhile, for second case, influent from both SF 2 mm and 33 μm will be used as reference for removal efficiency calculation. Dashed red line in Figure 4.1 represents the boundary condition for each cases. Both boundary condition will be used in the discussion of COD, BOD_5 , TN, TP, and TSS measured in this study.

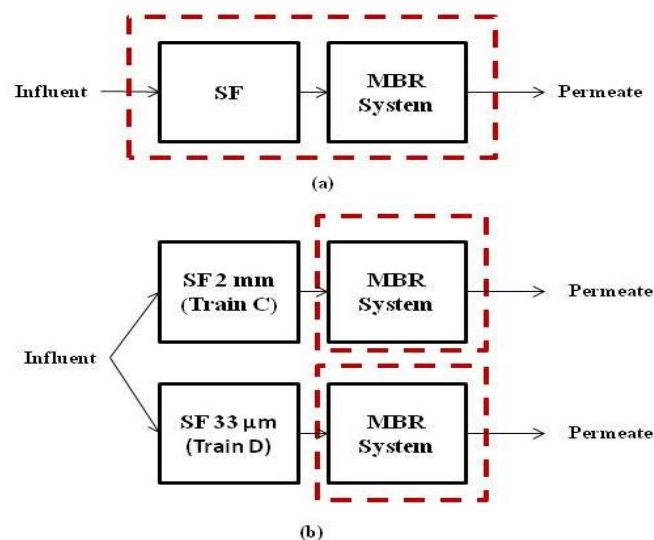


Figure 4.1 : Boundary conditions used in the discussions

4.3 COD Removal

COD removal for both boundary conditions are discussed in the following sections.

4.3.1 COD Removal – Case 1

Table 4.2 indicates that effluent TCOD concentration fluctuating around 32-33 mg/L in both treatment trains. The result is better than the discharge requirement set by Norwegian government, where it requires that TCOD concentration in the effluent must be at least 125 mg/L, or equivalent with 75 % of TCOD removal.

Table 4.2 : Case 1 - TCOD summary (Day 0-96)

Treatment Train	Average Influent TCOD (mg/L)	Average Permeate TCOD (mg/L)	Average TCOD Removal (%)
C (SF 2 mm)	513 ± 166	33 ± 7	93
D (SF 33 µm)		32 ± 7	93

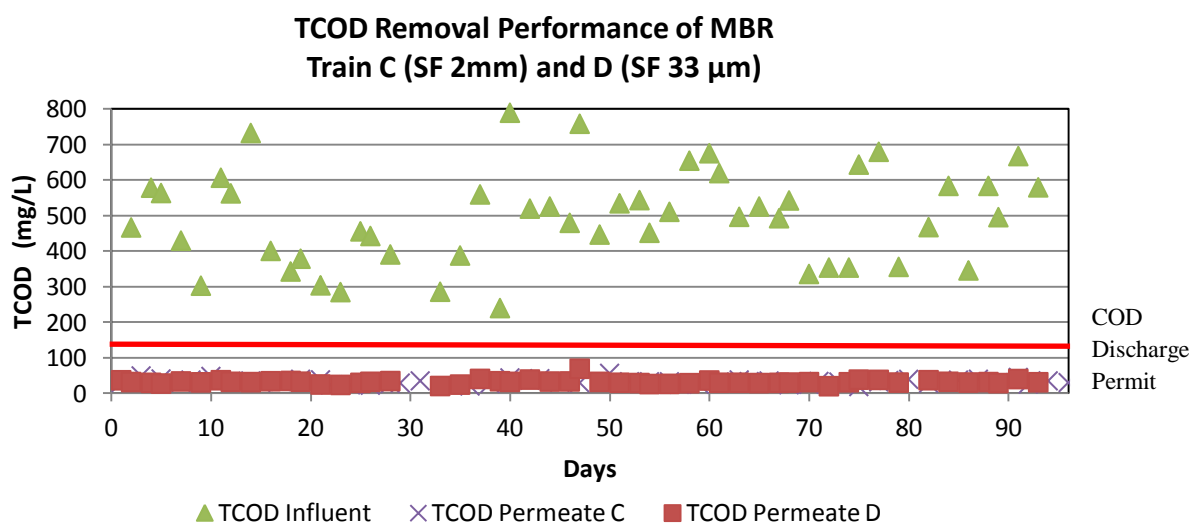


Figure 4.2: TCOD concentration removal in treatment Train C and D

Horizontal red line in the graph above represents the COD discharge permit. Figure 4.2 shows that even though the influent wastewater contains big range of TCOD concentration, both treatment trains can handle the variation of organic loading. TCOD percentage removal is stable, with approximately 93 % removal efficiency for treatment Train C and D. Both treatment trains do not need a long period of acclimatization for COD removal, as the permeates had showed good results since Day 1.

The location of aerobic tank after anoxic tank further ensure that any organic substrate not consumed in the anoxic zone would eventually be aerobically degraded in the aerobic tank. In addition, OUR results show that both system has high biological activity which could be one factor attributable to high organic removal. Treatment Train C and D have a specific oxygen uptake rate (SOUR) of 11 mgO₂/gVSS.h and 13 mgO₂/gVSS.h, respectively. These numbers are slightly higher than the reported SOUR in literature under similar operating conditions by Zielinska et al. (2012), which is around 0.7-10.3 mgO₂/gVSS.h. High SOUR could also indicate that the system contains high amount of readily biodegradable COD.

Furthermore, an additional of organic removal is also attributable to membrane filtration. As shown in Figure 4.3, sCOD concentration around 80-90 mg/L in the last tank can be cut down into around 32-33 mg/L in the permeate by the membrane barrier. The membrane also keeps the biomass concentration inside the system, further ascribing the system's capability to face fluctuation of influent organic loading.

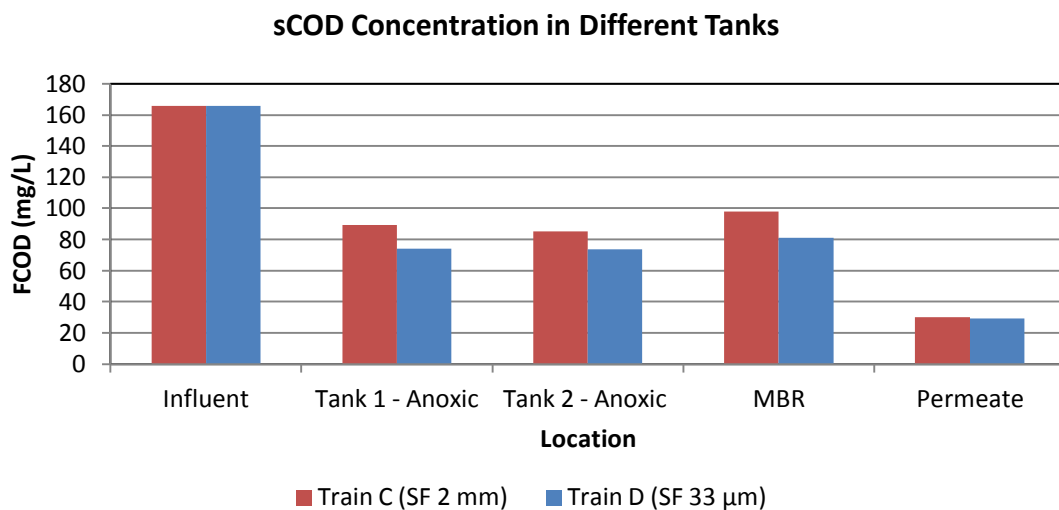


Figure 4.3: Average sCOD concentration in MBR system (Day 0-96)

sCOD concentration in every reactors are described in Figure 4.3. MBR has slightly higher sCOD concentration than tank C1, C2, D1, and D2, even though Galil et al. (2009) indicates that sCOD concentration should be the lowest in the last reactor because the carbon is supposed to be used as carbon source during denitrification in anoxic reactor. It should be noted that in several DO measurements, it was found that there were DO gradient in the MBR tank, where the surface has slightly higher DO

than the bottom of the reactor. It is possible that the bottom aerator could accommodate some anoxic bacteria, which would excrete EPS in order to protect themselves from their surroundings. EPS is major component of sCOD (Barker and Stuckey, 1999, Ni et al., 2010) but not a component of BOD, which explains why sCOD concentration is quite high in MBR tank, while BOD concentration is low (Figure 4.5). High SVI number for tank C3 and D3 (Table 4.15) could also be one indication that activated sludge in these reactors contain high percentage of EPS. To overcome this problem and bring back the fully mixed aerobic tank, an increase in DO concentration was applied to the experiment.

4.3.2 COD Removal – Case 2

In regard of influent coming from different SF mesh size, Table 4.3 shows that MBR in treatment Train D receives lower TCOD concentration due to smaller SF mesh size. To be precise, SF 33 μm in treatment Train D removes more COD in the primary treatment by 20% than SF 2 mm. Overall, the removal percentage in both cases are almost similar, around 91-93%, which can be meant that different mesh size on SF primary treatment did not have any negative effects on COD removal.

Table 4.3 : Case 2 - TCOD summary (Day 0-96)

Treatment Train	Average Influent TCOD (mg/L)	Average Permeate TCOD (mg/L)	Average TCOD Removal (%)
C (SF 2 mm)	513 \pm 166	33 \pm 7	93
D (SF 33 μm)	408 \pm 241	32 \pm 7	91

4.4 BOD₅ Removal

BOD₅ removal for both boundary conditions are discussed in the following sections.

4.4.1 BOD₅ Removal – Case 1

Table 4.4 and Figure 4.4 shows TBOD₅ removal summary, where the result from both treatment trains meet with the discharge requirement of 25 mg/L TBOD₅, or equivalent with 70-90 % of BOD₅ removal.

Table 4.4 : Case 1 - BOD₅ summary (Day 45-87)

Treatment Train	Average Influent TBOD₅ (mg/L)	Average Permeate TBOD₅ (mg/L)	Average TBOD₅ Removal (%)
C (SF 2 mm)	122 \pm 62	1.46 \pm 2.21	99

D (SF 33 μm)		1.12 ± 0.8	99
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TBOD₅ Removal Performance of MBR Train C (SF 2 mm) and Train D (SF 33 μm)

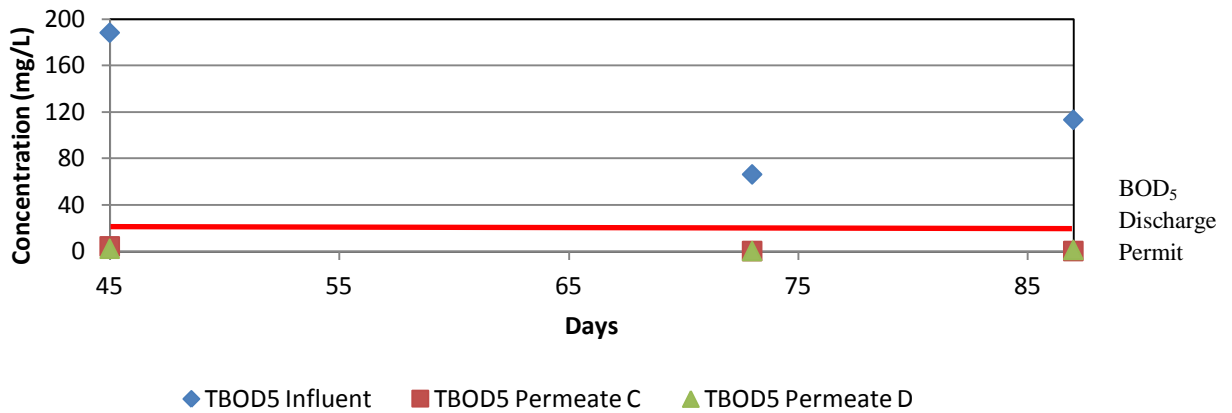


Figure 4.4 : TBOD₅ concentration removal in treatment Train C and D

Figure 4.5 shows that most of the biodegradable organic materials are consumed by the microorganism in the anoxic tank for denitrification. As a result, MBR tank is exposed to very low biodegradable organic loads. The extremely low concentration of BOD₅ in permeate also further ensures that the measurement process did not contaminated by nitrogenous oxygen demand. The latter is often regarded as a factor that can cause an inflation in BOD₅ results. Considering the fact that the nitrification component of the BOD₅ is generally at least 5-25 mg/L (Rich, 2003), the result from this study which revolves around 1-2 mg/L appears to impose no error in procedures.

