# Investigation of Performance of a Submerged Anaerobic Membrane Bioreactor (AnMBR) Treating Meat Processing Wastewater

by

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## AUTHOR'S DECLARATION

I hereby declare that I am the sole author of this thesis. This is a true copy of the thesis, including any required final revisions, as accepted by my examiners.

I understand that my thesis may be made electronically available to the public.

*Mohammed Abdullah Galib*

## ABSTRACT

Anaerobic membrane bioreactor (AnMBR) has grown popular as a means of sustainable biological treatment in the recent few decades due to less energy and space requirement, less sludge production, increased treatability, and methane production, which can make the system energy-positive; however, membrane cost and membrane fouling remain the major issues in its widespread use.

Slaughterhouse/meat processing wastewater poses threat to the environment and its release with a higher contaminant concentration than the discharge standards imposes surcharge fees on the plant. High organic strength and slowly degradable particulates in such wastewater make AnMBR a good choice for its treatment, as shown by few studies. However, those studies used membrane in a pressure-driven external configuration requiring high cross flow velocity and consuming more energy. Internally submerged vacuum driven membranes can operate on lower pressure and energy, while offering similar treatment potentials. Owing to the knowledge gaps, this study was conducted using an AnMBR in a submerged membrane configuration with the objectives to (a) assess the performance in terms of COD removal and biogas production at varying feed loads, (b) investigate the membrane performance in terms of achievable flux and fouling behaviour, and (c) establish baseline information on start-up and operating conditions for implementing larger scale reactors.

A bench-scale anaerobic reactor (5L) was set up, with a submerged ultrafiltration hollow fibre membrane (pore dia. 0.04µm, surface area 0.046 $m^2$). Sludge from a mesophilic anaerobic digester at a municipal wastewater treatment plant was used to inoculate the AnMBR, and wastewater from Conestoga Meat Packers, Woolwich, Ontario was used as the feed. The reactor was run on continuous mode at room temperature and neutral pH. The system performance was evaluated under three different operating conditions by varying the HRT (5d, 2d, and 1d), which simultaneously changed the membrane permeate flux. Intermittent pumping, surface scouring by biogas and weekly chemical cleaning were applied to minimize fouling of the membrane.

The feed solids concentration was seen to fluctuate widely (0.3-2.6 g/L) depending on the nature and extent of works in the plant; however, the reactor showed good stability and the MLSS was not affected significantly by the wide variation. The average MLVSS concentrations were 1.7±0.7, 1.8±0.3, and 2.1±0.2 g/L, in Phases I, II, and III respectively with a variation of less than 550 mg/L. With the incoming organic concentration (TCOD 0.6-4.9 g/L) varying widely like the solids, the average effluent COD in Phase I, Phase II and Phase III were 96±28, 170±36 and 373±76 mg/L,

giving very good COD removal efficiencies of 95±3.1%, 94±2.3% and 88±4.6%. The average organic loading rates (OLR) of 0.4±0.2, 1.4±0.4 and 3.1±1.1 kg COD/$m^3$/day were achieved in the three Phases. Results of this study were similar to or better than some of the earlier studies with similar wastewater in terms of percent COD removals and effluent COD concentrations.

The daily biogas production went up from 0.37±0.18 L/day in Phase I to 2.82±0.62 L/day in Phase III. The percentage of methane in the biogas remained consistently high at 72±4% throughout the study period. The specific methane yields were 0.24±0.16, 0.16±0.05 and 0.20±0.09 L $CH_4$/g $COD_{removed}$ in Phase I, II and III respectively, which are similar or slightly lower than the values (0.2 – 0.3 L $CH_4$/g $COD_{removed}$) reported by some of the earlier studies. VFAs/Alkalinity ratio of less than 0.2 was observed throughout the study which indicated the stability of the system. Extracellular polymeric substances (EPS) in the reactor sludge were 96.9±8.6, 100.2±8.5 and 105.2±5.0 mg/gVSS in Phases I, II and III, with a Protein/Carbohydrate ratio of 6.1-6.5. The increasing EPS concentrations contributed towards build-up of cake sludge on the membrane surface, which augmented membrane fouling.

Average measured membrane flux of 1.14±0.02, 3.15±0.04 and 6.15±0.37 LMH were observed during the three Phases. Measured flux were very close to the set flux throughout Phases I and II, and the first 15 days of Phase III, indicating that there was none or insignificant membrane fouling. This attested the success of membrane surface scouring with biogas and periodic membrane maintenance cleaning. However, with the progress of Phase III (at 1d HRT) the membrane became more fouled and declines in flux were experienced. Transmembrane pressure (TMP) at the end of Phase I, Phase II and at the beginning of Phase III were below 3 kPa. However, TMP higher than 40 kPa was observed towards the end of Phase III.

This lab-scale AnMBR was able to demonstrate the applicability and efficiency in treating meat processing wastewater with a submerged membrane and at ambient temperature. The produced biogas had high percentage of methane, suggesting its scope for being an energy positive process, though there is still potential to increase the specific methane production. From the results of this study, HRT of 2 days, SRT of 50-60 days and membrane permeate flux of 6 LMH are recommended. Periodic maintenance cleaning will help to reduce membrane fouling. The start-up and operational information from the successful performance of this lab-scale reactor can be used as baseline for implementation of a larger or pilot scale AnMBR treating similar wastewater.

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I wish to thank all the great people whose contributions have made my thesis a success; whether or not I address all of them in this short acknowledgement.

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Finally, I am ineffably grateful to my wife not only for her encouragements but also for shouldering added responsibilities while I pursued my degree, and to my son for his share of sacrifice. I like to dedicate this thesis to my parents, who have been a source of inspiration for me. All praise to the Almighty for keeping me healthy and allowing me to complete my research.

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## LIST OF ABBREVIATIONS AND NOMENCLATURE

AnMBR: Anaerobic membrane bioreactor

COD: Chemical oxygen demand

CSTR: Continuously stirred tank reactor

EDTA: Ethylenediaminetetraacetic acid

EPS: Extracellular polymeric substances

F/M: food-to-microorganism ratio

HRAR: High rate anaerobic reactor

HRT: Hydraulic retention time

LMH: Litres per square metre per hour, unit of permeate flux ($L/m^2/h$)

MLSS: Mixed liquor suspended solids

MLVSS: Mixed liquor volatile suspended solids

MWW: Municipal wastewater

OLR: Organic loading rate

SCOD: Soluble chemical oxygen demand

SMP: Soluble microbial product

SRT: Solids retention time

STP: Standard temperature and pressure

TCOD: Total chemical oxygen demand

TMP: Transmembrane pressure

TSS: Total suspended solids

UF: Ultrafiltration

VFA: Volatile fatty acid

VSS: Volatile suspended solids

# *Chapter 1*

# *Introduction*

# 1 INTRODUCTION

## *1.1 Introduction*

Development of human civilization has vastly advanced over the past few centuries, due largely to the industrialization processes. Food industry has also thrived along with many others and has evolved into a great contributor to the human sustainability as well as to the pollution that inadvertently comes with it. Meat processing industry, under the broader range of food industries is common to almost every country of the world. It generates a large amount of wastewater from the extensive use of water in different stages of production and cleaning with volumes from 0.4 to 3.1 $m^3$ per slaughtered animal (Saddoud & Sayadi, 2007). Effluent from meat processing plants and slaughterhouses is very harmful to the environment. It contains blood, animal fat, skin and meat particles, manure and pathogenic microorganisms, with blood being the major contributor in organic strength (Massé and Masse, 2000; López-López, et al., 2010). The total chemical oxygen demand (TCOD) and total suspended solids (TSS) in such wastewater are seen to be in the range of 2.2 – 20.1 g/L and 0.5 – 4.7 g/L respectively. Treatment of meat processing plant and slaughterhouse wastewater is very important for the sake of environmental protection and sustainability. Wastewater from slaughterhouses in Ontario is generally discharged in municipal sewers after some degree of primary or chemical pre-treatment at site. However, due to further requirement of treatment at municipal wastewater treatment plants, the industries require to pay a surcharge to dispose their wastewater (Mittal, 2006).

As meat processing wastewater contain mostly biodegradable organic materials, biological treatment process is considered to be most suited and economical for treating such wastewater. Conventionally, treatments like aerobic activated sludge process were applied to treat meat processing and slaughterhouse wastewater; however they have the problem of requiring large area and energy (for aeration). Anaerobic treatments (biological treatment in the absence of oxygen) like

covered anaerobic lagoon, anaerobic filter and up-flow anaerobic sludge blanket (UASB) have shown very good contaminant removal efficiency (over 90% COD removal) for different types of wastewater, including meat processing wastewater. In addition to having high organic strength and high solids, this type of wastewater contains slowly degradable particulate matters (Dereli, et al., 2012). These criteria make it very suitable to be treated with anaerobic membrane bioreactor (AnMBR), which offers the strengths of anaerobic treatment, while the use of membrane allows complete retention of solids (and biomass) in the reactor providing high solids retention time for the degradation of organics. AnMBR is an efficient and proven treatment technology that can help in reducing or eliminating the surcharge fees for companies and protect the environment by delivering effluent of acceptable quality through a sustainable process. AnMBR can offer better treatment performance than conventional anaerobic treatment, has smaller footprint than most other processes and has the added benefit of energy production. The major perennial concerns associated with AnMBR are the cost of membrane and membrane fouling.

