TREATMENT OF HIGH-STRENGTH ORGANIC WASTEWATER USING AN AEROBIC GRANULAR SYSTEM WITH BAFFLED MEMBRANE BIOREACTOR

by

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A thesis submitted in partial fulfillment of the requirement for the degree of Master of Engineering

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Abstract

Aerobic granular system was investigated to overcome disadvantages of conventional treatment system. Because of its prominent advantages, such as: high MLVSS concentration, good settling ability, etc. aerobic granular system could be applied for treating high strength wastewater. In this study, synthetic wastewater and basalts as carriers were used to cultivated aerobic granules in two kinds of reactor: Sequencing Batch Airlift Reactor (SABR) and Sequencing Batch Bubble Reactor (SBBR). The results showed that at OLR of 3 kgCOD/m^3.d, aerobic granules were formed simultaneously in both SBAR and SBBR within 1.5 months. In the next step aerobic granular system was increased OLR to 20 kgCOD/m^3.d to estimate the maximum and optimum OLR that system could suffer. It could be realized that after OLR exceed 20 kgCOD/m^3.d, aerobic granules were unstable and easily worn. Only at OLR of 15 kgCOD/m^3.d, aerobic granular system demonstrated stability and good treatment ability.

Experimental data proved that settling ability of aerobic granules was 10 fold better than that of activated sludge (SVI of aerobic granules was less than 25 mL/g, while that value in activated sludge was 220 mL/g). Moreover at optimum OLR aerobic granular system always maintained at MLSS of 11230 and 12780 mg/L in SBAR and SBBR, respectively. Matured granule size could reach to 1.5 mm in SBAR and 1.7 mm SBBR, and then it proportionally varied with the increase of OLRs. In term of compactness, settled biomass concentration displayed that aerobic granules posed high biomass concentration. At OLR 17.5 kgCOD/m^3.d, settled biomass concentration was 47.8 g/$L_{granule}$ in SBAR, and 53.7 g/$L_{granule}$ in SBBR. Although SBBR always presented better aerobic granulation, it predicted to cause more fouling than SBAR through bound EPS results.

The second objective of this study was to characterize effluent from aerobic granular system to evaluate fouling potential. By monitoring MLSS, EPS and MFI, it could be concluded that effluent from SBBR cause membrane fouling easier than that from SBAR.

The third objective of this study was to monitor fouling behavior of PVDF flat sheet membrane (pore size of membrane was 0.1 μm) in Baffled Membrane Bioreactors (MBR) connected to aerobic granular system. During 1.5 months of operation including 4 cycles, together with investigating EPS, MFI, it was concluded that bound EPS had influence on cake layer fouling whereas soluble EPS had significant impact on irreversible fouling.

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List of Abbreviations

COD	Chemical Oxygen Demand
EPS	Extracellular Polymeric Substances
HRT	Hydraulic Retention Time
MBR	Membrane Bioreactor
MF	Micro-filtration
MFI	Membrane fouling index
MLSS	Mixed Liquor Suspended Solids
MLVSS	Mixed Liquor Volatile Suspended Solids
NF	Nano-filtration
OLR	Organic Loading Rate
PN	Protein
PS	Polysaccharides
SBR	Sequencing Batch Reactor
SBAR	Sequencing Batch Airlift Reactor
SBBR	Sequencing Batch Bubble Reactor
SVI	Sludge Volume Index
TMP	Transmembrane Pressure
WW	Wastewater

Chapter 1

Introduction

1.1 Background

Dealing with industrial wastewater is still one of the greatest obstacles for manufacturers and environmentalists nowadays. For along time, biological process has been a major part in wastewater treatment systems, in which aeration unit is the most popular. However day by day these conventional treatment processes expose their limits. Microorganisms exist in the form of separated and fine flocs that make activated sludge have low biomass concentration and bad settling ability. This results in low volumetric loading rate (0.5 – 2 kgCOD/m^3.d) and requires large area for clarifiers that is not favorable in somewhere that is land is unavailable or high price. Thus requirement of novel technology for industrial wastewater treatment is necessity than ever.

From 1990s, many researchers have tried to overcome disadvantages of conventional activated sludge process, and aerobic granules were developed as one of optimal alternatives. Aerobic granular sludge in many ways are advantageous compared to that of conventional activated sludge in terms of regular and condensed microbial structure, good settling ability, high biomass retention and ability to withstand high organic loading rate (Tay et al., 2002). Besides they present excellent ability in resistance to shock loading and inhibitory or toxic compounds. Reactor configurations as well as operation option significantly contribute to aerobic granule formation; sequencing batch airlift reactor or bubble column reactor showed excellent ability in aerobic granulation (Beun et al., 1999) because of theirs good mixing condition by convection current in airlift reactor and completely turbulent in bubble reactor. Granules can be formed with or without carrier. However carrier material had more advantages due to rough surface which results in good potential for biofilm development. Study conducted by Tijhuis, et al.,1994 suggested the application of basalt as the support media. In sequencing batch airlift reactor granule size with bivalve shell as carrier can reach up to 4 mm with sludge volume index of 30 mL/g, and can stand at volumetric loading rate of 30 kgCOD/m^3.d (Thanh, 2004). In short word, aerobic granule reactor not only expresses high treatment ability but also compacted.

Effluent standard which is more stringent day by day and limitation of fresh water put more pressure on water usage of manufacturers. Many approaches have been applied to reduce water supply or reuse wastewater, and membrane bioreactor (MBR) appeared as one of the most efficient and easy applying approaches for reusing and reclaiming industrial wastewater. Applied after activated sludge processes, MBR could maintain high volumetric loading rate, good effluent quality, and less space requirement. However membrane fouling behavior reduces wide application of this technique. To overcome disadvantage or last operating time, baffled membrane bioreactor was proposed and investigated in this study. The renovation of settling unit combining with membrane put in the last chamber made solid-liquid separation process more efficient. This reactor could be an attractive application of membrane bioreactor that allows suspended solids settle which reduce fouling behavior and clarifies supernatant of aerobic granulation. This application had ability to maintain advantages of MBR and overcome its disadvantages, and will be a novel and promising process for handling industrial wastewater.

From the above advantages of both aerobic granules and baffled MBR, this study tried to find out the new technique for the applications of granule MBR in field of industrial

wastewater treatment. In this study, SBAR and SBBR were used to cultivate aerobic granules with synthetic wastewater and then effluent was further treated in baffled MBR and investigated fouling behavior.

1.2 Objectives of Study

The main objectives of this study focused on investigating aerobic granules, as well as supernatant, in two types of reactors and then coupling with baffled membrane bioreactor. These include:

- Cultivate aerobic granules in airlift and bubble column reactors with basalt as carrier.
- Characterize granules and effluent of above reactors at different loading rates.
- Coupling aerobic granule system with baffled membrane bioreactor to investigate the fouling potential.

1.3 Scope of Study

Airlift and bubble column reactor were chosen to form aerobic granules. Synthetic wastewater with glucose as sole carbon source simulating industrial wastewater was used for whole experiment. During granulation process, similar operating conditions were maintained in two reactors, such as: cycle time of about 3 hours/batch, COD influent, airflow rate of 4L/min. After aerobic granules appeared, some basic parameters were investigated to evaluate aerobic granules characteristics, such as: morphology (mean diameter), biomass concentration, biomass density, hydrophobicity, extracellular polymeric substances, and sludge volume index.

For further treatment after aerobic granule reactor, membrane bioreactor combined with baffled reactor was used to achieve high solid-liquid separation, and also further substrate removal. By this mean baffled membrane bioreactor could reduce significantly membrane fouling behavior. Parameters to be analyzed to examine fouling behavior were initial membrane resistance, membrane fouling index (MFI), extracellular polymeric substances (EPS), and transmembrane pressure.

- Cultivation of aerobic granules at a starting organic loading rate of 3 – 4 kg COD/(m^3.day) in airlift and bubble column reactor with basalts as a support media;
- Investigation of increasing organic loading rate with matured aerobic granules;
- Investigation of effluent characteristics from both aerobic granule reactors;
- Investigation of fouling behavior of aerobic baffled MBR.

Chapter 2

Literature Review

2.1 Introduction

Continuous depletion of fresh water nowadays forces environmental administrators shift from safely discharge into receiving body toward reuse and/or recycle treated wastewater. Furthermore the more industry develops the higher strength of organic matters in wastewater. Therefore, conventional treatment systems which have low organic loading rate (0.5 – 2 kgCOD/m^3.d) could not meet new requirement. To satisfy both dealing with higher strength industrial wastewater and reusing it, an alternative should be investigated.

More and more environment researches focus not only on increasing capacity but on compacting industrial wastewater treatment system. One of considerable innovations that can break through a new trend in wastewater treatment is application of aerobic granules. Tijhuis et al., 1994-a; Beun et al., 1999 & 2002; Tay et al., 2001 & 2002; Arrojo et al., 2004; Wang et al., 2004; Jang et al., 2003, Qin et al., 2004; Linlin et al., 2005; Liu et al., 2003; Yang et al., 2004; Zheng et al., 2004; Toh et al., 2003 were some early authors who found out application of aerobic granules in treating high strength wastewater. Effectively granular growth is just a special case of biofilm growth in which mature granules can stand for high organic loading rate, toleration with toxic substances and high settling ability. Granulation is not restricted to methanogens, granulation by acidifying bacteria, nitrifying bacteria, denitrifying bacteria, and aerobic heterotrophs have been observed. It is regularly hypothesized that the specific bacterial interactions are the main cause of the granulation. In aspect of operation system, discontinuous systems have more advantages than continuous one in cultivating aerobic granules (Beun et al., 1999 & 2002; Tay et al., 2004). Sequencing Batch Airlift Reactor (SBAR) and Sequencing Batch Bubble Reactor (SBBR) are preferred due to theirs good mixing conditions and high shear stress which are supposed as the main factors for aerobic granulation process.

Membrane bioreactor has become the most famous and convenient in treating or reusing industrial wastewater. However fouling behavior prevents further development of this new technology. This problem could be solved easily if membrane bioreactor is coupled with aerobic granulation whose effluent is low surplus biomass production. In addition baffled reactor could reduce considerable amount of suspended solid before wastewater comes to membrane bioreactor. Hence, this excellent but simple combination overcame disadvantages of membrane bioreactor.

This chapter reviews the literature on following area: aerobic granule formation and its characteristics, stimulating or inhibiting factors, reactor configurations for aerobic granulation; and MBR and its application in reclamation industrial wastewater.

2.2 Aerobic Granular Sludge Production

There are a number of factors affecting to formation of aerobic granules which can be considered as a complicated process. From the point of micro view, aerobic granulation is generally influenced by both physicochemical and biological processes. Besides it is insufficient if materials required for aerobic granulation, such as seed sludge, carbon source, and support material, are not mentioned. This part will review factors considered as initial requirements.

2.2.1 Cell Immobilization

Biofilm and granular sludge indeed can be regarded as different forms of cell immobilization. Hence it is essential to have a full understand of cell immobilization involving categories and formation process. Generally, cell immobilization can be roughly classified into three categories:

- Biofilm: microorganisms are immobilized or attached onto a solid surface, such as activated carbon, basalts, plastics, polymers, ceramics and others (Kwok et al., 1998).
- Microbial aggregates and granular sludge: microbial granulation can be regarded as a self-immobilization community of bacteria. Aerobic and anaerobic granules have been successfully developed (Beun et al., 1999; Tay et al., 2001).
- Entrapped microorganisms: microorganisms may be entrapped in hydrophobic gels of photo-crosslinked polymers or in other types of gels, such as polyacrylamide (Myoga et al., 1991).

Cell immobilization technology has been used in bioengineering and environmental engineering areas for decades. So far, it has been recognized that the formation of biofilms and microbial aggregates is a multiple-step process, to which physicochemical and biological forces make significant contributions (Beun et al., 1999). Based on previous studies, cell immobilization can be roughly described as a four-step process as follows (Liu et al., 2002):

Step 1: Physical movement to initiate cell-to-cell contact or bacterial attachment onto a solid surface. The forces involved in this step are:

- Hydrodynamic force
- Diffusion force
- Gravity force
- Thermodynamic force
- Cell mobility

Step 2: Initial attractive forces to keep stable bacteria solid surface and multicellular contacts. Those attractive forces are:

- Physical forces including Van der Waals forces, electrostatic forces, thermodynamic forces, hydrophobicity, and forces between filamentous bacteria.
- Chemical forces including hydrogen liaison, formation of ionic pairs, formation of ionic triplet, interparticulate bridge and so on.
- Biochemical forces including cellular surface dehydration, cellular membrane fusion.

Step 3: Microbial forces to make attached bacteria or aggregated bacteria mature

- Production of extracellular polymer such as exopolysaccharides etc.
- Growth of cellular cluster.
- Metabolic change and genetic competence induced by environment, which facilitate and further strengthen the cell-cell interaction and result in the high density of adhering cells.

Step 4: Steady state three-dimensional structure of microbial aggregate shape by hydrodynamic shear force. The outer shape and size of microbial aggregates are finally determined by the interactive strength between aggregate and hydrodynamic shear force, microbial species, and substrate loading rate and so on.

2.2.2 Seed Sludge

Many studies have proved that granules can be cultivated with seed sludge from aerobic sludge (Beun et al., 1998 & 2002; Tay et al., 2001; Jang et al., 2003; Arrojo et al., 2004; Wang et al., 2004; Qin et al., 2004; Schwarzenbeck et al., 2004) or from anaerobic sludge (Linlin et al., 2005). In the other word, seed sludge is not the limitation of cultivating aerobic granules.

2.2.3 Carbon Source

Aerobic granulation process can operate with synthetic with acetate or glucose as sole carbn source (Beun et al., 1999, 2002; Jang et al., 2003; Wang et al., 2004) or real industrial wastewater (Arrojo et al., 2004) which has different kinds of carbon sources. This means that granule can be formed with any biodegradable carbon sources, even with high suspended solid wastewater (Schwarzenbeck et al., 2004).

2.2.4 Support Media

Most of aerobic granulation researches concentrated on formation mechanisms, as well as characteristics of aerobic granules, and less examination on role of support media. Actually, an effective way of increasing the settling ability and enhancing the formation of the sludge granules is the use of support media, which act as carriers. Furthermore the carrier material has a rough surface which results in good potential for biofilm development. Various support media with different properties (listed in table 2.1), such as feature/structure, porosity, or inorganic portion, etc., have certain impacts to formation of aerobic granules.

Recent researches have been investigated role of carriers such as basalt (Tijhuis et al, 1994), anaerobic sludge (Linlin et al., 2004), bivalve shells (Thanh, 2004), etc. However fully understand function of carriers on aerobic granulation has not been examined.

Study conducted by Tijhuis, et al (1994), suggested the application of basalt as the support media, which is found solidified lava, a type of igneous rock mainly comprising of calcium rich feldspar and pyroxene. Calcium which is the main component of bivalve shells and basalt is supposed to play an important role in cultivation of aerobic sludge granule (Wang et al, 2004). In addition, other essential amount of inorganic components (K, Na, Mg, Fe, etc.) considerable contributes to aggregation of microorganism in aerobic granules (Thanh, 2004).

Table 2.1 Type of studied support media (modified from Harendranath et al., 1996)

Support materials	Morphological features	Macro-structure	Porosity	Surface area (m^2/ m^3)	Reference
Smooth					
P.V.C	Even surface; pore absent	Size, shape variable	0.96	140	Kenedy & Droste, 1983
P.A.C	Smooth surface; pores absent	Fine power	-	-	Ng et al, 1988
Porcelain	Smooth surface even; pore absent	Size, shape variable	-	-	-
Perspex					
Uneven					
Nylon	Densely distributed	Size, shape variable	-	-	-
Stone	Highly corrugated with randomly distributed crevices	Amorphous	0.42-0.53	-	Henze & Harremoes, 1983
Sand	Crests and troughs	~ 0.7 mm	0.38-0.43	2500-4000	Henze & Harremoes, 1983
Sintered glass	Rough, jagged surface, shallow pore up to 20μm width, loosely distributed	Open-pore structures	0.57	90,000	Anderson et al., 1994
Porous					
Basalt	Rough surface	0.26 mm	-	1150	Tijhuis et al., 1994
Bivalve shell (*)	Crests and troughs with deep pores, 10μm with densely distributed	Concave, convex	0.77-0.82		Henze & Harremoes, 1983
Granulated clay	A moisiac of particulates, 1-10μm width, pores of 5μm width, uniformed distributed	Amorphous aggregate	~ 0.7	53-397	Henze & Harremoes, 1983
Gravel	Pores of 5μm, loosely distributed, irregular ridges present	Amorphous	0.4	-	Henze & Harremoes, 1983
Ceramic	Thorny surface with polygonal pores 1-10μm width	Variable size and shape	0.6	274	Cordora & sinerriz, 1990
Refractory bridge	Crystalline, pointed structure and pores, densely distributed	Amorphous	-	149	Henze & Harremoes, 1983
Diatomaceous earth	Deep, minute pores 5μm width, densely distributed,	Variable size and shape	-	-	-
GAC	Rough surface with 3 dimensional pore distribution, pore size up to 250μm	Amorphous	0.6	5469	Henze & Harremoes, 1983
Limestone	Highly porous structures, composed of crystalline unit 2-5μm length, densely distributed deep pores 10 μm width	Variable size and shape	0.49	5,000 – 10,000	Henze & Harremoes, 1983
Sponge	Labyrinth of pores 200-500μm width, hexa or pentagonal in shape	Highly compressible variable size and shape	-	-	-

2.2.5 Reactor Configuration

Previous aerobic granulation researches showed that granules can be formed in batch system such as Sequencing Batch Airlift Reactor (SBAR), Sequencing Batch Reactor (SBR) and continuous system such as Biofilm Airlift Suspension Reactor (BAS). However most of researches favored batch system when culturing aerobic granules due to its good mixing conditions and easiness in control selection variables (HRT or settling time). SBAR is also preferred because of its simple and compacted design (high ratio of height to diameter – H/D). Common time to get mature granules was about 50 days. The comparison among types of reactor to cultivate granule showed in the table 2.2.

Table 2.2 Comparison between SBAR, SBBC and BAS

	SBAR (Beun et al., 2000)	**SBBC (Beun et al., 1999)**	**BAS (Tijhuis et al., 1994)**
System	Discontinuous	Discontinuous	Continuous
External clarifier	No	No	No
Riser	Need	Need	3 phase separator
Carrier	No	No	Yes
Selection variable	Settling time	Settling time	HRT
Detachment mechanism	Hydrodynamic conditions	Hydrodynamic conditions	Bare carrier conc.
Nitrification and denitrification	Yes	-	No denitrification
Granule density	ρ = 48g/Lgranules	ρ = 12g/Lgranules	ρ = 15g/Lgranules
Granule diameter	d = 1.0 mm	d = 2.0 mm	d = 0.350 mm ($d_{carrier}$ = 0.26 mm)

SBAR: Sequencing batch airlift reactor; SBBC: sequencing batch bubble column; BAS: Bio-film airlift suspension reactor;

From table 2.2, SBAR is more efficient because it can create granule with dense, smaller size so this type is suitable for this study. Another advantage in application of SBAR is old conventional activated sludge process can be upgraded for treatment improvement.

Table 2.3 compared required factors for aerobic granulation, i.e. carbon source, reactor type and formation time, from various researches.

Table 2.3 Effects of carbon source and reactor type on aerobic granulation

Author	Carbon source	Reactor type	Formation time
Beun et al., 2000	Acetate	SBAR	50 days
Beun et al., 2002	Acetate	SBAR	> 63 days
Etterer & Wilderer, 2001	Acetate; glucose & peptone	SBR	56 days
Jang et al. 2003	Glucose and acetate	SBR	50 days
Linlin et al., 2004	Acetate	SBR	50 days
Tay et al., 2002	Acetate	SBR	Cycle 42; 4h/cycle
Wang et al., 2004	Glucose	SBR	67 days
Tijhuis et al., 1994	Acetate	BAS	-

2.3 Aerobic Granular Sludge Formation

There are many hypotheses for aerobic granulation from different researches. Whether seed sludge from aerobic activated sludge or anaerobic sludge, generally matured granules spent 3 stages: acclimation, granulation, and maturation. Followings briefly describe aerobic granulation process from activated sludge to matured granules which was found out by many authors.

Wang et al. (2004) found that granules were first initiated as mycelial pellets in the reactor and began to accelerate growth, the 'granules initiated'. The corresponding period from the start-up operation to the 'granules initiated' was the sludge acclimation phase. The granulation phase was that corresponding from the 'granules initiated' to the granules matured point. In the other words, the granulation process initiated form the appearance of granules in the reactor. After reaching granule matured point, the granules were stable and dynamically balanced in the maturation phase. In this phase, the granular size was stable and changed slightly depending on operational conditions.

From the point of microbial view, the sludge inoculated in the SBR was a mixture of filamentous sludge with brown color, loose structure and difficult in settling. Since the settling time was kept short during operation, a washout of biomass took place. The sludge concentration in the reactor decreased from this wash out and more sludge was observed in the effluent because of bad settling ability. During this time, most of the sludge in the reactor changed to flocs. Over the next 56 days, the floc-like sludge gradually changed to granular sludge. After 67 days of operation, granular sludge began to appear whereas flocs still remained dominant in the reactor. The initial granular sludge with fluffy edges and small size were formed in the SBR. The small granules grew rapidly in the following weeks, while more floc-like sludge was washed out, resulting in the accumulation of the granules. Eleven weeks after inoculation, the sludge in the reactor was nearly completely granulized, and visually no suspended biomass was present. Due to the intensive mixing by aeration, the granular sludge became spherical with a smooth surface. The diameter of the

granular sludge increased to 6.0–9.0 mm. Most of the biomass was kept in the reactor due to the good settleability. The microscopic and SEM view of matured granules from Wang's research are showed in figure 2.1.

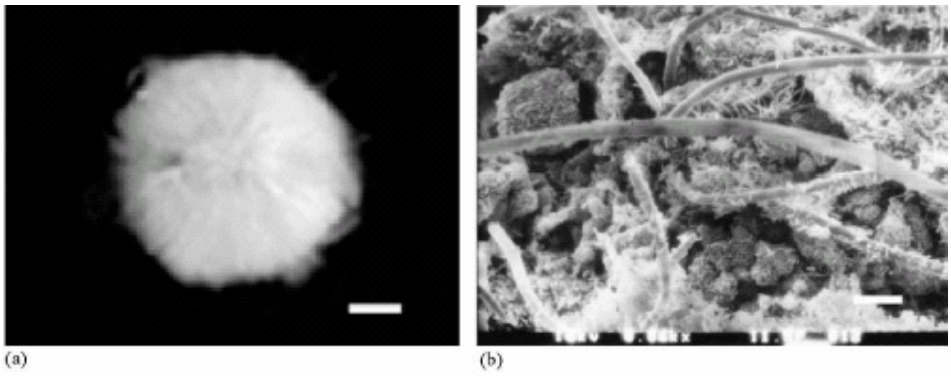


Figure 2.1 Microscopy images of mature granules after 120 days. (a) Microscope overview image, bar = 2 mm, (b) SEM of the granules surface, bar =11 µm.

The granule formation process in Wang's can be described as the figure 2.2.

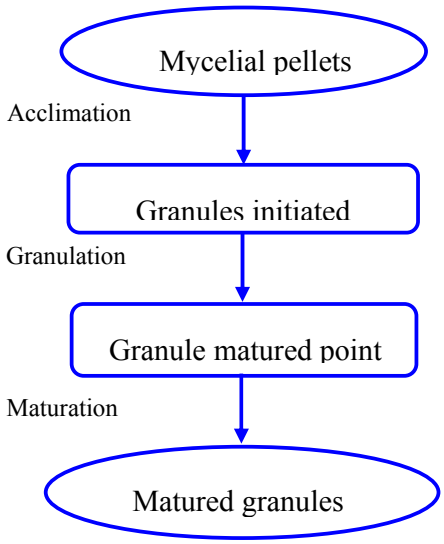


Figure 2.2 Formation process of aerobic granules (Wang et al., 2004).

Similar formation mechanism of aerobic granules, Jang et al., (2003) described granulation base on both microbial and physicochemical aspects (from activated sludge to matured granules). Firstly, the seed sludge was not in the form of large flocs, rather irregular and unstable filaments were dominant. The initial seed sludge for the SBR operation had size of 0.08-0.18 mm and SVI of 210-230 ml/g. The particles eventually started to join together to form biomass aggregates and the aerobic floc-like sludge form was accomplished within 10 days. Secondly, the aerobic floc-like sludge was heterogeneous mixed, with irregular and soft granules that started to appear around 30 days. Granulation of the seed sludge can be achieved through accumulation by interparticle bridging under a condition of turbulent flow mixing. The floc-like sludge changed gradually to granules over time. After 40 days

of operation, the aerobic floc-like sludge formed, the seed sludge in reactor was nearly granularized. At this time most of granules had an uneven surface and soft texture. Finally, the irregular granules became stable and smoother, round-shaped with a solid surface. After 50 days, granules formed with the size of 0.95-1.35 mm and SVI of 70-90 ml/g. Figure 2.3 illustrate the granulation process of Jang's research.

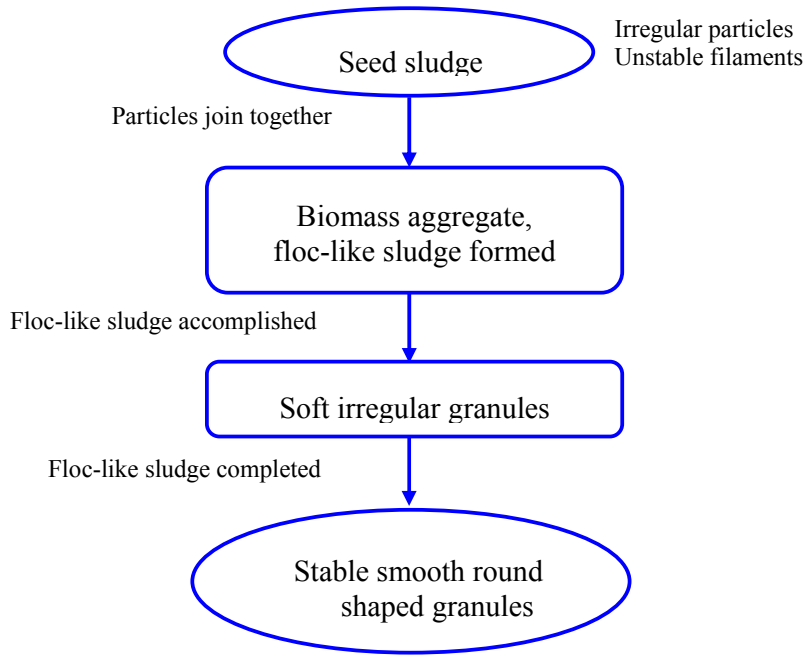


Figure2.3 Formation process of aerobic granules (Jang et al., 2003).

The morphological development from seed sludge to granules in Jang's research was recorded in figure 2.4.

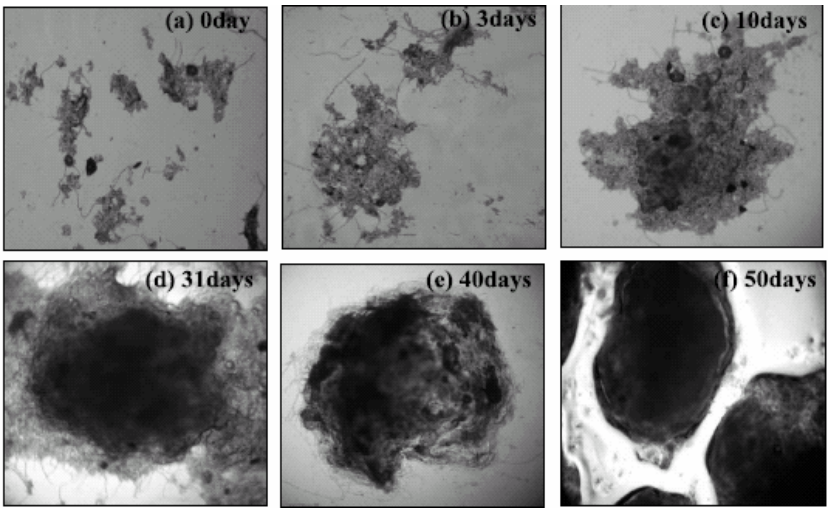


Figure 2.4 Time dependent development of granules, extending from the seed sludge to granules, of: (a) 0 day, seed sludge; (b) 3 days; (c) 10 days; (d) 31 days, flocs-like; (e) 40 days and (f) 50 days, granular sludge (Jang et al., 2003).

Although different in formation time, granulation in Etterer and Wilderer's research, (2001) also experienced in 3 main phases. They found that when keeping the short settling time, biomass in the SBR was washed out during the start-up period. First, the filamentous granules appeared after 10 to 15 days whereas flocs still remained dominant. In the following weeks granules accumulated; and three or four weeks after inoculation, biomass in reactor consisted of mainly aerobic granules. Due to the intensive mixing with suitable hydrodynamic shear force by aeration, granules became spherical with smooth surface. Besides, it indicated that fungi and filamentous organisms in general were present in the overall structure of the aggregates when observing granules by light microscope. However when using fluorescent in situ hybridisation (FISH), only filaments were possibly detected. The formation process of granules by these authors is shown in figure 2.5.

Based on the microscopic observations, Beun et al. (1999 & 2002) suggested a detailed description about the mechanism of aerobic granulation as the following scheme.

After inoculation with bacterial sludge, fungi become dominating. Fungi easily form mycelial pellets, with well settling ability and can be retained in the reactor. Bacteria do not possess that property and will be washed out almost completely. Therefore, during the start-up, the biomass in reactor will consist of mainly filamentous mycelial pellets. Due to the shear in the reactor, detachment of the filaments on the surface of pellets takes place and the pellets become more compact. The pellets grow out to diameter of 5-6 mm and then they lyses probably due to oxygen limitation in the inner part of the pellets. The mycelial pellets seem to function as an immobilization matrix in which bacteria can grow out to colonies. When the mycelial pellets fall apart due to lyses of inner part of the pellets, the bacterial colonies can maintain themselves because now they are large enough to settle. These microcolonies further grow out to granules, leading to eventually to bacterial dominated population in the reactor.

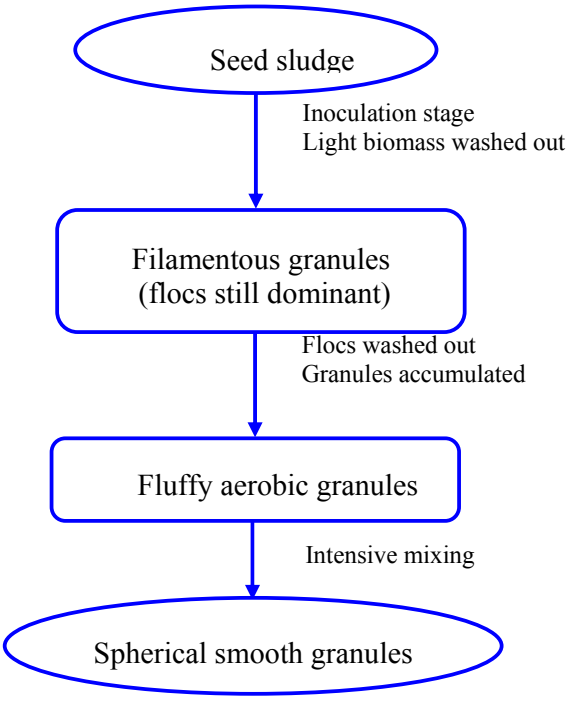


Figure 2.5 Formation process of aerobic granule (Etterer and Wilder, 2001).

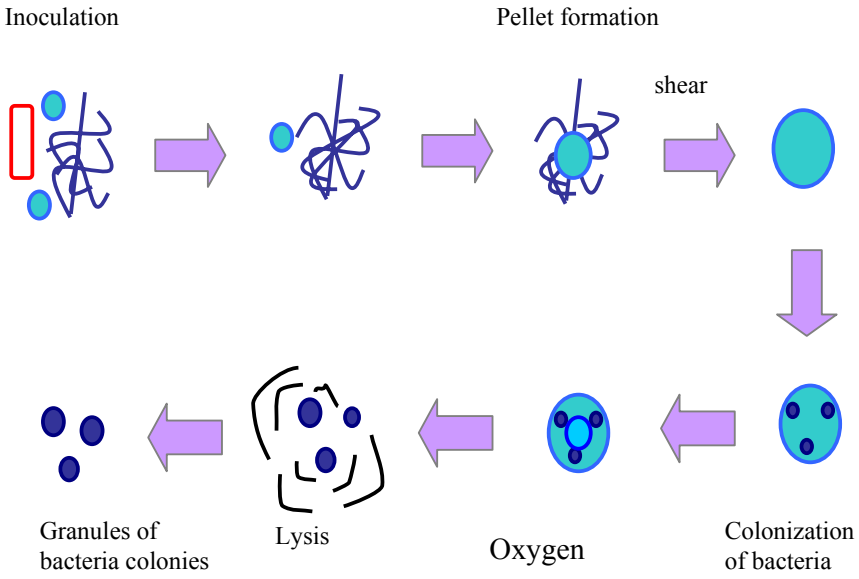


Figure 2.6 Mechanism of granulation of aerobic granule (Beun et al., 1998).

From some of typical above researches about aerobic granulation, it could be recognized that in spite of a little bit difference in flocs morphology and granulation time, matured aerobic granules require 3 basic phases, i.e.: acclimation, granulation, and maturation. Based on this clarification, it will easy to follow, as well as evaluate granulation process.