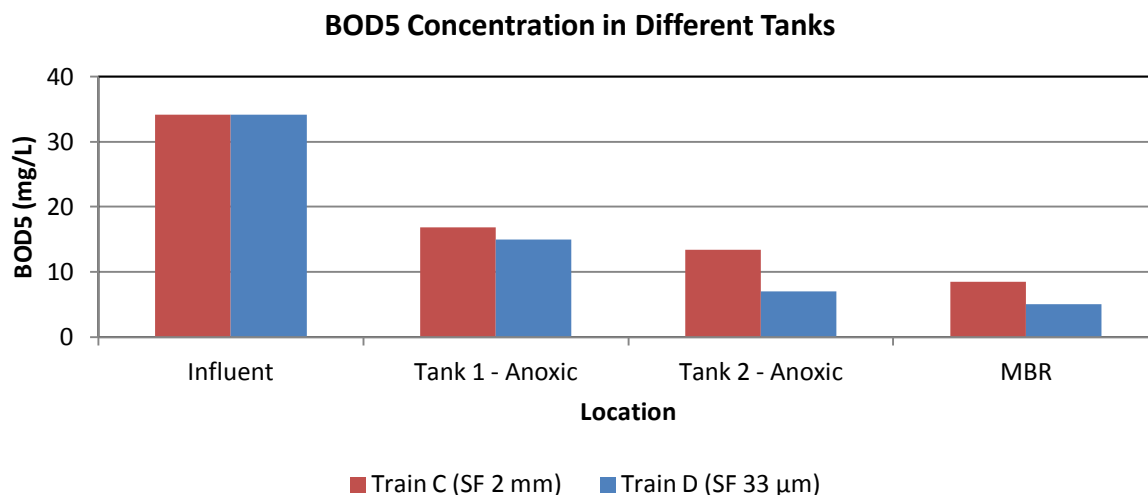


Figure 4.5: Average sBOD₅ concentration in MBR system (Day 45-87)

4.4.2 BOD₅ Removal – Case 2

From Table 4.5, it can be seen that SF 33 µm in treatment Train D removes more BOD₅ constituents than SF 2 mm by 52%. This pattern is similar with COD removal, where MBR system in treatment Train D receives less carbonaceous matter due to smaller mesh size in SF 33 µm. Overall removal percentage in both cases also suggest that different mesh size of SF primary treatment does not really substantial in BOD₅ removal.

Table 4.5 : Case 2 - BOD₅ summary (Day 45-87)

Treatment Train	Average Influent TBOD ₅ (mg/L)	Average Permeate TBOD ₅ (mg/L)	Average TBOD ₅ Removal (%)
C (SF 2 mm)	122 ± 62	1.46 ± 2.21	99
D (SF 33 µm)	58 ± 34	1.12 ± 0.8	98

4.5 Nitrogen Removal

Nitrogen removal for both boundary conditions are discussed in the following sections.

4.5.1 Nitrogen Removal – Case 1

Table 4.6 : Case 1 - TN summary (Day 0-96)

Treatment Train	Average Influent TN (mg/L)	Average Permeate TN (mg/L)	Average TN Removal (%)	Influent TCOD/TN Ratio
C (SF 2 mm)	43 ± 12	12 ± 4	73	12.3
D (SF 33 µm)		14 ± 5	68	9.7

As seen in Table 4.6, average TN effluent concentration are 12 mg/L and 14 mg/L for treatment Train C and D, respectively. These average numbers do not meet the discharge requirement of 10 mg/L TN, even though there were some days where the permeate effluent contains less than 10 mg/L TN (Figure 4.6). Fraction of soluble TN accounts for 72 % of TN after SF treatment. Nitrifiers were proven to be the limiting factor in overall nitrogen removal. They need 17 days for acclimatization for treatment Train C and 3 days for treatment Train D. Slower nitrification acclimatization in treatment Train C can be caused by higher MLSS and MLVSS in these reactors, thus decreasing oxygen diffusion in the tank and therefore slowing nitrifier growth. Denitrifiers, however, have worked optimally since Day 1 in both treatment trains.

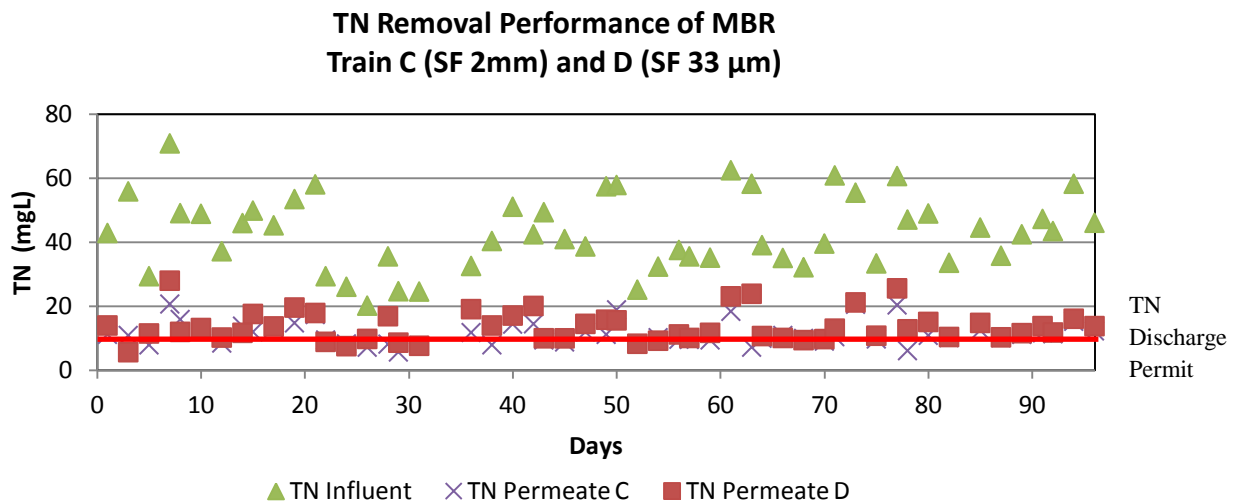


Figure 4.6: TN concentration removal in treatment Train C and D

Lower removal percentage in treatment Train D could originate from insufficient denitrification caused by TCOD/TN ratio in the system. Treatment Train C has influent TCOD/TN of 12.3, while treatment Train D only has TCOD/TN ratio of 9.7. SF 33 μ m produces lower incoming TCOD due its smaller mesh size than SF 2 mm. Wang et al. (2009) and Islam et al. (2009) both state that higher TCOD/TN ratio leads to higher TN removal efficiency, as COD can act as limiting factor for denitrification because of its role as electron donor in this process. As seen in Figure 4.7, treatment Train D has higher $\text{NO}_3\text{-N}$ than Train C in its permeate, suggesting that denitrification is hampered in this system.

Dissolved oxygen, another factor that can be potentially obstruct denitrification rate, has an average concentration of 0.01 mg/L in both treatment trains of anoxic tank. It means that oxygen imported by recycle streams from the aerobic reactor, if any, can not be a factor that hinder the denitrification process in the anoxic tank in treatment Train D.

While denitrification is a problem in treatment Train D, complete nitrification have been obtained successfully in both reactors, indicated by 97-99 % removal of $\text{NH}_4\text{-N}$ in aerobic MBR tank. High SRT, high bacteria concentration, and proper oxygen supply contributes to the thrive of slow-growing nitrifying bacteria in aerobic tank. Alas, there are several occasions when pH drops below 7 in the aerobic tank as the cause of nitrification process, resulting in the needs of constant addition of alkalinity.

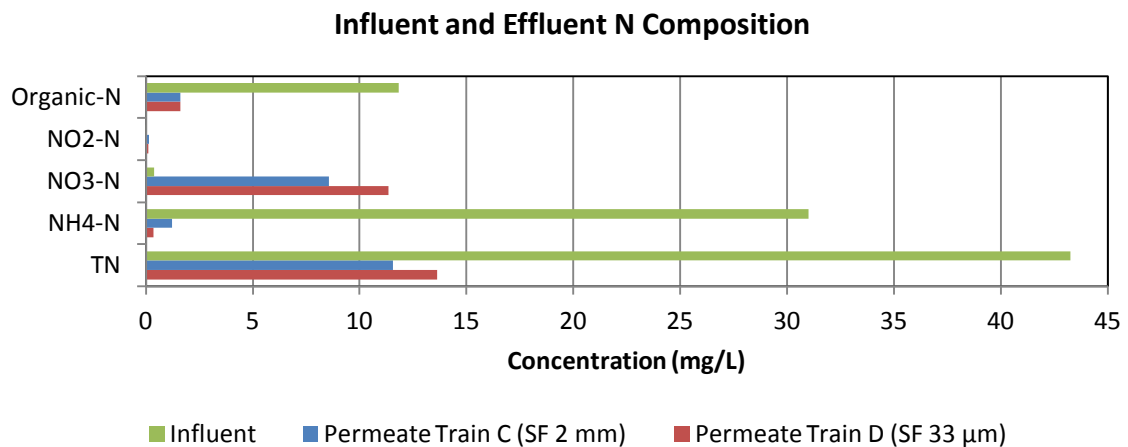


Figure 4.7: Average influent and effluent composition in treatment Train C and D (Day 0-96)

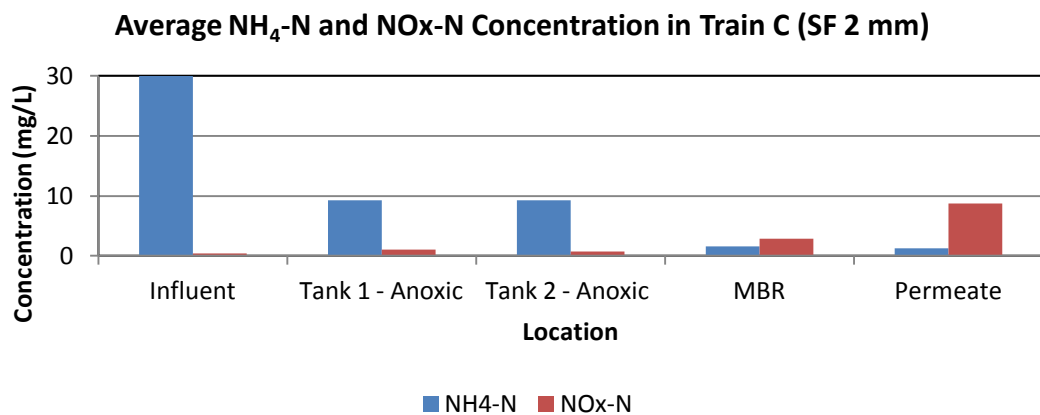


Figure 4.8: Average $\text{NH}_4\text{-N}$ and $\text{NO}_x\text{-N}$ concentration in treatment Train C (Day 0-96)