Despite the potential benefits of AnMBR only a limited number of studies were conducted in lab scale using this technology. A review of the literature revealed few publications on treatment of meat processing/slaughterhouse wastewater using AnMBR, and all of them used membrane in an external cross-flow configuration. Membranes installed in such configuration require higher energy for creating enough filtration pressure, and some studies have reported reduced biomass activity due to the high cross flow velocity and pressure. Membranes installed in internally submerged configuration and driven by vacuum pressure can also achieve similar treatment efficiencies to the external configuration, and operate on lower pressure and energy requirements. Thus, a knowledge gap exists regarding performance of AnMBR with a submerged membrane treating meat processing wastewater. Also, the impacts on biological process and membrane performance need to be assessed at different loading rates so as to determine the ideal long term operational conditions.

### *1.2 Objectives of research*

The overall goal of this study was to evaluate the applicability and performance of an AnMBR fitted with a submerged, vacuum driven hollow fibre membrane and fed with high-strength industrial wastewater.

The specific objectives of the study were to:

- Investigate the effects of different organic loading rates (OLR) on the COD removal efficiency
- Examine the changes in biogas production and level of methane in biogas with change of feed loading rates
- Compare the performance of this study (submerged AnMBR) with those of earlier studies (side-stream AnMBR)
- Evaluate the membrane performance in terms of achievable operating flux, frequency of fouling and effectiveness of chemical cleaning during the treatment of high-strength wastewater
- Establish baseline information on the start-up and operating conditions (e.g. SRT, HRT, pH, F/M ratio, OLR, membrane flux) that can be used for large or pilot scale reactors treating similar wastewater

### *1.3 Scope of research*

The research was conducted using a bench-scale membrane coupled anaerobic reactor in the Waterloo Environmental Biotechnology Laboratory at the University of Waterloo utilizing high-strength wastewater. The study is unique in the way that a submerged AnMBR was used to treat meat processing wastewater under low pressure and ambient temperature. The study primarily focussed on evaluating the performance of the reactor in connection to effluent quality, bio-energy

potential and membrane performance at three different organic loading rates (OLR) and hydraulic retention times (HRT). Effluent quality was determined in terms of organic removal, bio-energy potential was determined by monitoring biogas and methane production, and membrane performance was determined by observing flux and transmembrane pressure (TMP). The results of this study were compared to those obtained from other studies using anaerobic membrane reactor and similar wastewater. No evaluation of performance regarding removal of nutrients was performed, and no economic analysis of the system regarding self-sustainability and net energy production was conducted under this study; however, they are recommended as potential future works (Chapter 6).

### *1.4 Structure of the Thesis*

The thesis is arranged in six chapters. Chapter 1 briefly introduces the background problem and the suitable treatment method along with the objectives and scope of the research. Details on the evolution of anaerobic membrane treatment process, its uses, and performance results from past studies are presented in Chapter 2. Chapter 3 presents the materials and methods of the study, describing the experimental set up and analytical methods followed. The results are given in Chapter 4 along with discussion and comparisons with similar past studies. Chapter 5 summarizes the findings from the study, and some recommendations for potential future studies are presented in Chapter 6.

# *Chapter 2*

# *Background and Literature Review*

# 2 BACKGROUND AND LITERATURE REVIEW

## *2.1 Anaerobic biological process: its development and uses*

Biological treatment of wastewater is well established and widely applied across the globe in addition to the conventional physical and chemical treatment processes. Among biological processes, the anaerobic treatment process is considered to be the most promising and meets the desired criteria of being an environmentally friendly intervention while contributing towards sustainable environmental and social development (McCarty, 2001; Lettinga, et al., 1997). It has been applied towards wastewater and sludge treatment for over 100 years (McCarty & Smith, 1986). Basically, the anaerobic process is where the biological stabilization of organic matter takes place in the absence of oxygen and in the presence of anaerobic microorganisms, finally producing methane ($CH_4$) gas as a significant end product (Parkin & Owen, 1986). Although at early stages the anaerobic process was primarily used for treatment and stabilization of waste sludge, it has also been applied later in treating wastewater (Metcalf & Eddy, Inc., 2003).

The history and development of anaerobic biological process and its application for treatment of waste and wastewater is fascinating and at the same time too long to suit the scope of this paper. However, brief description of the important discoveries and progresses will be provided in this section. The early reported incidents of anaerobic process involve Volta's demonstration of "combustible air" forming from the sediments in lakes and ponds in 1776, and Reist's observation of methane release from decomposing manure in 1856 (McCarty, 2001). That this formation of methane production is due to degradation of organic matter through microbiological process was first stated by Bechamp in 1868, and the different biochemical reactions comprising that process were later reported by Omelianski in the 1890s and Sohngen in 1910 (Abbasi, et al., 2012). The first full-scale application of anaerobic treatment was for domestic wastewater, and is credited to Mouras, a Frenchman, and was called "Mouras' Automatic Scavenger" (Khanal, 2008). The

"Anaerobic filter" (AF) was introduced in the late 1880s in the form of a bed of sand at the Massachusetts Experimental Station, USA, and in the early 1890s in the form of a bed of stones as a hybrid system with a digester by W.D. Scott Moncrieff (McCarty, 2001). An Englishman, David Cameron, designed the "septic tank" in Exeter, England in 1895 that utilized methane gas as a source of energy for heat and electricity. The septic tank technology continued to be used in other parts of the world including USA and India (McCarty, 2001). In 1904, William A Travis developed the two-stage system with a separate solid digestion, which was modified in 1905 by Karl Imhoff of Germany. The first sludge-heating apparatus in a separate digestion tank was installed by the Ruhrverband at Essen-Rellinghausen plant in 1927. The process of heating and separate sludge digestion gained popularity and within the next few years its use spread throughout many large cities. Methane gas produced from such treatments was used for heating digesters, powering treatment plants and supplementing municipal gas supplies (Khanal, 2008).

Application of the anaerobic process in treating industrial wastewater and agricultural residues was studied extensively by Arthur M Buswell and his co-workers in the 1920; however, their studies faced an obstacle in application as the conventional single tank anaerobic digester had no provision of biomass separation for long solids retention time (SRT) (McCarty 2001). A widely known development came in 1969, when J C Young and P L McCarty reexamined AF for the treatment of soluble wastewater (Khanal, 2008). In 1950, G J Stander realized the importance of SRT, which had been the basis for development of "Clarigester" in South Africa. This new idea of increasing solids (biomass) retention time while keeping the microorganisms from escaping the reactor paved the way for development of high rate sludge bed reactors like the Upflow Anaerobic Sludge Bed process (UASB) and the Expanded Granular Sludge Bed process (EGSB) (Lettinga, 2001). A very successful new design of the anaerobic treatment processes, the Upflow Anaerobic Sludge Blanket (UASB) by G. Lettinga in the 1970s achieved vast improvement in liquid withdrawal and solids

retention and saw the growth of "anaerobic granular sludge", a dense conglomerate of microorganisms working together for efficient treatment while providing excellent biomass retaining capability in the reactor (McCarty, 2001; Abbasi, et al., 2012). UASB is a prime example of a "high rate" anaerobic reactor (HRAR), which can retain high viable biomass and can handle high organic loading rates (OLR) (Lettinga, 1995). Other good examples of HRAR are anaerobic fixed film reactor (AFFR), anaerobic fluidized bed reactor (AFBR), expanded granular sludge bed (EGSB), anaerobic filter and hybrid systems (Khanal, 2008).

While the developing countries (especially China, and some countries in South and South-east Asia) had been using the anaerobic technology from its early stage more for generation of fuel (biogas) than for treatment of waste, the developed countries used it more for the latter purpose until the mid 1970s, with most of the digesters being established in western Europe (Abbasi, et al., 2012). Although $CH_4$ produced from anaerobic digestion is utilized locally (domestic utilization, a single unit process in a treatment plant, etc.), the biogas cannot be injected in central gas distribution systems as it does not meet the required criteria because of having low pressure, low calorific value, high $CO_2$, high $H_2S$ and high water content (Lindeboom, et al., 2011). Fig 2.1 shows a picture of a conventional large-scale anaerobic digester.