2.4 Characteristics of Matured Aerobic Granules

Aerobic granules are supposed as a suspended spherical biofilm includes inert particles, degradable particles, microbial cells and extracellular polymeric substances (EPS). Aerobic granules compared with activated sludge have more advantages in term of settling ability, treatment ability, nitrification and denitrification, biomass concentration, etc. Due to these advantages, aerobic granules can operate with high strength wastewater, stand with shock loading and easy to separate from liquid. This part will regard to some typical characteristics of matured aerobic granules.

Granule Size

Granule size is a direct parameter to evaluate the growth and aging process in the microbial organizations. Mature granule size can vary from 0.5 to 9 mm. The granule size is very important for substrate, nutrients, oxygen accessibility and product releasing, which also has great impact on microbial viability, microenvironment and the microstructure of the microbial community. It also plays a significant role in the limitation of mass transport and diffusion, due to porosity in the granular structure, which diminish with the increase in size and age. Hence, granule size is the eminent factor in molding the physical performance and characteristics of aerobic granules (Linlin et al., 2004).

Density of Granules

Density of granules is equal to density of discrete bacterial cells but granules show much better settling properties because of their larger sizes (Guiot et al., 1993). So far, granule density, specific gravity and its size have a close relationship. However granules at rather big size can not maintain dense and strong structure that could make its density reduce.

Specific Gravity

In term of physical properties, specific gravity of the aerobic granules reflects the compactness of microbial community. The significant improvement of specific gravity for granular sludge indicated its highly compact structure (Tay et al., 2002). Activated sludge has density of 1.0008 kg/L, and matured granules could reach to an average density value of 1.0069 kg/L.

Settling Velocity

One of remarkable advantages of aerobic granules compared with activated sludge is high settling ability, which could compact treatment system. The settling velocity of cultivated aerobic granules is in the range of 22-60 m/h, the average is 38.4 m/h, compared with 72 m/h for anaerobic granules. Another parameter that is often used to assess settling ability of granules and activated sludge is SVI. Granules have SVI varying from 30 to 80 mL/g (Beun et al., 1999 & 2002; Tay et al., 2002), while SVI of activated sludge normally greater than 120 mL/g. However settling velocity decrease when granule size grows corresponding to the fact that water content in aerobic granule increases (Linlin et al., 2004).

Water Content

The water content in inoculated anaerobic granules is 92.7%; the water content in cultivated aerobic granules is about 94.3% (Linlin et al., 2004).

Biomass concentration

Difference with specific gravity which shows physical property, biomass concentration expresses density of biomass in matured granules which shows biomass compactness in term of biological view. Measured by settled biomass concentration, it could vary from 2.7 to 40g/$L_{granules}$ (Beun et al., 1999 & 2002). Obviously, the higher settled biomass concentration, the higher treatment ability. Biomass concentration proportionally increases with OLR.

VSS/SS Ratio

VSS/SS ratio shows the relative biological component. Compared to the compact granules, the loose and amorphous structure of flocs allowed them to have better access to nutrient and oxygen. Hence, these flocs contained a higher portion of active biomass which is reflected as higher VSS/SS ratios (Tay et al., 2005). It means that VSS/SS ratio in flocs is higher than that in aerobic granules. This ratio in flocs raise from 0.85 to 0.92 (Linlin et al., 2004), while in aerobic granule this ratio varied from 0.71 to 0.87; and in anaerobic granules this ratio is 0.57.

Biological Characteristics

Granulation is not restricted to methanogens, granulation by acidifying bacteria, nitrifying bacteria, denitrifying bacteria, and aerobic heterotrophs have been observed. Wang et al., 2004 found that rod bacteria were predominant in granules, and lots of cavities were present in matured aerobic granules. These cavities can enhance substrates transfer from the bulk liquid to granules and intermediate or by-product, product easily transfer form inside granules to the bulk (Tay et al., 2002). Thanh, 2005 proved that cocci, rod shape

bacteria, fungi, filamentous organisms coexisted in structure of granules in which rod shape and cocci bacteria were dominant. Microbial aggregation was linked together by coweb-like material.

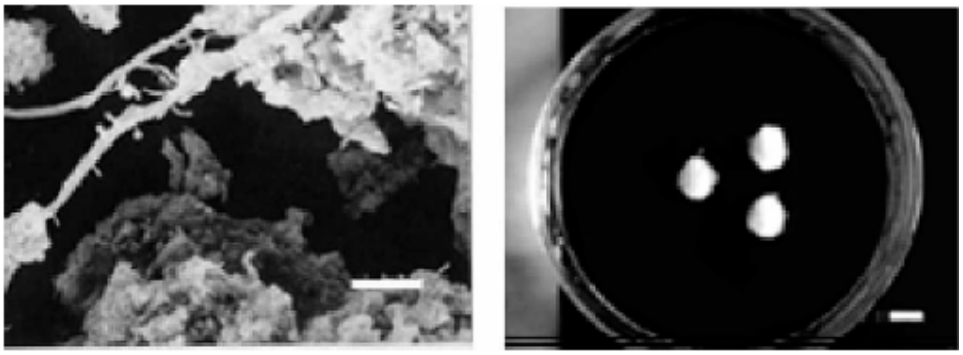


Figure 2.7 Microscopy image of the seed sludge (left), bar = 5 µm, filamentous sludge; aerobic granules (right) at steady-state, bar = 8 mm (Wang et al., 2004).

Table 2.4 below shows some main characteristics of matured granules in previous researches.

Table 2.4 Characteristics of matured granules

Author	**Granule diameter** (mm)	**SVI** (mL/g)	**Biomass density** ($gVSS/L_{granule}$)
Beun et al., 2000	1		48
Beun et al., 2002	2.5	-	60
Etterer & Wilderer, 2001	1.1-6.5	-	-
Jang et al. 2003	1-1.3	70-90	-
Linlin et al., 2004 (*)	1.2	30-40	-
Tay et al., 2002	0.35	50	-
Wang et al., 2004	6-9	40	-
Tijhuis et al., 1994	0.5		-

Comparison between aerobic granule sludge and conventional activated sludge

Granular sludge has more advantages than conventional activated sludge. Figure 2.8 show characteristic comparison between granular sludge and floc-like sludge.

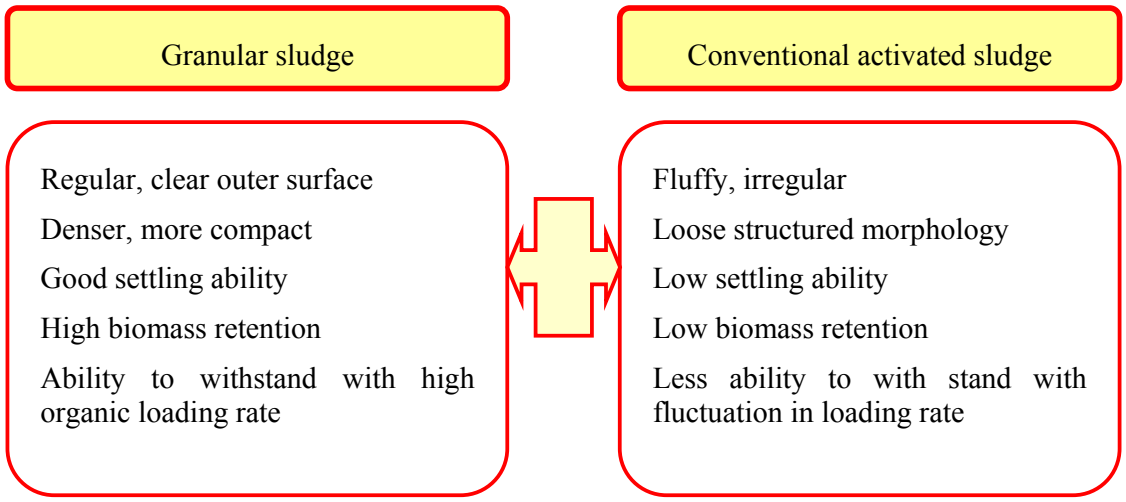


Figure 2.8 Characteristics of granular sludge and conventional activated sludge.

2.5 Factors Affecting Aerobic Granulation

Besides factors requiring for aerobic granulation mentioned above, there are many necessary considerations like operation conditions, stimulating or inhibiting substances that strongly contribute to formation of granules. In aspect of operation conditions, organic loading rate, hydrodynamic shear force, and settling time appear as main factors for selecting or growing granules. In aspect of environment, changes in hydrophobicity, EPS, and free ammonia concentration have significant effects to formation and structure of aerobic granules. Although these factors have not fully documented but some main factors has been surveyed by many authors as follows

2.5.1 Organic Loading Rate

Many studies showed that OLR played an important role in the cultivation of aerobic granules. For cultivating aerobic granules, proper high-strength wastewater is preferred (Moy et al. 2002). Tay et al., (2005) also proved that smooth, dense and stable biofilms can be obtained when the biological treatment systems are operated within an optimal range of OLR value. Tay et al. (2003) carried out study with organic loading rate of 8, 4, 1 kg COD/(m^3.day). The result is shown in the table 2.5.

Table 2.5 shows that optimum organic loading rate (OLR) for aerobic granulation is 4 kgCOD/m^3.day. This OLR has the stabilized granules with the size of 5.4 mm, roundness of 1.29, SOUR of 118 mg O_2/(mg VSS.h), SVI of 50 ml/g, COD removal rate of 99%. Too high or too low of OLR appeared to be unfavorable for the formation of a compact sludge bed, and further for maintaining the stability of reactor performance. Under the OLR of 1 kgCOD/m^3.day, only the patchy flocs were produced. If the OLR is higher than 8 kgCOD/m^3.day, both granules and fluffy flocs also co-existed. Granules cultivated with high OLR also contained a relatively smaller amount of EPS (Tay et al., 2003). In conclusion, proper OLR for cultivating aerobic granules should be around 4 kgCOD/m^3.d.

Table 2.5 The characteristics of aerobic granules/aggregates with superficial air velocity of 0.041 m/s (Tay et al., 2003)

Reactor	R1	R2	R3
Organic loading rate, kg COD/(m^3.day)	8	4	1
SOUR, mg O_2/(mg VSS.h)	148	131	82
Mean diameter by number, mm	8.8	5.4	4
Granule roundness	1.49	1.29	2.23
Specific gravity, kg/L	1.024	1.034	1.011
SVI, mL/g	65	50	138
COD removal efficiency	0.79	0.99	0.95
VSS/SS ratio, %	0.91	0.87	0.88

2.5.2 Mineral Cations

Mineral cations tend to complex with EPS, affecting bioflocculation, settling and dewaterability of the sludge (Liu and Fang, 2003). Therefore mineral cation concentration considerable contributes to characteristics of aerobic granules.

There are two bioflocculation models: double layer compression and cation bridging. In the cation-bridging model (Tezuka, 1969; Forster and Lewin, 1972; Bruus et al., 1992; Higgins and Novak, 1997a), cations serve as a bridge between negatively charged EPS of neighboring microbial cells. The bridging stabilizes the floc network and thus improves sludge bioflocculation, settling and dewater ability.

Wang et al., (2004) found that most of the metal elements in the sludge changed significantly during aerobic granulation (table 2.6). Calcium and potassium amount were increased in matured aerobic granules, while other elements decreased. Therefore, calcium may play an important role in the cultivation of aerobic granular sludge because it may create a matrix (Van der Hoek, 1987). Furthermore, calcium ion were also suggested either to stimulate granulation by neutralizing negative charges on bacterial surfaces as a result of relatively strong Van der Waals attractive forces, or to function as cationic bridges between bacteria since most of microorganisms are negatively charged at usual pH. Consequently, the calcium-induced cell fusion might initiate the formation of cell cluster, which acted as microbial nuclei of further granulation (Liu et al., 2003).

The change of granule color during granulation was probably due to the change of the biomass compositions, especially for decrease in the content of iron, magnesium, copper and cobalt in the sludge.

Table 2.6 The metal elements in the sludge (mg/g) (Wang et al., 2004)

Type	K	Na	Ca	Mg	Fe	Cu	Mn	Co	Zn
Seed sludge	9.60	9.16	30.20	5.93	26.40	0.23	0.37	0.024	1.12
Matured sludge	43.58	8.00	45.70	2.58	0.76	0.01	0.01	0.012	0.17

2.5.3 Hydrodynamic Shear Force

The formation, structure and metabolism of immobilized microbial community are closely associated with hydrodynamic shear force in reactors. More compact, stable and denser than biofilms, aerobic granules are formed at relatively high hydrodynamic shear force. In other words hydrodynamic shear forces created by superficial air velocity in the reactor have a significant effect on the formation, mass transfer, production of exopolysaccharides, metabolic/genetic behaviour and structure of granules.

Tay et al., (2001) observed that, in upflow sequencing batch reactor (USBR), at rather low air velocity (0.008 m/s) no granules but only fluffy flocs were observed (figure 2.9a). Aerobic granules were only formed at superficial air velocity above 1.2 cm/s (43.2 m/h). When superficial velocity is higher than 2.5 cm/s (0.025 m/s), regular shaped granules were successfully developed (figure2.9b).

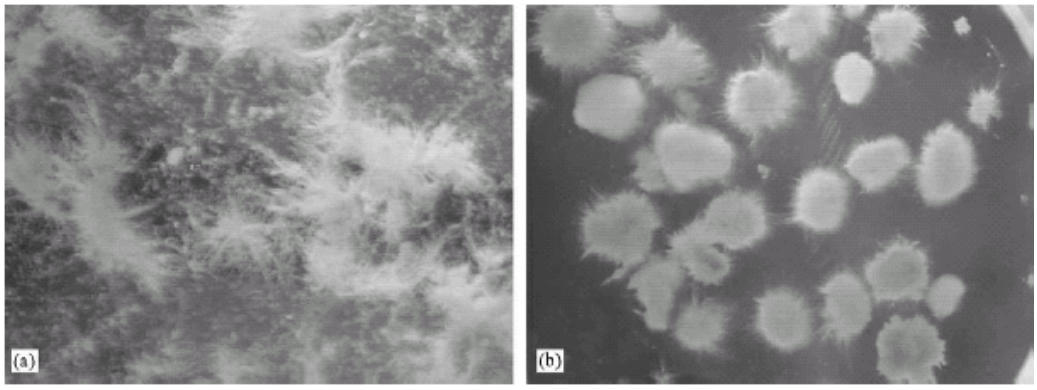


Figure 2.9 Bioflocs cultivated at a superficial air velocity of 0.008 m/s (a); and granules formed at a velocity of 0.025 m/s in USBR (b) (Tay et al., 2001).

From the point of settling ability, Liu et al., 2002 was found that increasing hydrodynamic shear force led to the increase in specific gravity and decrease in SVI of aerobic granules (figure 2.10). Higher granule density or lower SVI can ensure a more efficient biosolid-liquid separation in wastewater treatment systems.

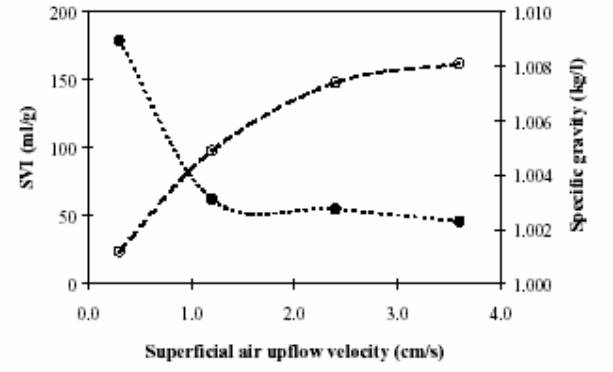


Figure 2.10 Effects of superficial air upflow velocity on the specific gravity and SVI of aerobic granules developed in USBR. (•): SVI; (■): specific gravity (Liu et al., 2002).

2.5.4 Settling Velocity

Settling velocity is the key factor to select and cultivate aerobic granules in reactor. Proper settling velocity will wash out fluffy flocs and retain high settling ability particles. In conventional activated sludge system, settling velocity of flocs usually less than 10 m/h but in the granular sludge system the settling velocity of granules must be greater than 10 m/h. In aerobic granule cultivation process, fraction of aerobic granules can be determined by settling time. The settling velocity of particles was usually chosen first to calculate settling time for system (Beun et al., 2002).

Settling time (h) = [settling height (m) / chosen settling velocity (m/h)]

If the settling velocity is chosen 10 m/h and settling height is 50 cm, the settling time will be 3 min. Table 2.7 shows relationship between settling velocity, superficial air velocity and diameter of granule in some recent researches.

Table 2.7 Sludge settling velocity, superficial air velocity of some research and diameter of granules

References	Beun et al., 2000	Etterer and Wilderer, 2001	Morgenroth et al., 1997	Jang et al., 2003	Wang et al., 2004
Settling velocity (m/h)	16.2	12.6-64.8	30-40	25.2-28.8	> 32.7
Superficial air velocity (m/h)	86.4	72	-	-	63
Granule size (mm)	1	1.1-6.5	2.35	1.1-1.3	6-9
Initial formation time (days)	30	56	40	30	67

The initial selected settling time must be considered carefully when cultivating aerobic granules because this will decide formation time of granules. If the settling time is too

high, it was observed that only flocculated biomass is formed. On the contrary, it did not lead to accumulate of sufficient granules in the reactor (Linlin et al., 2004). So the settling time must be chosen that the settling velocity of particle must be higher than 10 m/h for accumulating of granules in the reactor (Beun et al., 2000).

2.5.5 Cell Hydrophobicity

Hydrophobicity of cell surface has generally been considered to play an important role in the self-immobilization and attachment of cells to a surface, i.e. cell-to-cell attachment (Mahoney et al., 1987; Tay et al., 2002). Cell hydophobicity could be induced by culture conditions, and initiates cell-to-cell aggregation that is a crucial step towards biogranulation. More recent researches showed that cell hydrophobicity induced by culture conditions could serve as triggering force of aerobic granulation (Liu et al., 2003). When the bacteria became more hydrophobic, cell-to-cell adhesion was increased (Del et al., 2000). In fact, the physico-chemical properties of cell surface have profound effects on the formation of biofilms (Bossier and Verstraete, 1996; Zita and Hemansson, 1997; Kos et al., 2003).

In the sense of thermodynamics, microbial aggregation is driven by decreases of free energy. Thereby increasing the hydrophobicity of cell surfaces would cause a corresponding decrease in the excess Gibbs energy of the surface. The decreasing in free energy promotes cell-to-cell interaction and further serves as inducing force for cells to aggregate out of hydrophilic liquid phase.

Cell hydrophobicity depends on types of carbon source. Cell hydrophobicity measured by the method of microbial adhesion to hydrocarbon was 68% for glucose-fed aerobic granules, and 73% for acetate-fed aerobic granules, while the cell hydrophobicity of suspended seed sludge was only about 39%. It is obviously seen that the cell hydrophobicity of aerobic granules was nearly two times higher than that of the suspended seed sludge (Tay et al., 2003).

There is a significant difference in cell hydrophobicity of aerobic granules observed before and after granulation process. It increased from a value of 50.6% in the period before granulation to 75.1% after granulation, i.e. 50% higher than for aerobic granular sludge. It appears that the formation of aerobic granules is coupled to an increase in the cell hydrophobicity.

Some studies showed that starvation conditions could induce cell surface hydrophobicity that in turn facilitated microbial adhesion and aggregation (Watanabe et al., 2000). It is most likely that microorganisms can change their surface properties when faced with starvation, and such changes can contribute to their ability to aggregate.

In some researches cell hydrophobicity can be determined by contact angle (CA) measurement, microbial adhesion to hydrocarbon in forms of liquid or solid (Liu et al., 2004). Cell hydrophobicity was classified into three categories:

$CA > 90^o$	: hydrophobic surface
$50^o < CA < 60^o$	: medium hydrophobic surface
$CA < 40^o$	: hydrophilic surface

2.5.6 Extracellular Polymeric Substances

The abbreviation "EPS" has been used for "extracellular polymeric substance", "extracellular polysaccharides", "exopolysaccharides" and "exopolymer". However, the composition of the EPS matrix in biofilms and activated sludge flocs has been reported to be very complex, mainly consisting of polysaccharides, protein, nuclein acids, lipids, various heteropolymers and humic substances as table 2.8 (Wingender, 1999).

The exopolysaccharides can mediate both cohesion and adhesion of cells, and play a crucial role in maintaining structural integrity of biofilm matrix (Christensen, 1989; Fletcher and Floodgate, 1973; Tsuneda et al., 2001). EPS formed a three-dimensional gel-like, highly hydrated and often charged biofilm matrix, in which the microorganisms were embedded and more or less immobilized. In general, the proportion of EPS in biofilms could be varied between roughly 50 to 90% of the total organic matters (Wingender et al., 1999; Liu and Tay, 2002).

In granular sludge the content of polysaccharides is much higher than content of proteins (Liu and Tay, 2002). Vandevivere and Kirchman, (1993) also found that the content of exopolysaccharides in attached cells was 5-fold greater than that in free-living cells. These in turn imply that the polysaccharides would highly play an important role in attachment and self-immobilization processes. On the other hand, cellular proteins would be less important to the contribution of structure and stability of granule-associated bacteria.

Table 2.8 Composition of EPS (Wingender et al., 1999)

EPS	Principle component	Main types of linkage between subunits	Structure of polymer backbone
Polysaccharides	Monosaccharides Uronic acids Amino acids	Glycosidic bonds	Linear, branch
Proteins	Amino acids	Peptides bonds	Linear
Nucleic acids	Nucleotides	Phosphodiester bonds	Linear
Lipids (phosphor)	Fatty acids Glycerol Phosphate Ethanolamine Serine Chlorine Sugars	Ester bonds	Side-chains
Humic substances	Phenolic compounds Simple sugars Aminoacids	Ether bond, C-C bonds, peptide bonds	

EPS included bound EPS attaching to cell wall and soluble EPS suspending in bulk liquid. Carbonhydrate and protein were usually found as the major EPS component having a protein to carbonhydrate ratio between 0.2 and 5 (w/w) (Frolund et al., 1996). The ratio of polysaccharides to proteins (PS/PN) depends on hydrodynamic shear force (Tay et al., 2002), and inhibitor like ammonia (Yang et al., 2004). The higher superficial velocity is the higher PS/PN ratio is (Tay et al., 2004). In other words the higher ammonia concentration is the lower PS/PN ratio is (Yang et al., 2004).

2.5.7 Free Ammonia

When looking into the ability of developing aerobic granules for the simultaneous organics removal and nitrification, the role of free ammonia must be taken in account. Free ammonia is inhibitor for most of microbial community at a high concentration. Concentration of free ammonia calculated by the following formula depends on pH and temperature of wastewater.

$$FA(mg/L) = \frac{[NH_4 - N]x10^{pH}}{\exp[6334/(273+T)] + 10^{pH}}$$

Table 2.8 descript the effects of ammonia and N/C ratio on granule formation with optimum conditions for nitrification (pH = 7.8-8, D.O ≥ 2 mg/L) (Yang et al., 2004). It demonstrates that if the concentration of free ammonia is higher than 23.5 mg/L, aerobic granules could not be formed.

Table 2.9 Effects of free ammonia to aerobic granular sludge (Yang et al., 2004)

Reactor	R1	R2	R3	R4	R5
N/C ratio	5/100	10/100	15/100	20/100	30/100
Free ammonia (mg N/L)	2.5	9.2	18.0	23.5	39.6
Granular size (mm) after 4 weeks	0.51	0.32	0.25	-	-
Morphology	Smooth, regular shaped dense	Smooth, regular shaped dense	Less smooth than R1, R2	-	-

Effect of Free Ammonia on Cell Hydrophobicity

As mentioned above, cell hydrophobicity plays a positive role in the formation of biofilm and granules (Tay et al., 2001). Nevertheless hydrophobicity had inverse effect with free ammonia concentration. The cell hydrophobicity decreased from 70.6% to 40.6% with the increase of the free ammonia concentration from 2.5 mg/L to 39.6 mg/L (Yang et al., 2004) (figure 2.11). Consequently, low cell hydrophobicity resulted from the appearance of free ammonia

Effect of Free Ammonia on Production of Polysaccharides

Polysaccharides act as key factor in the cell immobilization (Flemming and Wingender, 2001). Extracellular polysaccharides can contribute to the formation and architect of biofilm, anaerobic and aerobic granules and their stability (Tay et al., 2001). The increase of free ammonia concentration, however, led to the decrease of synthesis of cell polysaccharides.

Research of Yang et al. (2004) expressed close relationship between PS/PN ratio and free ammonia concentration in figure 2.11. When ammonia concentration increases from 2.5 to 39.6 mg/L, the PS/PN ratio relatively decreases from 2.8 to 0.55 (PS/PN ratio of activated sludge is 0.55). It means that when ammonia concentration reaches to 39.6 mg/L granulation process can not progress.

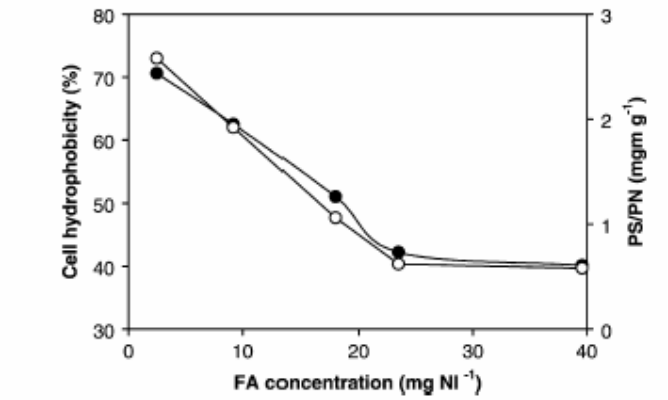


Figure 2.11 Effect of free ammonia on cell hydrophobicity and PS/PN ratio after four-week operation (•) hydrophobicity; (•) PS/PN ratio (Yang et al., 2004).

Effect of Free Ammnia on Activity of Heterotrophic and Nitrifying Bacteria

High free ammonia concentration exhibits the activities of nitrifying bacteria and also represses the energy metabolism of heterotrophs. Metabolic activities of heterotrophic bacteria are quantified by the specific oxygen utilization rate (SOUR). When free ammonia increases, SOUR decreases (Yang et al., 2004). Free ammonia inhibition threshold is 10-150 mg/L for Nitrosomonas and 0.1-4 mg/L for nitrobacter (Bae et al., 2001; Liu and Tay, 2001).

Deflocculation was observed when the aerobic microbial activities were exhibited (Wilen and Nielsen, 2000) while inhibition of energy-generating function would prevent the development of competence for cell aggregation (O'Toole, 2000). Consequently, the reduced microbial activity that results from the free ammonia inhibition is partially responsible for no aerobic granulation (Yang et al., 2004).

In conclusion, free ammonia causes significant influences to nitrification, cell hydrophobicity, production of extracellular polysaccharides, nitrifying activity

2.6 Application of MBR in Wastewater Treatment

2.6.1 MBR

Membrane bioreactor is a system that combines membrane filtration and biological process. Membranes are natural or artificial, two dimensional objects that separate fluids with different compositions from each other (Staude, 1992). The special quality of membranes allows the transport of only specific matters or materials groups. So membranes can be combined with water and wastewater treatment facilities.

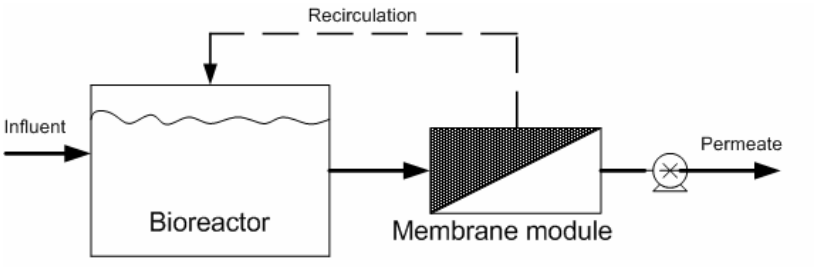
There are many different membrane processes resulted from the different demands on the separation process. Subdivision of the different processes occurs according to:

- The driving force behind the filtration process;
- The type of the inserted membrane;
- The kind of the matters to be separated.

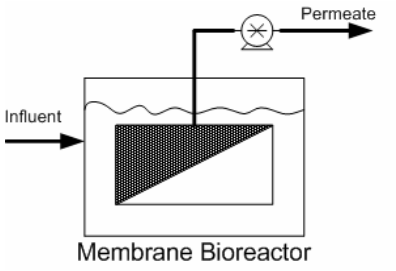
Combining membrane technology with biological reactors for treating wastewater has led to the development of three generic MBRs:

- Separation and retention of solids;
- Bubbleless aeration within bioreactor;
- Extraction of priority organic pollutants from industrial wastewater.

The first one, separation and retention of biosolids, is the most widely studied and has found full-scale applications in many countries (Visvanathan et al., 2000). Solid/liquid separation bioreactors employ micro- or ultrafiltration modules for the retention of biomass. The membranes can be placed in the external circuit of bioreactor or they can be submerged directly into bioreactor (figure 2.12).



a. Membrane in external circuit



b. Membrane in internal circuit

Figure 2.12 Solid/liquid separation MBR (Visvanathan et al., 2000).

A submerged membrane should be outer-skinned. In general, permeate is extracted by suction or, less commonly, by pressurizing the bioreactor. In the external circuit, the membrane can be either outer- or inner skinned, and permeate is extracted by circulating the mixed liquor at high pressure along the membrane surface.

Gas permeable porous membranes can be used to aerate the mixed liquor in the aeration tank by bubbleless oxygen mass transfer (Yasuda and Lamaze, 1972). In certain case, the membrane can act as support for biofilm development, with direct oxygen transfer through the membrane wall in one direction and nutrient diffusion from the bulk liquid phases into the biofilm in the other direction (Brindle and Stephenson, 1996). Because the membrane can form bubble-free or fine bubble mass transfer, the efficiency is very high.

Membranes have been finding wide application in water and wastewater treatment ever since the early 1960s when Loeb and Sourirajan invented an asymmetric acetate membrane for reverse osmosis. Many combinations of membrane solid/liquid separators in biological treatment processes have been studied since. The trends that led to the development of today's MBR are depicted in figure 2.13.

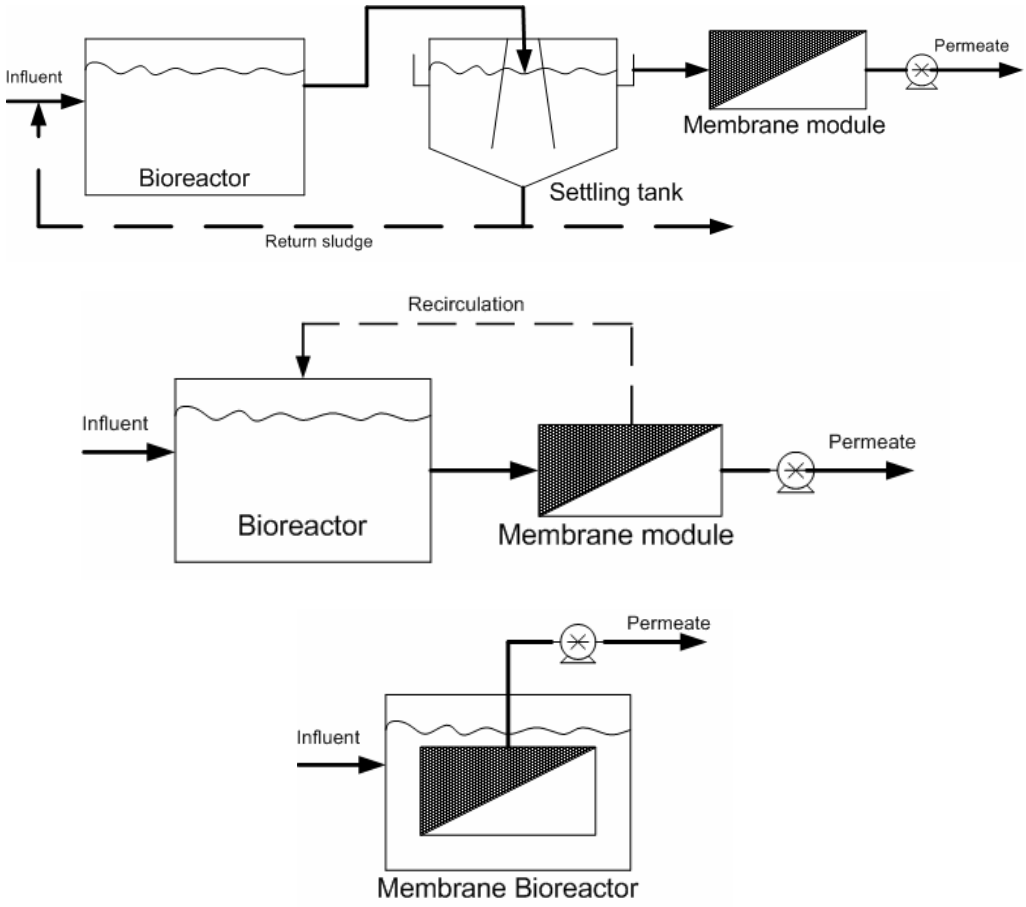


Figure 2.13 Trends in MBR development (Visvanathan et al., 2000)

2.6.2 Advantages of MBR

MBR process has been proved to have many advantages in comparison with conventional biological processes. The main advantages are high quality of treated water, small size of

treatment plant, less sludge production and flexibility of operation (Visvanathan et al., 2000).