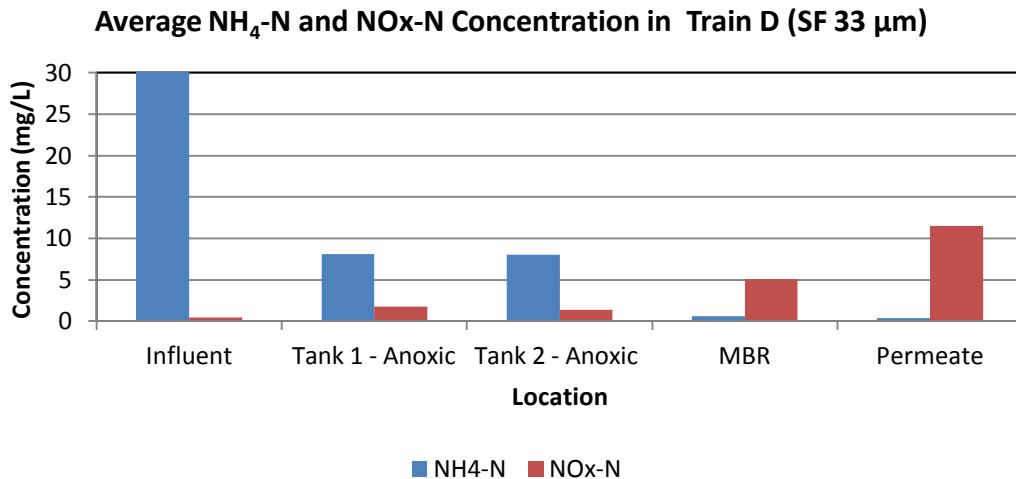


Figure 4.9: Average NH₄-N and NO_x-N concentration in treatment Train D (Day 0-96)

Figures 4.7, 4.8, and 4.9 show the components that build up TN and their concentration before and after the treatment, which can also demonstrate the nitrification-denitrification capability of both systems. Nitrite concentration is negligible as all of detected concentration in all reactors are below 0 mg/L NO₂-N, indicating full nitrification process. NH₄-N has the lowest concentration in aerobic tank, where nitrifiers use NH₄-N as electron donor and O₂ as electron acceptor to fully degrade NH₄-N into NO₃-N with 97-99 % efficiency. Several literatures (Galil et al., 2009; Kraume et al., 2005; Monti et al., 2006; Sun et al., 2013) also highlight the stable performance of MBR system in nitrification process over a wide range of operational conditions.

NO_x-N has most presence in aerobic tank and the least in anoxic tank, where denitrification depletes NO₃-N to N₂. Recirculation system from MBR tank to C1-D1 provides MLSS rich in NO₃-N, thus further increase the overall nitrogen removal. It also should be noted that both systems are able to degenerate and remove high amount of organic-N, as illustrated in Figure 4.7.

It is also possible that a simultaneous nitrification denitrification (SNDN) occurred during the period when the bottom of MBR tank undergo through anoxic condition, as discussed previously in section 4.3.1. SNDN is a condition where an oxygen diffusion limitation create anoxic zones within floc particles, facilitating simultaneous nitrification and denitrification process (Paetkau and Cicek, 2011). It explains why the nitrogen removal efficiency was almost stable during the whole

experiment period even though there were less NO₃-N recycled back to the anoxic tank in the period when DO was not properly mixed.

Overall, the average TN effluent concentration still exceeds the discharge requirement made by Norwegian government. Thus, carbon addition such as methanol are recommended to achieve TN effluent concentration less than 10 mg/L. Another way to enhance nitrogen removal is by increasing recycle ratio up to 3 or more, as reported by Lee et al. (2010) and Tan and Ng (2008). The latter reported that recycle ratio of 3, 5, and 10 under similar DO concentration with this experiment resulted in nitrogen removal efficiency of 80 %, 84 %, and 89 %, respectively. This study used recycle ratio of 2.

4.5.2 Nitrogen Removal – Case 2

Table 4.7 : Case 2 - TN summary (Day 0-96)

Treatment Train	Average Influent TN (mg/L)	Average Permeate TN (mg/L)	Average TN Removal (%)
C (SF 2 mm)	43 ± 12	12 ± 4	73
D (SF 33 µm)	43 ± 18	14 ± 5	66

The result in Table 4.7 is the only result where SF 33 µm does not contribute to any nitrogen removal in the first stage. The incoming TN to MBR system is similar between two trains, while permeate in treatment Train D is higher, as already discussed in the previous sub-chapters. Overall, TN removal percentage in Cases 1 and 2 are almost similar, with treatment Train C has the better removal percentage by 5-7 %.

4.5.3 Nitrification and Denitrification Rate

Specific nitrification rate (SNR) and specific denitrification rate (SDNR) data are calculated using Equation (3) and (4) and the results can be seen in Table 4.8 and Figure 4.10.

Table 4.8 : Nitrification and denitrification rate summary (Day 43-96)

Treatment Train	Specific nitrification rate (mgNO_x-N/gMLVSS.h)	High F/M specific denitrification rate (mgNO_x-N/gMLVSS.h)	Low F/M specific denitrification rate (mgNO_x-N/gMLVSS.h)
C (SF 2 mm)	1.30	2.76	1.49
D (SF 33 µm)	1.48	2.88	1.39

From the batch experiment, it is found that the nitrification rate in this experiment is slightly higher, but comparable to those reported in the literature, 0.91–1.12 mgNO_x-N/gMLVSS.h with domestic wastewater (Harremoës and Sinkjær, 1995). The little difference may be attributable to the different system configurations and operating conditions used in these studies. Nitrification rate between two treatment trains is not significantly different, as shown in Figure 4.10 below, as both treatment trains achieve 97-99 % NH₄-N removal rate. A further relation between nitrification rate and particle size distribution is discussed in section 4.8.

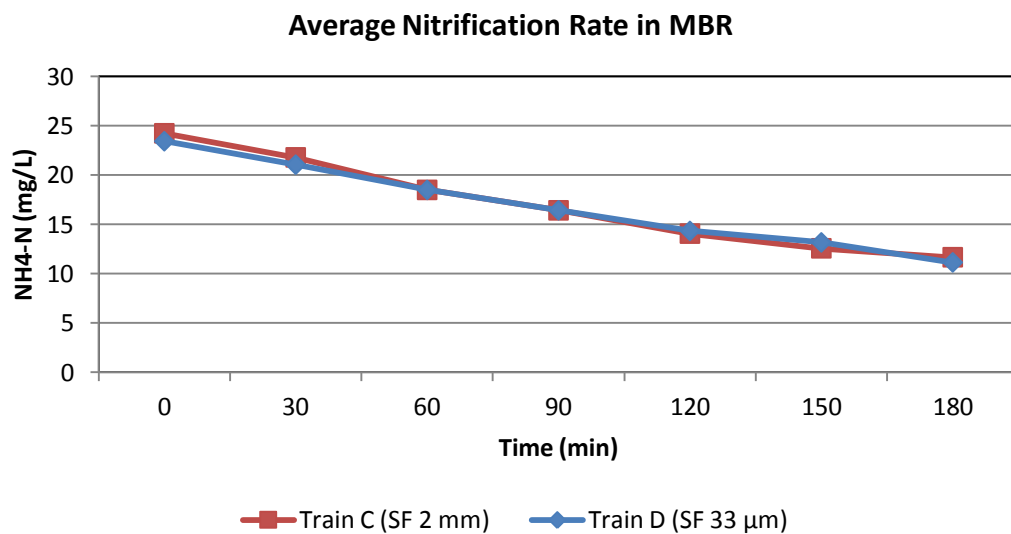


Figure 4.10: Result of batch nitrification rate (Day 43-96)

Result from batch denitrification rate, both from the high and low F/M ratio, also in accordance with literature from Kraume et al. (2005), which states that typical denitrification rate for municipal wastewater is 1-6 mgNO_x-N/gMLVSS.h.

Figure 4.11 shows low F/M ratio denitrification rate result, which indicates that microorganisms in treatment Train C are able to remove NO_x-N faster than the ones in treatment Train D. This is also supported by the NO₃-N data in Figure 4.7, where NO₃-N concentration in permeate treatment Train C is lower than the one in treatment Train D.

However, high F/M ratio denitrification data shown at Table 4.8 and Figure 4.12 imply that in a condition where there is an abundance of nutrient and carbon source, denitrification performance between two treatment trains are almost similar, between 2.7-2.8 mgNO_x-N/gMLVSS.h.

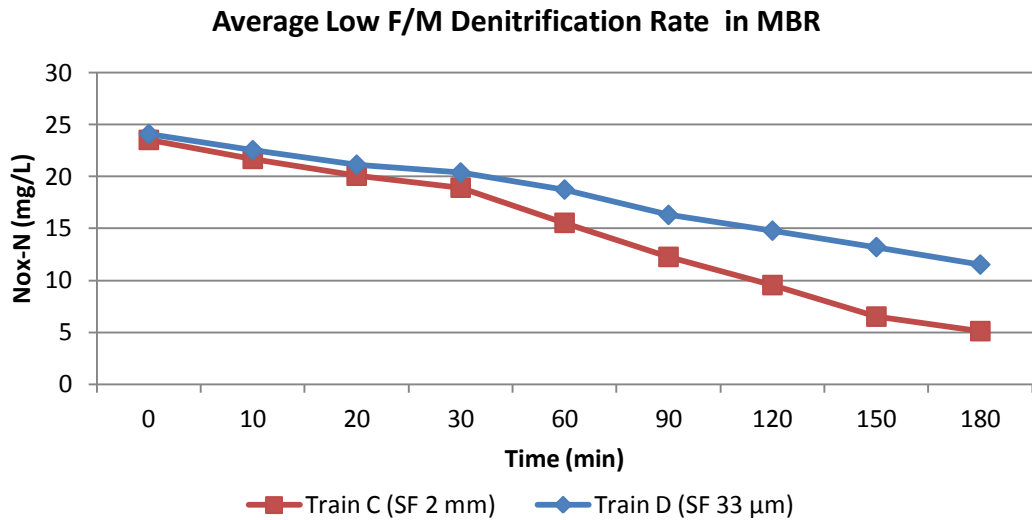


Figure 4.11: Result of batch low F/M denitrification rate (Day 43-96)