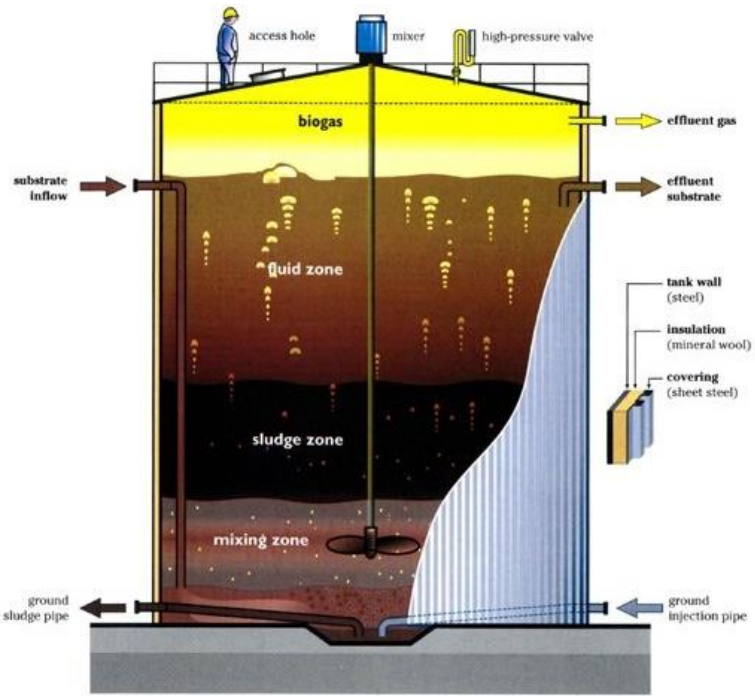


**Figure 2-1: A large scale completely mixed anaerobic digester (3AD, 2013)**

Conventionally, anaerobic digestion is carried out in a single digester, however, two-stage anaerobic digestion have also been in practice for several decades. The primary feature of the two-stage digestion is that it offers separate environmental and operational conditions for acidogenic and methanogenic populations to be maintained in two reactors. The acidogenic digester, having a lower preferable pH range would produce $CO_2$ and $H_2$, and the methanogenic digester is where the $CO_2$ and $H_2$ will be optimally utilized to produce $CH_4$ and $CO_2$ under favourable methanogenic conditions. Two-stage anaerobic digestion is mentioned to be more advantageous than single-stage in several ways, including higher rate of hydrolysis, higher rate of substrate conversion, higher gas yield, reduction of volatile solids, higher buffer capacity, and higher effluent quality (Ghosh, 1987).

Methane produced (captured) from wastewater serves several purposes for the benefit of human kind: supplying a clean source of energy that can be used for generating heat and electricity, reducing the requirement of fossil fuel, minimizing deforestation and reducing the emission of methane in the atmosphere (Rittmann & McCarty, 2012; Abbasi, et al., 2012). Although it is the

second most emitted greenhouse gas after carbon dioxide, methane causes twenty five times more global warming than carbon dioxide. It is estimated that 60% of current methane emissions occur from anthropogenic activity, and wastewater is the fifth largest source of anthropogenic $CH_4$ emission, which is more than 9% of the total emission. The meat and poultry, pulp and paper and fruits and vegetable industries contribute the largest quantity of wastewater and have high organic chemical oxygen demand (COD), thereby releasing a high volume of methane into the atmosphere. Fig 2.2 shows past and future projected methane emissions (as million tons of $CO_2$ equivalent) by the four leading $CH_4$ emitting countries and the rest of the world (adopted from USEPA, 2006). It is, therefore, crucial that wastewater be treated and $CH_4$ be captured by using the anaerobic treatment technology.

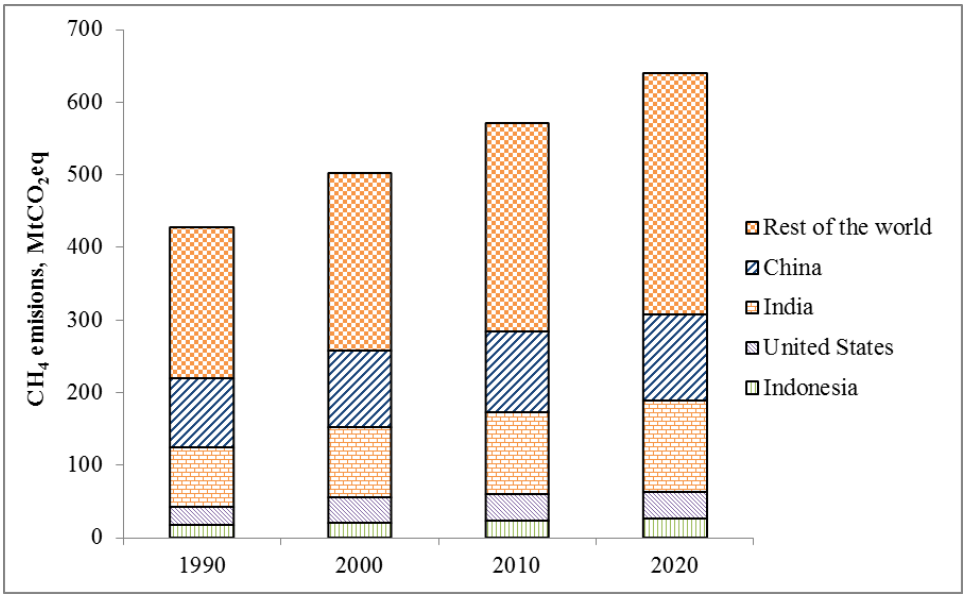


**Figure 2-2: Past, and projected, methane emission from wastewater**

Different types of anaerobic biological treatment are available, which are summarized in Table 2.1.

**Table 2-1: Major anaerobic treatment processes (Metcalf & Eddy, Inc., 2003)**

| Type of system | Common name | Use |
|---|---|---|
| Suspended growth | Anaerobic contact process | Carbonaceous BOD (CBOD) removal |
| | Anaerobic digestion | Stabilization, destruction of solids and pathogens |
| Attached growth | Anaerobic packed and fluidized bed | CBOD removal, waste stabilization, denitrification |
| Sludge blanket | Upflow anaerobic sludge blanket | CBOD removal (high-strength waste) |
| Hybrid | Upflow anaerobic sludge blanket / attached growth | CBOD removal |

Major advantages and disadvantages of the anaerobic treatment process over the conventional aerobic treatment process are listed in Table 2.2 (Lettinga, 1995; McCarty 2001; Metcalf & Eddy, Inc., 2003; Rittmann & McCarty, 2012, Skouteris, et al., 2012).

**Table 2-2: Advantages and disadvantages of anaerobic treatment**

| *Advantages* | *Disadvantages* |
|---|---|
| • High degree of waste stabilization<br>• Less biological sludge production<br>• No oxygen is required (hence less energy and cost for operation)<br>• Low nutritional requirement<br>• Methane is produced, which is a potential clean energy source<br>• Smaller reactor volume required<br>• Elimination of off gas air pollution<br>• Rapid response to feed addition after long period without feeding<br>• Capability of destroying most chlorinated hazardous compounds | • Longer start-up time needed<br>• High buffer required for pH control<br>• No nitrogen and phosphorus removal<br>• Slower growth rate of microorganisms<br>• More sensitivity to the adverse effects of environmental variables (pH, temperature)<br>• More susceptibility to upsets due to toxic substances<br>• Possibility of production of odour and corrosive gas<br>• Probable requirement of post-treatment to meet discharge standards |

### *2.2 Microbiology and biochemistry*

The overall anaerobic bioconversion of waste is a complex process involving many types of bacteria and archaea linked by several inter-related steps (Gujer & Zehnder, 1983; Parkin & Owen, 1986; Khanal, 2008). However, a simpler version of the process scheme has also been published (McCarty & Lawrence, 1969; Metcalf & Eddy, Inc., 2003; Grady, et al., 2011), describing it as a three stage process involving **hydrolysis**, **acidogenesis** and **methanogenesis**. A more recent trend mentions **acetogenesis** to precede methanogenesis, as shown in Fig 2.3.

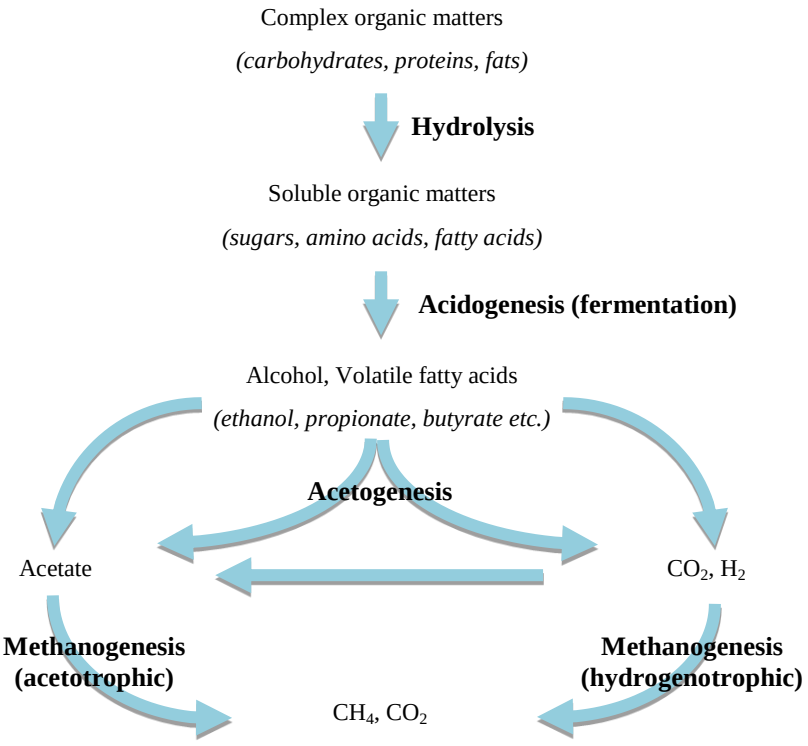


**Figure 2-3: Anaerobic digestion biochemical conversion pathways (adapted from Rapport, et al., 2008)**

*Hydrolysis*: Insoluble and complex organic materials must be solubilized for consumption by the microbes. Also, the large soluble organic molecules have to be broken down for easier transport through the cell membrane. These hydrolytic reactions and size reduction take place using extracellular enzymes like cellulases, amylases and proteases, produced by chemoheterotrophic

non-methanogenic bacteria (Speece, 1983). In this step, complex organic matters like carbohydrates, proteins and fats are converted to simple sugars, amino acids and fatty acids.