High rate decomposition: Treatment efficiency is also improved by preventing leakage of undecomposed polymer substances. If these polymers are biodegradable, they can be broken down with a reduction in the accumulation of substances within the treatment process. Other dissolved organic substances with low molecular weights, which can be eliminated by membrane separation alone, can be broken down and gasified by microorganisms or produced new bacteria cells. Most MBR studies indicate the effluent BOD5 is below 5 mg/L (Trouve et al., 1994; Buisson et al., 1997; Parameshwaran and Visvanathan, 1998) and based on reactor volume, MBR process is 15 to 20 fold higher in substrate conversion rate in comparison with conventional activated sludge process (Buisson et al., 1997).

Treated water quality: In conventional technology, treated quality strongly depends on settling ability in sedimentation tank. In MBR process, solid/liquid separation is conducted by membrane filtration. Therefore, the final effluent does not contain suspended matter, and almost bacteria. This enables the direct discharge of the final effluent into the surface water and the reuse of the effluent for cooling, toilet flushing, lawn watering or process water.

Flexibility in operation: Solid retention time (SRT) can be controlled completely independent from hydraulic retention time (HRT). So the system can be run at very long SRT providing favourable conditions for the growth of slow-growing microorganisms, which are able to degrade biorefractory compounds.

Compact plant size: Because the MBR process is independent upon sludge settling quality, high biomass concentration can be maintained up to 40 g/L in the reactor (Yamamoto et al., 1991). Therefore, the system can stand for high volumetric loading rate resulting in the reduced size of the bioreactor. In addition, secondary settling tank, filter, sludge thickener or post treatment for further BOD, SS removal are not necessary in MBR process, thus the plant becomes more compact.

Low sludge production rate: In real MBR sludge production rate is very low. Excess sludge from MBR process is much lower than that of conventional activated sludge process about one fifth fold (Buisson et al., 1997). Low F/M ratio and longer sludge age (from 50 to 100 days) in the reactor may be the reason for low sludge production rate. In addition, the microscopic observation on microorganism population indicates that with increased sludge age, reduction in filamentous bacteria increased rotifiers and nematodes (Praderier, 1996; Pliankarn, 1996).

Disinfection and odour control: In membrane filtration, the removal of bacterial and viruses can be achieved without any chemical addition (Pouet et al., 1994; Langlais et al., 1992; Kolega et al., 1991). All the process equipment can also tightly close, no odor dispersion occurs.

2.6.3 Membrane Fouling

Membrane fouling is one of the obstacles of this application because of flux reduction, cause of short membrane life and impairment of fractionation capability of membrane. As the resistance increases the flux will decline. This increase in resistance may be due to changes Rm, Rc or Rf or all of three (equation 2.2). If the flux decline is not reversible by simply alternating operating conditions, it is termed fouling (Fane et al., 1989). Membrane fouling can result from the precipitation of less soluble inorganic species (scaling),

adsorption of organic substances (organic fouling), and adhesion and growth of microbial cells at the membrane surface (biofouling).

Membrane fouling can result from the formation of a polarization cake layer and the plugging of membrane pores (figure 2.14).

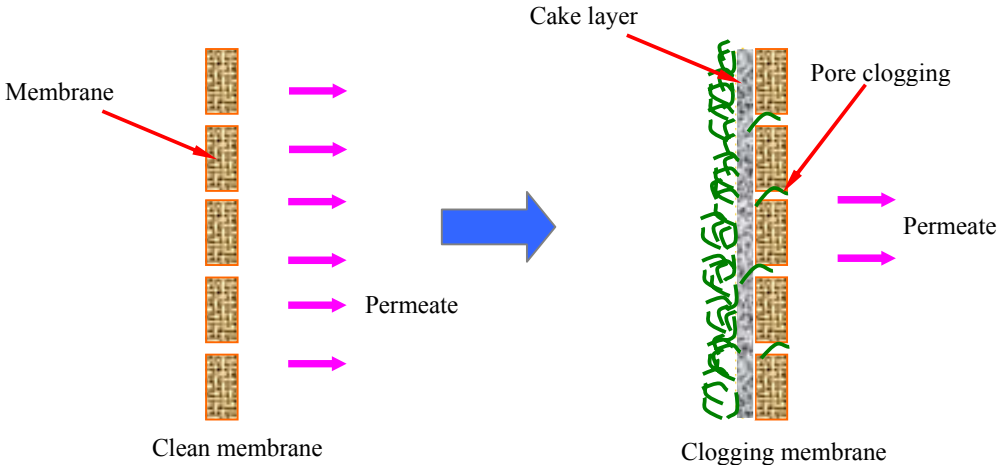


Figure 2.14 Membrane fouling.

Effect of membrane fouling on the decline of permeate flux can be explained by using the resistance-in-series model. According to this model, the relationship between permeate flux and transmembrane pressure (TMP) is described by equation 2.1.

$$J = \frac{\Delta P}{\mu * R_t} \qquad \text{(Eq. 2.1)}$$

Where:

J: Permeate flux ($m^3/m^2.s$)

ΔP: Transmembrane pressure (Pa)

μ: Viscosity of the permeate (Pa.s)

Rt: Total resistance for filtration (1/m)

$$Rt = Rm + Rc + Rf \qquad \text{(Eq. 2.2)}$$

Rm: Intrinsic membrane resistance

Rc: Cake layer resistance

Rf: Fouling resistance due to irreversible and pore plugging

Intensive researchers have been conducted to understand the mechanisms and causes of membrane fouling. Current trends of controlling membrane fouling are focus on (1) controlling the production of extracellular polymeric substances (EPSs) in bioreactor and (2) reducing the cake layer resistance.

EPSs are the substances excreted by microorganism. These compounds comprises of polysaccharides, protein, nucleic acid and lipid. EPS in microbial flocs have been reported as a major foulant in the membrane bioreactor system (Chang et al., 1996; Nagaoka et al., 1996) as they occupy the pores of the membrane. Among different approaches to control of

EPS production have been investigated is control of nutrient composition in the reactor and development attached growth for MBR system (Kim et al., 1998).

Characteristics of the cake layer play an important role in membrane fouling. Effects of cake layer characteristics can be described by the Carman-Kozeny equation as follows (Liew et al., 1995):

$$P_h = \frac{d_p^2 * \varepsilon^3}{180(1-\varepsilon)} \qquad \text{(Eq. 2.3)}$$

Where:

P_h: Hydraulic permeability through the cake layer

d_p: Particle diameter

ε: Porosity of the cake layer

Based on this model, one can derive that the greater the particle size and porosity, the higher permeability. Many attempts have been conducted to improve the permeability of the cake layer by the addition of filter aids such as metal-based coagulants (Chang and Benjamin, 1996), PAC (Pirbazari et al., 1996) into reactor. These filter aids are expected to form a dynamic cake layer on the membrane surface. The permeability of the dynamic cake layer is thought to be higher due to larger particle size and porosity. Schematic diagram of the dynamic cake layer is illustrated in figures 2.15. The porous layer also plays as a filter layer to retain soluble organic compounds preventing them to contact and plug in the membrane pores. Dan (2002) reported a different approach as developing a yeast culture for MBR system. Due to larger size of yeast cells in comparison with bacteria cells, the yeast cells play as a porous layer and therefore permeate flux can be enhanced.

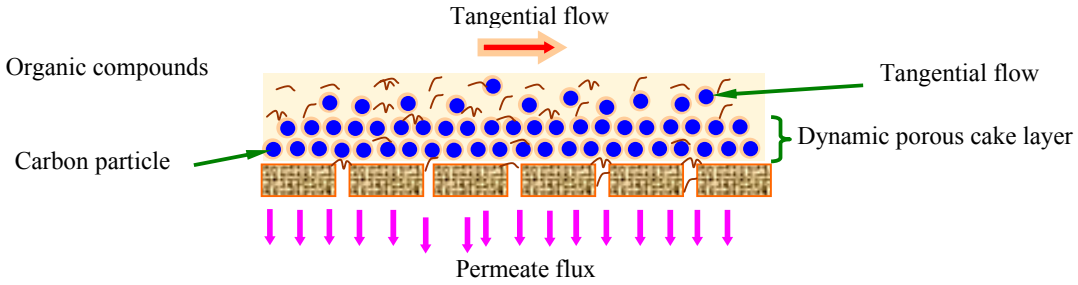


Figure 2.15 Schematic diagram of dynamic porous PAC layer in crossflow membrane filtration (Pirbazari et al., 1996).

Chapter 3

Methodology

3.1 Introduction

As mentioned in chapter 1, the objectives of this study are to focus on: (1) cultivating aerobic granules in both SBAR and SBBR, (2) characterizing granular sludge and effluent at different loading rates; and (3) combining aerobic granular system with membrane bioreactor to investigate membrane fouling. To satisfy above objectives, the experiment work was divided into 3 stages corresponding to 3 main objectives as following:

- The first stage: cultivating aerobic granules in both SBAR and SBBR with basalts as carriers at OLR from 3 to 4 kgCOD/m^3.d. Similar operational parameters in granulation process, such as: cycle time, air flow rate, settling time, etc., were maintained in both reactors. Concerned parameters like morphology, MLVSS, settled biomass concentration, SVI, and EPS were monitored to evaluate maturity of granules.
- The second stage: matured granules were applied with increasing OLR until granules perform overload. Parameters used in observing aerobic granulation were continued to analyze to investigate effectiveness of reactor and development of granules when increasing OLR. In addition, effluent from both aerobic granule reactors was characterized, such as MLSS, EPS, and membrane fouling index (MFI), to evaluate membrane fouling potential.
- The third stage: aerobic granule reactors were coupled with baffled membrane bioreactor. In this stage to evaluate effectiveness of MBR or its fouling behavior some parameters, such as transmembrane pressure, MFI, membrane fouling rate, and membrane resistance were investigated. From results optimum operation conditions for aerobic granule coupled with MBR system was proposed.

Figure 3.1 below describes whole experiment work of the study.

3.2 Experimental Runs

Whole study involves three experimental runs corresponding to three stage of the study. Entire experiment was conducted within 8 months including granule formation, investigation of aerobic granule sludge and effluent, and coupling aerobic granular system with Baffled MBR. To effectively fulfill experiment work within specific time, each stage was conducted as following schedule.

- The first stage of experiment ran in first 2 months. Result of the first stage was matured aerobic granules formed in both SBAR and SBBR.
- After getting matured granules, both reactors were applied increasing OLR, and simultaneously coupled with Baffled MBR. Therefore the second and third stage conducted almost simultaneously. These stages were the heart of the study and ran in 4 to 6 months.

Figure 3.2 below figures out graph of experimental work versus time.

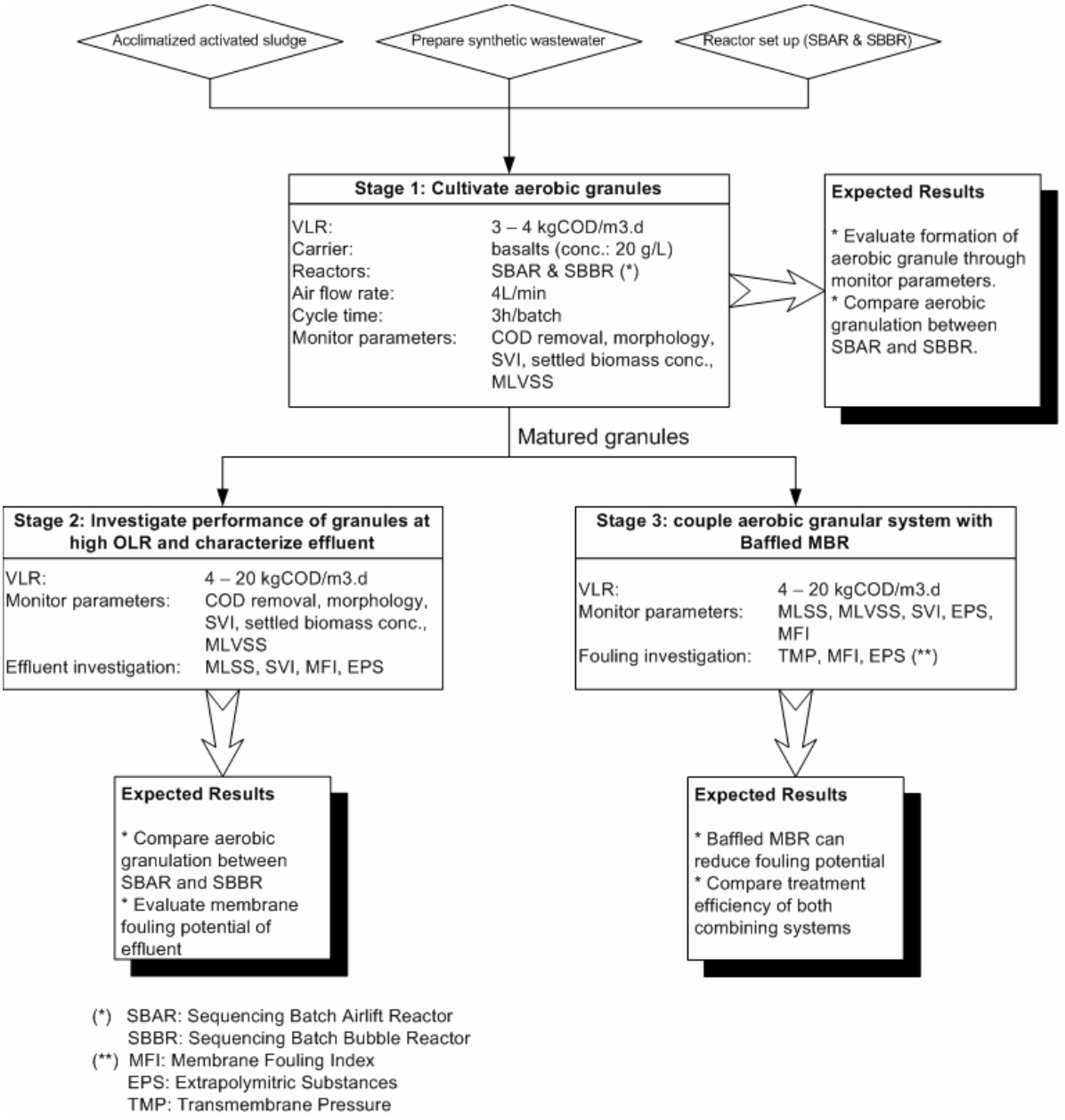


Figure 3.1 Experimental Investigation.

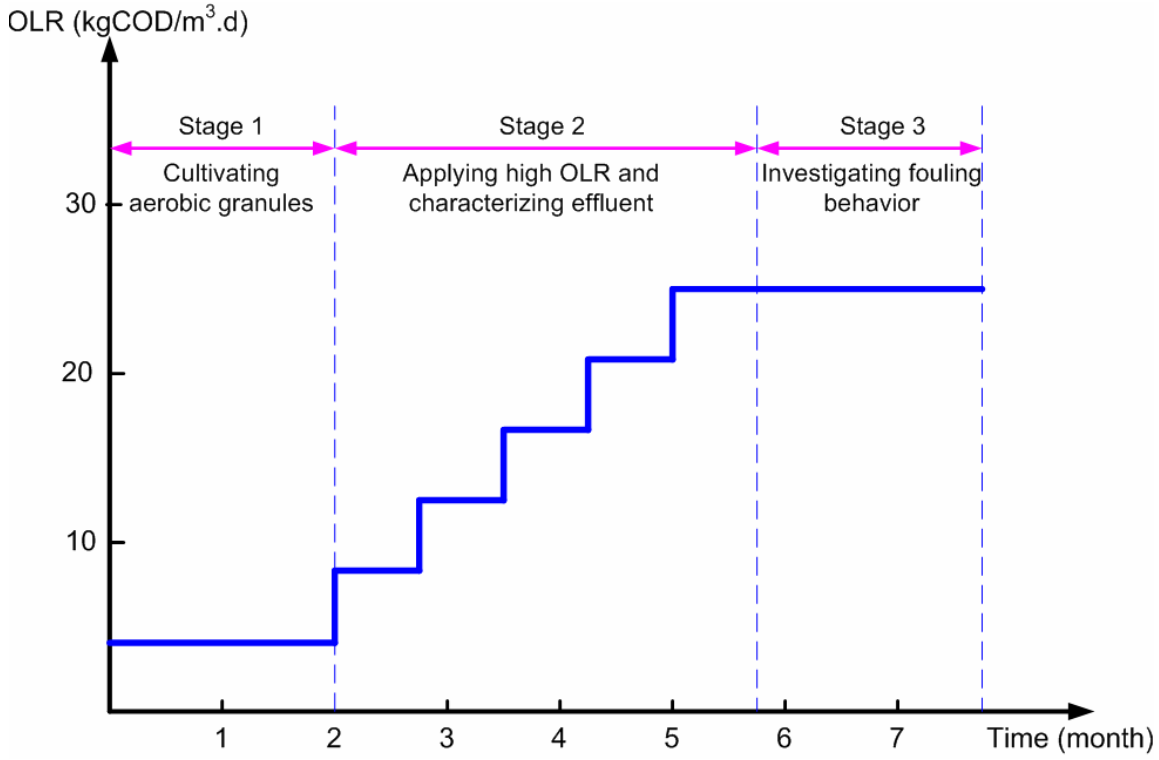


Figure 3.2 Experimental Process.

3.3 Aerobic Granular System

3.3.1 Feed Wastewater, Seed Sludge and Carriers

3.3.1.1 Feed Wastewater

In aerobic granulation of previous researches (Beun et al., 2000; Jang et al., 2003; Wang et al., 2004), glucose and acetate were usually chosen as carbon source for aerobic granule cultivation. In this study, synthetic wastewater consisting of glucose as sole carbon source was used for entire experiment. In cultivation process, COD concentration in influent was maintained about 600 mg/L. Nitrogen and phosphorous were added to avoid nutrient limitation for microbial development, ratio COD:N:P was kept at 100:5:1 during granulation. Furthermore other necessary elements are added to simulate industrial wastewater. The components of feed synthetic wastewater are expressed in table 3.1.

Trace element was added to satisfy the growth and assimilation of microorganisms. Trace solution was prepared as following formula: H_3BO_3: 0.15 g/L; $CoCl_2.6H_2O$: 0.15 g/L; $CuSO_2.5H_2O$: 0.03 g/L; $FeCl_3.6H_2O$: 1.5 g/L; $MnCl_2.2H_2O$: 0.12 g/L; $Na_2Mo_4O_{24}.2H_2O$: 0.06 g/L; $ZnSO_4.7H_2O$: 0.12 g/L; KI: 0.03 g/L (Wang et al., 2004).

In the second and third stage, when applying high OLR in both reactors, OLR was increased by proportional increasing glucose concentration in synthetic wastewater, as well as nitrogen phosphorous, and trace elements. OLR was raised gradually from low to high level. In spite of COD concentration increasing, trace element concentration was added with ratio of 1 mL/L synthetic wastewater. COD removal was used as a main factor to

evaluate withstanding of aerobic granules. If COD removal is higher 90%, aerobic granule reactors are stable at new OLR.

Table 3.1 The components of feed wastewater

Medium	Component	Concentration (mg/L)
Stock solution A	Glucose	664.3
Stock solution B (*)	$NaHCO_3$	270.0
Stock solution C	NH_4Cl	127.0
Stock solution D	KH_2PO_4	53.5
	$CaCl_2.2H_2O$	30.0
	$MgSO_4.7H_2O$	12.0
	$FeCl_3$	3.6

(*) Amount of $NaHCO_3$ used depends on pH value of feed wastewater

3.3.1.2 Seed Sludge

Activated sludge taken from Thammasat University's wastewater treatment plant at Pathumthani province, Thailand was acclimatized 1 week by synthetic wastewater before used for aerobic granulation. The characteristics of the seed sludge, such as morphology, MLVSS, SVI, EPS, etc., were determined prior start-up of lab scale experiment so that it was easy to compare changes in characteristics from activated sludge to aerobic granular sludge. The initially required seed sludge concentration (MLVSS) needed for starting reactor was totally 4000 mg/L. Hence at starting point sludge amount in the reactor was about 10g.

3.3.1.3 Carrier/ Support Media

In this study basalts were chosen as carriers for aerobic granulation process because of theirs rough surface and calcium-rich characteristics that can strongly support for adhesion and cohesion of microorganisms. Moreover, basalt acting as core of aerobic granules could remarkably increase settling velocity of granules. Therefore granule formation time was accelerated with basalt as carriers. With basalts acted as carriers in both SBAR and SBBR, one of objectives in this study was to observe and compare the granule formation in between SBAR and SBBR, as well as with granule formation from other researches.

Basalts were imported from Germany and prepared in AIT's ambient lab before used. Following is the basalt preparation procedure:

- Raw basalts with average diameter of 5 mm were ground to diameter from 212 to 300 μm (sieve No 50 and 70 were used after grinding to select the size);
- After grinding, basalts with desired size were washed with tap water to remove dirt;
- Then dry basalts at 105^0C in 24 hours and keep in dry place before used.

3.3.2 Reactor Design

Batch system has more advantage than continuous system in granule formation, therefore Sequencing Batch Airlift Reactor (SBAR) and Sequencing Batch Bubble Reactor (SBBR) were chosen in this study.

The airlift reactor had working volume of 2.5 L (figure 3.4). The internal diameter of the column is 6.2 cm, and total height is 120 cm. The riser with 90 cm in height, 4 cm in internal diameter was positioned at a distance of 6 cm from the bottom of the column. Effluent valve was 50 cm from the bottom of the reactor. Air was introduced by a fine bubble diffuser in the bottom of the riser, and whole fluid move up inside of the riser. After reaching the top of the riser, whole fluid move down outside, flow convection was made. An airflow meter was used to keep airflow rate constant at 4 L/min. The reactor was operated as a Sequencing Batch Airlift Reactor (SBAR).

The bubble column reactor had working volume of 2.5 L (figure 3.4). The internal diameter of the column is 6.2 cm, and total height is 120 cm. Effluent valve was 50 cm from the bottom of the reactor. Air was introduced by a fine bubble diffuser in the bottom and distributed through metal net with pore size of 0.1 mm. An airflow meter was used to keep airflow rate constant at 4 L/min. The reactor was operated as a Sequencing Batch Bubble Reactor (SBBR)

3.3.3 Operational Condition

The research was carried out at AIT's ambient lab. Two reactors, SBAR and SBBR, were acclimatized with conventional activated seed sludge within the first week. Then basalts were added to reactors to stimulate aerobic granulation. It is said that basalts have a rough surface and rich calcium which results in good potential for biofilm development (Wang et al, 2004). Thus use of calciferous basalt as support media is advantageous in granule formation. Basalt concentration was 20 g each working volume of reactor, it mean that total weight of basalts added to each reactor was 20 x 2.5 = 50 g.

These two reactors were operated with the same operational conditions to investigate the characteristics of granules from both reactors. Organic loading rate for cultivating aerobic granules was maintained at 3 – 4 kgCOD/m^3.d. The temperature to conduct experiment was at the ambient temperature from 28-35^oC, and the pH was kept at neutral pH = 7 ± 0.5 during feeding synthetic wastewater. Two reactors were well-mixed and highly turbulent with a liquid circulation.

.

Two reactors were operated in successive cycles of 3 hours each. At stable condition or when granules matured, one cycle consisted of 5 minutes feeding, 170 minute aeration, 3 minute settling, and 3 minute effluent withdrawal. The synthetic wastewater was stored in the feeding tank, then flew to level control tank before feed to reactors. The settling time was initially chosen such that only particles with a settling velocity higher than 10 m/h were effectively retained in the reactor. And all of filaments and flocs were washed out because of slow settling velocity (figure 3.3).

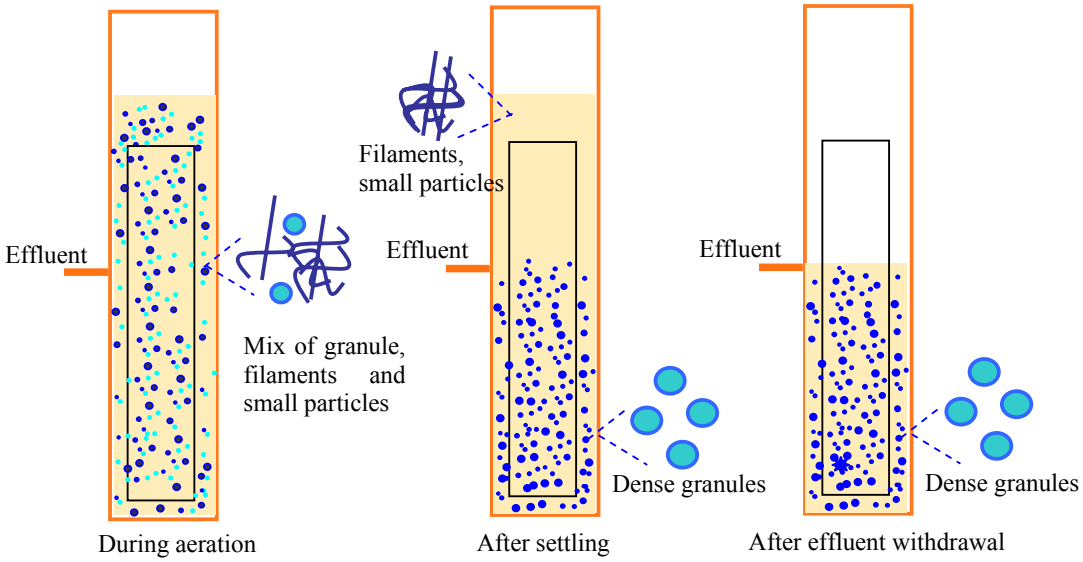


Figure 3.3 Selection of well settling, dense granules in the SBAR & SBBR.

During first 2 weeks of aerobic granule cultivation, the settling velocity was adjusted manually to prevent washout of all the biomass. The first 7 days previously called acclimation stage, settling time was maintained at 10 minutes. From day 8 to day 14, the settling time decreased from 10 minutes to 4 minutes and on day 15 the final settling time reached to 3 minutes. The H/D ratio (column height to column diameter) was high to improve selection of granules by difference in settling velocity. To encourage microorganism development on carrier, biofilm attached on reactor wall on the first days of operation was removed completely. Table 3.2 shows operational conditions for start-up of SBAR and SBBR

Figure 3.4 shows detail of aerobic granulation experiment set up.

Table 3.2 Operational conditions for the start-up of SBAR and SBBR

Day	Action	Remark
1 – 6	Inoculation with activated sludge Settling time of 10 minutes	-
8	Settling time of 9 min	-
9	Settling time of 8 min	Suspended biomass (bio flocs) was started to be washed out
10	Settling time of 7 min	-
11	Settling time of 6 min	Significant biomass growth on the wall of reactor
12	Settling time of 5 min. Removal of biomass growth from reactor wall	In both reactors, small granules were present
13	Settling time of 4 min	-
14	Settling time of 3 min	-
12 onward	Settling period 3 min	Granules became bigger in size

Source: Modified from Beun et al. (2002)

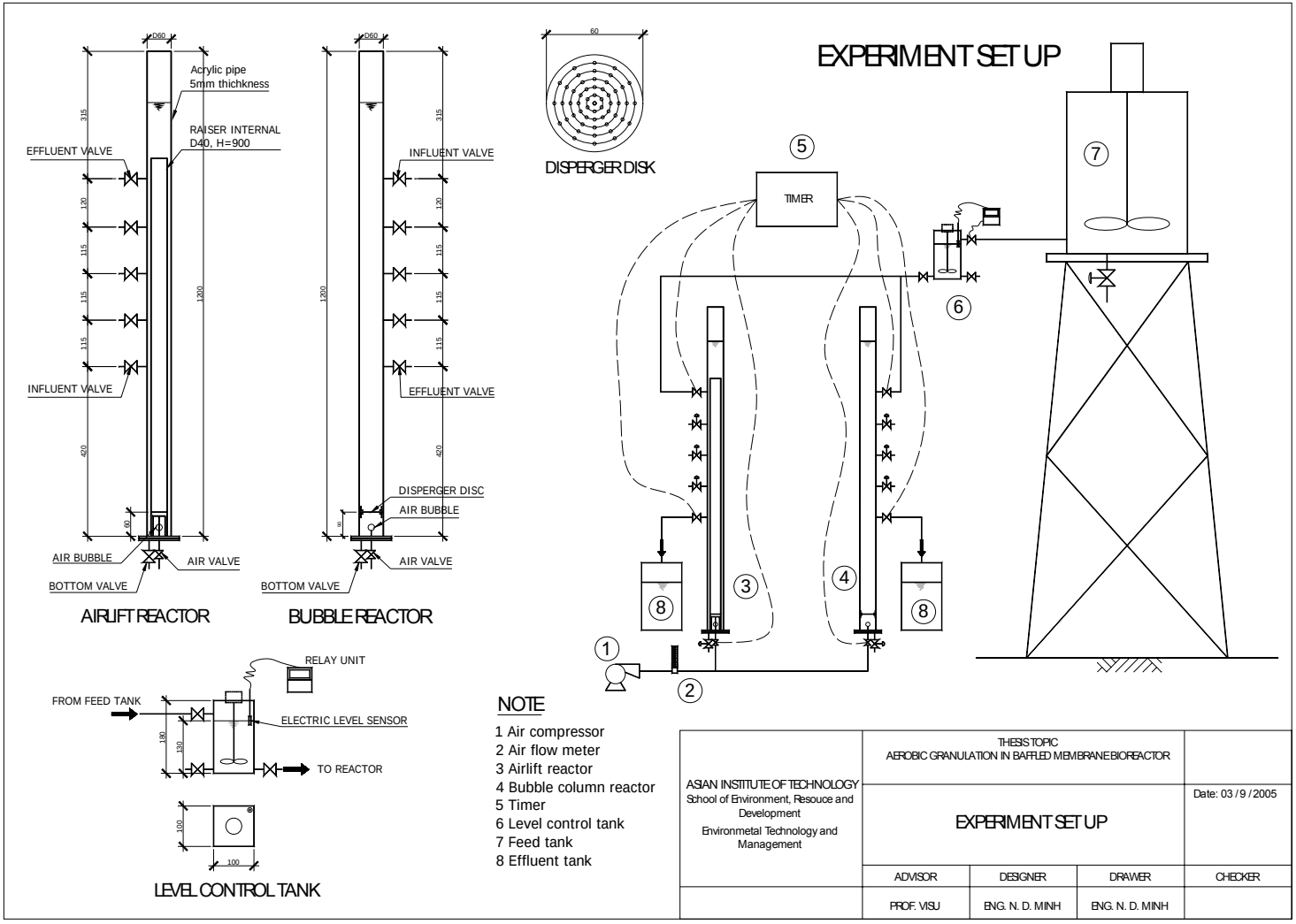


Figure 3.4 Detail of SBAR and SBBR and experiment set up.

3.4 Membrane System

3.4.1 Baffled MBR Design

Baffled MBR were constructed by acrylic sheet with working volume of 8.54 L (figure 3.5). HRT in baffled MBR was 12h. Baffled MBR which was operated continuously had 2 settling compartments and 1 membrane compartment which fit for membrane module. Vertical flow in baffled compartment allowed suspended solid settle and retain in sludge hopper. Sludge was withdrawn daily with the volume of about 500 mL/day. In membrane compartment air was supplied by a fine bubble diffuser to reduce cake layer formation on membrane surface. Membrane was flat sheet membrane with pore size of 0.1 µm.

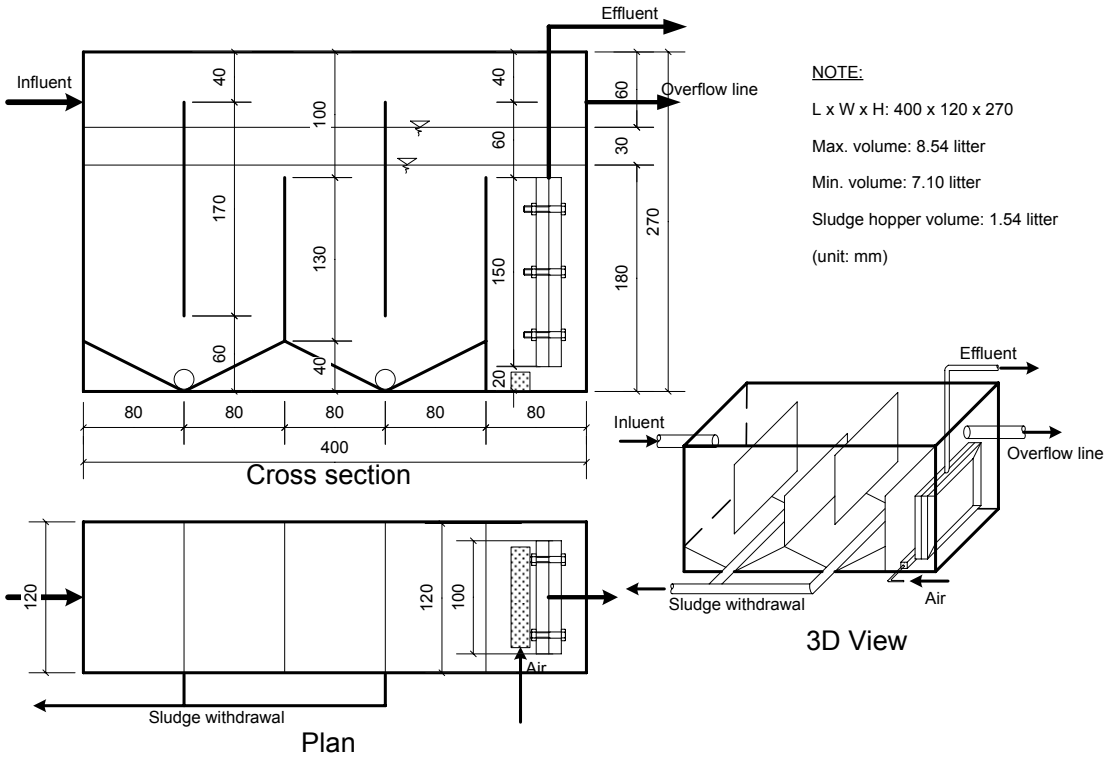


Figure 3.5 Design of Baffled MBR.