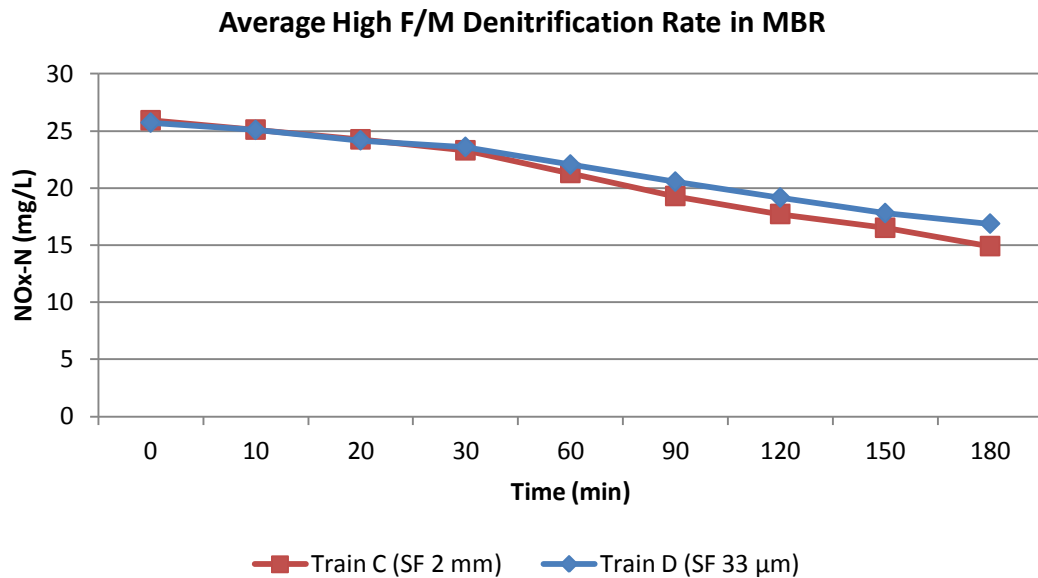


Figure 4.12: Result of batch high F/M denitrification rate (Day 43-96)

4.5.4 Denitrification Potential

To investigate the main cause of nitrogen removal, denitrification potential from wasted sludge and nitrification-denitrification process were calculated. Denitrification potential from wasted sludge was analyzed from cell assimilation of nitrogen in the system using Equation (2), while nitrogen removal from nitrification-denitrification was determined by the difference between the actual nitrogen removal with denitrification potential of wasted sludge.

Table 4.9 : Source of denitrification process (Day 0-96)

Treatment Train	Denitrification from Waste Sludge (mg N/day)	Denitrification from Nitrification-Denitrification (mg N/day)
C (SF 2 mm)	18.31	3946
D (SF 33 μ m)	15.44	3733

Table 4.9 shows denitrification potential from wasted sludge only account for less than 1% from actual total denitrification performed by the system. The number indicates that nitrogen loss in the process mostly caused by nitrification-denitrification rather than cell assimilation, as also previously stated by Tan and Ng (2008).

4.6 Phosphorus Removal

Phosphorus removal for both boundary conditions are discussed in the following sections.

4.6.1 Phosphorus Removal – Case 1

Table 4.10 : Case 1 - TP summary (Day 0-80)

Treatment Train	Average Influent TP (mg/L)	Average Permeate TP (mg/L)	Average TP Removal (%)	TCOD/TP Ratio after SF	TN/TP Ratio after SF
C (SF 2 mm)	4.62 \pm 1.79	0.99 \pm 0.8	78	124	9.5
D (SF 33 μ m)		0.94 \pm 0.7	79	105	9.5

During the research, phosphorus removal efficiency achieved were 78 % and 79 % for treatment Train C and D, respectively. Fraction of soluble TP accounts for 42-45 % of TP after SF treatment. As seen in Table 4.10 and Figure 4.13, the effluent concentration is in compliance with discharge requirement for TP, which requires the effluent from WWTP has TP concentration less than 1 mg/L TP.

**TP Removal Performanc of MBR
Train C (SF 2mm) and D (SF 33 μm)**

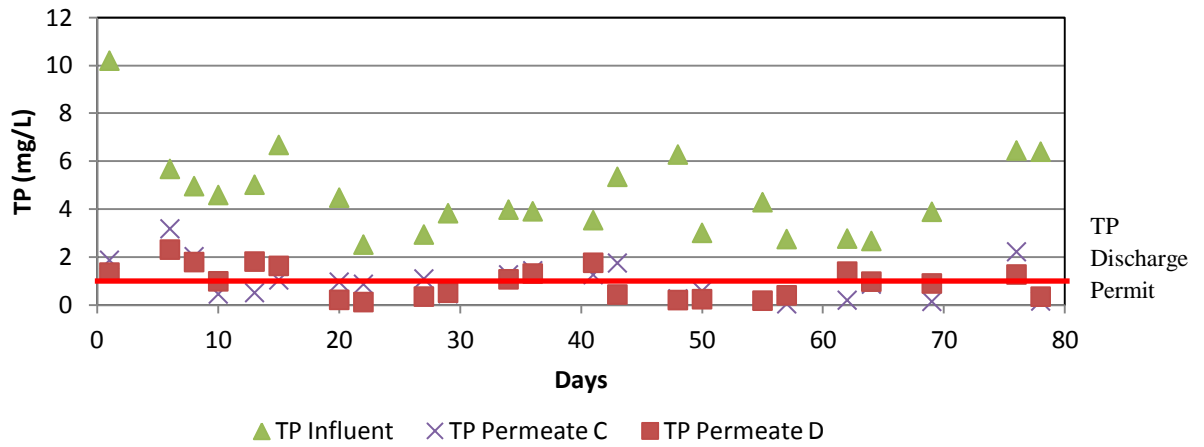


Figure 4.13: TP concentration removal in Train C and D

Even though the effluent concentration fulfills the discharge criteria, 78 % of removal that are achieved by both treatment trains can be categorized as low. A review article by Melin et al. (2006) points that TP removal by MBR system could be as high as 97 %. In this study, phosphorus removal is done by cell assimilation. Phosphorus is regularly incorporated into biomass by assimilation, as regular bacterial cells usually contain 2-3 % dry weight of phosphorus. Then, with wasting the sludge from MBR tank, phosphorus content in the system is also removed. However, assimilation process is limited by cell requirement of N : P content, a ratio of 5 : 1 (Strom, 2006). Data from treatment Train C and D indicate that N : P ratio in the system is 9.5 : 1 (Table 4.10), so it is likely that cell assimilation process in both systems does not go optimally because of phosphorus limitation.

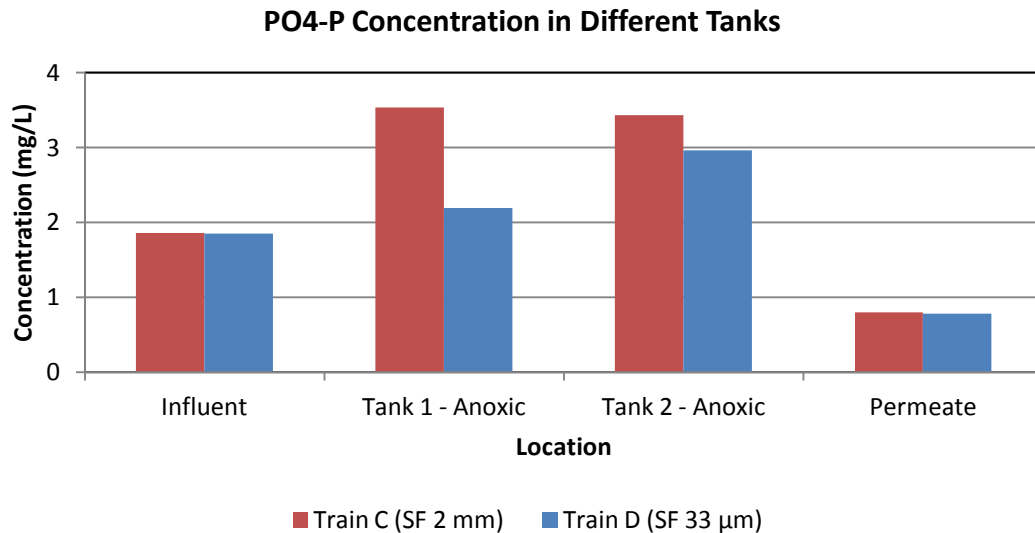


Figure 4.14: PO₄-P concentration in Train C and D (Day 0-80)

Figure 4.14 shows an abundance of phosphate concentration in the anoxic system compared to permeate, most likely caused by a secondary phosphorus release from biomass decay. High aeration could also be one factor that cause secondary phosphorus release, and this phosphate release is brought to anoxic tank by recirculation line from MBR tank. Removal of net biomass growth, or sludge wasting, is necessary to keep the system intact.

Several studies suggest that MBR system can easily support an Enhanced Biological Phosphorus Removal (EBPR) concurrently with nitrogen removal (Punrattanasin, 1997; Sun et al., 2013; Wang et al., 2009), with effluent phosphorus concentration less than 0.1 mg/L. EBPR can be achieved by growing phosphorus accumulating organisms (PAO), a certain group of microorganisms that, under specific condition, can facilitate removal of phosphorus from the wastewater. Under anoxic condition, they can create an energy store in the cell and in doing so release phosphate into the medium. In aerobic conditions they use the stored energy to reproduce, taking up more phosphate that will kept in the cells and stored as polyphosphate (Grote and Teacher, 2010). The phosphate in EBPR is removed in the waste sludge, which might have 5 % or more P (dry weight) as opposed to less than 3 % in non-EBPR sludges. In this experiment, waste sludge has less than 2 % phosphorus in dry weight, further confirming that phosphorus removal is solely come from cell assimilation.

EBPR also can be achieved in this system, if only TCOD/TP ratio can be decreased into around 25-40 for optimum EBPR (Punrattanasin, 1997). There are two factors affecting the efficiency of EBPR, i.e. COD limited or phosphorus limited (Galil et al., 2009). In this experiment, average TCOD/TP ratio found to be approximately 100 for both of the reactors (Table 4.10), indicating a process with phosphorus limitation, thus potential of the enhanced biological phosphorus removal was not fully expressed. It is because the higher the influent phosphorus concentrations, the more the phosphorus was released due to the more phosphorus available for PAO to accumulate as internal polyphosphate (Wang et al., 2009).

4.6.2 Phosphorus Removal – Case 2

Table 4.11 : Case 2 - TP summary (Day 0-80)

Treatment Train	Average Influent TP (mg/L)	Average Permeate TP (mg/L)	Average TP Removal (%)
C (SF 2 mm)	4.62 ± 1.79	0.99 ± 0.8	78
D (SF 33 µm)	4.44 ± 2.1	0.94 ± 0.7	77

From Table 4.11, it can be seen that SF 33 µm contributes in TP removal by 3.8% in the first stage than SF 2 mm. If comparing the removal percentage results in Cases 1 and 2, the results are also similar, between 77-79 % of phosphorus removal efficiency. In conclusion, different SF mesh size does not give substantial effect for phosphorus removal in both systems.

4.7 Suspended Solid Removal

Suspended solid removal for both boundary conditions are discussed in the following sections.

4.7.1 Suspended Solid Removal – Case 1

ZW-10 membrane used in this experiment provides very efficient solid removal, with 100 % efficiency in both treatment trains and corresponds with 0 mg/L TSS in the effluent, as shown in Table 4.12 and Figure 4.15. TSS effluent results meet the discharge criteria, which requires an effluent with 35 mg/L TSS or equivalent with 90 % TSS reduction from the influent.