*Acidogenesis (fermentation):* In acidogenesis, hydrolyzed organic compounds are fermented to long chain fatty acids (propionic, butyric and valeric acid) by anaerobic bacteria as in the previous stage, such as *Bacteroides*, *Clostridia* and *Bifidobacteria.*

*Acetogenesis:* Intermediates accumulated in acidogenesis are further fermented to acetate, $CO_2$ and $H_2$ and this specific step is called acetogenesis. $H_2$ production from the fermentative reaction is small compared to that from oxidation of volatile and long chain fatty acids to acetic acid (termed as 'anaerobic oxidation'). $H_2$ from this stage acts as the electron donor for homoacetogens or hydrogenotrohpic methanogens that allow partial pressure of $H_2$ to be extremely low. Then, acetogenesis reaction becomes thermodynamically feasible. The stoichiometry of acetate, $CO_2$ and $H_2$ formation from ethanol, propionate and butyrate along with the standard Gibbs free energy value ($\Delta G^{o/}$) are given below (McCarty & Smith, 1986):

**Ethanol**

$$CH_3CH_2OH(aq) + H_2O(l) = CH_3COO^-(aq) + H^+(aq) + 2H_2(g),\ \Delta G^{o/} = +9.65\ \text{kJ} \quad (2.1)$$

**Propionate**

$$CH_3CH_2COO^-(aq) + 2H_2O(l) = CH_3COO^-(aq) + 3H_2(g) + CO_2(g),\ \Delta G^{o/} = +71.67\ \text{kJ} \quad (2.2)$$

**Butyrate**

$$CH_3CH_2CH_2COO^-(aq) + 2H_2O(l) = 2CH_3COO^-(aq) + H^+(aq) + 2H_2(g),\ \Delta G^{o/} = +48.30\ \text{kJ} \quad (2.3)$$

*Methanogenesis:* Acetic acid, $H_2$ and some of the $CO_2$ are then used by methanogens, which are members of a strictly anaerobic domain called Archaea, to produce methane gas. Mainly two groups are involved in methanogenesis: aceticlastic methanogens that utilize acetic acid to form methane, and hydrogenotrophic methanogen that oxidize $H_2$ and reduce carbon dioxide to methane. The reactions that lead the conversion of fatty acids to acetic acid and $H_2$ are thermodynamically

unfavourable under standard conditions (note the positive $\Delta G^{o/}$ values in Eq. 2.1 – 2.3). When the partial pressure of $H_2$ is high, these reactions will not proceed, and only fermentation will occur. However, when partial pressure of $H_2$ is low (from its consumption by methanogens), the reactions can proceed. Thus, methane formation by methanogens keeps the partial pressure of $H_2$ low, thereby allowing the production of more $H_2$ and acetic acid from acidogenesis to be used for methane formation. Likewise, methanogens are obligately linked to the bacteria performing acidogenesis as the latter produce the carbon and energy sources required by the former. Such a relationship between these two microbial groups is called obligate syntrophy. Hydrogenotrophic methanogens are classified into three orders of the domain *Archaea*: *Methanobacteriales*, *Methanococcales*, and *Methanomicrobiales*, while all aceticlastic methanogens are of the order *Methanosarcinales*. Fig 2.4 shows a picture of methanogenic bacteria.

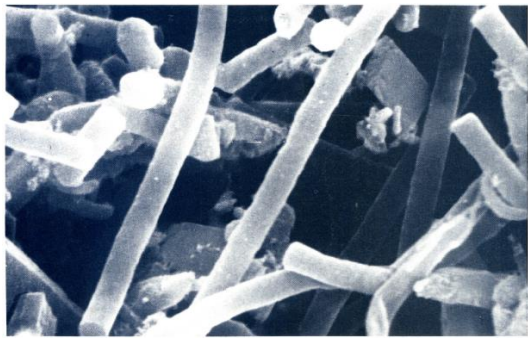

**Figure 2-4: Electron micrograph of methanogenic bacteria (Speece, 1983)**

The two steps that are most likely to be rate limiting in the anaerobic process are hydrolysis of complex organics and conversion of volatile acids to methane (Rittmann & McCarty, 2012). The stoichiometry of chemical reactions for methane conversion is given below:

**Acetotrophic methanogenesis**

$$CH_3COOH \rightarrow CO_2 + CH_4 \quad (2.4)$$

**Hydrogenotrophic methanogenesis**

$$CO_2 + 4\,H_2 \rightarrow CH_4 + 2\,H_2O \quad (2.5)$$

The flow of electron along the pathway of the anaerobic treatment process is shown in Fig 2.5 (McCarty & Smith, 1986), which depicts that 72% of the methane conversion happens from acetate cleavage and 28% results from reduction of $CO_2$ using $H_2$ as an energy source.

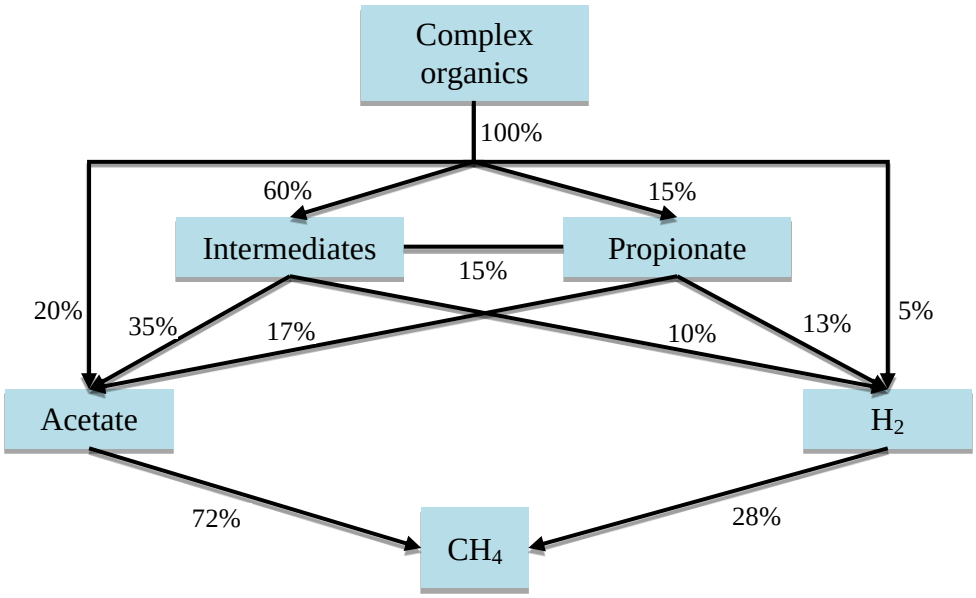


**Figure 2-5: Electron flow in the conversion of complex substrates to methane**

A list of essential features for favourable bacterial growth and efficiency of an anaerobic treatment process would include (McCarty, 1964; Parkin & Owen, 1986): 1. optimum retention time, 2. sufficient mixing (for bacteria-feed contact), 3. appropriate pH, 4. suitable temperature, 5. sufficient concentrations of required nutrients, 6. absence of toxic materials and 7. proper feed characteristics. Some of them are discussed further under the following section.

### *2.3 Operation and performance parameters*

The common operational parameters that are monitored or controlled are hydraulic retention time (HRT), solids retention time (SRT), temperature, pH and food to microorganism ratio (F/M).

The HRT and SRT are two of the fundamental parameters having profound impact on the operation and performance of an AnMBR. Longer HRT will necessitate a larger footprint (Smith, et al., 2012)

and shorter HRT will cause higher MLSS/MLVSS concentration due to higher OLR (Huang, et al., 2011). Longer SRT is generally helpful for retention of more biomass in a reactor; however it may also lead to higher biomass associated products (BAP), which are a part of SMPs in the bioreactor. This increase in SMP will result in higher effluent COD (Stuckey, 2012) and more membrane fouling in anaerobic membrane bioreactor (Huang, et al., 2011), which is discussed later. Usually an SRT of more than 20 days is applied for anaerobic wastewater treatment at $30^0$C, and higher SRT is required for lower temperatures (Metcalf & Eddy, Inc., 2003).

Due to the slow growth rate of microorganisms in anaerobic processes, temperature is a crucial parameter as it affects their growth rate as well as their performance (Rittmann & McCarty, 2012). The growth rate almost doubles with a rise of 10 $^0$C for a general mesophilic population operating in the range of 10 $^0$C to 35 $^0$C.

The acceptable range of pH for methanogens is generally 6.6 to 7.6 (McCarty, 1964). A pH value outside this range will have an unfavourable effect on process efficiency, and the system may take several weeks or months to recover. Maintaining the pH over 6.6 is also difficult in many circumstances. The intermediate organic acids produced during start-up, overload or unsteady periods can lower the pH and hinder methane production. Rittmann & McCarty (2012) express that the pH is governed by the concentrations of alkalinity in the reactor liquid (conventionally expressed in the unit of mg/L as $CaCO_3$) and carbon dioxide ($CO_2$) in the reactor headspace. It is assumed that $CO_2$ is in equilibrium between the gas phase and the liquid phase in the reactor, which is considered as the case in anaerobic systems.