3.4.2 Operational Condition

Two baffled MBRs were connected with SBAR and SBBR. Although aerobic granular system operated in batch option, baffled MBR was operated continuously. With membrane area was 6 x 11 = 66 (cm^2), and pore size of membrane was 0.1 µm, suction flow rate was chosen 3 mL/min. Peristaltic pump ran intermittently with cycle of 8 min on/ 4 min off, this option was chosen so that membrane fouling could be reduce due to continuously operation. Due to this operation, an extra amount of effluent from aerobic granular system was discharged.

3.4.3 Membrane Cleaning

After a long period of MBR operation, if transmembrane pressure (TMP) increases more than 80 kPa, membrane should be cleaned to ensure filtration flux. Cleaning membrane is an important step to recover its function after every experiment. Because membrane is a sophisticated and vulnerable structure, cleaning membrane requires a complicated technique. Combination between physical and chemical cleaning is necessary to remove completely cake layer, which are attached on membrane surface, and flocs that are stuffs in fine pore of membrane. Chemical concentration as well as pressure of water should be considered in cleaning process. In this study, PVDF flat sheet membrane was researched with membrane pore size of 0.1 μm. Cleaning membrane procedure was presented in detail in Appendix C.

3.5 Analytical Methods

As mention in chapter 1, this study focused on cultivating aerobic granules in SBAR and SBBR, then comparison of aerobic granulation between 2 reactors was made. However main objective of this study concentrated on investigating membrane behavior of system combining between aerobic granulation and membrane bioreactor.

3.5.1 Methods for Aerobic Granule Investigation

The development of aerobic granules was observed through some basic parameters, such as: COD removal efficiency, granule morphology, settling velocity, settled biomass concentration and MLVSS in reactors. These parameters were analyzed and made comparison to figure out relationships between them in aerobic granulation. Moreover, these parameters were used to compare granule formation process in SBAR and SBBR, and then optimum reactor was pointed out, particularly operation options for aerobic granulation.

3.5.1.1 Granule Morphology

Microscope at AIT's lab was used to observe the size development of granules in whole experiment. Morphology of granules including size and shape was observed and determined by microscope Olympus DF Plan 1X with magnification from 4 to 40x.

3.5.1.2 Settling Velocity

Free settling velocity of carriers and matured granules are determined by free-setting test. A plastic cylinder (6 cm in diameter and 90 cm in height) filled with clear liquid phase of synthetic wastewater is used for free-settling test. In practice, during settling time single granule will settle in reactor and reach their final settling velocity after passing the upper 30 cm of the water column. In free-settling test, granule is dropped freely in first 30 cm of the upper part of cylinder. In the rest 50 cm of cylinder, the settling time will be recorded manually with accuracy of ± 0.5 s. All settling test will be carried out twice and the average will be recorded (Etterer and Wilderer, 2001).

3.5.1.3 MLVSS

In this study, basalts were used as carrier to stimulate aerobic granule formation. Therefore to measure biomass concentration in reactor, basalts should be separated as initial requirement otherwise basalts shall affect to results. Tijhuis et al., 1994 suggested method

to analyze MLVSS of no-carrier granules by indirect way. Total organic compound (TOC) was chosen to indicate for biomass concentration. To get an exact value of biomass concentration (MLVSS), TOC in reactor and effluent were examined, and then difference of these values expresses MLVSS. A coefficient was proposed to convert TOC to MLVSS, which is supposed to have only organic matter. In this study Tijhuis's method was modified to separate carrier in granules before analyzing MLVSS. Hereafter is procedure and calculation method to analyze MLVSS in reactor.

TOC Analysis

- Take 30 mL of sample in centrifuge tube.
- Mix by using Ultra turax equipment in 10 – 15 seconds to separate biomass and carriers.
- Ultra sonic at 100 Watt, pulse 2 in 2 minutes to totally separate carriers out of biomass.
- Dilute sample from 20 to 40 times with deionized water.
- Mix completely by magnetic bar in 10 minutes.
- TOC – V_{CSN} Shimadzu (made in Japan) is used to measure TOC by analyzing TC and IC (TOC = TC - IC).

MLVSS Calculation

MLVSS in reactor was calculated indirectly through TOC in reactor and in effluent. Following equation used to convert TOC value to biomass concentration (MLVSS) is referred from Tijhuis et al., 1999.

$$MLVSS = 2.05 \times (TOC_{in\ reactor} - TOC_{in\ effluent})$$

3.5.1.4 Settled Biomass Concentration

Settled biomass concentration indicates concentration of biomass settling in 10 minutes. In aerobic granular system, there are 2 forms of biomass, i.e. suspended biomass and granule biomass. While suspended biomass has low settling velocity and easy to be washed out from reactor; granule biomass has high settling velocity and retain in reactor. Settled biomass concentration is used to represent granule biomass considered as total biomass of aerobic granules. Referred from related researches (Tijhuis et al., 1994, Beun et al., 1999) about aerobic granules, below settled biomass concentration procedure was modified to be suitable to this study.

- Take 50 mL of sample in centrifuge tube.
- Let the tube stand for 30 minutes to settle sludge, then record sludge volume.
- Decant supernatant.
- Mix sludge with 20 mL of DI water and separate biomass and carriers by Ultra turax in 10 – 15 second.
- Mix solution with 60 mL DI water to dilute biomass concentration for easy to separate biomass and carriers.
- Record volume of solution.
- Measure MLSS of solution as Standard method, then calculate amount of biomass in recorded solution.

- Settled biomass concentration is got by dividing biomass weight and volume of sample.

3.5.1.5 Other Parameters

Beside specific parameters for analyzing aerobic granular sludge, some basic parameters are also required, such as pH, DO, COD, SVI. Procedure for these parameters follows Standard method (APHA, AWWA, 1992). Table 3.3 below describes analytical methods, equipments of these parameters.

Table 3.3 Other measured parameters

Parameter	Analytical method	Equipment	Interference	Reference
pH		pH meter		APHA et al., 1989
DO		DO meter	H_2S, N_2, etc.	APHA et al., 1989
COD	Dichromate closed reflux		NO_2^-, Cl^-, Br^-, F^-	APHA et al., 1989
SVI			High sludge conc.	APHA et al., 1989

3.5.2 Methods for Membrane Investigation

There are many factors affecting to membrane fouling. However this study concentrated on effect of aerobic granular system on membrane fouling. When combining with aerobic granular system, the most concern supposed to cause membrane fouling was bio flocs and EPS. Bio flocs were washed out from aerobic granule reactors was in the form of suspended solid indicated by MLSS. While bio flocs caused fouling due to cake forming, EPS could cause pore clogging, especially polysaccharides (PS). Therefore to evaluate fouling behavior from effluent of aerobic granular system, membrane fouling index (MFI) and EPS were chosen as main parameters. And to get a detailed understand of membrane fouling, different types of MFI (filtered and unfiltered), and EPS (bound and soluble) were examined. This part will present detailed protocols for analyzing MFI and EPS

3.5.2.1 Extrapolymeric Substances

There are 2 major categories contributing to EPS, i.e. carbonhydrate (polysaccharides - PS) and protein (PN). It assumes that EPS exist in bulk liquid in 2 different categories: bound EPS and soluble EPS (figure 3.6). Bound EPS is supposed to be a web-like structure that helps to attach cell together, in which PS keep main role. However both bound and soluble EPS function as mian factor in membrane fouling.

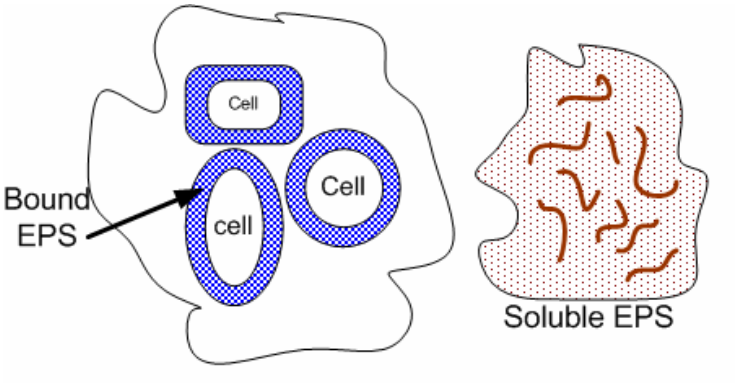


Figure 3.6 Bound and soluble EPS.

While soluble EPS could be easily separated in bulk liquid by centrifuging, bound EPS need an extraction process to completely detach cell. Therefore to analyze bound EPS, extraction is the initially required step. Then extracted solution was continued to analyzed PS and PN. Following is the extraction and analysis procedure.

Extraction Procedure

- Take 30 mL of sample.
- Centrifuge at 4000 rpm in 20 minutes to separate sludge and supernatant
- While supernatant is considered as soluble EPS, sludge is continued to extract to get bound EPS.
- Mix completely sludge with 50 mL of NaCl 0.9%.
- Heat solution at 80^0C in 1 hour, and then leave extracted solution cool to room temperature.
- Record extracted solution for calculating afterward.
- Centrifuge extracted solution at 4000 rpm in 20 minutes.
- Supernatant is bound EPS, and sludge is considered as cell.

Figure 3.7 below expresses entire extraction procedure

PS Analysis

The procedure for EPS-PS analysis

- Pipet 2 mL of sample into tube; if dilution is needed, adjust sample volume and DI water volume so that total volume is 2 mL.
- Add 1 mL of phenol solution 5% and 5 mL of concentrated sulphuric acid.
- Allow the tubes to stand 10 min.
- Vortex and place in water bath for 15 min to cool to room temperature.
- Read A490 after 2 minutes but before 1 hour.

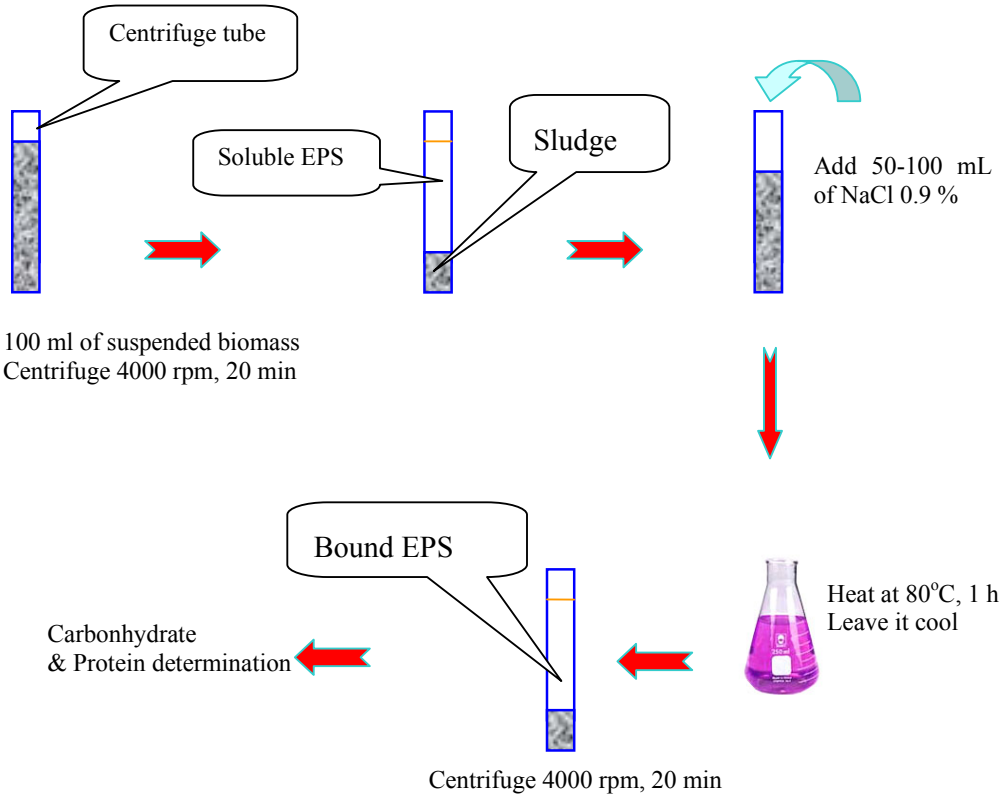


Figure 3.7 Scheme of EPS determined sample preparation.

PS Calculation (mg PS/g VSS)

Extracted solution	X (L)	has	PS	= AX	mg PS
MLVSS:				= B	mg/L
Sample has	C (L)	so	MLVSS	= BC	mg

$$PS(\frac{mg}{gVSS}) = \frac{PS(mg)}{MLVSS(mg)} x1000 = \frac{AxX}{BxC} x1000$$

PN Analysis

The procedure for EPS-PN analysis

- Pipet 0.5 mL of sample into tube; if dilution is needed, adjust sample volume and DI water volume so that total volume is 0.5 mL
- Add 2.5 mL solution C (*)
- Vortex and let stand at room temperature for 5-10 min
- Add 0.25 mL Solution D (*) and vortex
- After 20-30 min, read A750

Solution A: 100 mL of (0.5 g $CuSO_4.5H_2O$ + 1 g $Na_3C_6H_5O_7.2H_2O$);

Solution B: 1000 mL of (20 g Na_2CO_3 + 4 g NaOH);

Solution C: 1 mL of solution A + 50 mL of solution B;

Solution D: 10 mL of Folin-Ciocalteu phenol reagent

PN Calculation (mg PN/g VSS)

0.5 mL sample has	PN		=	A	mg/L
Extracted solution	X (L)	has protein	=	AX	mg Protein
MLVSS:			=	B	mg/L
Take mixed liquor	C L	so MLVSS	=	BC	mg

$$PN(\frac{mg}{gVSS}) = \frac{PN(mg)}{MLVSS(mg)} x1000 = \frac{AxX}{BxC} x1000$$

3.5.2.2 Membrane Fouling Index

Membrane fouling index (MFI) presents fouling behavior of membrane and determined by volume of filtrate versus with time to volume ratio. So the unit of MFI is T/L^6. In this research, MFI is measured by Stainless Steel Pressure Filter Holder SM16249 – made in Germany.

- Adjust air flow rate from the compressed nitrogen container to create 1 bar by 3 valves V1, V2, V3 (figure 3.11). Close V2, V4 and adjust V1 and V3 to get constant pressure of 1 bar maintaining in system.
- Install MFI measurement equipment and membrane;
- Prepare distilled water and real samples with volume of 200 mL each;
- Fill sample into Filter Holder;
- Insert membrane (*) and other membrane support layers as figure 3.8
- Connect air line between air container and MFI equipment;
- Prepare a balance with beaker 250 mL for weighing filtrate;
- Pour samples and tightly close filling port of MFI equipment;
- Activate weighing software to start data recording;
- Open valve V4, read weight, start counting time at the moment;
- Read value every 30 seconds until 10 minutes or at the time of constant weight;
- Finish and close valve V1 to stop gas supplied from cylinder, and open V2 to release air and close valve V4 before reinstalling Filter Holder;
- Reinstall and clean equipment.

(*) Membrane that used for distilled water can be used for real sample.

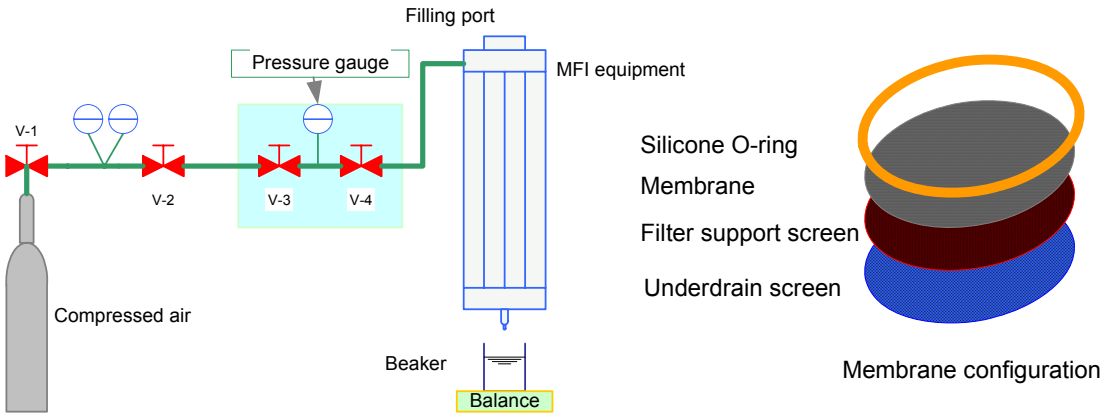


Figure 3.8 MFI measurement set-up.

3.5.3 Sampling Points and Frequency

A detailed schedule of sampling, including sampling points and frequency, was drawn out to standardize analytical process, as well as accurate experiment results. Depending on expected results, sampling points were proposed, and then result was used to evaluate and make conclusion. Table 3.4 exposes sampling points together with frequency of entire experiment.

Table 3.4: Sampling points and frequency

Sampling point	Analytical parameters	Frequency
Influent	• COD	Every 2 days
In SBAR and SBBR	• Granule morphology • SVI, MLVSS, settled biomass conc. • Soluble EPS (PS & PN), bound EPS (PS & PN)	Two times at each OLR (5, 7.5, 10, 12.5, 15, 17.5, 20, etc. kgCOD/m^3.d)
Effluent of aerobic granule reactor	• SVI, MLSS, MLVSS • Bound EPS (PS & PN), soluble EPS (PS & PN) • MFI (filtered & unfiltered)	Two times at each OLR (5, 7.5, 10, 12.5, 15, 17.5, 20, etc. kgCOD/m^3.d)
In settling compartment	• MLSS, MLVSS • Bound EPS (PS & PN), soluble EPS (PS & PN) • MFI (filtered & unfiltered)	Two times at each OLR (5, 7.5, 10, 12.5, 15, 17.5, 20, etc. kgCOD/m^3.d)
In membrane compartment	• MLSS, MLVSS • Bound EPS (PS & PN), soluble EPS (PS & PN) • MFI (filtered & unfiltered)	Once per week.
Effluent of Baffled MBR	• Bound EPS (PS & PN), soluble EPS (PS & PN) • MFI (filtered & unfiltered)	Once per week.

Chapter 4

Results and Discussion

This chapter contains results of aerobic granulation, characteristics of effluent from aerobic granular system, and effects of supernatant of aerobic granular system on membrane fouling, especially on operation of baffled membrane bioreactor.

In aerobic granulation process, parameters affecting formation of aerobic granules were investigated, i.e. hydrodynamic configuration (airlift and bubble reactors), settling time, pH, DO, MLVSS, settled biomass concentration, SVI. To cultivate aerobic granules, OLR of 3 – 4 kgCOD/m^3.d, basalt carriers, and SBAR and SBBR were selected at the start-up stage. After matured granules formed at this loading rate (one month from the beginning), OLR in both reactor was gradually increased up to 25 kgCOD/m^3.d. Granules in both SBAR and SBBR were compared during whole experiment.

In line with analyzing factors directly relating to aerobic granule formation, effluent from both reactors at different OLR were examined, which included SVI, MLSS, MFI, and EPS. The objective of this observation is to evaluate inlet quality for later assessing treatment efficiency of settling unit (baffled reactor) as well as membrane fouling behavior.

The last stage of experiment was to combine aerobic granular systems, SBAR & SBBR, with baffled membrane bioreactors. The main objective of this study was to optimize aerobic granular system coupling with membrane filtration for reuse and reclamation of industrial wastewater.

4.1 Aerobic Granulation Process in SBAR and SBBR

Aerobic granulation is a process of dynamic selection in which environmental and operational options, both biological and physical, favor the cultivation of bacteria that can form aggregates. These factors include the microbial production of extracellular polymers, and the introduction proper shear stress (Etterer, 2000). In addition, filamentous microorganisms such as *Methanothrix* are known to play an important role in binding the granular component together (Jang et al., 2003). The process of aerobic granulation could be clearly divided into three phases of acclimation, multiplication, and maturation.

Activated sludge taken from a domestic wastewater treatment system was seeded into both SBAR and SBBR. Both reactors were started up with 4000 mg/L of activated sludge. The first week is called inoculation period in which the settling time was kept at 10 minutes. Organic loading rate selected to form aerobic granules was 3 kgCOD/m^3.d with influent concentration was 720 mgCOD/L. At initial time of inoculation period, the COD removal efficiency in both reactors was low (around 78%). From observation by using a short settling time of 10 minutes, a small amount of seed sludge was washed out of reactors. In initial period, especially in acclimation phase, to maintain required MLVSS in both reactors for aerobic granulation, this washed-out sludge was recovered in both reactors. The reactor biomass was dominated by flocs during the initial week or inoculation phase, and microscopy images showed that the biomass morphology evolved from suspended sludge or filamentous bacteria. After the first week of inoculation, aerobic granulation process was taken into account. The acclimation phase began from day 1 to day 14, when the seed sludge was in harmony with treatment system and developed into flocs. However in first 2 weeks after reactor startup, COD removal efficiency raised from 80 to 87%. On day 15, tiny granular activated sludge was observed on the wall of both SBAR and SBBR.

The end of the acclimation phase was marked by the appearance of aerobic granules within reactors.

Multiplication phase which initially consist of the transformation from flocs to granules. As the floc-like sludge grew and gradually changed to granules, stable COD removal was obtained. The average COD effluent in both SBAR and SBBR was from 50 – 80 mg/L, with the removal efficiency often over 95%. Multiplication phase was characterized by a significant increase in biomass concentration in the reactors. During this phase the MLVSS increased substantially from 4000 mg/L to 7000 mg/L, and the reactor favored the growth and proliferation of aerobic granules. Compared to the flocs, the granules had a smooth surface and a distinct shape with little or no filament attached. The young granules in the multiplication phase contained a variety of cell morphotypes, including filamentous microorganisms, rods, and cocci, embedded in an extracellular polymeric matrix (Morgenroth et al., 1997). Multiplication phase lasted in 3 weeks from the begining of acclimation phase.

After 5 week operation, granular sludge was started to mature and stable in reactor. From then onward, maturation phase started, the number of granular sludge increased, its size increased gradually, and its structure was denser as well. The stabilization of biomass took place during the maturation phase. At this stage, both SBAR and SBBR were dominated by the matured granular sludge. Granules have been referred to as "well-flocculated sludge" often display a regular shape with a well defined surface. The mature granules in the maturation phase had a thicker and more compact. Maturation phase took nearly 1 weeks. In general, formation of aerobic granules from activated sludge to matured granules took around 1.5 months.

Hereafter are changes in some basic parameters, such as COD removal efficiency, morphology, settling ability, MLVSS and EPS, during aerobic granulation process.

4.1.1 COD Removal Efficiency

In this experiment, synthetic wastewater (prepared from chemicals) was chosen for examination; and soluble COD could indicate for major substrate. Therefore soluble COD was analyzed to evaluate COD influent, effluent, as well as for calculating COD removal efficiency. In whole aerobic cultivation process, OLR of both reactors was fixed at 3 – 4 kgCOD/ m^3.d.

In sludge inoculation period, both SBAR and SBBR were initially maintained MLVSS of 4000 mg/L. At this first stage COD removal efficiency was around 78%, with COD influent was kept at 400 mg/L, however COD removal efficiency slightly increased to 80%. After that, sludge acclimation phase was marked by the continuous increase in COD removal efficiency. MLVSS in both reactors was raised during acclimation phase that could be the reason for COD removal efficiency increased. COD removal efficiency went upto 95%, with COD effluent was around 30 – 50 mg/L. When granules appeared, aerobic granulation process started maturation phase. In this last phase of aerobic granule cultivation, COD removal in both SBAR and SBBR was stable at 95 – 97%. Granules in this phase increase in size, biomass concentration, and dense in structure, the aerobic granulation process was almost agreed with that in research of Linlin et al., 2005. Figure 4.1 and 4.2 demonstrates change in COD removal efficiency and COD effluent in SBAR and SBBR, respectively.

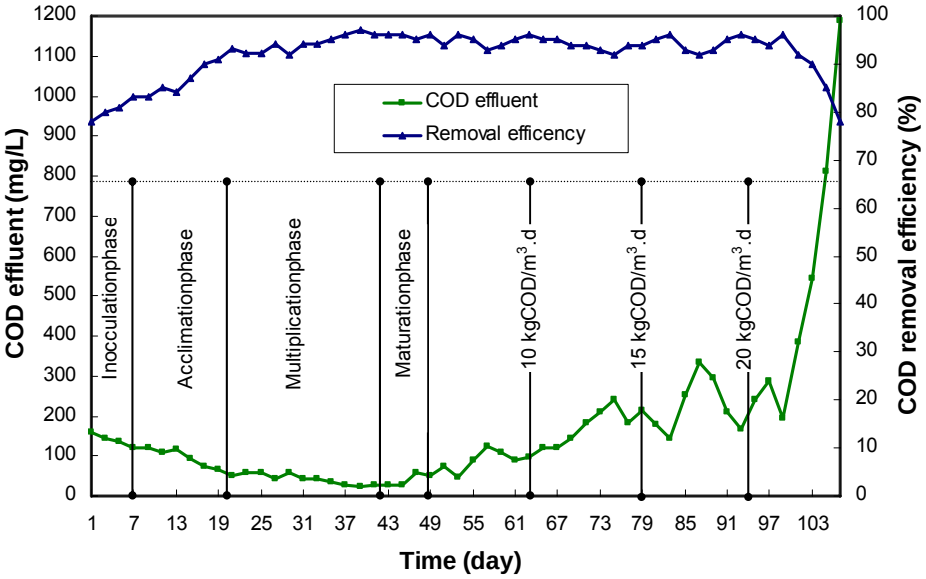


Figure 4.1 COD removal efficiency during aerobic granule cultivation in SBAR.

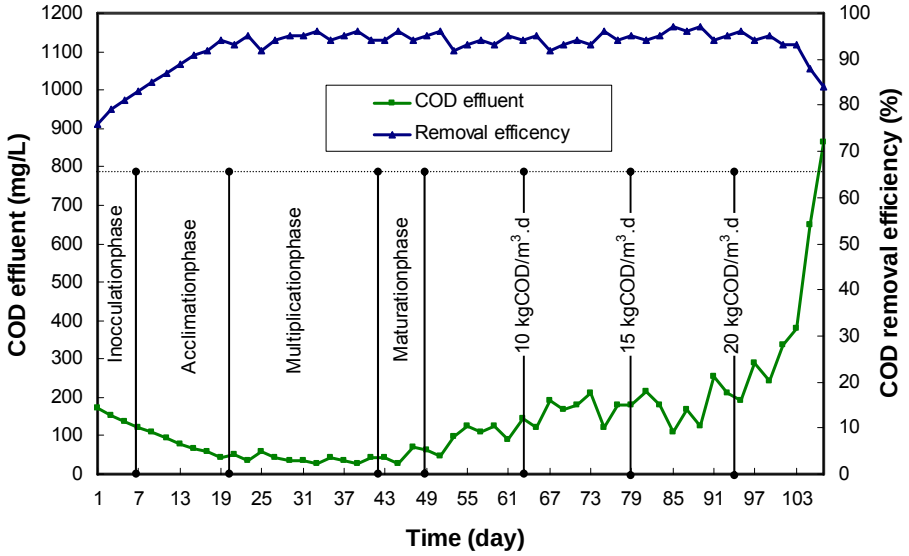


Figure 4.2 COD removal efficiency during aerobic granule cultivation in SBBR.

When comparing aerobic granulation process between SBAR and SBBR, from figure 4.1 and 4.2, it could be seen that SBBR had better treatment efficiency than SBAR, because granules in SBBR appeared and reached matured point earlier. However the difference in formation time is not much and the COD removal efficiency was relatively similar.

After granule matured, OLR was simultaneously increased in both reactors. All conditions, like air flow rate, cycle time, etc., that were applied for cultivating aerobic granules was hold the same when OLR increased. Each level of OLR was run in 1 weeks and COD removal efficiency was used as main factor to evaluate the stability of treatment system. If

aerobic granular system got COD removal efficiency higher than 90%, it could be considered as sufferable. Figure 4.1 and 4.2 shows COD effluent and removal efficiency in whole experiment, from cultivating aerobic granules to OLR of 22.5 kgCOD/m^3.d. In term of COD removal, both aerobic granular systems, SBAR and SBBR, performed similarly. It could be seen from figure 4.1 and 4.2 that aerobic granular system was quite sustainable at OLR from 5 – 20 kgCOD/m^3.d. This result completely corresponded with conclusion of other aerobic granule researches (Thanh, 2005; de Bruin et al., 2004) that is: aerobic granular systems can stand for high OLR (15 – 20 kgCOD/m^3.d).

4.1.2 Granule Morphology

Bio-particle density and diameter have been recommended as suitable descriptors to quantitatively describe the granulation process (Toh et al., 2003). Seed sludge had fluffy, irregular and loose structure morphology, as shown in figure 4.3. The color of sludge gradually changed from black brown to light yellow and lastly to dark yellow during granulation process,

Morphology of aerobic granules was recorded at the beginning of acclimation phase at which aerobic granules started to change form activated sludge to granules and then grow up in multiplication phase. Beginning of granulation formation was the combination of separated sludge under high shear stress; this phenomenon was also described in Liu et al.'s study, 2002. End of acclimation phase was marked by the appearance of some of granules; however these are weak connection. Diameter of initial granules was 0.5 mm.

Multiplication phase observed the change in structure of granules. From loose structure, granules gradually develop to strong and dense structure. Not only change in structure, granules also qualitative multiplied (Wang et al., 2004). The morphology of the granules showed obvious changes during 3 weeks of multiplication phase.

In the last phase, maturation phase, granules varied their structure to more perfect and stronger. Microscopic examination showed that the morphology of the mature granular sludge was nearly spherical (around 1.4 mm in diameter) with a clear outline, and had a strong structure. Experiences with aerobic granulation in both SBAR and SBBR pointed out that the surface morphology, the density and the thickness of biofilms was the net result of interaction between biomass growth and detachment processes (Liu et al., 2002). Compared to the looser and more amorphous flocs, granules are denser, firmer, and more compact, and can withstand compression.

In the second experiment, applying high OLR to aerobic granular system, there was not much change in structure of granules except their size. The development in size of granules will be discussed later. Figure 4.3 exposed morphology of granules at different phase (from activated sludge to matured granules).

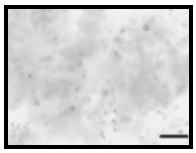
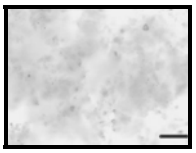
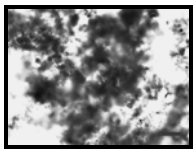
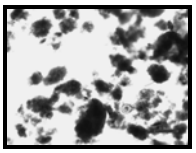
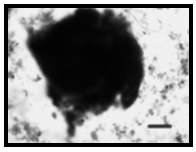
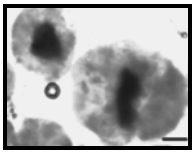
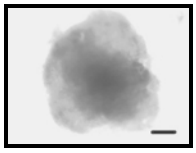
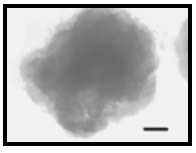
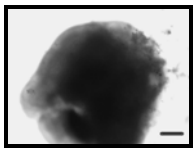
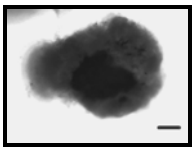
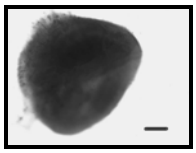
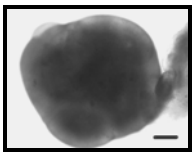
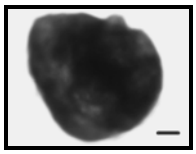
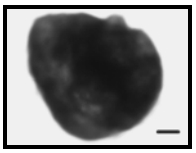
	SBAR	SBBR
Inoculation phase (bar scale of 0.5 mm) * Seed sludge (Activated sludge)		
Acclimation phase (bar scale of 0.5 mm)		
* Granules after 2 week		
* Granules after 3 week		
Multiplication phase (bar scale of 0.2 mm)		
* Granules after 4 weeks		
* Granules after 5 weeks		
* Granules after 6 weeks		
Maturation phase * Mature granules		

Figure 4.3 Development of granular sludge at different phases.

Size of sludge was one of the main factors to distinguish activated sludge and granular sludge. In aerobic granule cultivation, the most change was granule structure in which dispersed sludge gather to from aggregate flocs, then granules. Size of granules in cultivation process strongly increased between acclimation and multiplication phases. Size of mature granules came up to 2 mm.

After granules matured, high OLR was applied to both SBAR and SBBR. From then onward, size of granular sludge simultaneously increased with increasing OLR. At OLR of 5 kgCOD/m^3.d, granule size reached 1.8 mm. Compared to granules in SBAR, granules in SBBR was a little bigger. At OLR of 10 kgCOD/m^3.d granule size in SBAR and SBBR was 3 and 3.2 mm, respectively. Granule size proportionally develop with the increasing of OLR. When OLR went to 20 kgCOD/m^3.d, granule size in both reactors got 4 mm in diameter. However an increase in floc size can lead to a decrease in density and an increase in porosity and these parameters will in turn influence reactor performance. It is hypothesized that due to onset of problems associated with diffusion limitation of oxygen and nutrient into the granule interior lead to breakage of granules (Toh et al., 2003). In conclusion, good granules should have sufficient diameter that can get high conversion of COD, nitrogen, and also excellent settling ability. Figure 4.4 displays development of granule size in whole experiment.