Table 4.12 : Case 1 - Suspended solid summary (Day 0-96)

Train	Average Influent TSS (mg/L)	Average Permeate TSS (mg/L)	Average TSS Removal (%)
C (SF 2 mm)	269 ± 116	0 ± 1.8	>99
D (SF 33 μm)		0 ± 0.5	>99

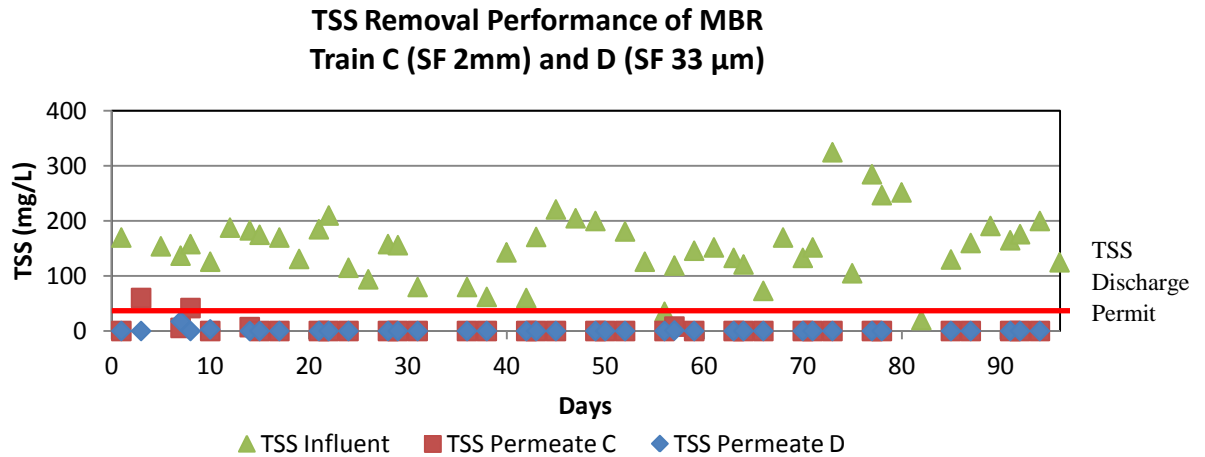


Figure 4.15: Suspended solid percentage removal for Train C and D

MLSS and MLVSS concentrations for every reactors are shown in Figure 4.16 and 4.17. During 96 days of experiment, MLSS concentration fluctuated more or less over 4000 mg/L for anoxic tank and 6000 mg/L for aerobic reactors, with MLVSS accounts for approximately 66 % of total MLSS. Higher MLSS and MLVSS concentration in treatment Train C is due to higher food concentration that goes to the reactors, as SF 2 mm passes more organic matter and nutrients than its SF 33 μm counterpart in treatment Train D.

MLSS Concentration in Different Tanks

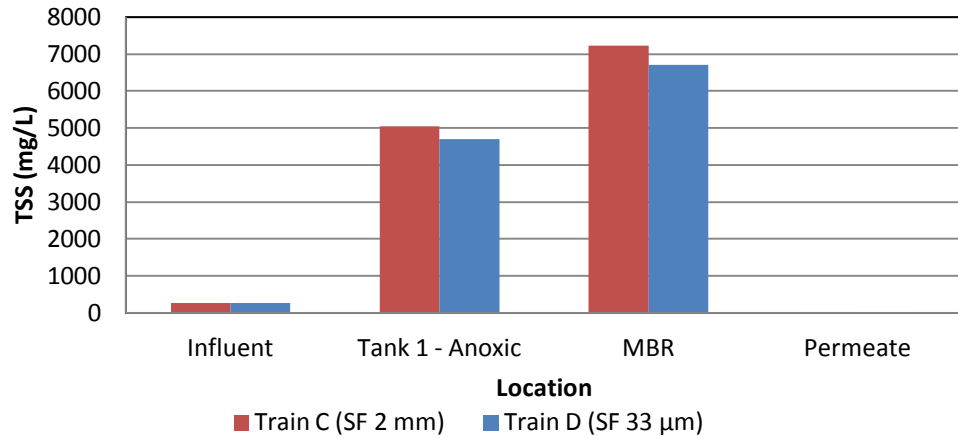


Figure 4.16: Average MLSS concentration in treatment Train C and D (Day 0-96)

MLVSS Concentration in Different Tanks

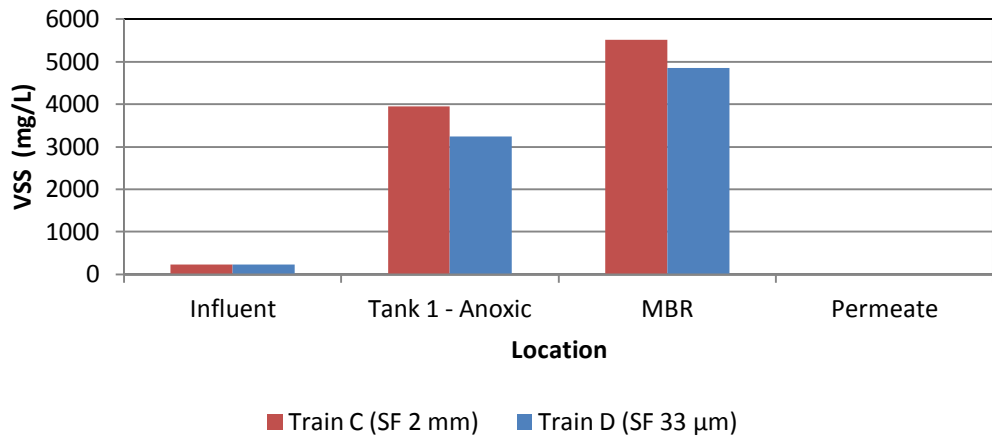


Figure 4.17: Average MLVSS concentration in treatment Train C and D (Day 0-96)

4.7.2 Suspended Solid Removal – Case 2

Table 4.13 shows that SF 33 µm removes more suspended solids than SF 2 mm by 38%, a result that is in accordance with previous parameters such as COD, BOD₅, and phosphorus.

Table 4.13 : Case 2 - Suspended solid summary (Day 0-96)

Treatment Train	Average Influent TSS (mg/L)	Average Permeate TSS (mg/L)	Average TSS Removal (%)
C (SF 2 mm)	269 ± 116	0 ± 1.8	>99
D (SF 33 µm)	168 ± 96	0 ± 0.5	>99

As an upstream treatment for MBR, here SF plays a necessary role in protecting membrane integrity by reducing a significant amount of suspended solids before the MBR system. When comparing to the full scale MBR plants in Europe, most of them use two stage screening equipment with 2-6 mm cut-off at the first stage and 0.5-3 mm cut off at the second stage (Hai, 2013). In this case, SF as upstream treatment for MBR is proven to be more advantageous because it provides high quality effluent with low TSS concentration with only one stage of treatment.

4.8 Particle Size Distribution (PSD)

Table 4.14 shows that average particle diameter in treatment Train C is slightly higher than treatment Train D as a direct cause from bigger mesh size. Further observation in Figures 4.18 and 4.19 indicates that SF 2 mm also gives broader range of overall particle diameter compared to its counterpart, especially at macroparticles range (above 100 μm). The lack of macroparticles in treatment Train D also contributes to smoother membrane operation, as it decreases the risk of cake formation and particle disposition on membrane surface (further explained in section 4.11). Particles in the influent wastewater are reported to have high correlation with suspended matter, biological entities, and adsorbed organics and chemicals (Wu et al., 2009). Thus, PSD analysis is in accordance with COD, BOD₅, and TSS data from previous sections, where treatment Train C receives more particles than Train D due to SF 2 mm placed before the anoxic tank, and therefore has more COD, BOD₅, and TSS concentration.

Table 4.14 : Particle size distribution summary (Day 63-96)

Treatment Train	Average particle diameter after SF (μm)	Average particle diameter in Aerobic/MBR tank (μm)
C (SF 2 mm)	42	44
D (SF 33 μm)	33	42

Particle Size Distribution - Frequency Distribution

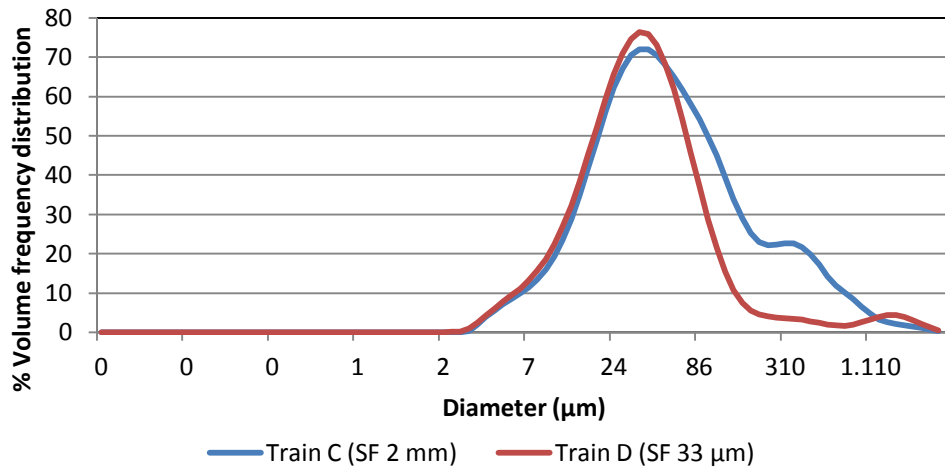


Figure 4.18: PSD frequency distribution after SF treatment in Train C and D (Day 63-96)

Particle Size Distribution - Cumulative Distribution

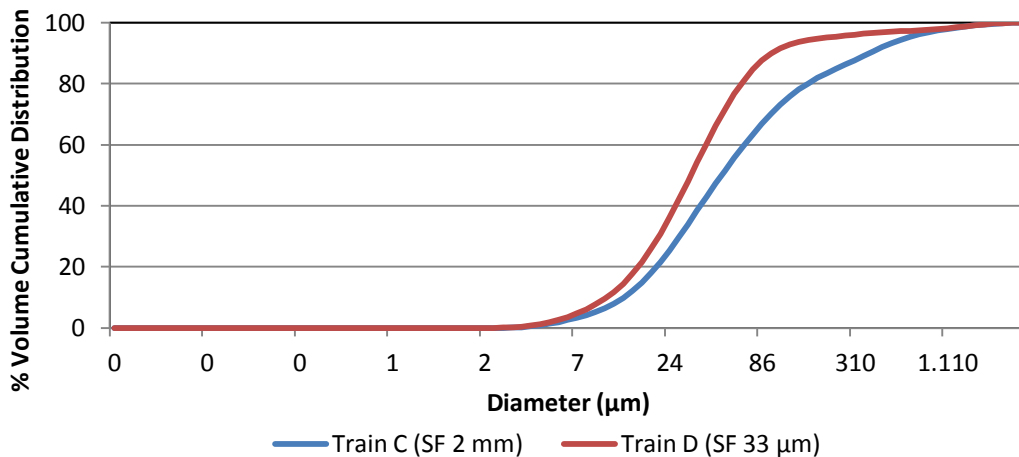


Figure 4.19: PSD cumulative distribution after SF treatment in Train C and D (Day 63-96)

Table 4.14 also gives the average particle diameter for activated sludge in the MBR/aerobic tanks. The results, 44 µm for treatment Train C and 42 µm for treatment Train D, are reported to be smaller than average particle size in conventional activated sludge of 77 µm (Chen et al., 2010). There are several factors that likely to cause this behaviour. First, violent turbulence produced from air scouring in the MBR system can break down the floc and particle of activated sludge. Second, tangential flow

along the membrane can also create significant shear stresses that decrease the diameter of particle. The amount of recirculation is also reported to be one of the factor affecting particle size in activated sludge system (Wu et al., 2009).