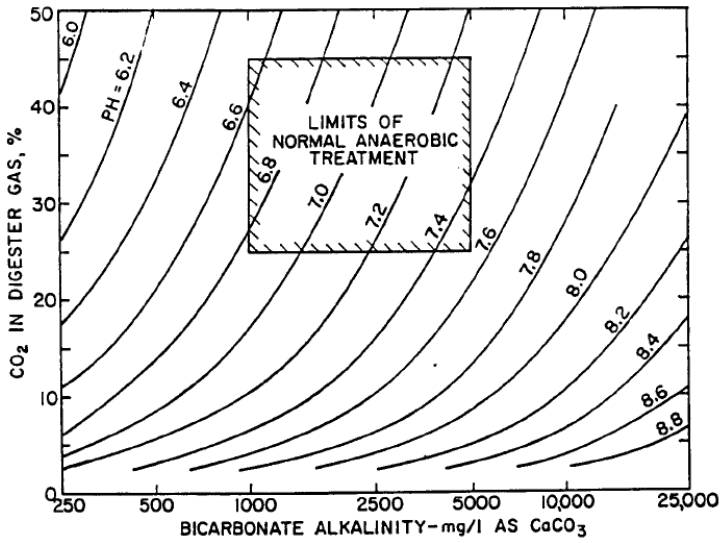


**Figure 2-6: Relationship among reactor pH, bicarbonate alkalinity and the percentage of carbon dioxide in the gas phase near 35$^0$C (McCarty, 1964)**

For the normal percentage of $CO_2$ in a reactor (25–45%), a bicarbonate alkalinity of 500-900 mg/L as $CaCO_3$ is required to maintain the pH over the minimum desired value (Figure 2.6). A higher percentage of $CO_2$ will mean a higher requirement of alkalinity. One of the most effective chemicals that can be used to control pH is sodium bicarbonate. It has several advantages over other available means to raise pH (e.g. lime): it is relatively inexpensive if purchased in bulk; it is non-reactive with $CO_2$ and hence does not create a vacuum; it can be dissolved in water for easy mixing and application; a small quantity is required; and its addition does not cause a toxic condition or excessive high pH (McCarty, 1964).

F/M is a controllable parameter that can significantly influence system performance. A higher F/M value results in higher amounts of EPS, SMP and fine particles in a system, and accelerates membrane fouling in AnMBRs (Liu, et al., 2012).

The common performance indicators and analytical parameters measured are COD of influent and effluent (with the prime objective of measuring the COD removal percentage, also termed as treatment efficiency), OLR (also called COD loading rate), biogas/methane yield and volatile fatty

acids (VFA)/Alkalinity ratio. COD is a universal measure of pollutant strength. It is fundamentally the amount of electrons available in a certain organic compound (also referred as electron equivalents), expressed as the amount of oxygen required to accept those electrons when the compound is completely oxidized to carbon dioxide and water (Grady, et al., 2011). Low COD concentration of effluent (mg/L) would therefore indicate very little organic compounds remaining in the effluent and ascertain the treatment efficiency of a system. The OLR is simply the measure of total amount of organic load (or COD) a reactor can handle in a day, usually normalised with the total working volume of the reactor.

In methanogenesis, carbon is reduced to its most reduced oxidation state, $CH_4$, by utilizing the electron equivalents in organic matter (COD), resulting in "waste stabilization" (Rittmann & McCarty, 2012). As one mole of $CH_4$ contains 8 electron equivalents and 1 electron equivalent is comparable to 8 g of $O_2$ (COD), each mole of $CH_4$ contains 64 g of COD. As the volume of $CH_4$ at standard temperature and pressure (STP, $T=0^0C$ and P=1atm) is 22.4 L, each g of COD stabilized would theoretically generate 0.35 L of $CH_4$ gas at STP.

The A/TIC, i.e. the acids (A) to total inorganic carbon (TIC) ratio is an established indicator for the process stability inside a reactor. The less the ratio, the less stressed the reactor is. Some researchers mention the desired ratio to be less than 1 while some mention it to be less than 0.3. The A/TIC is calculated by dividing the VFA by the alkalinity (Eq. 2.6):

$$\frac{A,mg/L}{TIC,mg/L} = \frac{VFA,mg\ COD/L}{Alkalinity,mg\ CaCO3/L} \tag{2.6}$$

Significant changes of the A/TIC-ratio indicate disruption of the system stability so that a counter-measurement step can be taken (decrease or increase of feed quantity, addition of buffer capacity) at the appropriate time (Heeb, 2009).

### *2.4 Anaerobic membrane bioreactor*

Retention of solids (biomass) is a pivotal feature that greatly enhances the performance of a biological treatment process, especially for the anaerobic process because of the slow growth rate of methanogens (Stuckey, 2012). Use of membrane allows complete retention of biomass in the reactor, thereby decoupling the solids retention time (SRT) from HRT (Liao, et al., 2006). The benefits of using a membrane in biological reactors are multi-faceted: suspended solids in the effluent is close to zero; effluent is substantially disinfected (usual membrane pore size is less than 0.1 μm); complete biomass retention allows separating SRT from HRT; substantial reduction of reactor size (because of concentrated biomass) and increase in organic loading rate is made possible (Santos, et al., 2011; Lin, et al., 2013). Retention of biomass in the reactor also facilitates the development of many slow growing microorganisms required for degradation of complex organics and may enhance hydrolysis of particulates; in addition, many active extracellular enzymes can also be retained that can create an active environment for microbial biochemical reactions (Cicek, et al., 2001).

Originally commercialized as a 'side-stream' process in the early 1970s, the membrane separation process saw its growth of successful application in aerobic biological wastewater treatment after it was introduced as an 'immersed' process since the early 1990s (Judd, 2008; Cote, et al., 2012). The side-stream and immersed configurations are described later in this chapter. The first commercial membrane bioreactors (MBR) were developed by Dorr-Oliver in the late 1960s (Cote, et al., 2012) and combined flat-sheet UF membranes (in a 'side-stream' configuration) with a conventional activated sludge process for application to ship-board sewage treatment (Judd & Judd, 2011). Trailing the success of membrane technology in aerobic processes, it was also incorporated in anaerobic wastewater treatment processes (Liao, et al, 2006; Stuckey, 2012).

The concept of using membrane filtration with anaerobic treatment of wastewater was reportedly first applied by Grethlein in 1978 to treat septic tank effluent in an external cross-flow membrane set-up. Dorr-Oliver developed the first commercially available AnMBR that treated high strength whey processing wastewater. In the last two decades, research on AnMBR has increased substantially with studies on membrane materials, membrane fouling and foulants, membrane cleaning and fouling management strategy. The advantages of this process over the conventional anaerobic systems and aerobic MBR systems are widely established and acknowledged. Among those, the most prominent ones are total biomass retention, increased treatability, lower sludge production, a smaller footprint and net energy production (Lin, et al., 2013).

### *2.4.1 Membrane configurations in AnMBR*

There are two basic types of membrane configurations in an AnMBR, namely side-stream (or external), where the membrane modules are contained in a separate vessel from the reactor, and submerged (or immersed), where the membrane module(s) are installed in the reactor itself (Judd & Judd, 2011, Skouteris, et al., 2012). Both of these configurations can be operated under 'pressure' mode or 'suction' mode, putting the total number of configurations to four (Visvanathan & Abeynayaka, 2012). Most of the commercial applications are seen to follow the submerged configuration, due to lower associated energy requirements, whereas most of the AnMBR which are set up for research used the side-stream configuration (Liao, et al., 2006). The side-stream configuration offers the benefits of more hydrodynamic control of fouling, easy replacement of the membrane without disturbing the microorganisms in the main reactor, and higher fluxes (Lin, et al., 2013). The two basic reactor configurations are shown in Fig. 2.7.