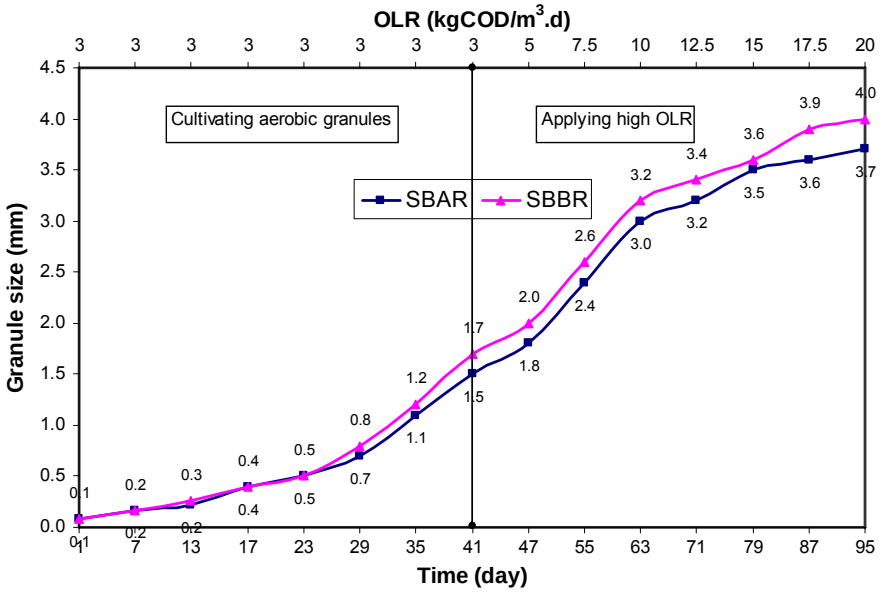


Figure 4.4 Granule size development.

4.1.3 Granule Settling

Settling ability is the critical factor to establish well settling particles in the reactors (Wang et al., 2003). The time allowed for settling, or settling time, is used to enforce a certain particle settling velocity, with which a selection can be made between suspended or flocculated biomass (low settling velocity) and granules (high settling velocity). Low settling particles like suspended biomass and filaments shall be washed out with the effluent.

The initial seed sludge for the SBAR and SBBR operation had SVI of 220 mL/g, and the median floc size of 0.01 – 0.08 mm. Granulation of seed sludge could be achieved through accumulation by interparticle bridging under a condition of turbulent flow mixing.

By observation, granules started to form when sludge had better settling ability. In week 3 after inoculating sludge into reactors, SVI of sludge reduced step by step. It means that, from the end of acclimation phase SVI of sludge was 126 and 112 mL/g in SBAR and SBBR respectively. In week 4, there was a significant change in SVI value in both reactors when some of small granule appeared in reactors. SVI in SBAR and SBBR was around 110

mL/g from the start of week, and then decreased to 65 and 54 for SBAR and SBBR. It could be said that, SVI value was one of instant indicators to realize formation of aerobic granules. And SVI display a remarkable advantage of aerobic granules over activated sludge. After 5 weeks of operation, the seed sludge in the reactors was nearly granularzed. SVI in reactors was reached 50 mL/g, and settling time in both reactors was only 2.5 minutes. From then onward, SVI of granular sludge in both reactors was always less than 50 mL/g. SVI was quite stable during applying high OLR. Figure 4.5 presents analytical result of SVI in whole experiment.

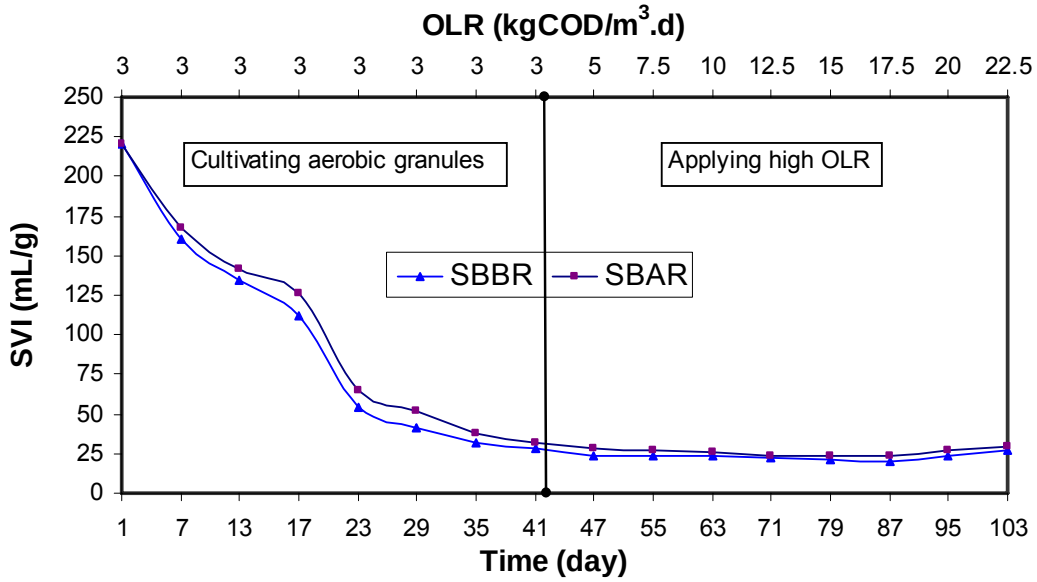


Figure 4.5 SVI of SBAR and SBBR.

Figure 4.5 shows that SVI in SBBR was lower than that of SBAR. It means that granular sludge in SBBR performed better settling ability than sludge in SBAR did. Minimum SVI that granular sludge could reach was 20 mL/g in SBBR and 24 mL/g in SBAR. An important factor that strongly contributed to reduce SVI or enhanced settling ability of sludge was carrier (basalt) which was role as core in granule (Thanh, 2005). It could be observed from figure 4.5 that although increasing high OLR leaded to increasing granule size, the SVI of sludge did not vary much between low and high OLR.

When OLR went to over 20 kgCOD/m^3.d, granule size was larger 4 mm, therefore density of granule decreasd. As a result, SVI of granular sludge increased. However due to big size, granule could not maintain substrates and oxygen diffusion into inner layer, granule was broken by itself.

4.1.4 Biomass Concentration (MLVSS) in Reactors, Settled Biomass Concentration

During start-up the biomass concentration of the reactor content increased from 4000 to 5000 mg/L. However in spite of biomass concentration increasing, MLVSS in both SBAR and SBBR were also remarkable reduce during acclimation phase due to wash out (Tay et al., 2002). Effectively granular growth is just a special case of biofilm growth because it is a need for microorganisms to grow in a granule otherwise they will be washed out due to low settling ability. In these SBAR and SBBR reactors, the biomass grew as well as granules improved settling ability, which allows the accumulation of high amounts of active biomass in the reactors.

Small granules were first observed at the end of acclimation phase. At this period, a competition of microorganism occurred due to short settling time (3 minutes). Flocs that had low settling velocity were washed out from reactors; therefore only flocs had high settling velocity retained in reactors and then develop to granules. The same observation was depicted in research of Beun et al., 2002. From inoculation to 3 weeks afterward, biomass concentration in both SBAR and SBBR slowly decreased. The minimum MLVSS in reactors could reach 2300 mg/L. However, from week 3 to maturation phase, MLVSS in reactors raised. When granules reached mature point, MLVSS in reactors got 11200 mg/L in SBAR and 12400 mg/L in SBBR. When OLR increased from 5 to 22.5 kgCOD/m^3.d, MLVSS in both reactors was positively proportional to the increasing of OLR. Figure 4.6 draws changes of MLVSS in SBAR and SBBR.

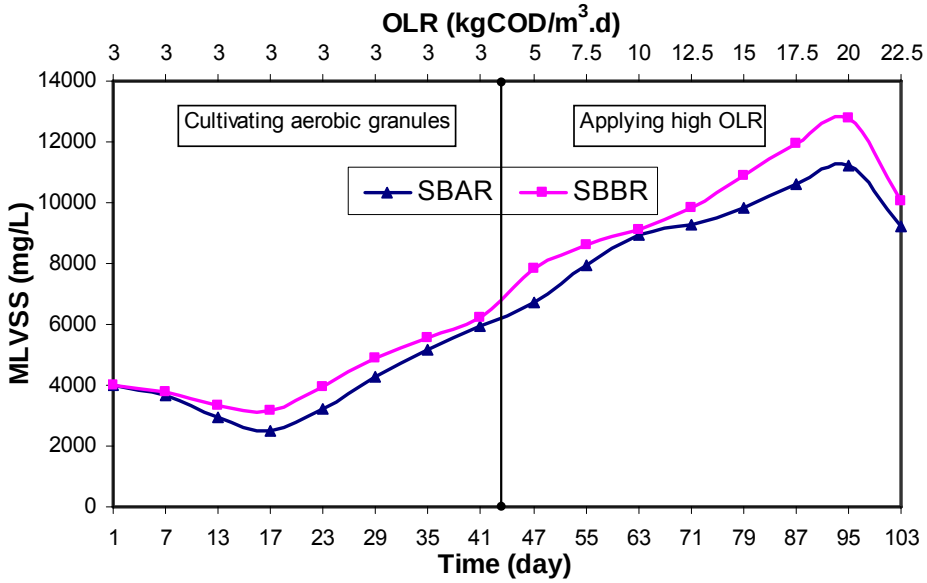


Figure 4.6 Biomass concentration.

From figure 4.6, it could be realized that MLVSS in SBBR was always higher than that in SBAR because SBBR provided better surface area for settling, and sludge in SBBR could settle better. When OLR came over 20 kgCOD/m^3.d MLVSS in both reactors suddenly decreased. It could be explained that at this point, granules had size of over 4 mm; hence they could not maintained structure because of limitation substrate and oxygen diffusion. So most of granules was themselves broken and washed out from reactors. Due to this constraint, application of high OLR was stopped and aerobic granular system was maintained at 15 kgCOD/m^3.d which was considered as stable OLR for aerobic granules.

If biomass concentration represent for total biomass per volume unit of bulk liquid in reactors, settled biomass concentration was a special parameter only used for granular sludge to evaluate total weight of biomass per volume unit of granules. The higher settled biomass concentration, the more compacted granules. Results from this experiment pointed out that when increasing OLR, the settled biomass concentration increased as well.

Settled biomass concentration was nearly equal to MLVSS from the beginning of experiment. 3.8 g/L_{sludge}. When granules appeared at the end of acclimation phase, MLVSS in reactors was not equivalent to settled biomass concentration. From then onward, settled biomass always had considerable growth compared to MLVSS. After 3 week, settled biomass concentration reached 12g/$L_{granules}$ and firmly rose to 29 g/$L_{granules}$ until granules

matured at week 6. SBBR also expressed better performance in term of settled biomass concentration. This value in SBBR was higher than that in SBAR during entire aerobic granulation process.

When applying high OLR, both settled biomass concentration in SBAR and SBBR simultaneously grew. Figure 4.7 exposed changes in settled biomass concentration in both reactors.

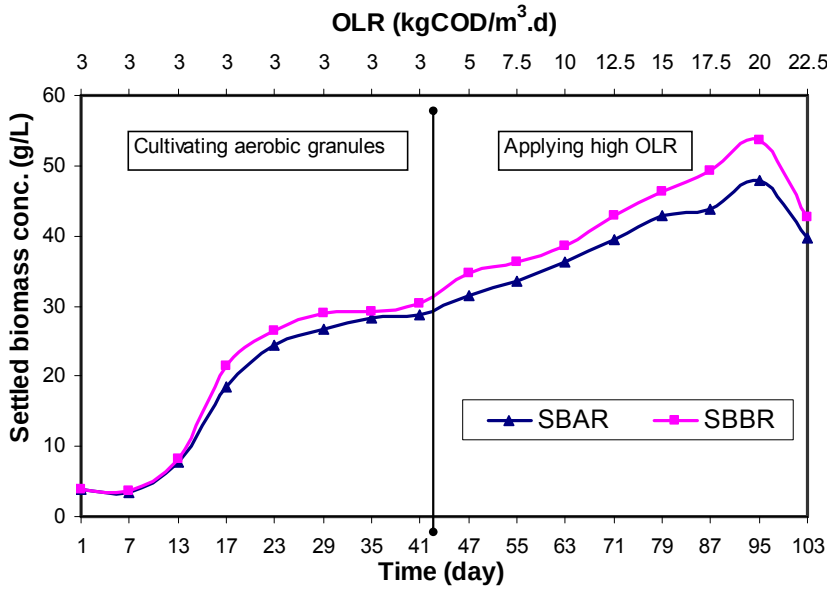


Figure 4.7 Settled biomass concentration.

From figure 4.7, it could be easily seen that from day 23 to day 41, settled biomass concentration increased not much. It means that at this stage biomass formed granules was quite stable, it means that aerobic granules was almost matured. When OLR reached 22.5 kgCOD/m^3.d, settled biomass concentration in both reactors suddenly decreased. At this time, a lot of granules were broken due to big size and could not maintain structure. As a result, amount of granules in SBAR and SBBR significantly reduced.

4.1.5 Bound EPS

From many researches about aerobic granules, it is hypothesized that bound EPS was the main factor contribute to the formation of aerobic granules (Zheng et al., 2004, Liu et al., 2003). Function as frame structure in granules, EPS helped to link and retained microorganisms in condense structure called granules. In EPS, polysaccharides (PS) play a major role in granules, compared with protein (PN) (Thanh, 2005). Therefore EPS result figured out overall view of development of granules in aerobic system. The EPS result from this study shows that when granules were more matured, PS was higher and PN slightly reduced. Figure 4.8 and 4.9 displayed results of PS and PN, respectively.

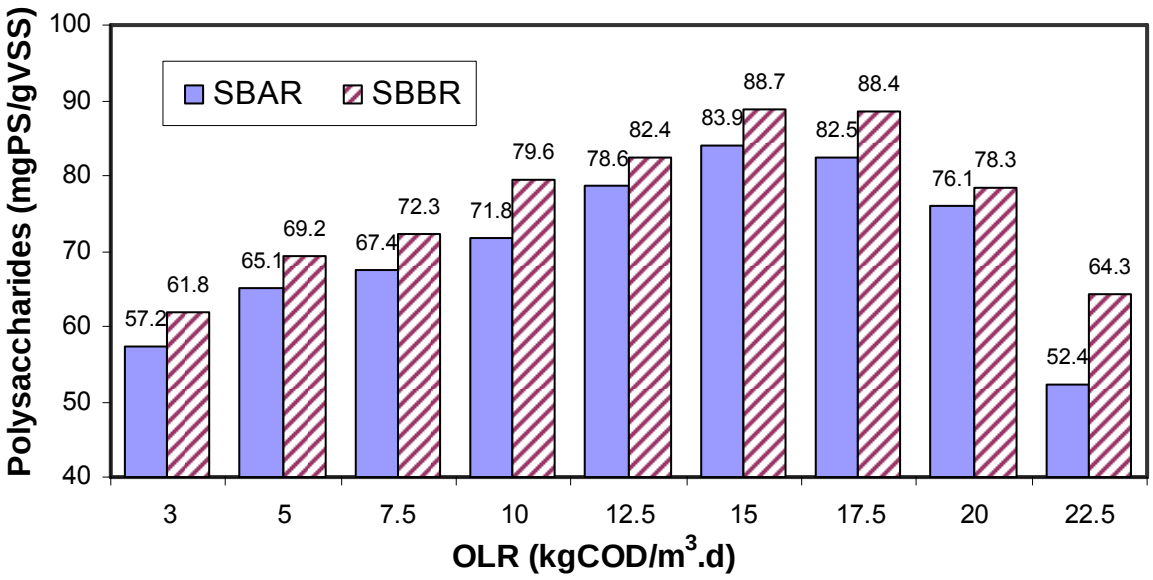


Figure 4.8 Bound EPS -PS in SBAR and SBBR.

Figure 4.8 shows changes of PS in aerobic granules. It could be confirmed that PS had positive effects in formation of aerobic granules. However, when OLR ranged from 12.5 to 17.5 kgCOD/m^3.d, PS did not vary much. But when OLR went over 17.5 kgCOD/m^3.d, PS reduced clearly. This proved that, aerobic granules were stable when OLR ranged from 10 to 17.5 kgCOD/m^3.d.

While there was difference of PS between initial granules and mature granules, PN did not express so much changes. It means that PN does not take part in aerobic granulation process. Totally opposite with PS value, PN in matured granules was lower than that in initial granules. In conclusion, PN presented reverse effect with aerobic granulation.

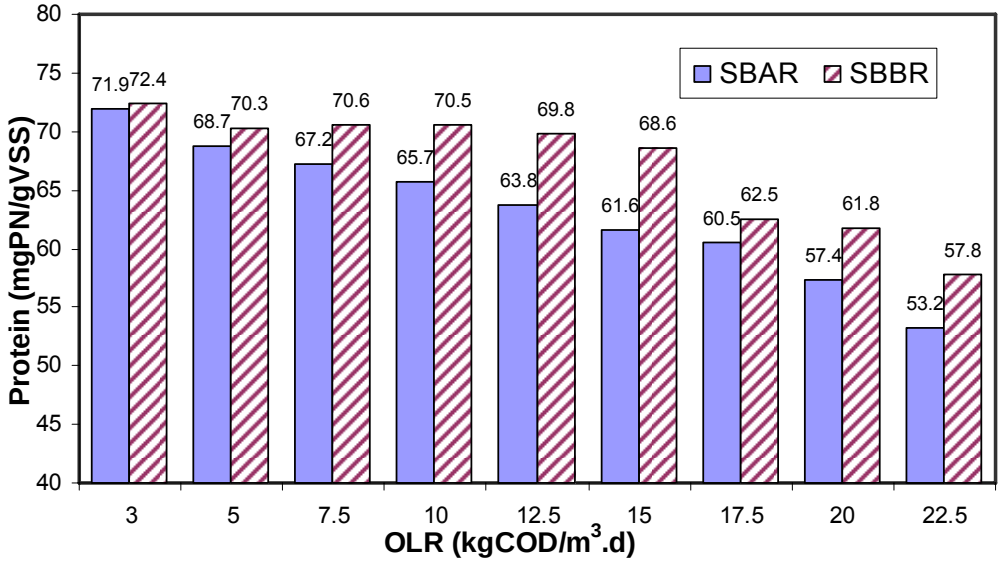


Figure 4.9 Bound EPS – PN in SBAR and SBBR.

4.2 Effluent Characterization in SBAR and SBBR

The second objective of this study was to investigate characteristics of effluent from aerobic granular systems, for evaluating potential when combining aerobic granular system with baffled membrane bioreactor. Due to its advantage nature of aerobic granular system over activated sludge system, aerobic granular system was able to couple with membrane for further dealing or for reclamation of industrial wastewater. For effluent from aerobic granular system, parameters needed to monitor was MLSS, sludge settling ability (SVI), bound and soluble EPS, and MFI.

4.2.1 Effluent MLSS

MLSS or biomass concentration in effluent was observed and performed close relationship with aerobic granulation in both SBAR and SBBR. Changes of MLSS in effluent could be used to evaluate phases in aerobic granulation.

In acclimation phase, a large amount of biomass was washed out from reactors since low settling ability, as a result MLVSS in SBAR and SBBR was significantly decreased. There was only 2510 and 3170 mg/L of MLVSS remaining in both SBAR and SBBR, respectively at OLR of 3 kg COD/m^3.d. At acclimation phase, MLSS in effluent or washed out biomass raised to 1120 mg/L in SBAR and 680 mg/L in SBBR (see Table B-1 and figure 4.10). Since carriers sometimes came together with washed out biomass during withdrawing, they was selected and recovered to reactors in order to maintain required carrier for aerobic granulation. End of acclimation phase was marked by the appearance of small aerobic granules, as well as sludge settling ability increased considerably. Therefore, MLSS in effluent reduced to 600 mg/L and 400 mg/L in SBAR and SBBR, respectively. Form then until granule matched matured point, MLSS in effluent gradually decreased. At the end of maturation phase, only 200 mg/L of MLSS was found out in effluent because most of biomass generated was formed granules, only small amount of excess was washed out.

When applying high OLR in both reactors, excess sludge washed out from aerobic granular system was very little and relatively stable. When OLR varied from 5 to 15 kgCOD/m^3.d. MLSS in effluent fluctuated from 140 to 200 mg/L because biomass generated attached to granules to make them denser and stronger. This result totally agreed with the morphology development of granules when OLR changed from 5 to 15 kgCOD/m^3.d. However from OLR of 17.5 kg/m^3.d washed-out sludge from aerobic granular system was progressively raised. The higher OLR, the more sludge was generated and washed out from reactors. Figure 4.10 displayed curve trend of MLSS in whole experiment.

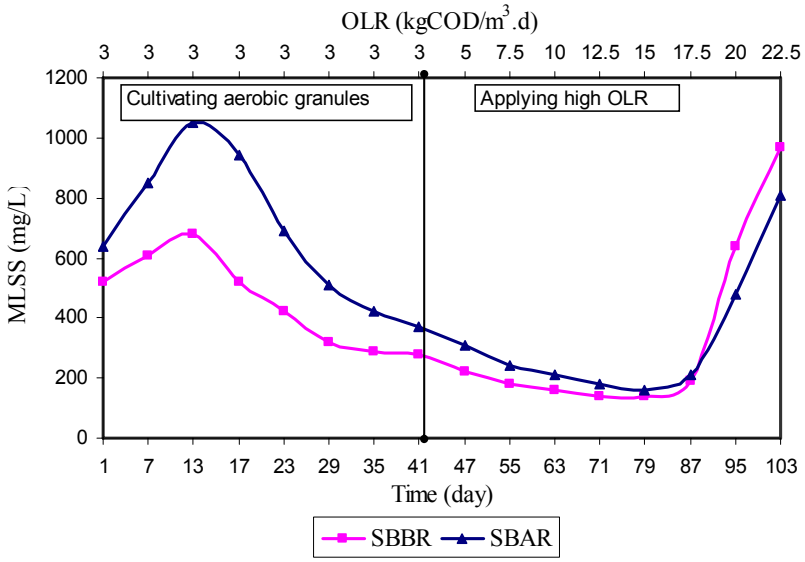


Figure 4.10 Change of MLSS in effluent in SBAR and SBBR.

According to figure 4.10, it could be seen that there was an extreme increase in MLSS in effluent of both SBAR and SBBR when OLR came over 20 kgCOD/m^3.d. Observation from operation showed that at OLR of 20 kgCOD/m^3.d, big granules (diameter was higher 3.5 mm) started to be broken because they could not maintained substrate and oxygen diffusion in the inner parts. Thanh (2005) also figured out the same phenomenon when increasing in granule diameter. Corresponding to COD results in this stage that did not reach 90% of removal efficiency, it could be assumed that both SBAR and SBBR could not stand at OLR of 20 kgCOD/m^3.d.

Although MLSS in effluent did not directly attribute to fouling behavior of membrane in MBR due to being maintained in baffled units, it also played certain impacts. Baffled MBR was designed to retain suspended sludge washed out from aerobic granular system (SBAR and SBBR), therefore only non settling sludge reach the membrane chamber or MBR. Results of MLSS in effluent could be used as baseline data to evaluate operation ability of baffled reactor which was regarded as settling unit. Moreover, sludge from effluent was further examined SVI to compare settling ability between sludge from SBAR and SBBR.

4.2.2 Settling Ability of Sludge from Effluent

Due to combining aerobic granular system with baffled MBR, SVI was used to evaluate settling ability of sludge in effluent. Most of sludge washed out from both SBAR and SBBR was immature granules or detached flocs that could be regarded as excess sludge. However when OLR exceeded 20 kgCOD/m^3.d, washed out sludge contained some aerobic granular sludge. Sludge from effluent was examined settling ability by measuring SVI based on standard method (APHA, 1992). Figure 4.11 presents overall view of SVI variation in whole experiment.

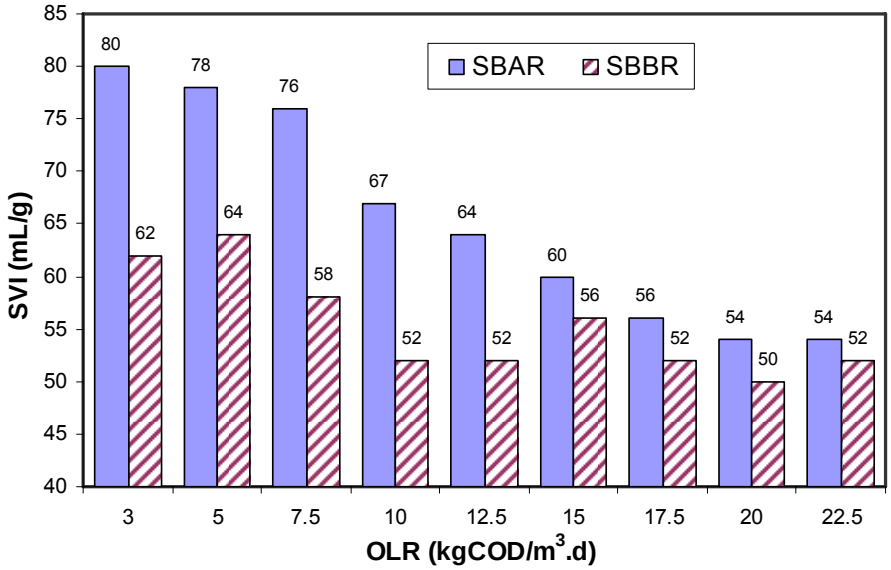


Figure 4.11 Variation of SVI in sludge from effluent of SBAR and SBBR.

Figure 4.11 indicates that there was not much difference in SVI of sludge in effluent at different OLRs. Although sludge form effluent of aerobic granular systems was suspended sludge, it performed well settling ability compared with activated sludge. Between SBAR and SBBR, sludge from effluent of SBBR always demonstrated stability and better settling ability than that of SBAR. Comparing settling ability of granules in SBAR and SBBR, it was obviously to realize that granules in SBBR had better settling ability than that of SBAR. Hence sludge from effluent of SBBR, both suspended and granular sludge, consequently settled better than that of SBAR.

The conclusion for MLVSS and settling ability of sludge in effluent of SBAR and SBBR could be stated as follows:

- At the same OLR, amount of sludge from effluent of SBBR was lower than that of SBAR due to more sludge washed out from reactor in SBBR.
- Sludge in effluent of SBBR performed better settling ability than that of SBAR.

4.2.3 Effluent EPS

Bound and soluble EPS from effluent of SBAR and SBBR was monitored to define membrane fouling potential, as well as calculate removal efficiency of PS and PN in baffle unit and MBR. The rest of PS and PN that could not be removed by biological process in baffled MBR would be the main reason of fouling behavior in membrane. Besides that, EPS was investigated to figure out whether soluble or bound EPS would significantly contribute to fouling behavior of membrane.

4.2.3.1 Soluble EPS of Effluent

In this section, both PS and PN of soluble EPS were monitored to evaluated fouling potential of effluent from aerobic granular systems. It was hypothesized that EPS, especially PS, was the main cause of membrane fouling (Judd, 2004). Contrary to bound EPS which was attached in sludge and formed net structure for granule formation, soluble EPS dissolved in bulk liquid (Hoa, 2002). Soluble EPS was only consumed by biological

process, while bound EPS might be removed by settling sludge. Therefore it could be presumed that soluble EPS causes certain effects on pore membrane clogging, and bound EPS will contribute to cake layer clogging.

Soluble EPS at different OLRs was examined then combined with membrane behavior to figure out correlation between soluble EPS and membrane fouling. Figure 4.12 and 4.13 presents graphs of EPS-PS and EPS-PN versus OLRs respectively.

Figure 4.12 and 4.13 obviously depict that both PS and PN in effluent of SBBR were higher than that of SBAR. In accordance with COD removal in both aerobic granular systems mentioned above, treatment efficiency of SBBR was often higher than that of SBAR so that PS and PN in effluent of SBBR would be higher. It can be deduced that effluent from SBBR could have more effect on membrane fouling, especially pore membrane clogging.

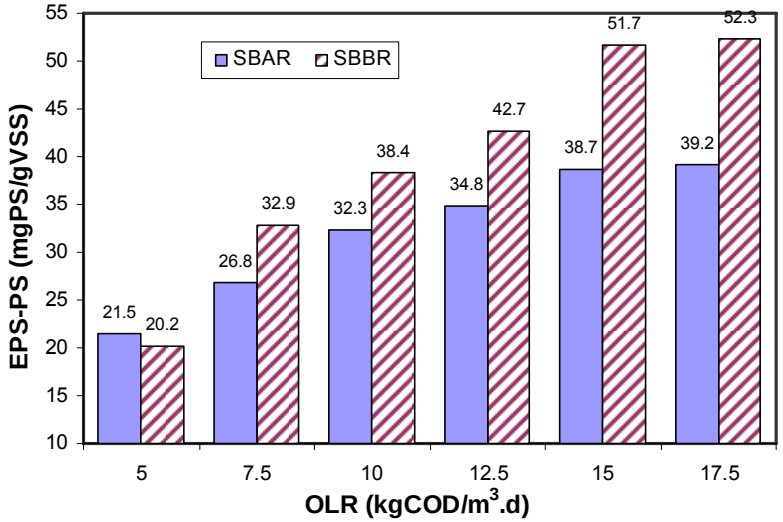


Figure 4.12 Soluble EPS-PS of effluent at different OLRs.

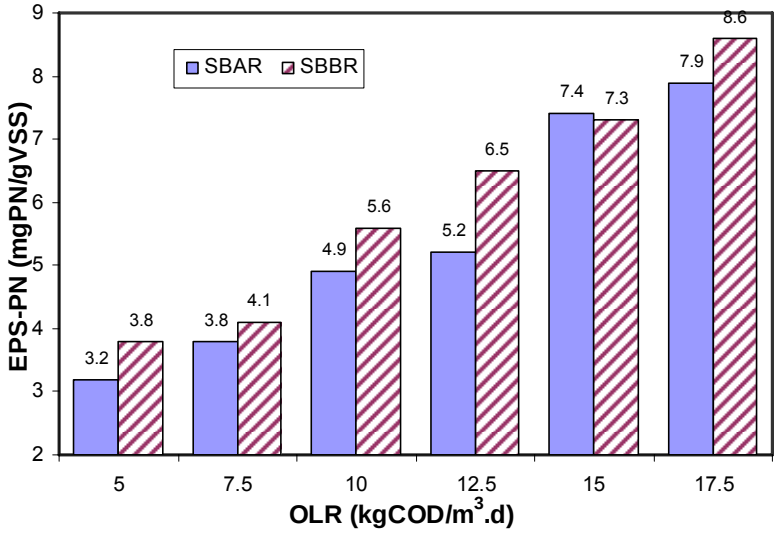


Figure 4.13 Soluble EPS-PN of effluent at different OLRs.

4.2.3.2 Bound EPS of Effluent

Whereas soluble EPS dissolved in bulk liquid, almost bound EPS attached to microorganism or it could be said that suspended sludge consists of bound EPS. Therefore bound EPS together with suspended sludge initially cause membrane fouling by cake layer formation, and then further develop to pore membrane clogging. In order to minimize membrane fouling due to bound EPS, reduce MLSS or suspended solid concentration in MBR was optimum solution because bound EPS directly correlated with suspended sludge. Bound EPS from effluent of aerobic granular system was investigated to firstly evaluate removal efficiency of bound EPS in baffled MBR, and then compare with soluble EPS in membrane fouling.

Most of sludge washed out from aerobic granular system was suspended sludge which displayed low settling ability compared with granular sludge. Although some granular sludge could be found in effluent of aerobic granular system, these were only immature or broken granules. Hence characteristics of sludge from effluent of aerobic granular system were quite similar to activated sludge. However when increasing OLR in aerobic granular system, it could be seen that some of matured granules was washed out. Because the higher OLR the more granules were formed, when amount of granules reached effluent valve, the excess matured granules would be washed out from the system. Therefore, from OLR of 17.5 kgCOD/m^3.d, some matured granules could be observed in effluent. This could be indicated that, at OLR of 17.5 kgCOD/m^3.d, both SBAR and SBBR match the balance point, and the amount of granules in aerobic granular system reached balance because the more granules were formed the more granules were washed out.

Sludge washed out from aerobic granular system was retained in baffled membrane bioreactor. Once a day, sludge was withdrawn to reduce development to anaerobic condition of retained sludge, as well as to minimize sludge in MBR causing membrane fouling. Due to sludge hopper volume of 1.54 L and sludge occupied two third (2/3) of sludge hopper, the amount of 800 ml of sludge was taken out everyday by bottom valves in baffled MBRs.

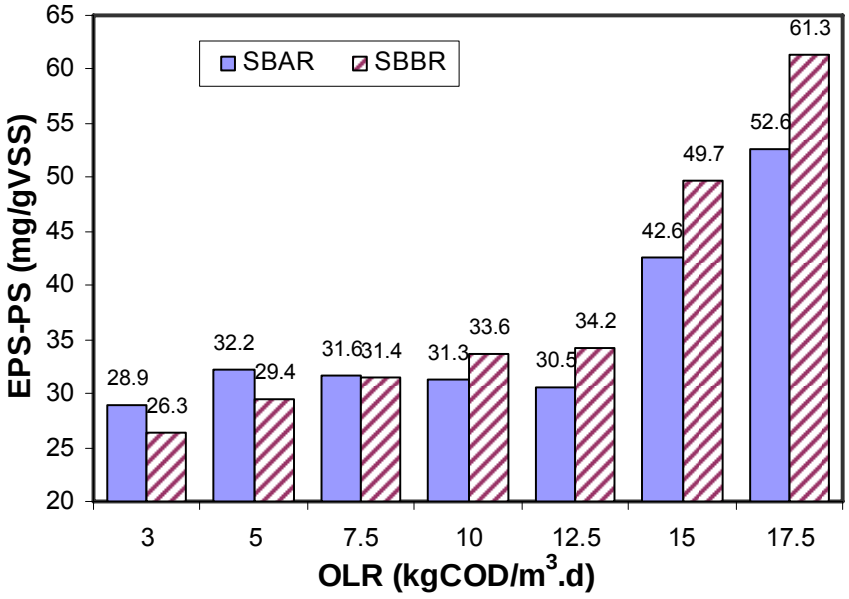


Figure 4.14 Bound EPS-PS of effluent at different OLRs.