There are also a slight change of average particle diameter after SF and in the MBR/aerobic tank, where particle in the latter sampling point exhibits a larger size in dimension. This can be contributed from bacterial activity in the anoxic and aerobic tank that can flocculate the small particles into larger flocs (Wu et al., 2009).

Furthermore, Zhang et al. (1997) found in his research that particle size affects SNR of the system, where SNR was decreased with increased particle or floc size. This theory is in accordance with this study, where treatment Train C, which has larger particle size, also has slightly slower SNR compared to treatment Train D. It is due to the fact that the smaller floc size is, the larger ratio of perimeter to area is. Smaller floc or particle also might have less limitation in oxygen transfer. However, this does not apply to denitrification and organic removal, as their rate are more dependent on other factor than particle size, such as carbon sources (Zhang et al., 1997).

4.9 Sludge Characteristics

Sludge settleability and production are two main characteristics of sludge that is observed in this experiment.

4.9.1 Sludge Volume Index

Table 4.15 shows SVI for both trains are averaged between 170-250 mL/g, which refers to bulking sludge condition and poor settleability. For reference, a good sludge settleability should be around 80-150 mL/g. This poor condition most likely due to abundance of filamentous bacteria, although other factors like hydraulic overload, EPS concentration, floc surface properties, shape, size, and flocculating ability may also contribute to sludge settleability (Jassby et al., 2014). Figures 4.20, 4.21, and 4.22 depict the comparison between normal sludge and sludge with bulking and foaming problem in this experiment.

Table 4.15 : Sludge volume index summary (Day 0-96)

Treatment Train	SRT (days)	Average SVI Index (mL/g)
C1 (SF 2 mm) – Anoxic	14	235
C3 (SF 2 mm) – MBR	14	175
D1 (SF 33 μ m) – Anoxic	17	244
D3 (SF 33 μ m) – MBR	17	172



Figure 4.20: Sludge without bulking and foaming in tank D1 (Day 95)



Figure 4.21: Bulking sludge in tank C1 (Day 95)



Figure 4.22: Foaming in tank C3 (Day 95)

Filamentous bacteria, with *M. parvicella* as the most important filamentous species in biological nutrient removal, are likely to be main factor of the problem encountered in this research. They mostly thrive in anoxic condition with DO concentration less than 1.5 mg/L O₂ and high concentration of ammonium (Martins et al., 2004), which can explain why C1-D1 tank have higher SVI number than C3-D3. In Europe, filamentous organism population also depends heavily on seasonal pattern with a maximum activity in winter/early spring (Eikelboom et al., 1998), which corresponds with this research's timeline. Several methods that can be introduced to overcome bulking sludge problem are chlorination, use of skimming devices, or create a condition that favour floc formers over filaments by introducing selectors that produce a substrate (food) gradient in the tank (Grote and Teacher, 2010). Microscopy results of filamentous bacteria found in the sample are shown in Figure 4.23 and 4.24 below, where the filaments are clearly seen in both magnification..

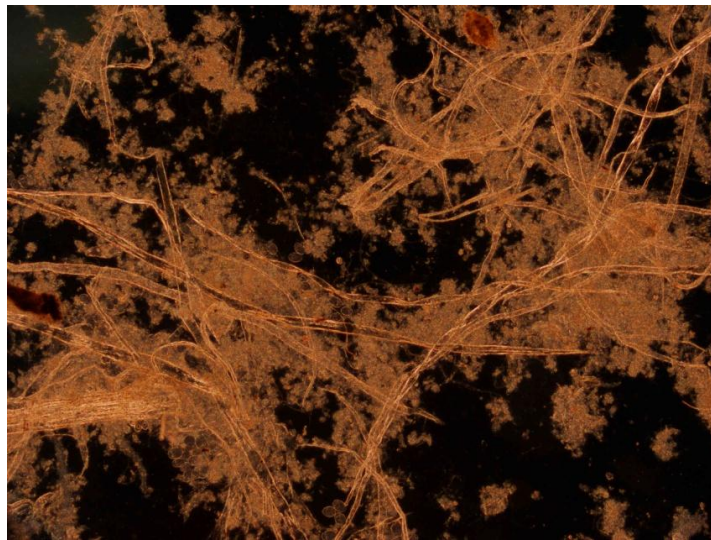


Figure 4.23: Filamentous bacteria in 4x magnification

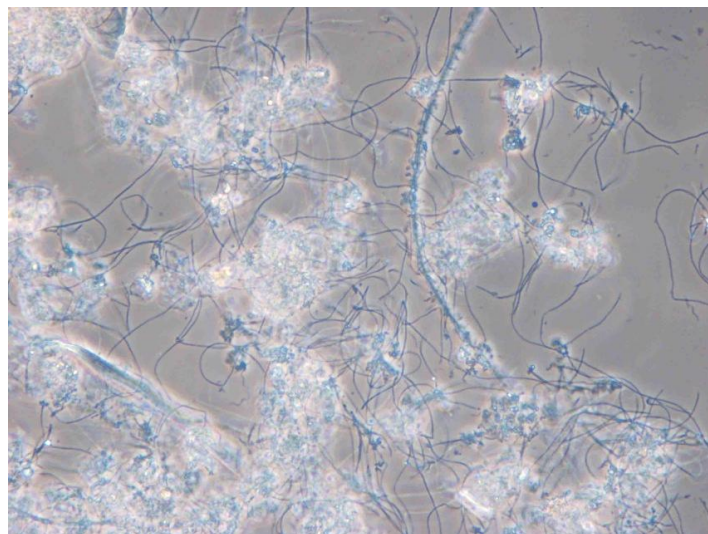


Figure 4.24: Filamentous bacteria in 40x magnification

4.9.2 Biosolids Production

Table 4.16 shows average growth rate of biomass in both train as calculated by Equation (1),

$$Y_{obs} = \frac{Q_w \times MLSS}{Q_i \times (TCOD_{in} - TCOD_{out})}$$

Table 4.16 : Observed yield summary (Day 0-96)

Treatment Train	Observed yield (mg MLSS/mg COD)
C (SF 2 mm)	0.35
D (SF 33 μ m)	0.4

Lower biomass yield in treatment Train C likely due to higher MLSS concentration in that system, which leads to substrate limitation and therefore low sludge yield. This sludge yield number is slightly higher than the reported yield observed in typical MBR system, which is around 0.25-0.38 mg MLSS/mg COD (Monti et al., 2006; Tan and Ng, 2008). A previous discussion about high SOUR number might be attributable to higher biomass yield than the theoretical one.

4.10 pH, DO, and Temperature

Table 4.17 presents the summary of pH, DO, and temperature in treatment Train C and D during the experiment.

Table 4.17 : pH, DO, and temperature summary (Day 0-96)

Treatment Train	Average pH in Anoxic Tank	Average pH in MBR Tank	Average DO in Anoxic Tank (mg/L O ₂)	Average DO in MBR Tank (mg/L O ₂)	Average Temperature Overall (°C)
C (SF 2 mm)	7.31 ± 0.11	7.12 ± 0.16	0.01 ± 0.01	3.75 ± 1.46	18.2 ± 2.45
D (SF 33 μ m)	7.24 ± 0.14	7.04 ± 0.23	0.01 ± 0.01	4.07 ± 1.21	18.1 ± 2.41

Low standard deviation for every parameter suggest that no major environmental change happens during the experiment. MBR tanks automatically have lower pH than anoxic tanks due to the nitrification activity. DO concentration in MBR tanks is more than 3 mg/L due to air scouring, which is installed to prevent cake layer formation in membrane structure. Treatment Train D has slightly higher DO concentration compared to treatment Train C, because lower MLSS concentration in

this system (Figure 4.16) provides easier O₂ transfer and diffusion than a system with higher MLSS quantity.

4.11 Membrane Performance

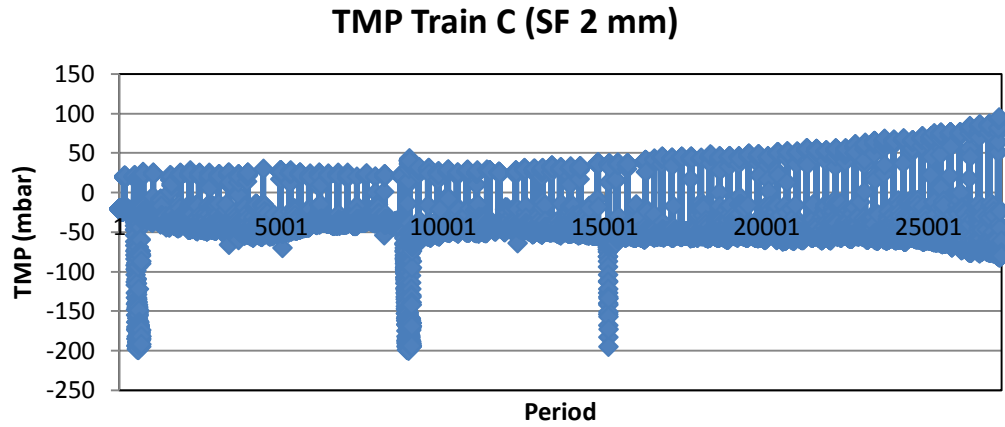
Based on Table 4.18, TMP for treatment Train C is reported higher than treatment Train D, with average of 50 mbar compared to 28 mbar for permeate flux, and 39 mbar to 26 mbar for backflushing flux . The experiment is operated as constant permeate flux mode of 6 L/h, which provide an operational mechanism with less risk of fouling, so there is no flux change during the experiment period.