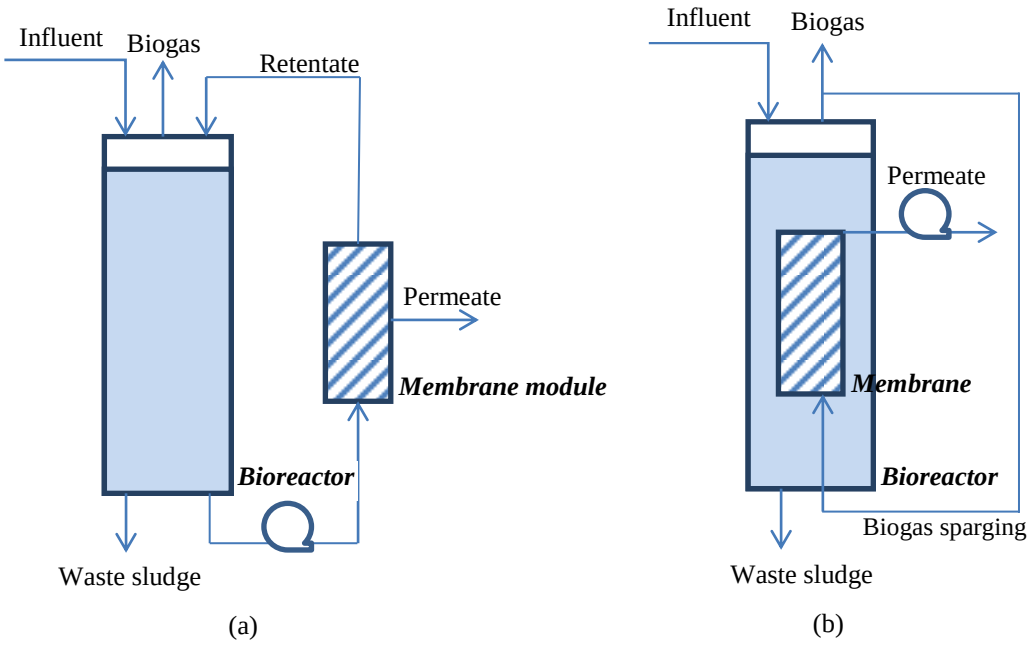


**Figure 2-7: Schematic of membrane configuration in AnMBR (a) side-stream (external) and (b) submerged (immersed)**

Another configuration of the AnMBR discussed recently is the sequential membrane reactors used in two-stage anaerobic reactor set-up, with the first reactor using a coarse membrane with larger pore size (Stuckey, 2012). Similar to the set-up of a two-stage anaerobic digester, a two-stage AnMBR will have the first reactor as the hydrolytic/acidogenic reactor and the second one as the methanogenic reactor, as depicted in Fig. 2.8.

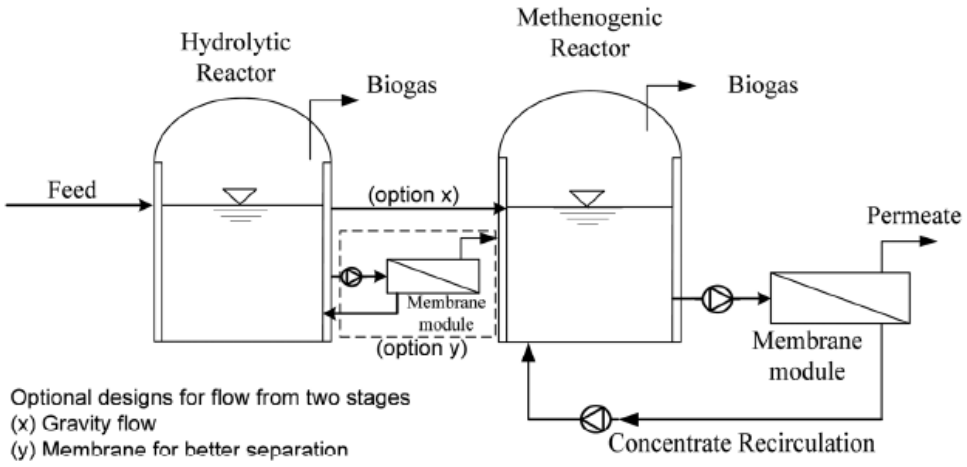


**Figure 2-8: Two-stage AnMBR configuration (Visvanathan & Abeynayaka, 2012)**

### *2.4.2 Flux, transmembrane pressure and permeability in AnMBR*

The most common parameters to monitor membrane performance are flux, transmembrane pressure (TMP) and permeability. Depending on the mode of operation, proper functioning of the membrane can be characterized by monitoring changes in flux (Equation 2.7) or TMP (Equation 2.8). During the constant pressure mode of operation, flux is calculated with the monitored flow rate data using Eq. 2.7. Under the constant flow rate mode of operation, TMP is calculated from the pressure data using Eq. 2.8. The permeability is used to express the membrane performance irrespective of mode of operation and calculated using Eq. 2.9:

$$Flux\ (J) = \frac{Permeate\ flow\ rate,\ \ L/h}{Membrane\ surface\ area,\ \ sq.m} \tag{2.7}$$

$$TMP = \left(\frac{Pressure_{feed} + Pressure_{concentrate}}{2}\right) - Pressure_{permeate} \tag{2.8}$$

$$Permeability = \frac{Flux}{TMP} \tag{2.9}$$

The units used for flux, TMP and permeability are L/m$^2$/h (LMH), kPa and LMH/bar, respectively. The term Fouling Index is sometimes of interest to show the change of flux or TMP value over a period of time. It is usually expressed as kPa/min, and calculated using Eq. 2.10:

$$Fouling\ Index\ (FI) = \frac{TMP_{final\ time} - TMP_{initial\ time}}{Final\ time - Initial\ time} \tag{2.10}$$

For every membrane operation, there is a critical flux. If permeate is pumped over the critical flux value, the rise of TMP will be unsatisfactorily high, performance of the membrane will be impeded and risk of membrane fouling will be increased. Nevertheless, a perfectly non-fouling operation is practically not possible, but operating below the critical flux has shown to cause a slow linear increase in TMP, without significant fouling (Liao, et al, 2006; Meng, et al., 2009). The critical flux is a function of sludge concentration and characteristics, and membrane characteristics. There is

more than one method to determine critical flux of a membrane, the most common being to measure the immediate rise of TMP against a step increase in flux. Usually critical flux is calculated with fresh or cleaned membrane. The critical flux of a membrane will decrease over time due to irreversible fouling. Membrane fouling is further discussed in the following sections.

### *2.4.3 Membrane fouling: mechanisms, types and contributing factors*

Although membrane systems can achieve high COD removals, a major hurdle that impedes their performance and reduces the flux is membrane fouling, which can be simply defined as the deposition of materials on membrane surface ('cake layer formation') or clogging of membrane pores ('pore blocking') (Akram & Stuckey, 2008; Meng, et al., 2009; Visvanathan & Abeynayaka, 2012).

According to the types of material accrued on the membrane, fouling can be classified as biofouling, organic fouling or inorganic fouling (Liao, et al., 2006; Meng, et al., 2009; Lin, et al., 2013), though all of these three fouling mechanisms usually occur together. Biofouling happens through the attachment of biological (cell or cell derived) components with the membrane surface under the mechanisms of pore clogging, sludge cake formation, and adsorption of extracellular polymeric substances (EPS). Pore clogging is caused when cell debris that are of identical size to the pore opening accumulate in membrane pores thereby reducing passage for filtration. If the shear flow on the membrane surface is inadequate, a thick cake layer of biomass forms by attachment to the polymeric surface and results in major hydrodynamic resistance (Choo & Lee, 1996). The extent of cake deposition depends in part on the concentration of solids, so in a CSTR configuration, high mixed liquor suspended solids (MLSS) in a reactor will increase sludge cake formation. The third type of biofouling is caused by extracellular polymeric substances (EPS) and soluble microbial products (SMP) as they are adsorbed and accumulated on the membrane surface (Meng, et al., 2009, Lin, et al., 2009). EPS are generally defined as large polymeric material that surround the microbial

cell surface and can be extracted by using chemical and physical methods. SMP are defined as microbial products released into the bulk solution as a result of the cell lysis, the hydrolysis of EPS, as well as of the interaction of microorganisms with their surroundings. Therefore, while EPS are of extracellular origin, SMP originate from cell lysis and decay (Aquino, et al., 2006). Lin, et al. (2011a) showed that bound EPS was the main support that kept the sludge floc on the membrane surface. They also found much higher concentration of EPS in cake sludge than in bulk sludge of a submerged AnMBR, suggesting EPS as the major reason for cake layer formation and high specific filtration resistance of cake sludge. In a study, Aquino & Stuckey (2002) found internal fouling of the membrane was greatly caused by SMP released from endogenous decay.

Organic fouling is caused by the adsorption and aggregation of different organic components in the bulk sludge, such as colloidal particles and soluble organics like EPS and SMP. EPS and SMP can also be considered under organic category, as EPS can exist as soluble organics and SMP can derive from lysis of feed in addition to biological sources (Laspidou & Rittmann, 2002). Colloids can cause pore clogging and they are found to be a major foulant of both MF and UF membranes especially with the use of pressure-driven, external cross-flow filtration where the shear stress liberates more colloids (Choo & Lee, 1996). In an AnMBR with high OLR, the residual (untreated) COD is higher and membrane flux is lower. The absolute residual COD affects fouling, therefore operating at higher SRT can help reduce the residual COD and hence the organic fouling caused by it (Liao, et al., 2006).

The most common inorganic fouling was found to be the precipitate of struvite, a phosphate mineral formed by the following equation (Visvanathan & Abeynayaka, 2012):

$$Mg^{2+} + NH_4{}^{+} + PO_4{}^{3-} + 6H_2O \rightarrow MgNH_4PO_4 \bullet 6H_2O \qquad (2.11)$$

This inorganic precipitation (Eq. 2.11) also has a role in prolonged fouling in AnMBR as it causes increased hardening of the cake layer (Choo & Lee, 1996). Precipitation of other phosphate and calcium salts is possible, especially on inorganic membranes. Inorganic ions of Mg, Al, Fe, Ca, and Si have been seen to combine with organic particles to form a gel layer that covers the membrane surface. Unlike the aerobic systems, concentration of ammonia and carbonate are higher in anaerobic systems due to higher loads, protein hydrolysis and carbonate buffer chemistry; hence precipitation with these ions can occur (Liao, et al, 2006; Stuckey, 2012).