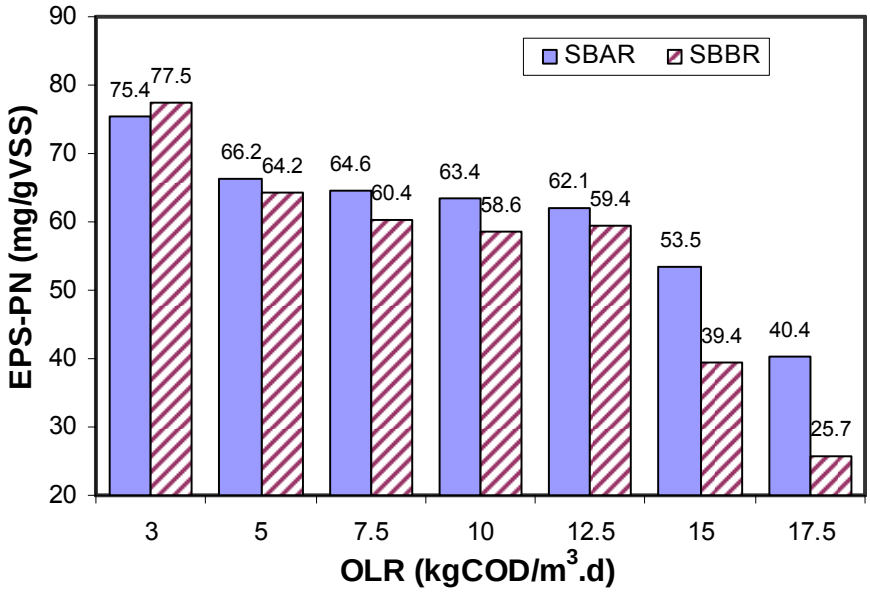


Figure 4.15 Bound EPS-PN of effluent at different OLRs.

Results of bound EPS in sludge from effluent depicted that when OLRs varied from 3 to 12.5 kgCOD/m^3.d, both bound PS and PN did not expressed much changes, or lightly reduced. From OLR of 12.5 kgCOD/m^3.d, bound PS and PN of sludge illustrated proportionally with OLRs. Figure 4.14 and 4.15 exposed correlation of PS and PN with OLR respectively.

Figure 4.14 and 4.15 present that PS from effluent of both SBAR and SBBR was not much different, and similarly happened to PN. PS of sludge in effluent of SBBR was a little bit higher than that of SBAR because aerobic granules appeared sooner in SBBR and also performed better characteristics. In contrary, PN in SBAR was often higher than that in SBBR because contribution of PN in structure of aerobic granules was better in SBBR. In term of structure, it could be explained that structure of aerobic granules in SBBR was stronger and denser than granules in SBAR, so PS of granules in SBBR was higher, and PN of granules in SBBR was lower than that in SBAR.

From the results of EPS (both soluble and bound) in effluent of aerobic granular systems, it could be concluded that:

- Soluble EPS, both PS and PN, in effluent of SBBR was higher than that of SBAR, corresponding to result of aerobic granulation process. These data proved that effluent from SBBR could induce more pore membrane clogging compared with that from SBAR
- Although bound PN in effluent of SBBR was lower than that of SBAR, bound PS in effluent of SBBR was always higher and could promote membrane clogging due to cake layer formation. Because in cake layer membrane clogging, PS plays main function.

4.2.4 MFI

A good indicator to compare fouling potential at different OLRs, as well as between SBAR and SBBR was membrane fouling index (MFI). The higher MFI value the more fouling potential. Although MFI could not provide clear explanation for membrane fouling, it

easily supplied relative comparison in membrane fouling behavior from different samples by comparing with MFI of clear water with those of sample or among MFIs of different samples. Under same test condition (200 mL of sample, 1 atm of pressure, membrane pore size of 0.45 μm) MFI which was the slope of the plot of time per volume versus permeate volume implied filtering ability of sample through 0.45 μm membrane. Figure 4.16 schematically gives out MFI of effluent of SBAR and SBBR at different OLRs.

Figure 4.16 clearly shows that both effluent form aerobic granular systems were progressively influenced by OLRs, and fouling potential of effluent from SBBR was always higher than that of SBAR. It illustrated that under same operation conditions of MBR, effluent from SBBR would easier cause fouling than effluent from SBAR. When OLR of 5 kgCOD/m^3.d, MFI of effluent in SBAR and SBBR was $110*10^3$ and $181*10^3$ s/L^6, these values were almost double at OLR of 7.5 kgCOD/m^3.d. There was significant increase in MFI of effluent from SBAR compared with slightly rise of that in SBBR. At OLR of 17.5 kgCOD/m^3.d, MFI was 15 times folder in effluent of SBAR and 11 times folder in effluent of SBBR than MFI at OLR of 5 kgCOD/m^3.d. This could be stated that MFI was drastically growth after OLR of 5 kgCOD/m^3.d.

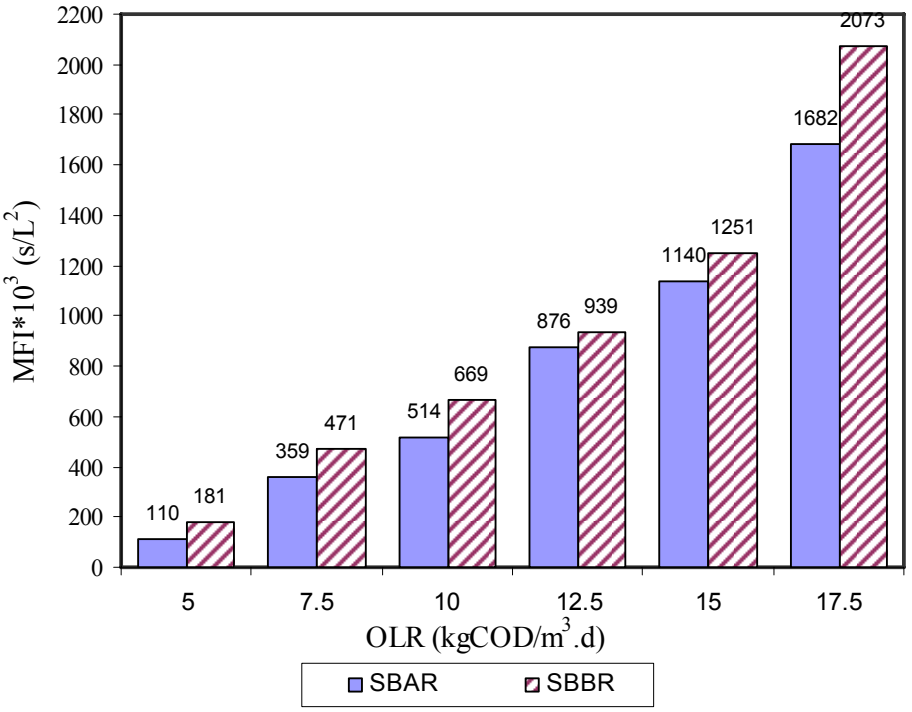


Figure 4.16 MFI of effluent at different OLRs of SBAR and SBBR.

4.3 Baffled Membrane Bioreactor

As mentioned in chapter 3, Baffled Membrane Bioreactors (Baffled MBR) was a combination of settling unit and MBR. Vertical baffled unit function as settling unit, and a flat sheet membrane was installed in the last chamber of Baffled MBR. Both SBAR and SBBR were directly connected to baffled MBRs, it means that effluent from aerobic

granular system was influent of baffled MBR. In this study baffled MBR1 was connected with SBAR, and baffled MBR2 was connected with SBBR.

4.3.1 Sludge Removal Efficiency in Baffled Unit

Thanh's result (2005) showed that if membrane was directly submersed in aerobic granular system, it could damage granules and membrane fouling was still unsolvable. An optimum sequence suggested by Thanh (2005) for combining membrane with aerobic granulation was aerobic granular system, settling unit and MBR. Therefore baffled MBR coupled with aerobic granular system was investigated, and expected to mitigate membrane fouling. Beside that baffled MBR could considerably minimize treatment system area and operational problems that separated system faced.

Baffle unit in this study had two chambers including four vertical baffles (Figure 3.5). The first chamber received effluent from aerobic granular system. After gravitationally passing through the first chamber, wastewater then came to the second one before flowed to MBR. Both chambers had sludge hoppers to contain sludge which was withdrawn everyday depending on amount of washed out sludge. In order to evaluate sludge removal efficiency of baffled unit, MLSS was examined before entering and after leaving baffled unit. The difference of MLSS between 2 points was the amount of sludge retained in sludge hopper. Figure 4.17 and 4.18 orderly displayed variations of MLSS in baffled MBR1 and baffled MBR2 when OLR in aerobic granular system prolonged from 7.5 to 17.5 kgCOD/m^3.d.

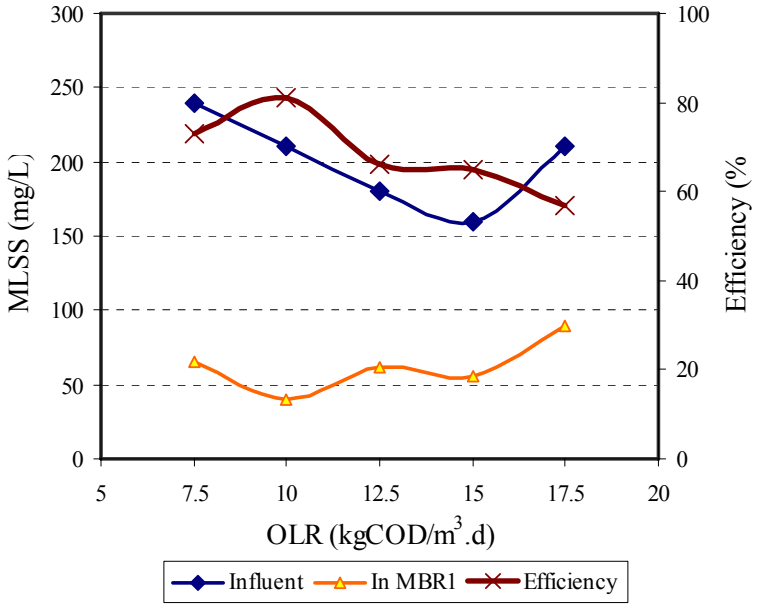


Figure 4.17 Sludge removal efficiency in Baffled MBR1.

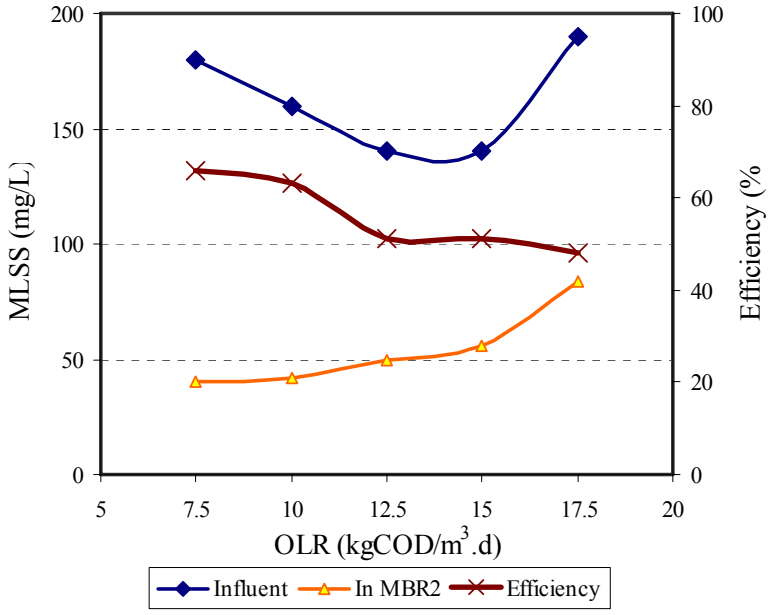


Figure 4.18 Variation of MLVSS in Baffled MBR2.

Figure 4.17 and 4.18 present when OLR in aerobic granular system increased from 7.5 to 15 kgCOD.m^3.d, the MLSS in influent (effluent of aerobic granular system was influent of baffled MBR) respectively decreased. This could be understood that generated biomass in aerobic granular system made aerobic granules bigger and denser. So with OLR from 7.5 to 15 kgCOD/m^3.d, aerobic granular system was quite stable and washed out sludge was little. However at OLR of 17.5 kgCOD/m^3.d, MLSS in effluent of aerobic granular system drastically increased due to breaking of some granules. In conclusion, when OLR was greater than 17.5 kgCOD/m^3.d, aerobic granules was affected and generally reduced treatment ability.

Removal efficiency in figure 4.17 and 4.18 clearly indicated that more than 50% of washed out sludge was retained in baffle unit. In baffled MBR1 (connected to SBAR) removal efficiency varied from 57 to 81% whereas in baffled MBR2 (connected to SBBR) removal efficiency changed from 48 to 66%. Moreover when comparing effluent from SBAR and SBBR it could be concluded that washed out sludge from SBAR had better settling ability than that from SBBR.

Actually figure 4.17 and 4.18 did not completely represent for sludge removal efficiency in baffled units because all washed out sludge after leaving baffled remained in MBR, therefore biomass concentration in both MBR1 and MBR2 progressively raised in line with operation time. Hence removal efficiency in both baffled MBR was only utilized as a relative evaluation.

After OLR in aerobic granular system varied from 7.5 to 17.5 kgCOD/m^3.d, optimum OLR for operation of aerobic granular system was 15 kgCOD/m^3.d at which effluent MLSS was stable. Membrane experiment was conducted in 1.5 month. Figure 4.19 depicts the change of MLSS in baffled MBRs at constant OLR in aerobic granular system.

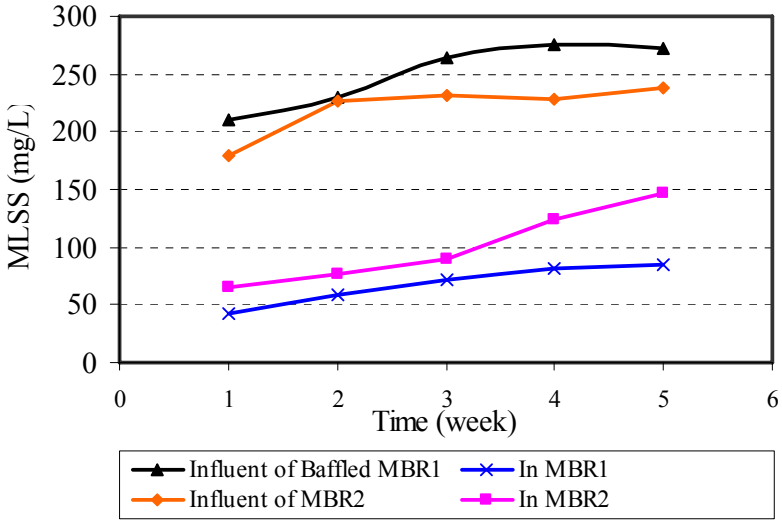


Figure 4.19 MLSS in of Baffled MBR1, 2.

Figure 4.19 obviously shows that MLSS in membrane chambers (or MBR) kept on increasing together with operation time. This could be comprehended that sludge accumulated in MBR because membrane was in dead-end option. Despite biological degradation continuously carried out under aerated conditions, sludge deposit in MBR continued. In MBR1, MLSS was slowly increased in 5 weeks while in MBR2 MLSS was stable in first 2 weeks and considerably rose in later 2 weeks. The more sludge in MBR the easier membrane fouling. This could imply that membrane in MBR2 was more possible to fouling than that in MBR1. Comparing MLSS in influent and in MBR, figure 4.19 again proved that sludge from SBAR get better settling ability than that in SBBR.

4.3.2 EPS Variation in Baffled MBR

EPS was hypothesized as the main factor that contributes to membrane fouling (Cho et al., 2004). Therefore objective of this study was to figure out whether soluble or bound EPS considerably attributed to membrane fouling. Variation of EPS (both soluble and bound) was monitored in baffled MBRs to find out effects of different EPSs on membrane fouling. While bound EPS was the key bridging factor for aerobic granulation and directly influence fouling due to cake layer formation, soluble EPS was regarded as dissolved substrate and mostly affect on pore membrane clogging. Following parts will present in details EPS variation in baffled MBRs.

4.3.2.1 Bound EPS

EPS was the frame structure for biomass attachment (Hoa, 2002), therefore bound EPS, especially bound PS, correlated with sludge (both suspended and granular sludge) and simultaneously varied with MLSS concentration in baffled MBR. Figure 4.20 indicates bound EPS (PS and PN) in Baffled MBR1.

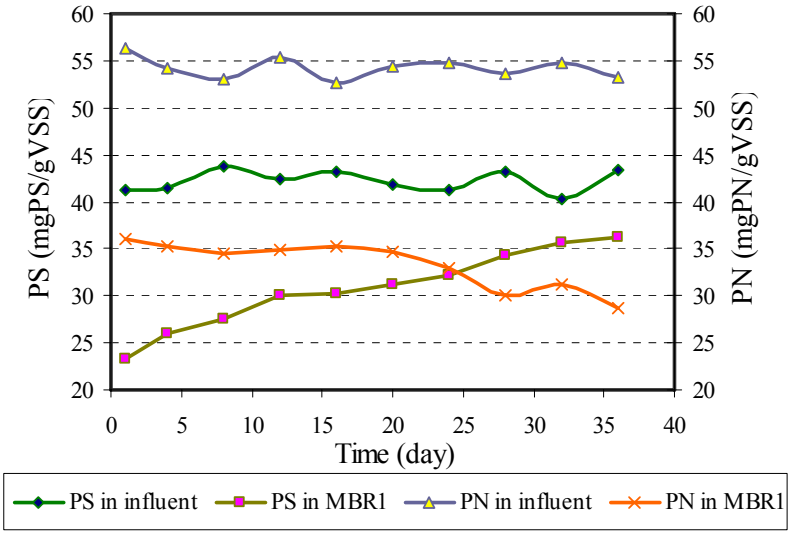


Figure 4.20 Bound PS and PN in Baffled MBR1.

According to figure 4.20, it could easily conclude that at constant OLR of 15 kgCOD/m^3.d, bound PS and PN were rather stable because amount of washed out sludge from aerobic granular system was stable. PS varied around 55 mgPS/gVSS, while PN changed from 40 to 45 mgPN/gVSS. It could be seen that PS concentration in MBR1 progressively increasing due to accumulation of biomass in MBR. At initial time of operation, biomass in MBRs was little. However the longer operation times the more biomass concentration in MBR1 was accumulated. As a result, bound PS increased with operation time.

In contrast to PS in MBR, PN had trend of reduction. This could be explained that PN did not directly relate to biomass network, and PN was degraded by biological process in aerated MBR. Therefore PN in baffled MBR was slowly decreased.

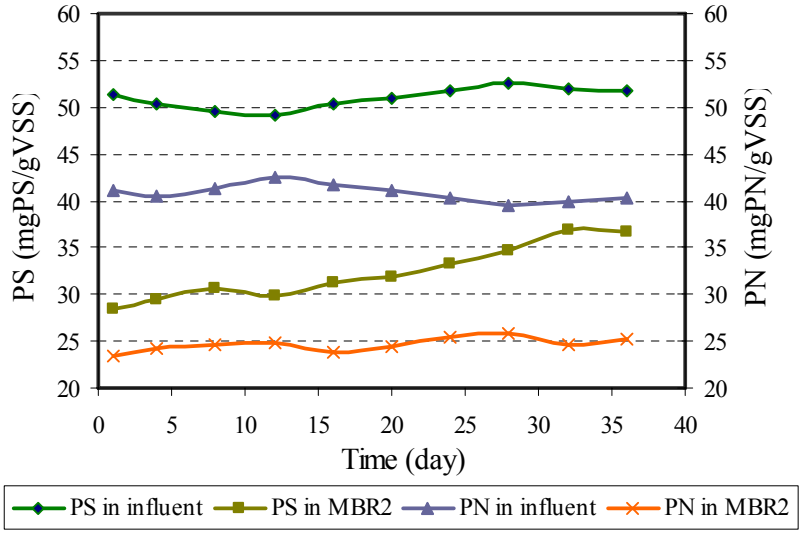


Figure 4.21 Bound PS and PN in Baffled MBR2.

Compared with influent bound PS of baffled MBR1, influent bound PS of MBR2 was higher. The result of effluents from SBAR and SBBR also figured out bound PS in SBBR was always higher than that in SBAR. Consequently, PS remained, as well as accumulated in MBR2, was higher than that in MBR1. Variation of PN in baffled MBR2 was rather similar to that in MBR1. PN in MBR2 was nearly a half of PN in MBR1. Because both PN in influent and PN assimilation in MBR2 was stable, PN concentration in MBR2 did not indicate an accumulation.

In conclusion, in both MBR there was a gradual increase of bound PS whereas bound PN was quite stable and low concentration. This could be assumed that PS could have certain affects on membrane fouling.

4.3.2.2 Soluble EPS

On contrary to bound EPS, soluble EPS was not influenced by MLSS because soluble EPS did not directly correlate with biomass. Like a substrate diluting in bulk liquid, soluble EPS was also digested in aeration condition by biological process, so soluble EPS could not accumulate in aerated MBR if there was sufficient aeration. Therefore soluble EPS (both PS and PN) could have less effect on fouling. The profile data about soluble EPS in baffled MBR1 and baffled MBR2 was plotted in figure 4.22 and 4.23, respectively.

Figure 4.22 and 4.23 obviously show that treatment efficiency of membrane in MBR1 was high (more than 80%). Compared with bound EPS, soluble EPS was treated better in MBR. Treatment ability in MBR was caused by 2 factors, i.e. biological assimilation as aeration reactor, and filtration by membrane. Despite soluble PS was always higher than soluble PN, both PS and PN almost got same concentration in permeate. It could be comprehended PS and PN concentration in MBR1 did not cause much impact on membrane filtration, or permeate quality only depended on pore size of membrane. With pore size of 0.1 µm, PS and PN in permeate was always less than 4 mg/L.

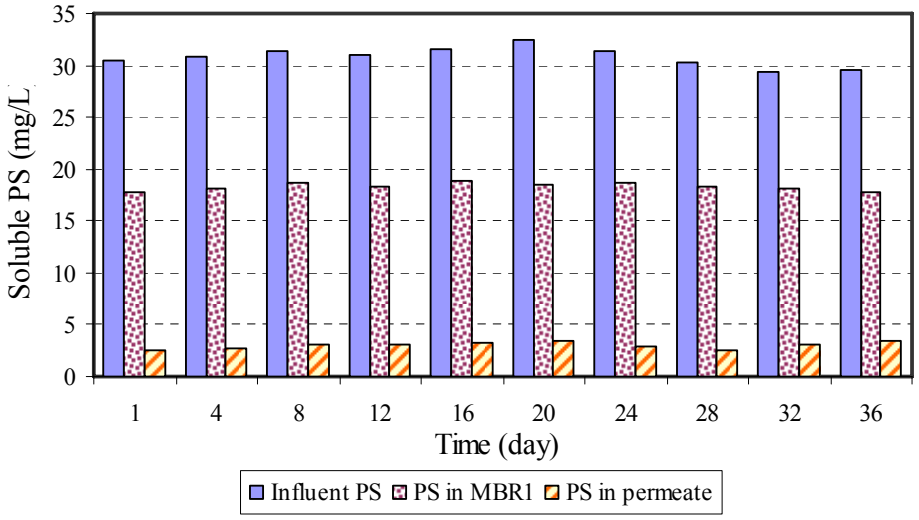


Figure 4.22 Soluble PS in Baffled MBR1.

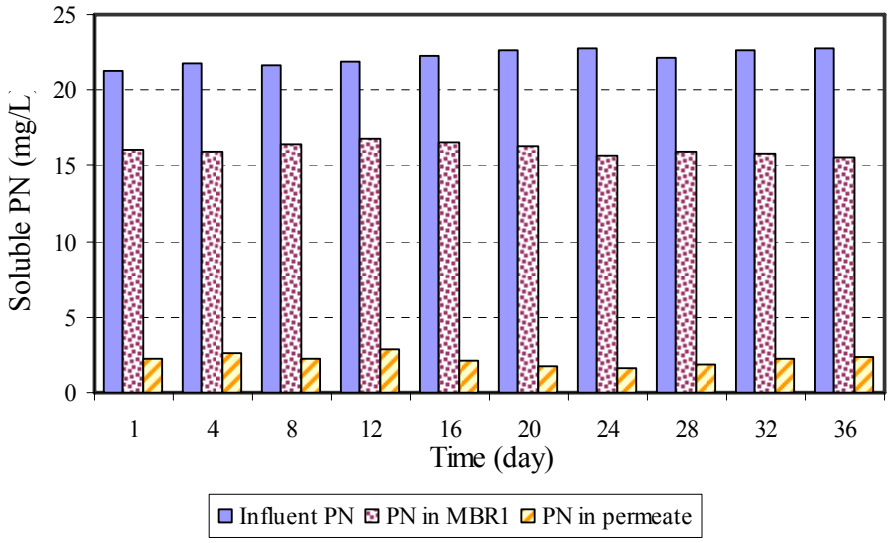


Figure 4.23 Soluble PN in Baffled MBR1.

Although soluble EPS in influent of baffled MBR1 was a little higher than that of baffled MBR2, soluble EPS in baffled MBR2 was expressed the different variation which plotted in figure 4.24 and 4.25.

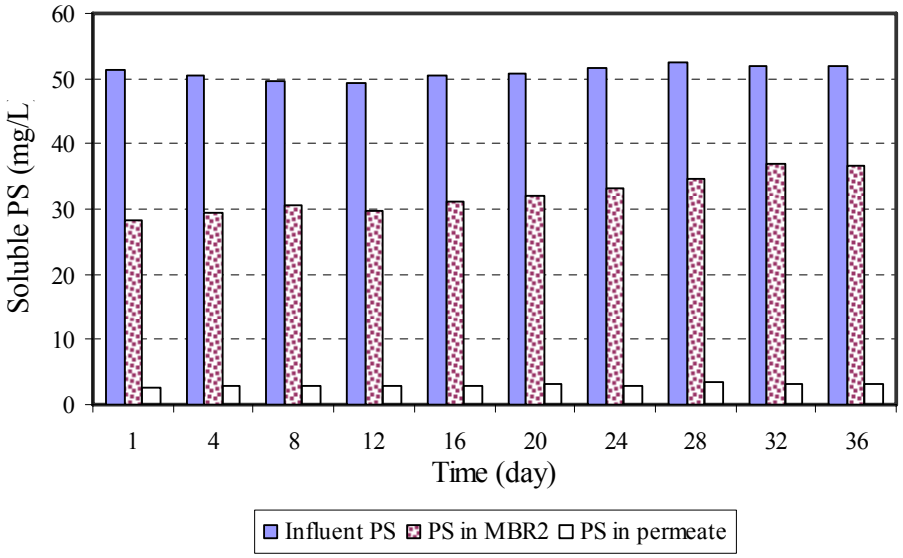


Figure 4.24 Soluble PS in Baffled MBR2.

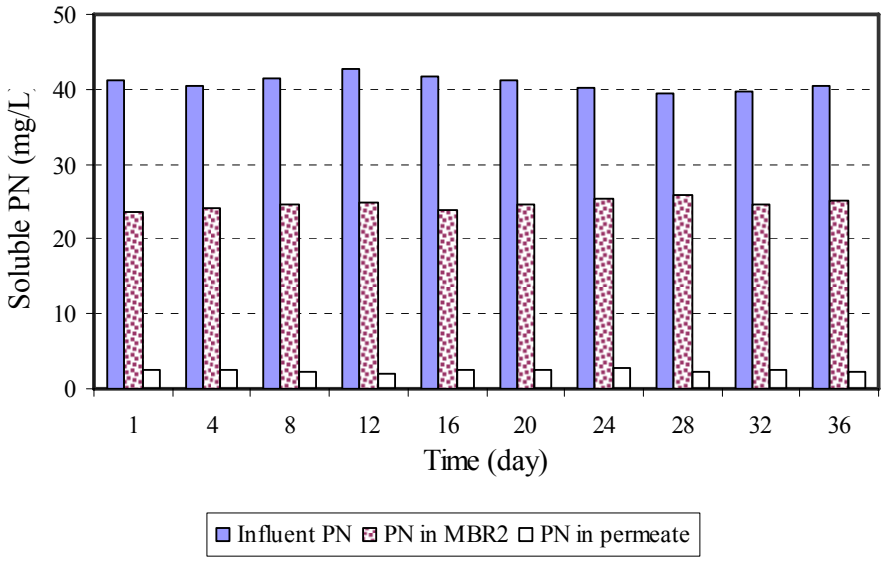


Figure 4.25 Soluble PN in Baffled MBR2.

Similarly to soluble EPS results in baffled MBR1, soluble EPS (PS and PN) in baffled MBR2 was table in influent, in MBR1 and in permeate. With membrane pore size of 0.1 μm, permeate quality did not impact by influent concentration, especially substrate concentration in membrane chamber. However soluble PS and PN still contributed to membrane fouling. Compared with results in MBR1, it could be observed that PS and PN in MBR2 were higher than that in MBR1. While average value of soluble PS in MBR1 was 17 mg/L, that value in MBR2 got average of 30 mg/L. In term of soluble PN, whereas value in MBR1 was only 16 mg/L, that in MBR2 was over 21 mg/L. Due to soluble EPS dissolved in bulk liquid, it particle size was mostly smaller than that of bound EPS. As a result, soluble EPS could be the main factor affecting on pore membrane fouling.

4.3.3 MFI

As mentioned in characterization of effluent from aerobic granular systems, MFI was employed to relatively evaluate fouling potential of different samples. MFI could be combined with EPS and MLSS results to properly interpret fouling behavior of membrane in MBR1 and MBR2. Graph in figure 4.24 below illustrates variation of MFIs from influents to permeate in baffled MBR1 and baffled MBR2.

From figure 4.26 it could be clearly seen that MFI value in MBR2 was much higher than that in MBR1. Although both Baffled MBRs got the same MFI in influent, MFI in MBR2 was drastically increased in membrane chamber. When linking information with bound EPS, especially bound PS, it could be explained that high bound PS might be the main reason for membrane fouling.

Permeate from both Baffled MBRs was almost similar. Due to very small pore size of membrane (0.1 μm), most of substrate was retained in MBRs and permeate quality nearly reached pure water quality (MFI of DI water was $0.2*10^3$ s/L^2). In conclusion, flat sheet membrane with pore size of 0.1 μm could be applied for water reuse and reclamation, especially in dealing with industrial wastewater.

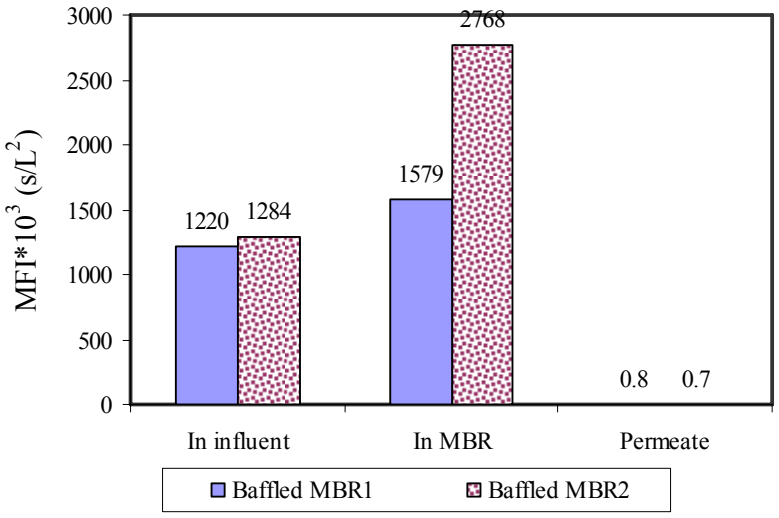


Figure 4.26 MFI at influent, MBR and permeate from Baffled MBR1, 2.

4.3.4 Particle Size Distribution in MBR

This experiment was conducted to examine particle size in membrane chambers. It was presumed that fouling in membrane was caused by 2 main factors. The first one namely cake layer fouling or clogging was made due to particles whose size was bigger than pore size of membrane (Ognier et al., 2002). These particles would directly attach on membrane surface when filtrating or form cake layer, and then reduce pore size of membrane. Membrane fouling because of cake layer could be recovered by pressurized water. The second one was pore fouling or irreversible fouling which was the stick of particles on pore of membrane (Stephenson et al., 2000). This kind of fouling was more difficult in recover than cake layer fouling. In order to remove pore fouling, chemicals need to be applied; recovery percentage in good condition was often 80%.

Particle size distribution test might figure out percentage of different particle sizes in bulk liquid and mean diameter of particles. The results from particle size distribution test shall be compared with pore size of membrane because particle size on membrane chamber affected directly to membrane fouling. In addition to that, particle size distribution was possibly used to explain whether cake layer fouling or irreversible fouling would significantly contribute to membrane fouling. The results from particle size distribution from membrane chamber of Baffled MBR1 and 2 were presented in figure 4.27 as follows.