Table 4.18 : Transmembrane pressure summary (Day 0-96)

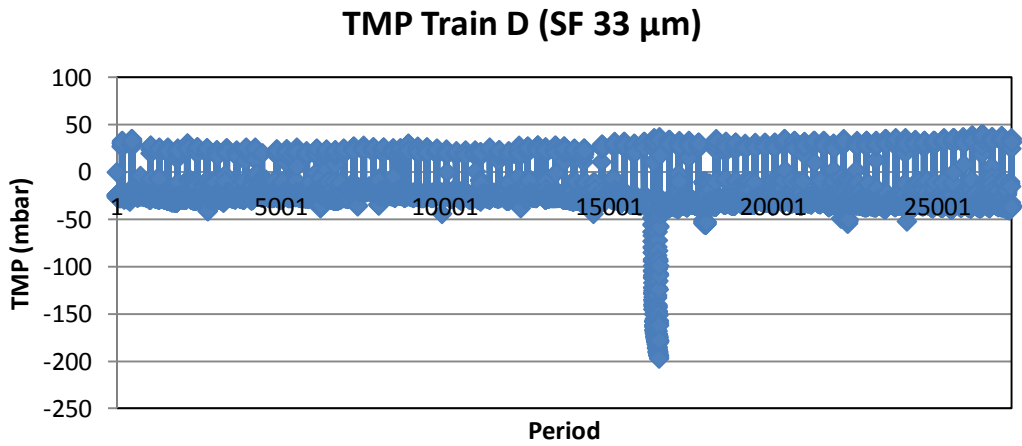
Treatment Train	Average Permeate TMP (mbar)	Average Backflushing TMP (mbar)
C (SF 2 mm)	50	39
D (SF 33 μm)	28	26

Figure 4.25 shows a TMP graph that is taken from control panel every five minutes from the first day of experiment. Positive number indicates backflushing pressure, while negative number indicates permeate pressure. Figure 4.25(a) indicates that treatment Train C consistently has higher TMP from the beginning of the experiment. There are three sudden peaks in treatment Train C's TMP, suggesting a rapid clogging, cake formation, or particle disposition on the membrane surface. Higher concentration of potential foulant from biological matters, solid, organic, and inorganic matter in treatment Train C could be a main cause of the sudden TMP increase in this system. However, this rapid fouling is only temporary, as as numerous high pressure backpulsing can decrease the TMP again to some extent.

On the contrary, treatment Train D only experience one sudden peak in the last quarter period of the experiment, as seen in Figure 4.25(b). This fact indicates that treatment Train D could be more cost effective in long-term operation because of the lower chance of rapid fouling in this system.



(a)



(b)

Figure 4.25: (a) TMP in treatment Train C (Day 0-96) (b) TMP in treatment Train D (Day 0-96)

Figure 4.25(a) also shows the slow increase of backflushing TMP from the start of the experiment. This is an indication of concentration polarization development on the initial period. This period is followed by slow, linear TMP rise which may correspond to an accumulation of EPS and other products of bioactivity, either deposited from the bulk liquor or produced in biofilms on the membrane surface (Miller et al., 2014; Zhang et al., 2006).

Further observation on Figure 4.26 and 4.27 suggest that TMP fluctuation in early days of treatment Train C and overall in treatment Train D generally follows MLSS concentration variation during the experiment. This means that EPS-bound that is produced and excreted by the microorganisms affects the degree of cake formation

in the membrane surface (Cosenza et al., 2013; Le-Clech et al., 2006; Zhang et al., 2006). Nevertheless, starting Day 30, a decrease of MLSS in treatment Train C did not proportionally followed by decrease of TMP. It is possible that fouling that occurred in treatment Train C is irreversible from Day 30, thus the TMP kept getting higher slowly regardless the change of concentration in MLSS. This phenomenon also observed in treatment Train D, although in a later time, specifically after Day 70. This means that in a long term operation, membrane in treatment Train C would have to be maintained more frequently with chemical cleaning rather than membrane in treatment Train D.

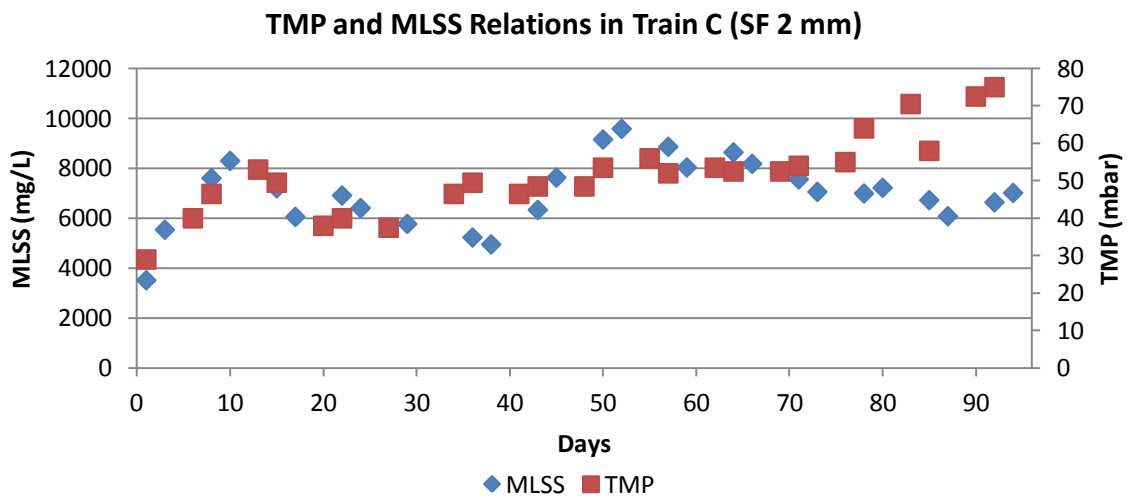


Figure 4.26: TMP and MLSS in treatment Train C (Day 0-96)

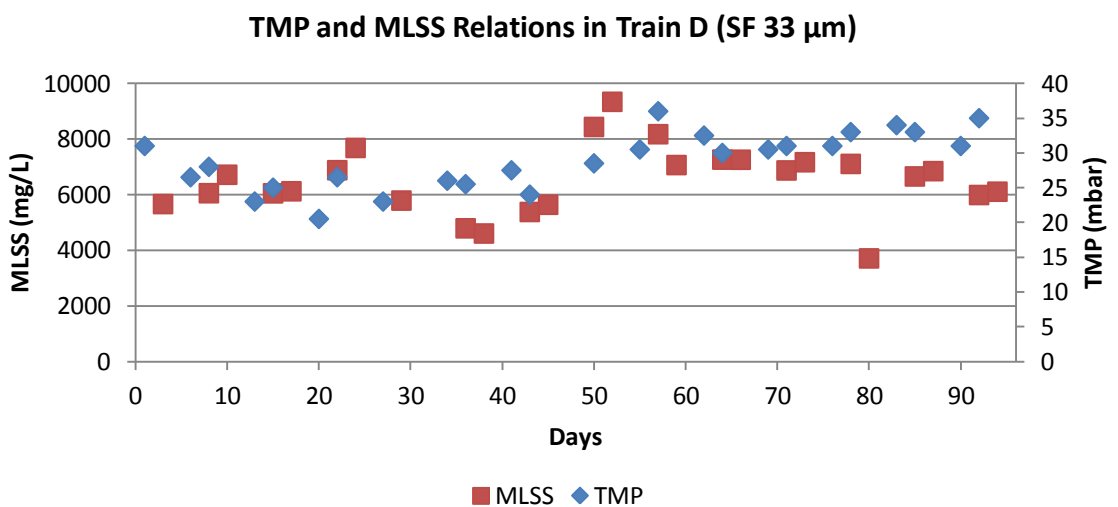


Figure 4.27: TMP and MLSS in treatment Train D (Day 0-96)

Overall membrane performance during the experiment was satisfying, with no excessive fouling observed, only four temporary rapid fouling. During 90 days of the experiment, membrane cleaning by chemical had not been practiced because the permeate TMP had not reached 300 mbar yet. Periodic backflush every 30 seconds for every 570 seconds of permeate flux is effective in preventing the build up of permanent cake on the membrane surface. Air scouring in MBR system also plays significant role in preserving the membrane integrity.

CHAPTER V

CONCLUSIONS AND FUTURE WORKS

5.1 Conclusions

This study investigated the performance of Salsnes Filter as primary treatment prior to hollow fiber membrane bioreactor for biological nitrogen removal. Two mesh sizes were investigated as a primary treatment, SF 2 mm (treatment Train C) and 33 μm (treatment Train D). Effluent from both treatment trains then compared and analyzed to determine which option provides the best solution for organic, nutrient, and solid removal. Nitrification, denitrification, and organic removal rate were also accessed, as well as the performance of the membrane. Two boundary conditions were used, the first investigated SF and MBR as the whole system, and the last reviewed the effect of different particle size to the MBR system.

For both boundary conditions, a combination between Salsnes Filter and HFMB was able to achieve high quality effluent. In both treatments trains, organic matter was successfully removed with 91-93 % and 98-99 % efficiency for COD and BOD₅, respectively. A higher actual SOUR than theory implicates that there were high biological activities in both systems, resulting in extensive utilization of organic matter and therefore high removal efficiency. Furthermore, solid removal consistently reached more than 99 % efficiency, mostly attributable to membrane filtration. Phosphorus removal was about 78-79 %, with bacterial cell assimilation accounts as major cause of the removal. All effluent concentrations mentioned above met the discharge requirement of urban wastewater set by Norwegian government.

Biological nitrogen removal process was hampered in treatment Train D, which causes only 66-68 % nitrogen removal compared to 73 % in treatment Train C. Lower TCOD/TN ratio in treatment Train D could be the main factor of low efficiency, as less COD concentration affects the availability of electron donors for denitrification process in the MBR system. Denitrification rate result also confirms this situation, where treatment Train D has lower denitrification rate than Train C. TN effluent concentrations fluctuated around 12-14 mg/L in both treatment trains, slightly higher than the discharge criteria.

While denitrification was hindered in treatment Train D, nitrification occurred successfully in both reactors, with 97-99 % $\text{NH}_4\text{-N}$ removal efficiency. Treatment Train C had slightly slower nitrification rate due to average bigger floc size in this system. Specific nitrification and denitrification rate results for both trains were comparable to the theory for similar operating condition. Nitrification and denitrification were also proven to be the main causes of nitrogen removal compared to biological cell assimilation.

The second boundary condition also proved that SF 33 μm provides additional contribution of parameter removal in the first stage of the treatment, with 20 %, 52 %, 4 %, and 38 % more removal than SF 2 mm for COD, BOD_5 , TP, and TSS, respectively.

HFMB used in the experiment has not been cleaned and replaced since its first usage. Extensive membrane fouling has not been observed, even though there were several sudden TMP increase as a result of rapid clogging in treatment Train C. Treatment Train D, on the other hand, can maintain the membrane integrity until the last days of experiment due to smaller fraction of organic and inorganic matters in its influent. Treatment Train C also suffers from higher TMP during the period of the experiment compared to treatment Train D. In a long term operation, membrane in treatment Train C would need a more frequent maintenance cleaning to overcome this performance problem.

5.2 Recommendations for future research

A future research, particularly an economic-related one, could be implemented to decide which option is the most cost effective choice for full scale plant, whether it is SF 2 mm (treatment Train C) or SF 33 μm (treatment Train D). Treatment Train C would need a more frequent maintenance cleaning, while treatment Train D would need an additional external carbon source or higher recycle ratio to overcome its denitrification problem. As the outcome of other parameters are similar, economic calculation of the problems above could determine the final choice of treatment.

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APPENDICES

A.1 Standard Methods for Examination of Water and Wastewater 2540 D Total Suspended Solids

1. General Discussion

a. Principle: A well-mixed sample is filtered through a weighed standard glass-fiber filter and the residue retained on the filter is dried to a constant weight at 103 to 105°C. The increase in weight of the filter represents the total suspended solids. If the suspended material clogs the filter and prolongs filtration, it may be necessary to increase the diameter of the filter or decrease the sample volume. To obtain an estimate of total suspended solids, calculate the difference between total dissolved solids and total solids.

b. Interferences: See Section 2540A.2 and Section 2540B.1. Exclude large floating particles or submerged agglomerates of nonhomogeneous materials from the sample if it is determined that their inclusion is not representative. Because excessive residue on the filter may form a water-entrapping crust, limit the sample size to that yielding no more than 200 mg residue. For samples high in dissolved solids thoroughly wash the filter to ensure removal of dissolved material. Prolonged filtration times resulting from filter clogging may produce high results owing to increased colloidal materials captured on the clogged filter.