Bulk sludge concentration and particle size distribution also have profound effect on membrane fouling. The studies of Lin et al. (2011a; 2011c) indicate the significance of smaller flocs having higher filtration resistance over the bulk sludge due to 1.5 times higher bound EPS and significant variation of microbial community structure in smaller flocs. Membrane permeability is affected by concentration of biomass and distribution of particle size (Choo & Lee, 1996). However, among all forms of fouling in AnMBRs, cake formation was identified as the most dominant feature contributing to membrane fouling (Jeison & van Lier, 2006, Lin, et al., 2009; Charfi, et al., 2012).

Whether or not the accumulated foulants can be cleaned (removed) and membrane can return to its pre-fouling stage (reversed) is the criteria for another type of classification for membrane fouling, which can be mentioned as removable fouling, irremovable fouling and irreversible fouling (Meng, et al., 2009). The fouling that can be removed easily by physical means (e. g. backwashing) can be termed removable. The irremovable fouling can not be removed by physical process and requires chemical cleaning. Removable fouling is analogous to reversible fouling, as termed by some other researchers, and the foulants are loosely bound, forming the cake layer. Irremovable fouling is the result of strongly bound foulants causing pore blocking. The irreversible fouling is the permanent fouling and deterioration of the membrane material over time (in part due to chemical exposure

while cleaning), which can not be recovered and will continue until the end of membrane operation life. Fig. 2.9 illustrates the three types of fouling discussed here.

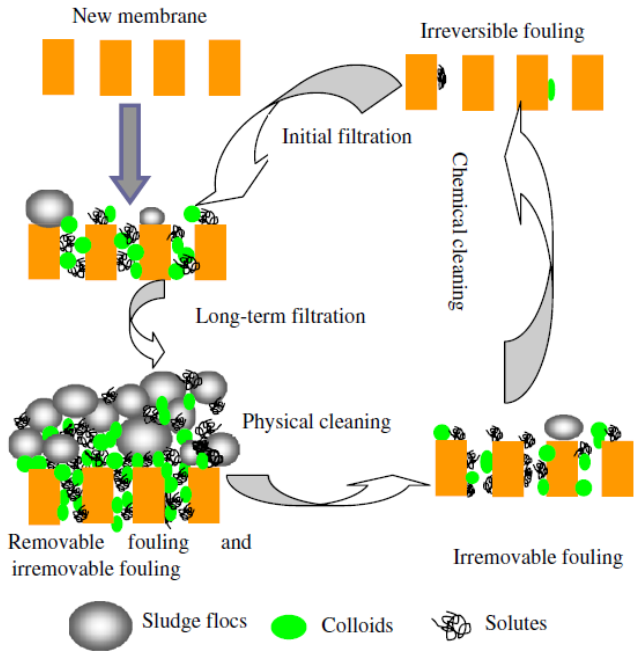


**Figure 2-9: Schematic illustration of removable, irremovable and irreversible fouling (Meng, et al., 2009)**

Many empirical and theoretical models have been devised to describe the membrane fouling phenomena; "resistance-in-series" model is considered the simplest of them all (Chang, et al., 2002):

$$J = \frac{TMP}{\eta . R_t} \tag{2.12}$$

$$R_t = R_m + R_c + R_f \tag{2.13}$$

where J = permeate flux; TMP = transmembrane pressure; η = dynamic viscosity of the permeate; $R_t$ = total membrane resistance; $R_m$ = intrinsic membrane resistance; $R_c$ = (reversible) cake resistance caused by the cake layer deposited over the membrane surface; and $R_f$ = (irreversible) fouling resistance produced by adsorption of dissolved matter (pore narrowing) and/or pore blockage within the membrane (plugging). According to this model the flux is inversely

proportional to the total resistance, the latter being the sum of individual, supposedly discrete resistances. While several researchers have identified the individual resistance components in their work, some prefer to quote a single resistance value including all resistances posed other than that of the clean membrane (Chang, et al., 2002).

The factors affecting membrane fouling can be both biotic and abiotic, and can be listed as parameters relating to: membrane materials, biomass characteristics, feedwater characteristics, reactor operating conditions and membrane operating conditions (Le-Clech, et al., 2006; Dereli, et al., 2012; Charfi, et al., 2012, Stuckey 2012). Membrane fouling eventually affects process performance, as shown in Fig. 2.10.

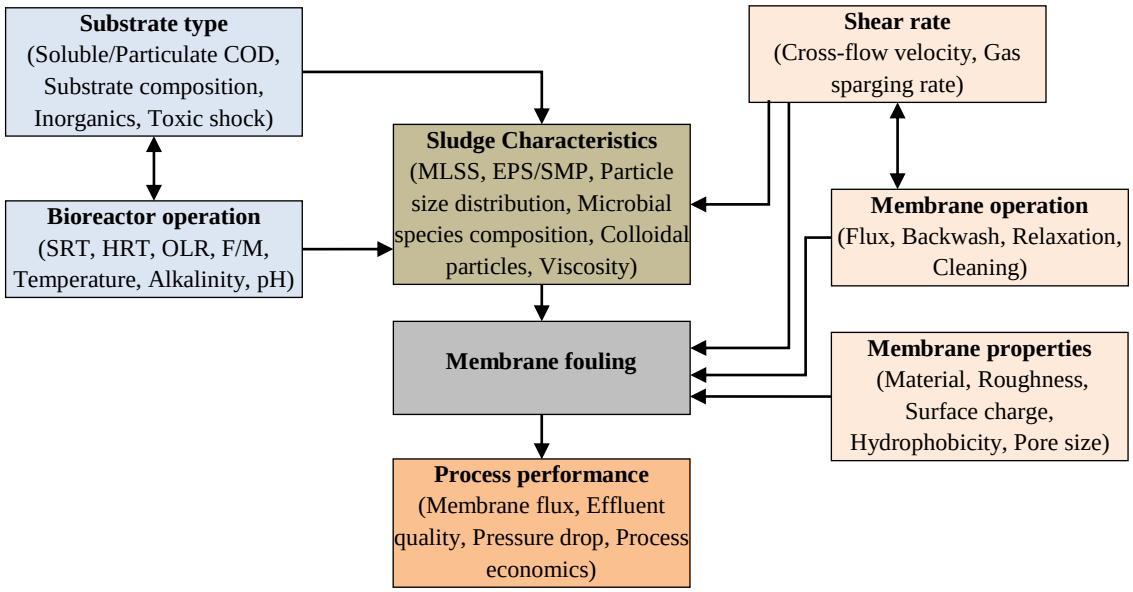


**Figure 2-10: Interactions between fouling parameters, membrane fouling and process performance**

### *2.4.4 Membrane fouling: controlling and cleaning*

Extensive review of publications on membrane fouling and their control has been performed by Meng, et al. (2009) and Lin, et al. (2013). Both the groups have specified direct relations between the contributing factors and their impact on membrane operation and fouling from the findings of those researches, as summarized in Table 2.3.

**Table 2-3: Relationship between various fouling factors and membrane fouling**

| Condition/Factor | Effect on membrane fouling |
|---|---|
| *Sludge condition* | |
| MLSS | - MLSS ↑ → TMP ↑, fouling potential ↑<br>- MLSS ↑ → normalized permeability ↓<br>- MLSS ↑ → cake resistance ↑ , specific cake resistance ↓ |
| Viscosity | - Viscosity ↑ → membrane permeability ↓<br>- Viscosity ↑ → membrane resistance ↑ |
| F/M | - F/M ↑ → fouling rates ↑, Protein in foulants ↑<br>- MLSS (2–3 g/L): F/M ↑ → irremovable fouling ↑<br>- MLSS (8–12 g/L): F/M ↑ → removable fouling ↑ |
| EPS | - polysaccharide ↑ → fouling rate ↑<br>- bound EPS influences on specific cake resistance<br>- bound EPS ↑ → membrane resistance ↑<br>- loosely bound EPS contributes to most of the filtration resistance of sludge |
| SMP | - SMP is more important than MLSS in regards to fouling<br>- SMP↑→filtration resistance↑<br>- SMP ↓→ fouling index ↓<br>- High-MW protein and carbohydrate material↑→internal fouling↑<br>- SMP↑→flux ↓ |
| Particle size | - amount of small flocs↑→filtration resistance↑<br>- floc size↓→specific cake resistance↑ |
| Microbial community | - some bacteria play a pioneering role in cake formation<br>- filamentous bacteria ↑ → sludge viscosity ↑<br>- bulking sludge could cause a severe fouling<br>- filamentous bacteria ↓→ cake resistance ↓ |
| *Operating condition* | |
| SRT | - SRT ↓ → fouling ↑<br><br>- SRT↑→sludge activity↓, SMP↑→dTMP/dt↑<br>- SRT↑→MLVSS↑, floc size↓→irreversible fouling↑ |
| HRT | - HRT↓→EPS↑, SMP↑→cake resistance↑<br>- HRT↓→biopolymers↑, floc size↓→specific cake resistance↑<br>- HRT↓→biomass concentration↑<br>- HRT↓→dTMP/dt↑ |
| Hydrodynamic condition | - gas sparging rate ↑ → permeability ↑<br>- gas sparging rate↑→ flux↑<br>- gas sparging time↓→TMP↑<br>- bubble-induced shear reduces fouling significantly<br>- air/gas backwashing is preferable for fouling control<br>- larger bubbles are preferable for fouling control<br>- air/gas scouring can prolong membrane operation |
| Permeate flux | - sub-critical flux mitigates fouling<br>- permeate flux↑→long-term operation period↓<br>- permeate flux↑→cake formation rate↑, fouling rate↑ |
| Temperature | - temperature↑→viscosity↓, COD removal↑, flux↑ |
| *Membrane characteristics* | - MWCO↑, surface roughness↑→flux decline↑<br>- pore size↑→attainable flux↓<br>- PEI membrane fouled faster than PVDF membrane coated with PEBAX |

*F/M: food-to-microorganism ratio (kg COD/kg MLVSS/day)

Adjustment and/or modification of the factors presented in Table 2.3 may allow controlling the extent of membrane fouling by creating favourable conditions, as shown by the schematic illustration in Fig. 2.11 (adapted from Meng, et al., 2009).