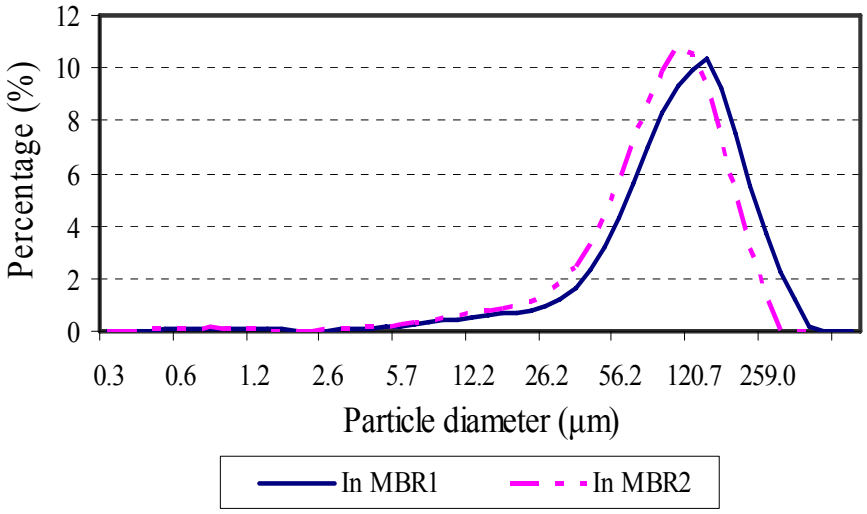


Figure 4.27 Particle Size Distribution in membrane chamber of Baffled MBR1, 2.

Mean diameter of particle size in MBR1 and MBR2 was concluded in table 4.1.

Table 4.1: Mean size diameter of particles in MBR1, 2

Parameters	Unit	MBR1	MBR2
Mean diameter	µm	97.35	91.31
Standard deviation	-	1.06	0.92
Uniformity	-	0.5113	0.4787

4.3.5 Fouling Behavior in MBR1 and MBR2

Under fixed permeate flux, transmembrane of flat sheet membrane increased day by day due to both cake layer and pore (irreversible) fouling. Used as an indicator for membrane fouling during operation, transmembrane pressure was measured in kPa by digital manometer. When reaching pressure of 80 kPa in suction pipeline, membrane was considered as fouling and stopped for cleaning. After cleaning, if filtration flux covered 80% under the same initial transmembrane pressure, membrane could be reused for filtration otherwise a new flat sheet membrane was replaced. Fouling started to happen from the initial time of operation, thus sometimes permeate flux should be adjusted properly to maintain constant permeate. Transmembrane pressure increased everyday and was sufficiently monitored to adjust suction pump.

Besides monitoring fouling behavior of membrane operation, digital manometer was employed to calculate different kinds of membrane resistances, such as: intrinsic resistance, cake resistance, and irreversible resistance.

4.3.5.1 Initial Resistance

Initial resistance test was conducted before applying membrane to MBR. By filtering DI water at difference filtration fluxes and recording the corresponding TMPs, this experiment helped to figure out intrinsic membrane resistance. Any flat sheet membrane was examined initial resistance test before applying in MBRs, and the results showed that initial resistance test were almost similar for every flat sheet membrane. This means that at the beginning both membranes had the same initial resistance. Table 4.2 shows typical result of initial resistance measurement of flat sheet membrane applied to MBR1 and MBR2. (Membrane area is 6 x 11 = 66 cm^2 = 0.0066 m^2).

From the table 4.2, graph on figure 4.28 shows the relationship between filtration flux and TMP, then trend line was drawn to find out membrane resistance. The membrane resistance was derived from the slope of the linear curve of ΔP versus J. With dynamic viscosity of pure water is 0.798*10-3 Pa.s (or N.s/m^2), initial membrane resistance was calculated as following:

$$R_m = \frac{0.0276(kPa)x1000(\frac{Pa}{kPa})x3600(\frac{s}{h})x1000(\frac{L}{m^3})}{(\frac{L}{m^2.h})x0.798x10^{-3}(Pa.s)} = 1.245x10^{11}(m^{-1})$$

In conclusion, intrinsic resistance of flat sheet membrane with pore size of 0.1 μm was 1.245*10^{11} m^{-1}. This result was used as baseline to evaluate recovery of membrane after cleaning, as well as to calculate cake layer resistance, and irreversible resistance if total resistance was known.

Table 4.2 Profile data of initial membrane resistance

Flux (mL/min)	Specific flux (L/m^2.h)	TMP (kPa)
3.8	34.5	5.6
4.8	43.6	7.6
5.5	50.0	8.9
5.9	53.6	10.9
6.6	60.0	12.6
8.2	74.5	17.5

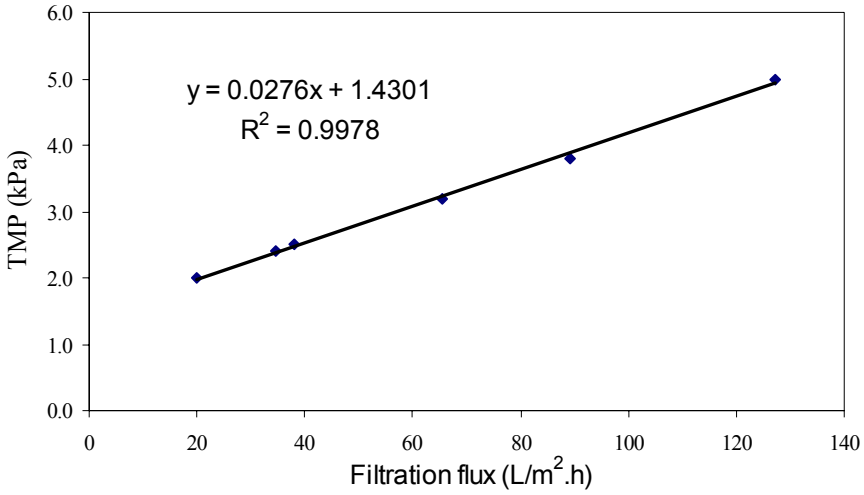


Figure 4.28 Initial membrane resistance

4.3.5.2 Fouling Behavior in MBR1 and MBR2

Flat sheet membrane was installed to membrane module and immersed into MBRs. A peristaltic pump (or suction pump) was used to suck permeate with flow rate of 3 mL/min. Operation cycle of membrane was 8 minutes on and 2 minutes off, and was repeated in 24 hours. Equivalently filtration flux of membrane was maintained at 21.82 L/m^2.h. Transmembrane pressure was observed to evaluate fouling behavior of membrane. During operation, transmembrane pressure kept on increasing due to cake layer formation and pore clogging, thus filtration flux in both membranes was accordingly adjusted in order to keep filtration flux at constant of 3 mL/min. Membrane fouling occurred when TMP came over 80 kPa. At that time, membrane was stopped for cleaning. Figure 4.29 presents the variation of TMP versus time in MBR1 and MBR2.

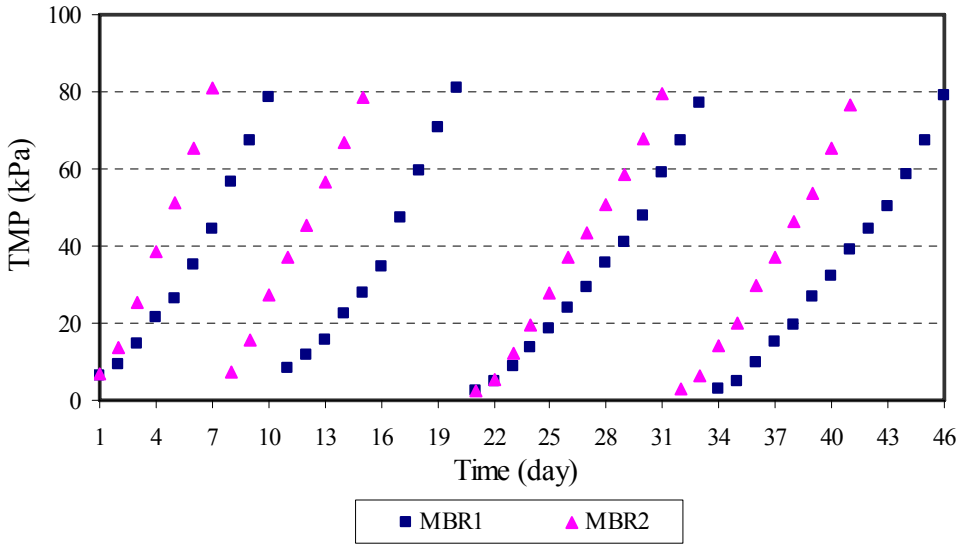


Figure 4.29 Fouling behavior in MBR1 and MBR2.

From figure 4.28, 4.29 and table 4.1 it could be seen that mean particle size in both MBRs was almost similar, it was 97.35 and 91.31 μm in MBR1 and MBR2, respectively. This result showed that particle size of sludge in bulk liquid was nearly 1000 fold bigger than pore size of membrane (0.1 μm). Consequently, cake layer had significant influence on membrane fouling because in membrane chamber there was a large amount of particles (more than 90%) whose size was bigger than pore size of membrane.

Although the membrane experiment was conducted in 1.5 months, the figure 4.29 clearly pointed out that membrane in MBR2 was easier to foul than that in MBR1. In entire 4 cycles of each MBR, average time of membrane operation in MBR2 was 9 days while this value in MBR1 extended to 12 days. When connecting this result with EPS and MFI, it could concluded that

- Bound PS had more significant on cake layer fouling while bound PN expressed less influence.
- Soluble EPS (both PS and PN) mainly contributed to pore fouling which was more important than cake layer fouling. In general, soluble EPS had considerable impact on membrane filtration, especially membrane fouling.

4.3.5.3 Membrane Cleaning and Membrane Resistance

When transmembrane pressure came over 80 kPa, membrane was stopped and disconnected for cleaning. Cleaning procedure based on instruction of manufacturer was modified according to experiment conditions. Before starting cleaning process, membrane was examined total resistance which includes cake and pore resistance. This step was considered as initial step for determining cake and pore resistances. Membrane module was taken out from MBR and a tank containing DI water was used to measure membrane resistances. Process of determine membrane resistance similar as measuring initial membrane resistance. After finishing measuring total membrane resistance, cake layer was removed by using properly pressurized flux. Then membrane was continued to measure resistance caused by pore and intrinsic resistances. Cake resistance was the difference of total and pore and intrinsic resistance. The next step of cleaning procedure was pore cleaning which included caustic and acid washing subsequently. And the last step that also essential for closing cleaning process was sanitation washing in which 150 ppm chloride solution was circulated to remove residue chemicals. Similar as previous step, after cleaning membrane was determined resistance. Clean membrane resistance was supposed to get 80% of initial resistance and pore resistance was the difference of total, cake and cleaned membrane resistance. Table 4.3 presents results of membrane resistance after sequencing steps.

Result from table 4.3 obviously points out that in both MBRs, pore clogging (or irreversible fouling) mainly contributes in fouling of membrane. Due to high aeration on membrane surface in MBR, cake layer formation significantly reduces and only participated 31.8% (MBR1) and 37.4% (MBR2) in total membrane resistance.

Between results in MBR1 and MBR2, it could be observed that cake layer resistance in membrane of MBR2 was higher while irreversible resistance was lower than that that of MBR1. When combing with EPS results, it could be explained that soluble EPS contributed to pore fouling and bound EPS had more effect on cake layer fouling. And between pore and cake layer fouling, pore fouling was more serious and quickly reduced filtration ability of membrane.

Table 4.3 Membrane Resistances

Resistance	Value (m^{-1})	Percent (%)
Membrane in MBR1 (connected with SBAR)		
Total resistance (R_T)	$3.156*10^{12}$	
Intrinsic resistance (R_m)	$1.245*10^{11}$	3.9
Cake layer resistance (R_c)	$1.179*10^{12}$	37.4
Irreversible resistance (R_f)	$1.852*10^{12}$	58.7
Membrane in MBR2 (connected with SBBR)		
Total resistance (R_T)	$2.593*10^{12}$	
Intrinsic resistance (R_m)	$1.245*10^{11}$	4.8
Cake layer resistance (R_c)	$8.245*10^{11}$	31.8
Irreversible resistance (R_f)	$1.644*10^{12}$	63.4

4.4 Comparison between results in this study and in previous study (Thanh, 2005)

Some parameters were selected to compare results in this study and Thanh's study. Table 4.4 below displayed the comparison between this study and Thanh's study (2005).

Form table 4.4, it could be concluded that SBAR with bivalve shell as carrier performed better for aerobic granulation compared with SBBR and basalts as carrier.

Table 4.3 Comparison between this study and Thanh's study (2005)

	This study		Thanh's study
Aerobic granulation			
Reactor	SBAR	SBBR	SBAR
Carriers	Basalt	Basalt	Bivalve shell
Maximum OLRs ($kgCOD/m^3.d$)	20	20	30
Granules size (mm)	1.5 – 3.7	1.7 – 4.0	0.5 – 4.0
SVI (mL/g)	24 - 32	21 - 28	18
MLVSS (mg/L)	11230	12780	> 12000
Settled biomass concentration ($mg/L_{granule}$)	28 - 47	30 - 53	20 – 62
Effluent characterization			
MLSS (mg/L)	160 - 1000	140 – 180	300 – 1200
Soluble PS (mgPS/gVSS)	8.6 – 39.2	9.4 – 52.3	5 – 30
Soluble PN (mgPN/gVSS)	2.4 – 7.9	2.7 – 8.6	0.5 – 25
$MFI*10^3$ (s/L^2)	110 - 1682	181 - 2073	130 - 1018
Membrane bioreactor			
Membrane type	PVDF flat sheet	PVDF flat sheet	Ceramic hollow fibre
Operation time	12	9	7 – 10
Main factor causing fouling	Pore fouling (58 – 63%), soluble EPS	Pore fouling (58 – 63%), soluble EPS	Cake layer (68.4%)

Chapter 5

Conclusions and Recommendations

5.1 Conclusions

This study investigated aerobic granular system combining with baffled MBR in order to treat high strength organic wastewater. The main research objectives were divided into 3 parts which had close relationship, i.e. forming aerobic granules in SBAR and SBBR, characterizing effluent from aerobic granular systems at different OLRs, and monitoring membrane fouling in baffled MBRs. The conclusion based on whole experiment results was summarized as follows:

For aerobic granulation in SBAR and SBBR

1. Aerobic granules could be formed at OLR of 3 kgCOD/m^3.d in both SBAR and SBBR, and it took 45 days to get matured granules.
2. Matured granule size was 1.5 and 1.7 mm in SBAR and SBBR, respectively. When applying high OLR, granule size proportionally increased with the increase of OLRs.
3. In term of settling ability, matured granules had advantages over activated sludge. SVI of aerobic granules was 24 mL/g in SBAR and 20 mL/g in SBBR, while SVI of activated sludge was 220 mL/g.
4. After aerobic granules matured, MLVSS in reactor positively influenced with the increase of OLRs. At OLR of 20 kgCOD/m^3.d, MLVSS in reactor could reach 11230 mg/L and 12780 mg/L in SBAR and SBBR, respectively. This proved that aerobic granular system was able to treat high strength organic wastewater.
5. Settled biomass concentration of aerobic granules varied proportionally with OLRs. Settled biomass concentration in SBAR could obtain 47.8 g/$L_{granule}$ and in SBBR was 53.7 g/$L_{granule}$, whereas this value in activated sludge was only 3.8 g/L.
6. Aerobic granular system could treat high strength organic wastewater with OLR varied from 5 to 17.5 kgCOD/m^3.d. When OLR was over 20 kgCOD/m^3.d, aerobic granules was unstable and broken. Optimum OLR for aerobic granular system was 15 kgCOD/m^3.d.
7. In aerobic granulation process, SBBR performed better than SBAR because bubble column configuration could reduce clogging, as well as provide better settling area compared with SBAR.
8. Bound EPS results in SBAR and SBBR showed that PS and PN in SBBR was always higher than that in SBAR. When OLRs increased from 5 to 17.5 kgCOD/m^3.d, PS increased also while PN decreased. Bound EPS results implicated that SBBR had more fouling potential than SBAR.

For characterizing effluent from aerobic granular system

1. MLSS in effluent causes certain effects on membrane fouling due to cake layer formation. Despite of high OLRs, MLSS varied from 180 to 310 mL/g in effluent SBAR, and from 140 – 220 mL/g in that of SBBR. However when OLR was more than 20 kgCOD/m^3.d, there was a large amount of MLSS consisting of worn

granular sludge. It was suggested that OLR of 15 kgCOD/m^3.d was optimum for aerobic granular system.

2. SVI of sludge was examined to evaluate settling ability of washed out sludge. Although most of washed out sludge was suspended, it had better settling ability than activated sludge. Sludge from effluent of SBBR got SVI lower than that of SBAR.
3. In term of soluble EPS, both PS and PN in effluent of SBBR displayed higher value than that of SBAR, and simultaneously increased with the increase of OLRs.
4. While bound PN seemed to gradually reduce with increasing OLRs, bound PS was quite stable at OLRs less than 15 kgCOD/m^3.d, and it drastically increased when OLR greater than 15 kgCOD/m^3.d. The variation of bound PS and PN was mostly impacted by MLSS in effluent.
5. MFI was a general indicator for fouling potential. MFI results in this study showed that effluent from SBBR induced more fouling than that from SBAR, as well as higher OLR in aerobic granular system caused more fouling potential. It could be inferred that soluble EPS had more influence on membrane fouling, especially irreversible fouling.

For investigating membrane fouling in baffled MBRs

1. MLSS increased together with operation time due to MLSS accumulation in membrane chambers. MLSS removal efficiency in baffled unit was about 60 – 80%.
2. At OLR of 15 kgCOD/m^3.d in aerobic granular system, bound PS in both MBRs was increased with operation time, while bound PN was stable or slightly decreased due to biological assimilation.
3. Soluble EPS in baffled MBRs presented different trend with bound EPS. Soluble PS was 17 and 31 mg/L in MBR1 and MBR2, respectively. Soluble PN in MBR1 and MBR2 was maintained at 16 and 22 mg/L, respectively. In general, soluble EPS in MBR2 was always higher than that in MBR1.
4. Particle size distribution in both membrane chambers almost similar, it was 97.35 and 91.31 μm in MBR1 and MBR2, respectively. Particle size was nearly 1000 fold higher than pore size of membrane and significant contributed to membrane fouling.
5. During 4 operation cycles of membrane, average operation time of membrane in MBR1 was 12 days while in MBR2 was 9 days of operation. Fouling behavior of membrane figured out that pore size fouling contributed remarkable compared with cake layer fouling.

5.2 Recommendations

This study again enlarged knowledge about aerobic granulation which was expected as innovative technology for wastewater treatment. Furthermore, combining aerobic granular system with baffled MBR was promising trend in water reuse and reclamation. In order to fully understand about aerobic granulation and its effects on membrane fouling, future researches should consider the following recommendations:

1. Investigate aerobic granulation with real wastewater, especially high strength wastewater like industrial wastewater, leachate food processing, etc.
2. When operating aerobic granulation system, major problem was clogging of pipe line (inlet, out let, diffuser, etc.) due to aerobic granules. Therefore special equipments for high suspended wastewater should be applied.
3. Investigate save energy reactors replacing for Airlift and Bubble reactors which consumed a lot of energy by supplying high air velocity.
4. Investigate mechanism that damage aerobic granules. It was predicted that granule size, substrate and oxygen diffusion, or bound EPS strongly correlated to formation and disruption of aerobic granules. Furthermore, future study might investigate on stability of aerobic granule under shock loading, or high nutrient and heavy metal concentration.
5. Investigate variation of EPS base on effects of nutrient in aerobic granular system because EPS had close relationship to membrane fouling.
6. In order to successful coupling aerobic granular system with MBR, future study should investigate method to control EPS in membrane chamber. It could be intensify aeration in MBR.

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Appendix A

Experimental result of aerobic granulation in SBAR and SBBR

Table A-1 COD results in SBAR and SBBR

Day	Influent	OLR	SBAR		SBBR	
			Effluent	Removal Eff.	Effluent	Removal Eff.
	(mg/L)	(kgCOD/m^3.d)	(mg/L)	(%)	(mg/L)	(%)
1	712	3.0	158	78	173	76
3	718	3.0	144	80	151	79
5	702	2.9	137	80	137	81
7	730	3.0	122	83	122	83
9	736	3.1	122	83	108	85
11	734	3.1	108	85	94	87
13	706	2.9	115	84	79	89
15	708	2.9	94	87	65	91
17	710	3.0	72	90	58	92
19	732	3.0	65	91	43	94
21	738	3.1	50	93	50	93
23	742	3.1	57	92	36	95
25	694	2.9	57	92	58	92
27	692	2.9	43	94	43	94
29	722	3.0	58	92	36	95
31	720	3.0	43	94	36	95
33	746	3.1	43	94	29	96
35	738	3.1	36	95	43	94
37	748	3.1	29	96	36	95
39	712	3.0	22	97	29	96
41	706	2.9	29	96	43	94
43	724	3.0	29	96	43	94
45	743	3.1	29	96	29	96
47	1172	4.9	60	95	71	94
49	1240	5.2	50	96	62	95
51	1236	5.1	73	94	48	96
53	1240	5.2	48	96	96	92
55	1810	7.5	91	95	127	93
57	1792	7.5	126	93	108	94
59	1810	7.5	109	94	127	93
61	1805	7.5	90	95	90	95
63	2389	9.9	96	96	143	94
65	2400	10.0	120	95	120	95
67	2405	10.0	120	95	192	92

69	2402	10.0	144	94	168	93
71	3010	12.5	181	94	181	94
73	3001	12.5	210	93	210	93
75	3005	12.5	240	92	120	96
77	3012	12.5	181	94	181	94
79	3587	14.9	215	94	179	95
81	3598	15.0	180	95	216	94
83	3605	15.0	144	96	180	95
85	3602	15.0	252	93	108	97
87	4186	17.4	335	92	167	96
89	4216	17.5	295	93	126	97
91	4204	17.5	210	95	252	94
93	4205	17.5	168	96	210	95
95	4796	20.0	240	95	192	96
97	4816	20.0	289	94	289	94
99	4810	20.0	192	96	241	95
101	4805	20.0	384	92	336	93
103	5420	22.5	542	90	379	93
105	5400	22.5	810	85	648	88
107	5405	22.5	1189	78	865	84

Calculation of Organic Loading Rate (OLR) from COD influent concentration:

Volume of reactor : 2.5 L

Withdraw/ feeding volume : 1.3 L

Cycle time : 3 h/batch

Batches per day : 8 batches

OLR is calculated as following

$$OLR(kgCOD/m^3.d) = \frac{COD\_\inf luent(\frac{mg}{L})*10^{-6}(\frac{kg}{mg})*1.3(\frac{L}{batch})*8(\frac{batch}{day})}{2.5(L)*10^{-3}(\frac{m^3}{L})}$$

Table A-2 Characteristics of aerobic granules in whole experiment

Day	OLR	Granule size		SVI		Biomass conc.		Settled biomass conc.		EPS - PS		EPS - PN	
		SBAR	SBBR	SBAR	SBBR	SBAR	SBBR	SBAR	SBBR	SBAR	SBBR	SBAR	SBBR
	($kgCOD/m^3.d$)	(mm)		(mL/g)		(mg/L)		($g/L_{granule}$)		(mgPS/gVSS)		(mgPN/gVSS)	
1	3	0.08	0.08	220	220	4000	4000	3.8	3.8	-	-	-	-
7	3	0.16	0.17	168	160	3640	3760	3.4	3.6	-	-	-	-
13	3	0.22	0.26	142	134	2970	3350	7.8	8.2	-	-	-	-
17	3	0.40	0.40	126	112	2510	3170	18.4	21.5	-	-	-	-
23	3	0.50	0.50	65	54	3240	3920	24.3	26.4	-	-	-	-
29	3	0.70	0.80	52	41	4270	4870	26.8	29.0	-	-	-	-
35	3	1.10	1.20	38	32	5190	5530	28.2	29.3	-	-	-	-
41	3	1.50	1.70	32	28	5930	6210	28.7	30.4	57.2	61.8	71.9	72.4
47	5	1.80	2.00	28	24	6720	7830	31.4	34.7	65.1	69.2	68.7	70.3
55	7.5	2.40	2.60	27	23	7950	8590	33.6	36.2	67.4	72.3	67.2	70.6
63	10	3.00	3.20	26	23	8970	9120	36.2	38.6	71.8	79.6	65.7	70.5
71	12.5	3.20	3.40	24	22	9260	9830	39.5	42.8	78.6	82.4	63.8	69.8
79	15	3.50	3.60	24	21	9840	10870	42.8	46.4	83.9	88.7	61.6	68.6
87	17.5	3.60	3.90	24	20	10590	11920	43.8	49.2	82.5	88.4	60.5	62.5
95	20	3.70	4.00	27	23	11230	12780	47.8	53.7	76.1	78.3	57.4	61.8
103	22.5			30	27	9240	10070	39.6	42.7	52.4	64.3	53.2	57.8

Appendix B

Characterization of Effluent from SBAR and SBBR

Table B-1 Characteristics of Effluent from SBAR and SBBR

VLR	MLVSS		SVI		Bound PS		Bound PN		Soluble PS		Soluble PN		MFI	
	SBAR	SBBR	SBAR	SBBR	SBAR	SBBR	SBAR	SBBR	SBAR	SBBR	SBAR	SBBR	SBAR	SBBR
($kgCOD/m^3.d$)	(mg/L)		(mL/g)		(mgPS/gVSS)		(mgPN/gVSS)		(mgPS/gVSS)		(mgPN/gVSS)		(s/L^6)	
3	850	610	212*	196*	28.9*	26.3*	75.4*	77.5*	8.6*	9.4*	2.4*	2.7*	-	-
3	1050	680	-	-	-	-	-	-	-	-	-	-	-	-
3	940	520	-	-	-	-	-	-	-	-	-	-	-	-
3	690	420	-	-	-	-	-	-	-	-	-	-	-	-
3	510	320	-	-	-	-	-	-	-	-	-	-	-	-
3	420	290	-	-	-	-	-	-	-	-	-	-	-	-
3	370	280	80	62	-	-	-	-	-	-	-	-	-	-
5	310	220	78	64	32.2	29.4	66.2	64.2	21.5	20.2	3.2	3.8	110	181
7.5	240	180	76	58	31.6	31.4	64.6	60.4	26.8	32.9	3.8	4.1	359	471
10	210	160	67	52	31.3	33.6	63.4	58.6	32.3	38.4	4.9	5.6	514	669
12.5	180	140	64	52	30.5	34.2	62.1	59.4	34.8	42.7	5.2	6.5	876	939
15	160	140	60	56	42.6	49.7	53.5	39.4	38.7	51.7	7.4	7.3	1140	1251
17.5	210	190	56	52	52.6	61.3	40.4	25.7	39.2	52.3	7.9	8.6	1682	2073
20	480	640	54	50	-	-	-	-	-	-	-	-	-	-
22.5	810	970	54	52	-	-	-	-	-	-	-	-	-	-

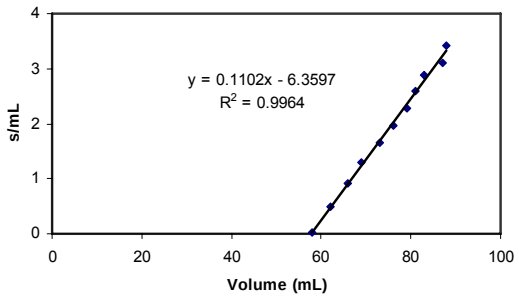
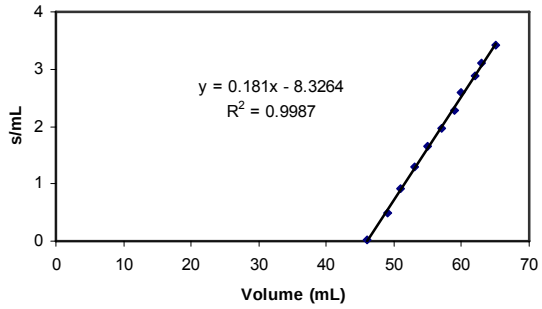
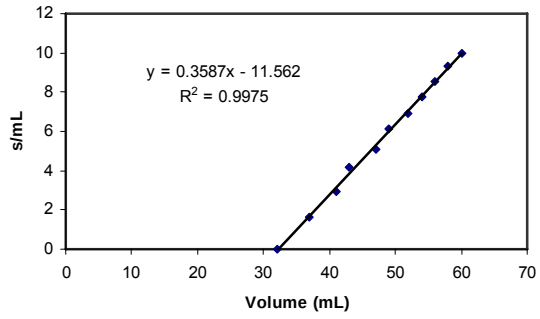
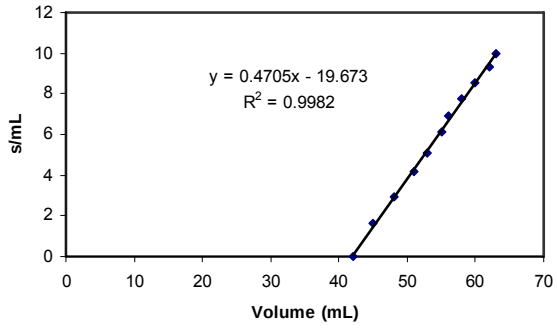
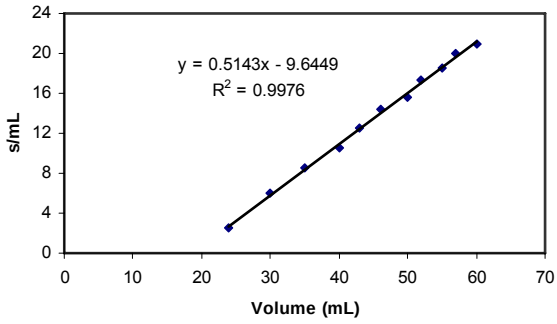
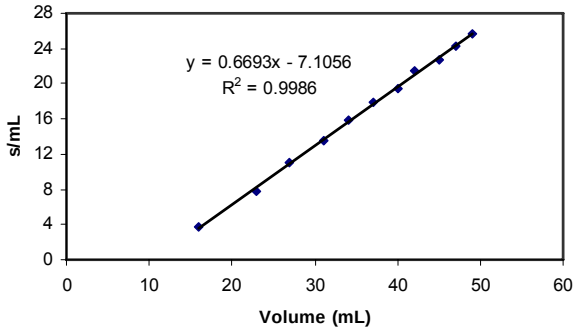
(*) value of seed sludge (activated sludge)

Profile data of MFI in effluent at different OLRs.