2. Apparatus

Apparatus listed in Section 2540B.2 and Section 2540C.2 is required, except for evaporating dishes, steam bath, and 180°C drying oven. In addition: *Aluminum weighing dishes.*

3. Procedure

a. Preparation of glass-fiber filter disk: If pre-prepared glass fiber filter disks are used, eliminate this step. Insert disk with wrinkled side up in filtration apparatus. Apply vacuum and wash disk with three successive 20-mL portions of reagent-grade water. Continue suction to remove all traces of water, turn vacuum off, and discard washings. Remove filter from filtration apparatus and transfer to an inert aluminum weighing dish. If a Gooch crucible is used, remove crucible and filter combination. Dry in an oven at 103 to 105°C for 1 h. If volatile solids are to be measured, ignite at

550°C for 15 min in a muffle furnace. Cool in desiccator to balance temperature and weigh. Repeat cycle of drying or igniting, cooling, desiccating, and weighing until a constant weight is obtained or until weight change is less than 4% of the previous weighing or 0.5 mg, whichever is less. Store in desiccator until needed.

b. Selection of filter and sample sizes: Choose sample volume to yield between 2.5 and 200 mg dried residue. If volume filtered fails to meet minimum yield, increase sample volume up to 1 L. If complete filtration takes more than 10 min, increase filter diameter or decrease sample volume.

c. Sample analysis: Assemble filtering apparatus and filter and begin suction. Wet filter with a small volume of reagent-grade water to seat it. Stir sample with a magnetic stirrer at a speed to shear larger particles, if practical, to obtain a more uniform (preferably homogeneous) particle size. Centrifugal force may separate particles by size and density, resulting in poor precision when point of sample withdrawal is varied. While stirring, pipet a measured volume onto the seated glass-fiber filter. For homogeneous samples, pipet from the approximate midpoint of container but not in vortex. Choose a point both middepth and midway between wall and vortex. Wash filter with three successive 10-mL volumes of reagent-grade water, allowing complete drainage between washings, and continue suction for about 3 min after filtration is complete. Samples with high dissolved solids may require additional washings. Carefully remove filter from filtration apparatus and transfer to an aluminum weighing dish as a support. Alternatively, remove the crucible and filter combination from the crucible adapter if a Gooch crucible is used. Dry for at least 1 h at 103 to 105°C in an oven, cool in a desiccator to balance temperature, and weigh. Repeat the cycle of drying, cooling, desiccating, and weighing until a constant weight is obtained or until the weight change is less than 4% of the previous weight or 0.5 mg, whichever is less. Analyze at least 10% of all samples in duplicate. Duplicate determinations should agree within 5% of their average weight. If volatile solids are to be determined, treat the residue according to 2540E.

4. Calculation

where:

$$\text{mg total suspended solids/L} = \frac{(A - B) \times 1000}{\text{sample volume, mL}}$$

A = weight of filter + dried residue, mg, and

B = weight of filter, mg.

5. Precision

The standard deviation was 5.2 mg/L (coefficient of variation 33%) at 15 mg/L, 24 mg/L (10%) at 242 mg/L, and 13 mg/L (0.76%) at 1707 mg/L in studies by two analysts of four sets of 10 determinations each. Single-laboratory duplicate analyses of 50 samples of water and wastewater were made with a standard deviation of differences of 2.8 mg/L.

A.2 Standard Methods for Examination of Water and Wastewater 2540 E Fixed and Volatile Solids

1. General Discussion

a. Principle: The residue from Method B, C, or D is ignited to constant weight at 550°C. The remaining solids represent the fixed total, dissolved, or suspended solids while the weight lost on ignition is the volatile solids. The determination is useful in control of wastewater treatment plant operation because it offers a rough approximation of the amount of organic matter present in the solid fraction of wastewater, activated sludge, and industrial wastes.

b. Interferences: Negative errors in the volatile solids may be produced by loss of volatile matter during drying. Determination of low concentrations of volatile solids in the presence of high fixed solids concentrations may be subject to considerable error. In such cases, measure for suspect volatile components by another test, for example, total organic carbon (Section 5310). Highly alkaline residues may react with silica in sample or silica-containing crucibles.

2. Apparatus

See Section 2540B.2, Section 2540C.2, and Section 2540D.2.

3. Procedure

Ignite residue produced by Method 2540B, C, or D to constant weight in a muffle furnace at a temperature of 550°C. Ignite a blank glass fiber filter along with samples. Have furnace up to temperature before inserting sample. Usually, 15 to 20 min ignition are required for 200 mg residue. However, more than one sample and/or heavier residues may overtax the furnace and necessitate longer ignition times. Let dish or filter disk cool partially in air until most of the heat has been dissipated.

Transfer to a desiccator for final cooling in a dry atmosphere. Do not overload desiccator. Weigh dish or disk as soon as it has cooled to balance temperature. Repeat cycle of igniting, cooling, desiccating, and weighing until a constant weight is obtained or until weight change is less than 4% or 0.5 mg, whichever is less. Analyze at least 10% of all samples in duplicate. Duplicate determinations should agree within 5% of their average weight. Weight loss of the blank filter is an indication of unsuitability of a particular brand or type of filter for this analysis.

4. Calculation

$$\text{mg volatile solids/L} = \frac{(A - B) \times 1000}{\text{sample volume, mL}}$$
$$\text{mg fixed solids/L} = \frac{(B - C) \times 1000}{\text{sample volume, mL}}$$

where:

A = weight of residue + dish before ignition, mg,

B = weight of residue + dish or filter after ignition, mg, and

C = weight of dish or filter, mg.

5. Precision

The standard deviation was 11 mg/L at 170 mg/L volatile total solids in studies by three laboratories on four samples and 10 replicates. Bias data on actual samples cannot be obtained.

A.3. Standard Norge NS-EN 14702-1 for Characterisation of sludges – settling properties – Part 1 : Determination of settleability

I Scope

This document specifies a method for the determination of the settleability of sludge suspensions. This document is applicable to sludge suspensions from storm water handling, urban wastewater collecting systems, urban wastewater treatment plants, treating industrial wastewater similar to urban wastewater (as defined in Directive 91/271 EEC), and water supply treatment plants. This method is also applicable to sludge suspensions from other origin.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 872, Water quality — Determination of suspended solids — Method by filtration through glass fibre filters

EN 12880, Characterization of sludges — Determination of dry residue and water content

EN 1085:1997, Waste water treatment— Vocabulary

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 1085:1997 and the following apply.

3.1 settleability

ability of sludge solids to separate from water by sedimentation under gravity

3.2 settled sludge volume

volume of the sludge solids suspension after settling under specified conditions [7360, EN 1085:1997]

3.3 sludge volume index

sludge volume divided by the concentration of dry matter by mass in the sludge [7370, EN 1085:1997]

4 Principle

The settled sludge volume and the sludge volume index are determined by a 30 mm settling of a sludge suspension.

5 Interferences

In order to avoid modifying the settling process, the sludge/water mixture must not be too strongly shaken. The settling process can be disturbed by the walls of the vessel and the mutual interference between individual flocs, particularly when the proportion of sludge volume is high (greater than 250 ml/l). In such cases a new sample is prepared by dilution as described under 7.2. If the dissolved solids content is low and can be neglected in comparison to dry matter content, the total solids content should be determined and used for calculations. Interference also occurs when there are fairly

large temperature differences between the temperature of the sample and the ambient temperature as a result of convection and formation of gas bubbles. With differences of more than 5 °C it is advisable to place the settling cylinder with the sample in a bucket filled with the sample fluid.

6 Apparatus

6.1 graduated cylinder, nominal volume 1000 ml, made of glass or transparent plastic, diameter 60 mm to 70mm. NOTE : In cases where the sludge volume after 30 mm is less than 50 ml, an Imhoff cone of a volume of 1 L may be used.

6.2 scoop, nominal volume 1 L

7 Procedure

7.1 General

A representative sample of a sludge suspension is taken by a scoop and immediately poured into the graduated cylinder up to the 1000 ml mark. For this purpose, a scoop holding 11 up to the edge should be used; this avoids possible settling in the scoop. Once the sample has stood for 30 mm in one place without shaking, the sludge volume is read off at the surface level of the sludge (sludge-water interface). The determination shall be repeated if the sludge volume is greater than 250 ml/l. For this purpose, the new sample shall be first of all diluted with water taken from the standing water of a sludge suspension or from water run off from a settling basin, in a volume ratio 1:1, 1:2 or 1:3. The value then read off for the sludge volume is multiplied by the dilution factor 2, 3 or 4 for the evaluation. Homogenize the diluted sample by turning the closed cylinder two times overhead. In reporting the result, the dilution used is that at which the value goes below 250 ml/l for the first time. Determination shall be performed in duplicates.

7.2 Determination of solids concentration

7.2.1 If the total dissolved solids concentration is less than 10 % of total solids, the concentration by mass of dry matter in sludge (g/l) has to be determined following EN 12880.

7.2.2 If the total dissolved solids concentration is higher than 10 % of total solids, the concentration by mass of dry matter in sludge (g/l) has to be determined following EN 872.

8 Expression of results

The settled sludge volume V in ml/l is obtained as the sludge volume after settling divided by the volume of the initial sludge sample used. The sludge volume index is calculated from the equation:

$$SVI = \frac{V_s}{MLSS}$$

Where SVI = sludge volume index in milliliters per gram (mL/g)

NOTE 1 In technical literature this parameter is often named SVI.

V_s is the sludge volume in millilitres per litre (ml/l) after 30 mm settling, taken as an average of at least two measurements;

MLSS is the concentration of solids in sludge, in grams per litre (g/l)

NOTE 2 Values rounded to the nearest 10 ml/l are given for the proportion of sludge volume. If the sample has to be diluted, the sludge volume shall be read off in the diluted sample and the dilution factor shall be given in brackets after the reported value.

EXAMPLE 1 Original sample: Proportion of the settled sludge volume 180 ml/l

EXAMPLE 2 Diluted sample: Proportion of the settled sludge volume 510 ml/l (170 ml after 3 times dilution). Values rounded to the nearest 1 ml/g are reported for the sludge volume index.

EXAMPLE 3 Sludge volume index 145 ml/g

9 Precision

The repeatability standard deviation ranges from 0,066 ml/g (0,2 %) for digested sewage sludge, to 0,287 ml/g (1,2 %) for waterworks sludge, and to 4,370 ml/g (3,0 %) for activated sewage sludge.

Mean value is 1,574 ml/g (2,2 %). Minimum precision is 3,0%.

The reproducibility standard deviation ranges from 0,131 ml/g (0,3 %) for digested sewage sludge, to 0,52 ml/g (2,2 %) for waterworks sludge, and to 7,304 ml/g (5,1 %) for activated sewage sludge.

Mean value is 2,652 ml/g (3,8 %). Minimum precision is 5,1 %.