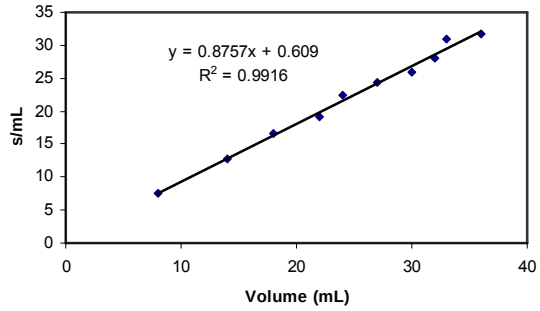
	Effluent from SBAR		Effluent from SBBR	
Time (s)	Volume (mL)	t/v (s/mL)	Volume (mL)	t/v (s/mL)
	OLR of 5 kgCOD/m^3.d			
1	58	0.017	46	0.017
30	62	0.484	49	0.484
60	66	0.909	51	0.909
90	69	1.304	53	1.304
120	73	1.644	55	1.644
150	76	1.974	57	1.974
180	79	2.278	59	2.278
210	81	2.593	60	2.593
240	83	2.892	62	2.892
270	87	3.103	63	3.103
300	88	3.409	65	3.409
	OLR of 7.5 kgCOD/m^3.d			
1	32	0.031	42	0.031
60	37	1.622	45	1.622
120	41	2.927	48	2.927
180	43	4.186	51	4.186
240	47	5.106	53	5.106
300	49	6.122	55	6.122
360	52	6.923	56	6.923
420	54	7.778	58	7.778
480	56	8.571	60	8.571
540	58	9.310	62	9.310
600	60	10.000	63	10.000
	OLR of 10 kgCOD/m^3.d			
1	24	2.500	16	3.750
3	30	6.000	23	7.826
5	35	8.571	27	11.111
7	40	10.500	31	13.548
9	43	12.558	34	15.882
11	46	14.348	37	17.838
13	50	15.600	40	19.500
15	52	17.308	42	21.429
17	55	18.545	45	22.667
19	57	20.000	47	24.255
21	60	21.000	49	25.714

	Effluent from SBAR		Effluent from SBBR	
Time (s)	Volume (mL)	t/v (s/mL)	Volume (mL)	t/v (s/mL)
	OLR of 12.5 kgCOD/m^3.d			
1	8	7.500	14	4.286
3	14	12.857	19	9.474
5	18	16.667	23	13.043
7	22	19.091	26	16.154
9	24	22.500	29	18.621
11	27	24.444	32	20.625
13	30	26.000	33	23.636
15	32	28.125	36	25.000
17	33	30.909	38	26.842
19	36	31.667	39	29.231
			42	30.000
	OLR of 15 kgCOD/m^3.d			
1	16	3.750	23	2.609
4	22	10.909	28	8.571
8	28	17.143	33	14.545
12	32	22.500	37	19.459
16	36	26.667	40	24.000
20	40	30.000	43	27.907
24	42	34.286	46	31.304
28	45	37.333	49	34.286
32	48	40.000	51	37.647
	OLR of 17.5 kgCOD/m^3.d			
1	16	3.750	26	2.308
4	21	11.429	29	8.276
8	25	19.200	32	15.000
12	29	24.828	35	20.571
16	32	30.000	37	25.946
20	35	34.286	40	30.000
24	37	38.919	42	34.286
28	39	43.077	43	39.070

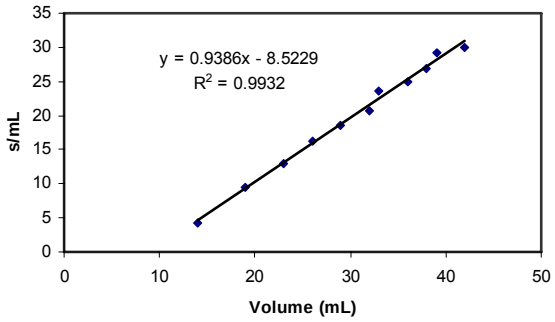
Graph from profile data of MFI

Effluent from SBAR	Effluent from SBAR
At OLR of 5 kgCOD/m^3.d 	At OLR of 5 kgCOD/m^3.d 
At OLR of 7.5 kgCOD/m^3.d 	At OLR of 7.5 kgCOD/m^3.d 
At OLR of 10 kgCOD/m^3.d 	At OLR of 10 kgCOD/m^3.d 

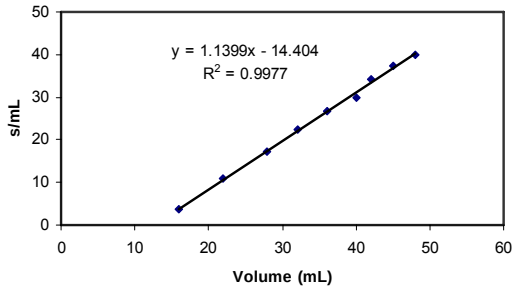
At OLR of 12.5 kgCOD/m^3.d



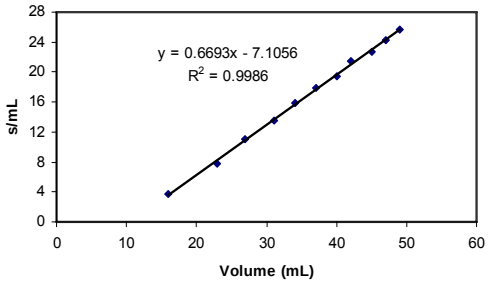
At OLR of 12.5 kgCOD/m^3.d



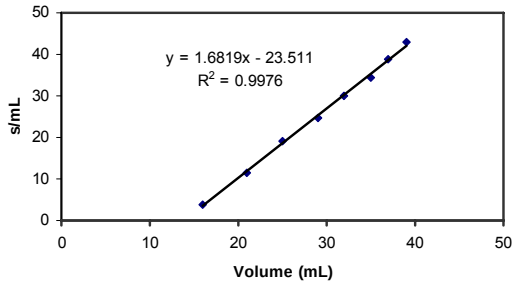
At OLR of 15 kgCOD/m^3.d



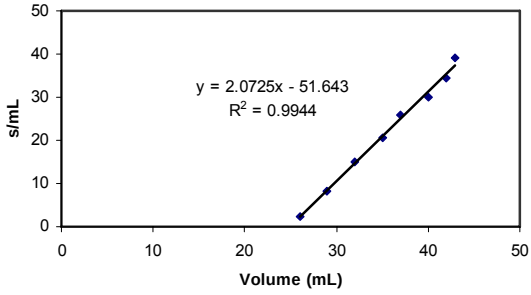
At OLR of 15 kgCOD/m^3.d



At OLR of 17.5 kgCOD/m^3.d



At OLR of 17.5 kgCOD/m^3.d



Appendix C

Membrane Cleaning Procedure

(Used for PVDF 0.1 flat sheet membrane)

1. Required Equipments

- Tank with volume of 3L
- Peristaltic pump
- pH meter

2. Required Chemicals

- Sodium hydroxide (NaOH) 30 – 50%
- Nitric acid (HNO3) 10%
- Sodium hypochloride (NaClO) solution with concentration of 150 mgCl2/L

3. Cleaning Procedure

There are 4 steps in cleaning process including: initial flush, alkaline washing, acid washing, and final sanitation. Time required for whole cleaning process is about 2 hours.

Initial flush (conducted in MBR)

- Use clean water to flush membrane surface in order to remove any remaining build-up.
- This step finish when membrane performs a clean surface.

Alkaline (caustic) wash (conducted in a 3L tank)

- Put membrane module in a 3L tank containing clean water
- Use a pump to suck water from membrane and circulate to the tank (figure below)
- Slowly add sodium hydroxide (NaOH) until pH achieve 10.8 – 11.0 (do not exceed 11.0)
- Circulate alkaline (caustic) solution for 30 minutes
- Flush membrane with clean water to finish this step

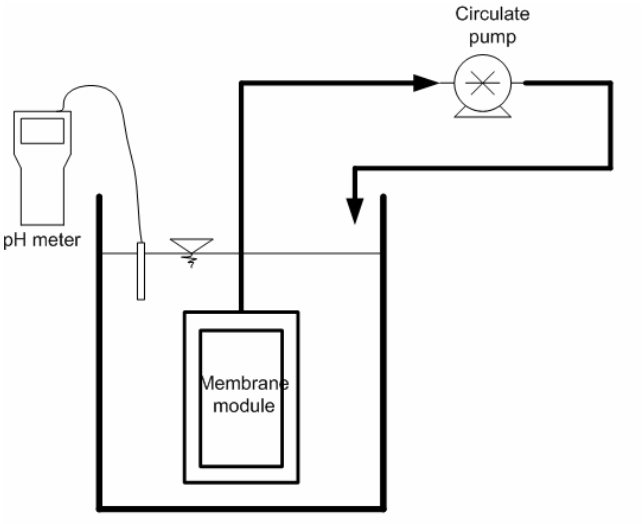
Acid wash (conducted in 3L tank)

- Put membrane module in a 3L tank containing clean water
- Use a pump to suck water from membrane and circulate to the tank
- Slowly add nitric acid (HNO3) until pH achieve 2.0 – 2.5 (do not below 2.0)
- Circulate acid solution for 30 minutes
- Flush membrane with clean water to finish this step

Final sanitation (conducted in 3L tank)

- Put membrane module in a 3L tank containing sodium hypochloride solution 150 ppm (do not exceed 180 ppm)
- Use a pump to suck water from membrane and circulate to the tank
- Slowly add sodium hydroxide (NaOH) until pH achieve 10.8 – 11.0 (do not exceed 11.0)

- Circulate alkaline/chloride solution for 20 minutes
- Flush membrane with clean water to finish this step



Appendix D

Monitoring Results in Baffled MBRs

Table D-1 MLSS in Baffled MBRs at different OLRs

OLR	MLSS in Baffled MBR1			MLSS in Baffled MBR2		
(kgCOD/m^3.d)		(mg/L)			(mg/L)	
	Influent	Baffled	MBR	Influent	Baffled	MBR
7.5	240	100	65	180	62	40
10	210	78	40	160	60	42
12.5	180	94	62	140	68	50
15	160	96	56	140	68	56
17.5	210	150	90	190	98	84

Table D-2 MLSS in Baffled MBRs at OLR of 15 kgCOD/m^3.d

Time	MLSS in Baffled MBR1		MLSS in Baffled MBR2	
	(mg/L)		(mg/L)	
	Influent	MBR	Influent	MBR
week 1	210	42	180	65
week 2	230	58	226	76
week 3	264	72	232	90
week 4	276	82	228	124
week 5	272	84	238	146

Table D-3 Bound EPS result in Baffled MBRs at OLR of 15 kgCOD/m^3.d

Time	Baffled MBR1				Baffled MBR2			
	Bound PS		Bound PN		Bound PS		Bound PN	
(day)	(mgPS/gVSS)		(mgPN/gVSS)		(mgPS/gVSS)		(mgPN/gVSS)	
	Influent	In MBR	Influent	In MBR	Influent	In MBR	Influent	In MBR
1	41.2	23.2	56.4	36.1	51.3	28.4	41.1	23.5
4	41.5	25.9	54.2	35.2	50.4	29.5	40.5	24.2
8	43.8	27.6	53.1	34.4	49.6	30.7	41.4	24.6
12	42.5	30.0	55.4	34.9	49.2	29.8	42.6	24.8
16	43.1	30.3	52.6	35.2	50.4	31.2	41.7	23.9
20	41.8	31.3	54.3	34.6	50.9	31.9	41.2	24.5
24	41.3	32.1	54.8	33.0	51.7	33.3	40.3	25.4
28	43.2	34.3	53.6	30.1	52.6	34.7	39.4	25.9
32	40.2	35.7	54.7	31.2	51.9	36.8	39.8	24.7
36	43.4	36.3	53.2	28.7	51.8	36.6	40.4	25.2

Table D-4 Soluble EPS result in Baffled MBRs at OLR of 15 kgCOD/m^3.d

Time	Baffled MBR1						Baffled MBR2					
	Soluble PS			Soluble PN			Soluble PS			Soluble PN		
(day)	(mg/L)			(mg/L)			(mg/L)			(mg/L)		
	Influent	In MBR	Permeate	Influent	In MBR	Permeate	Influent	In MBR	Permeate	Influent	In MBR	Permeate
1	30.5	17.8	2.6	21.3	16.1	2.3	51.3	28.4	2.5	41.1	23.5	2.4
4	30.8	18.2	2.8	21.8	15.9	2.6	50.4	29.5	2.8	40.5	24.2	2.5
8	31.4	18.6	3.1	21.6	16.4	2.2	49.6	30.7	2.9	41.4	24.6	2.3
12	31.1	18.3	3	21.9	16.8	2.8	49.2	29.8	3.0	42.6	24.8	2.1
16	31.5	18.9	3.2	22.3	16.5	2.1	50.4	31.2	2.8	41.7	23.9	2.4
20	32.4	18.5	3.4	22.6	16.3	1.8	50.9	31.9	3.1	41.2	24.5	2.6
24	31.4	18.6	2.9	22.8	15.7	1.6	51.7	33.3	2.9	40.3	25.4	2.8
28	30.2	18.4	2.6	22.2	15.9	1.9	52.6	34.7	3.4	39.4	25.9	2.3
32	29.3	18.1	3	22.6	15.8	2.2	51.9	36.8	3.2	39.8	24.7	2.5
36	29.5	17.7	3.4	22.7	15.6	2.4	51.8	36.6	3.1	40.4	25.2	2.2

Table D-5 Profile Data of MFI in Baffled MBRs.

In influent

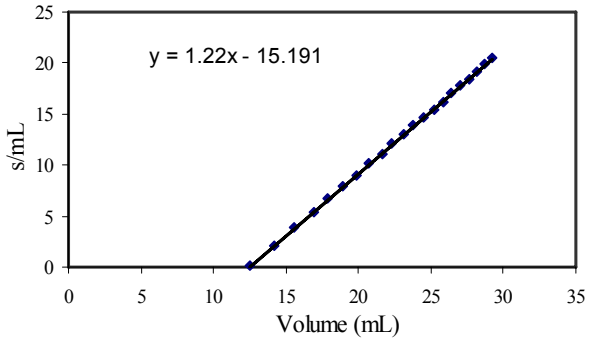
	Baffled MBR1		Baffled MBR2	
Time (s)	Volume (mL)	t/v (s/mL)	Volume (mL)	t/v (s/mL)
Influent				
1	12.5	0.080	4.8	0.208
30	14.2	2.113	7.8	3.846
60	15.6	3.846	9.5	6.316
90	16.9	5.325	11.0	8.182
120	17.9	6.704	12.3	9.756
150	18.9	7.937	13.4	11.194
180	19.9	9.045	14.4	12.500
210	20.7	10.145	15.3	13.725
240	21.6	11.111	16.3	14.724
270	22.3	12.108	17.0	15.882
300	23.1	12.987	17.8	16.854
330	23.8	13.866	18.5	17.838
360	24.5	14.694	19.3	18.653
390	25.2	15.476	19.9	19.598
420	25.9	16.216	20.6	20.388
450	26.4	17.045	21.2	21.226
480	27.0	17.778	21.8	22.018
510	27.6	18.478	22.4	22.768
540	28.2	19.149	23.0	23.478
570	28.7	19.861	23.6	24.153
600	29.2	20.548	24.0	25.000

In MBR and permeate

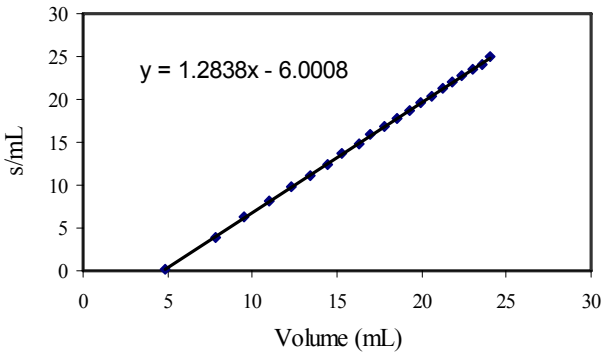
	Baffled MBR1		Baffled MBR2	
Time (s)	Volume (mL)	t/v (s/mL)	Volume (mL)	t/v (s/mL)
In MBR				
1	8.4	0.119	6.4	0.2
30	10.2	2.941	7.8	3.8
60	11.6	5.172	8.9	6.7
90	12.8	7.031	9.7	9.3
120	13.8	8.696	10.5	11.4
150	14.8	10.135	11.2	13.4
180	15.6	11.538	11.8	15.3
210	16.4	12.805	12.5	16.8
240	17.2	13.953	13.1	18.3
270	17.7	15.254	13.5	20.0
300	18.6	16.129	14.1	21.3
330	19.2	17.188	14.6	22.6
360	19.8	18.182	15	24.0
390	20.4	19.118	15.5	25.2
420	21.0	20.000	15.9	26.4
450	21.6	20.833		
480	22.1	21.719		
510	22.6	22.566		
540	23.0	23.478		
570	23.7	24.051		
600	24.1	24.896		
Permeate				
1	22.6	0.044	32.5	0.031
5	64.0	0.078	83.0	0.060
10	96.0	0.104	117.0	0.085
15	123.0	0.122	146.0	0.103
20	146.0	0.137	163.0	0.123
25	162.0	0.154	188.0	0.133
30	178.0	0.169	198.0	0.152
35	193.0	0.181		
40	210.0	0.190		

Graph form Profile Data of MFI

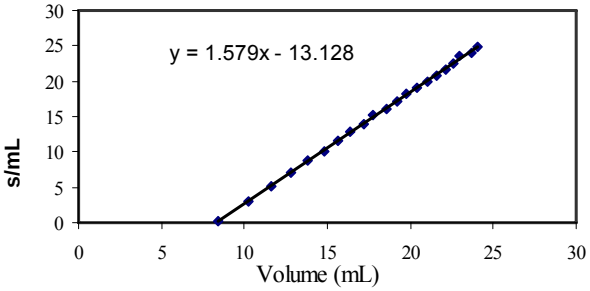
MFI of influent of Baffled MBR1



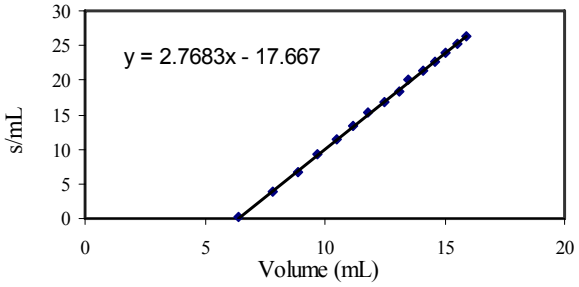
MFI of influent of Baffled MBR2



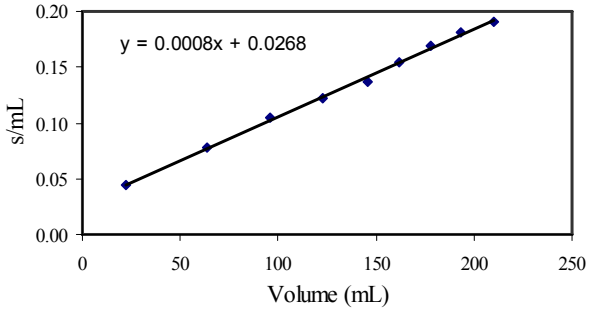
MFI of in MBR of Baffled MBR1



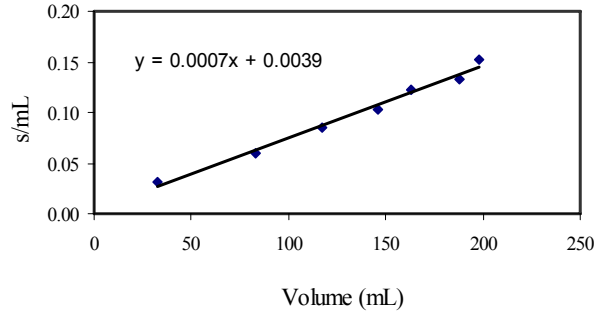
MFI of in MBR of Baffled MBR2



MFI of permeate of Baffled MBR1



MFI of permeate of Baffled MBR2



Appendix E

Monitoring Results in Transmembrane Pressure in Baffled MBRs

Table E-1 Profile data of TMP in MBR1, 2.

Day	TMP in MBR1	TMP in MBR2	Remark
1	6.8	6.1	
2	13.6	9.5	
3	25.4	14.8	
4	38.7	21.7	
5	51.2	26.4	
6	65.6	34.9	
7	81.0	44.2	Cleaning membrane 1
8	7.5	56.7	
9	15.6	67.3	
10	27.3	78.4	Cleaning membrane 2
11	37.2	8.1	
12	45.5	11.6	
13	56.7	15.7	
14	66.6	22.3	
15	78.5	27.9	
16	-	34.6	
17	-	47.2	
18	-	59.4	
19	-	70.7	
20	-	81.2	
21	2.6	2.3	Insert new membrane
22	5.2	4.8	
23	12.4	8.7	
24	19.7	13.5	
25	27.8	18.4	
26	36.9	23.9	
27	43.3	29.1	
28	50.8	35.7	
29	58.6	41.2	
30	67.8	47.6	
31	79.4	58.8	
32	2.8	67.3	New membrane in MBR1
33	6.4	76.9	
34	14.3	2.7	New membrane in MBR2
35	20.1	5.1	
36	29.7	9.6	
37	37.2	15.2	
38	46.3	19.4	
39	53.8	26.7	
40	65.6	32.1	
41	76.5	38.9	
42	-	44.6	
43	-	50.3	
44	-	58.6	
45	-	67.2	
46	-	78.9	

Appendix F

Results of Particle Size Distribution in MBR1, 2

Particle Size Distribution in MBR1

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Klong Luang, Pathumthani 12120 Thailand
Tel : (662)564-6500 Fax : (662)564-6501-5
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Result: Analysis Report

Sample Details

Sample ID: Sample 1_1
Sample File: TS1334
Sample Path: D:\DATASIZE\TECHNI~1\TECH_49\
Sample Notes: Dispersion medium : DI water
Treatment : Stir medium
Remark : MBR1_Aialift

Run Number: 2
Record Number: 129

Measured: Fri Mar 24 2006 8:53AM
Analysed: Fri Mar 24 2006 8:53AM
Result Source: Analysed

System Details

Range Lens: 300RF mm
Presentation: 3OHD
Analysis Model: Polydisperse
Modifications: None

Beam Length: 2.40 mm
[Particle R.I. = (1.5295, 0.1000); Dispersant R.I. = 1.3300]

Sampler: MS1

Obscuration: 21.4 %
Residual: 0.514 %

Result Statistics

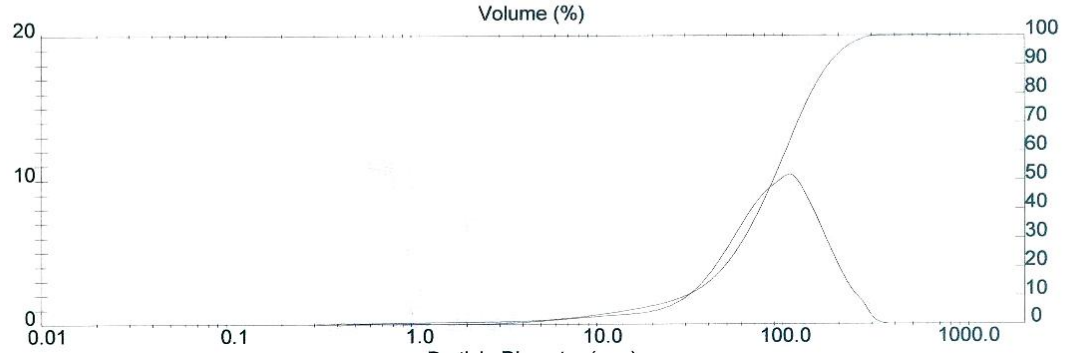
Distribution Type: Volume
Mean Diameters:
D [4, 3] = 97.58 um

Concentration = 0.1207 %Vol
D (v, 0.1) = 30.21 um
D [3, 2] = 30.16 um

Density = 1.000 g / cub. cm
D (v, 0.5) = 88.82 um
Span = 1.648E+00

Specific S.A. = 0.1989 sq. m / g
D (v, 0.9) = 176.56 um
Uniformity = 5.113E-01

Size Low (um)	In %	Size High (um)	Under%	Size Low (um)	In %	Size High (um)	Under%
0.05	0.00	0.06	0.00	6.63	0.40	7.72	2.49
0.06	0.00	0.07	0.00	7.72	0.47	9.00	2.95
0.07	0.00	0.08	0.00	9.00	0.54	10.48	3.49
0.08	0.00	0.09	0.00	10.48	0.60	12.21	4.09
0.09	0.00	0.11	0.00	12.21	0.66	14.22	4.74
0.11	0.00	0.13	0.00	14.22	0.72	16.57	5.46
0.13	0.00	0.15	0.00	16.57	0.81	19.31	6.28
0.15	0.00	0.17	0.00	19.31	0.97	22.49	7.24
0.17	0.00	0.20	0.00	22.49	1.23	26.20	8.47
0.20	0.00	0.23	0.00	26.20	1.65	30.53	10.13
0.23	0.01	0.27	0.02	30.53	2.29	35.56	12.42
0.27	0.03	0.31	0.04	35.56	3.16	41.43	15.58
0.31	0.04	0.36	0.09	41.43	4.29	48.27	19.87
0.36	0.06	0.42	0.15	48.27	5.60	56.23	25.47
0.42	0.08	0.49	0.22	56.23	7.00	65.51	32.47
0.49	0.10	0.58	0.32	65.51	8.29	76.32	40.76
0.58	0.10	0.67	0.43	76.32	9.30	88.91	50.06
0.67	0.11	0.78	0.53	88.91	9.96	103.58	60.02
0.78	0.09	0.91	0.63	103.58	10.34	120.67	70.36
0.91	0.08	1.06	0.71	120.67	9.21	140.58	79.56
1.06	0.06	1.24	0.77	140.58	7.47	163.77	87.04
1.24	0.05	1.44	0.81	163.77	5.53	190.80	92.57
1.44	0.04	1.68	0.85	190.80	3.72	222.28	96.29
1.68	0.04	1.95	0.89	222.28	2.27	258.95	98.57
1.95	0.04	2.28	0.93	258.95	1.24	301.68	99.80
2.28	0.05	2.65	0.98	301.68	0.20	351.46	100.00
2.65	0.07	3.09	1.05	351.46	0.00	409.45	100.00
3.09	0.11	3.60	1.16	409.45	0.00	477.01	100.00
3.60	0.15	4.19	1.31	477.01	0.00	555.71	100.00
4.19	0.20	4.88	1.51	555.71	0.00	647.41	100.00
4.88	0.26	5.69	1.77	647.41	0.00	754.23	100.00
5.69	0.32	6.63	2.09	754.23	0.00	878.67	100.00



Particle Size Distribution in MBR2

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Klong Luang, Pathumthani 12120 Thailand
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Result: Analysis Report

Sample ID: Sample 2_2
Sample File: TS1334
Sample Path: D:\DATASIZE\TECHNI~1\TECH_49\
Sample Notes: Dispersion medium : DI water
Treatment : Stir medium
Remark : MBR2_Bubble

Sample Details
Run Number: 1
Record Number: 140

Measured: Fri Mar 24 2006 9:51AM
Analysed: Fri Mar 24 2006 9:51AM
Result Source: Analysed

System Details

Range Lens: 300RF mm
Presentation: 3OHD
Analysis Model: Polydisperse
Modifications: None

Beam Length: 2.40 mm
[Particle R.I. = (1.5295, 0.1000); Dispersant R.I. = 1.3300]
Sampler: MS1

Obscuration: 20.8 %
Residual: 0.503 %

Result Statistics

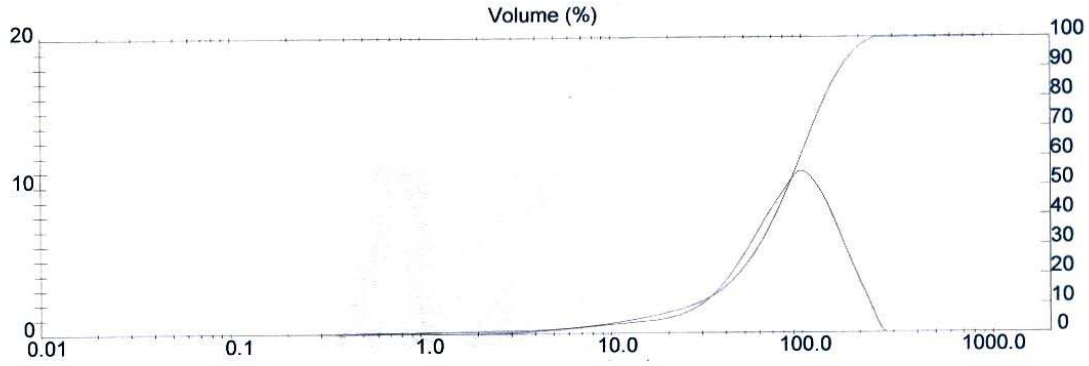
Distribution Type: Volume
Mean Diameters:
D [4, 3] = 91.27 um

Concentration = 0.1140 %Vol
D (v, 0.1) = 27.94 um
D [3, 2] = 30.94 um

Density = 1.000 g / cub. cm
D (v, 0.5) = 85.73 um
Span = 1.569E+00

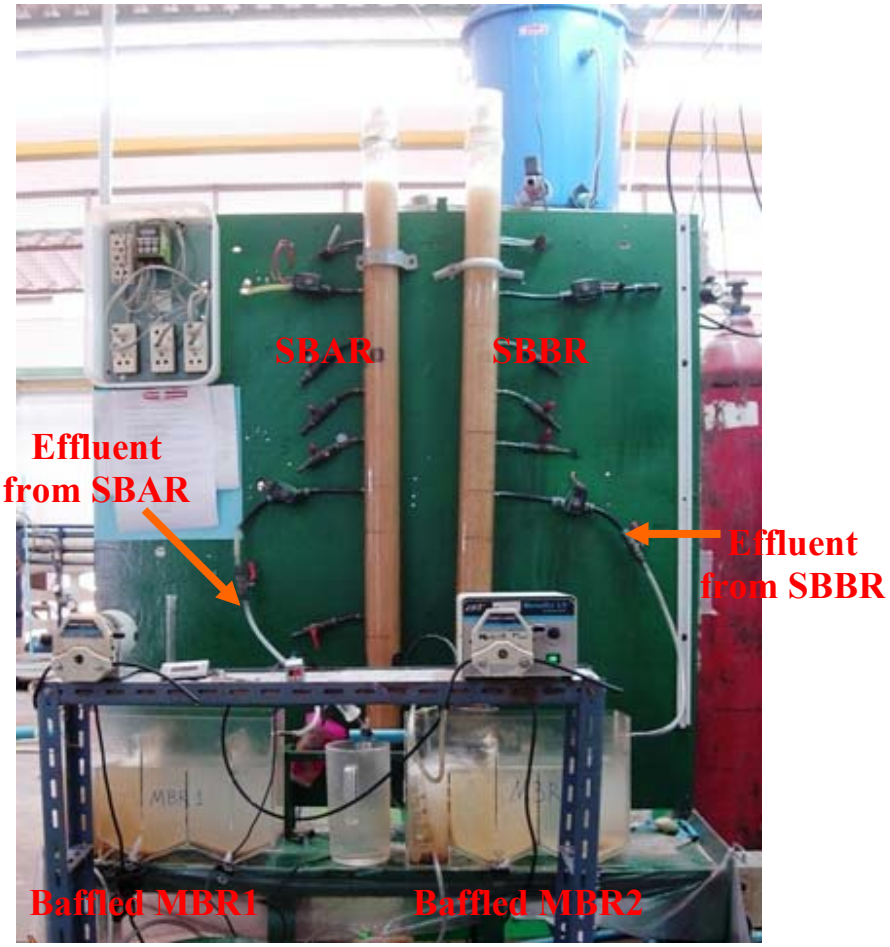
Specific S.A. = 0.1939 sq. m / g
D (v, 0.9) = 162.40 um
Uniformity = 4.787E-01

Size Low (um)	In %	Size High (um)	Under%	Size Low (um)	In %	Size High (um)	Under%
0.05	0.00	0.06	0.00	6.63	0.43	7.72	2.55
0.06	0.00	0.07	0.00	7.72	0.51	9.00	3.07
0.07	0.00	0.08	0.00	9.00	0.60	10.48	3.66
0.08	0.00	0.09	0.00	10.48	0.68	12.21	4.34
0.09	0.00	0.11	0.00	12.21	0.75	14.22	5.10
0.11	0.00	0.13	0.00	14.22	0.83	16.57	5.93
0.13	0.00	0.15	0.00	16.57	0.93	19.31	6.86
0.15	0.00	0.17	0.00	19.31	1.09	22.49	7.96
0.17	0.00	0.20	0.00	22.49	1.36	26.20	9.31
0.20	0.00	0.23	0.00	26.20	1.78	30.53	11.09
0.23	0.00	0.27	0.00	30.53	2.41	35.56	13.50
0.27	0.01	0.31	0.01	35.56	3.28	41.43	16.78
0.31	0.02	0.36	0.03	41.43	4.41	48.27	21.18
0.36	0.05	0.42	0.08	48.27	5.74	56.23	26.92
0.42	0.07	0.49	0.15	56.23	7.17	65.51	34.09
0.49	0.10	0.58	0.25	65.51	8.56	76.32	42.65
0.58	0.12	0.67	0.36	76.32	9.80	88.91	52.45
0.67	0.13	0.78	0.49	88.91	10.87	103.58	63.32
0.78	0.11	0.91	0.60	103.58	10.50	120.67	73.82
0.91	0.09	1.06	0.69	120.67	9.23	140.58	83.05
1.06	0.06	1.24	0.75	140.58	7.30	163.77	90.35
1.24	0.04	1.44	0.79	163.77	5.18	190.80	95.53
1.44	0.03	1.68	0.83	190.80	3.22	222.28	98.74
1.68	0.03	1.95	0.86	222.28	1.26	258.95	100.00
1.95	0.04	2.28	0.90	258.95	0.00	301.68	100.00
2.28	0.05	2.65	0.95	301.68	0.00	351.46	100.00
2.65	0.07	3.09	1.02	351.46	0.00	409.45	100.00
3.09	0.11	3.60	1.13	409.45	0.00	477.01	100.00
3.60	0.16	4.19	1.29	477.01	0.00	555.71	100.00
4.19	0.21	4.88	1.50	555.71	0.00	647.41	100.00
4.88	0.28	5.69	1.78	647.41	0.00	754.23	100.00
5.69	0.35	6.63	2.12	754.23	0.00	878.67	100.00



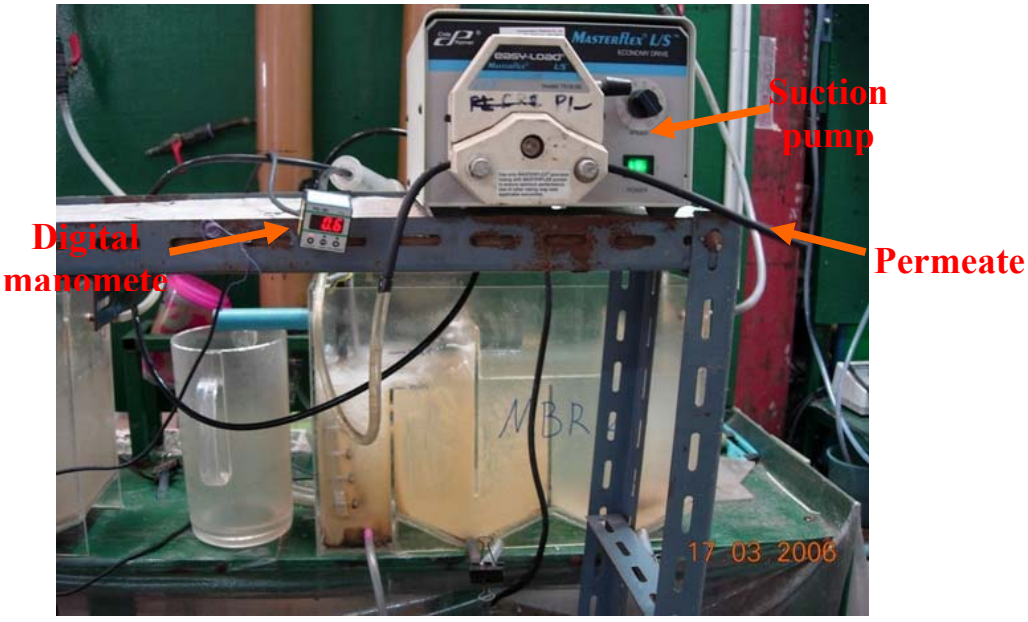
Appendix G

Photos of Experiment Work



Experimental model

Flat sheet membrane module



Baffled MBR