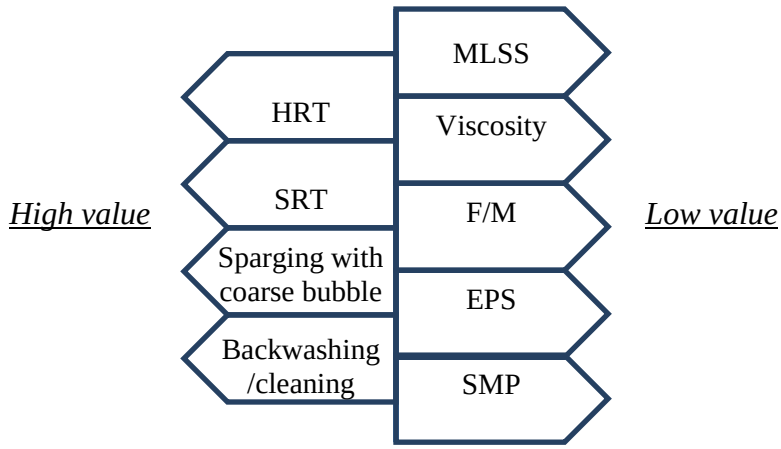


**Figure 2-11: Favourable conditions mitigating membrane fouling**

Application of an adsorbent/precipitant (e. g. activated carbon, cationic polymers, biopolymers, EDTA, metal salts) has been studied by several researchers to improve membrane performance and to prevent fouling (Stuckey, 2012). Powdered activated carbon (PAC) has been the most popular "flux enhancer" used in membrane reactors due to increase in floc size and decrease in soluble and colloidal organics, while some other absorbents that were used are zeolite, bentonite, vermiculite and *Moringa oleifera* (Lin, et al., 2013). Akram & Stuckey (2008) reported that addition of PAC in an AnMBR resulted in a substantial flux improvement in addition to an increase in treatment performance (maximum COD removal of 98% at 6h HRT). Pre-treatment of feed (e.g. filtration, pH control, ion exchange) has also proven to be beneficial for membrane performance and especially, control of inorganic fouling (Meng, et al., 2009).

An effective operational measure to control membrane fouling is intermittent pumping of permeate (on and off for a specific period of time in repeated cycles) that provides a "relaxation" for the membrane, instead of continuous pumping. A common practice usually applied along with this step

is the sparging of biogas from the reactor through the bottom of the membrane module so as to create a shear force that would help alleviate cake formation on the membrane surface. The intermittent permeation and gas bubbling have proven to be very effective in minimizing membrane fouling, providing more than ten times longer filtration life than without them (Cerón-Vivas, et al., 2012).

Interventions in the form of physical and chemical cleaning of the membrane help remove the cake layer and pore blockage, as also discussed in the previous section. Physical cleaning is usually performed by reverse pumping air/gas or backwashing by permeate/clean water through the membrane. Chemical cleaning is performed by backwashing with acid solution (citric, oxalic, nitric) with a pH of around 2 to remove inorganic foulants, and chlorine solution (NaOCL) with a pH of 10 to 12 to remove organic foulants. Chemical cleaning has some disadvantages as frequent cleaning can decrease the membrane material lifetime, and especially in the case of external/side-stream membranes, it requires taking the membrane off-line and creates chemical waste to be dealt with (Zhang, et al., 2007). In addition to the regular maintenance cleaning, a recovery cleaning is performed by soaking the membrane in chemical solutions used for maintenance cleaning, when the fouling is too severe to continue normal operations.

### *2.5 Application of AnMBR for treatment of different wastewater types*

Anaerobic membrane treatment technology has been applied to treat wastewater of a wide range of varieties comprising both synthetic and real wastewater, and increasingly more attention and efforts towards its treatment potentials have been conferred by individuals and groups in the last decade (Stuckey, 2012; Lin, et al., 2013). The trend is likely to continue, and may be accredited to firstly, the increasing stringent requirements and options for reuse of industrial wastewater effluent, and secondly, the growing cost of operation and labour as opposed to the declining cost of membrane equipment.

### *2.5.1 Synthetic wastewater*

Use of synthetic wastewater to perform treatment experiments with AnMBR at the laboratory scale is common. It is due to the fact that AnMBR, especially submerged configuration is a relatively new idea and testing with different operating conditions and influent loads are more convenient with this type of feed. Different substrates that have been used for this purpose include glucose, starch, molasses, peptone, yeast and volatile fatty acids (VFAs). The COD removal efficiencies were generally over 95%, with applied OLRs of less than 10 kg COD/$m^3$/day and lower biomass concentration than large scale reactors and HRARs. The reason for not operating at very high OLRs or biomass concentration is the requirement of long term operation for such studies in order to investigate performance variation and membrane fouling phenomena (Lin, et al., 2013).

### *2.5.2 Municipal wastewater treatment*

From the early stages of biological treatment, aerobic process has been preferred for application towards treatment of municipal wastewater (MWW) treatment, and not anaerobic due to some major features of the latter process: 1. heating of the reactors to mesophilic (30-40 $^0$C) or thermophilic (50-60 $^0$C) temperatures, 2. requirement of long SRT for the slow-growth microorganisms and 3. effluent quality not meeting the discharge standards without post-treatment (Smith, et al., 2012). Introduction of membrane technology has taken the aerobic treatment systems even further ahead, with 4400 installations by the top three suppliers alone (Kubota, Mitsubishi Rayon, and Zenon (now GE)) as of 2009 (Judd & Judd, 2011). However, the combination of HRAR and membrane technology can overcome the shortfalls of biological treatment at low temperatures, treating low strength wastewater at short HRTs, longer retention of biomass and higher quality effluents. It also offers the added advantages of lower sludge generation, net energy production and elimination of the huge cost for aeration associated with aerobic treatment. Reviews of several studies on anaerobic membrane treatment of MWW revealed that they can be operated at very high

SRT, low HRT (as low as 3 hours), and can achieve a maximum of 99% removal of COD and >99% removal of suspended solids (Lin, et al, 2011b; Smith, et al., 2012). Though it achieved high removal of contaminants, one bottleneck of the anaerobic treatment is that the removal of nutrients (nitrogen and phosphorus) is usually very low. This can be beneficial, though, especially in areas of water scarcity where this treated water may be used for agricultural or landscape irrigation utilizing both the water and the nutrients; in a broader sense this would also reduce fossil fuel consumption because of using N and P from wastewater instead of manufactured fertilizers (McCarty, et al., 2011). Removal of nutrients is possible by the coupling of AnMBR with conventional aerobic nutrient removal treatment, though it faces challenge because of low COD:N and COD:P ratio in AnMBR effluent (Smith, et al., 2012). However, one of the exciting recent developments by the use of anaerobic membrane system is the enhancement of nitrogen removal potential by anaerobic ammonia oxidation (Anammox) process. Anaerobic and aerobic processes were applied in combination to achieve high COD (>90%) and nitrogen (>95%) removal, while introduction of membrane in an anaerobic sequential batch reactor under a separate study increased Anammox activity by 19 times, proving that retention by membrane of the slow growing organisms were beneficial in this case (Stuckey, 2012). Presence of trace contaminants, such as endocrine disrupting chemicals (EDC) and pharmaceutically active compounds (PhAC) has become of significant interest in recent years. Although the treatment capability of these elements by anaerobic digestion is low (10-48%), bioaugmentation and relatively longer HRT (30 days) can improve the removal efficiency. Economically, the odds are in favour of anaerobic treatment, as both the UASB and AnMBR have lower electricity usage and hence less operational cost than their aerobic counterparts. Operational cost can even be fully offset by using recovered biogas (Liao, et al., 2006). Therefore, AnMBR process can be a suitable technology for MWW treatment as long as good performance and economic operation of membrane are ensured. Anaerobic digestion is widely applied for stabilization of municipal sewage sludge. COD removals are generally lower for sewage

treatment (71-74%), even by UASB, due to the presence of refractory COD and inert solids compared to food and beverage wastewaters. However, AnMBRs, incorporating membrane with anaerobic process, were able to remove more than 90% of COD and close to 100% of suspended solids in the effluent. AnMBRs also achieved high COD removal in treating night soil and sludge heat treatment liquor (Liao, et al., 2006).

### *2.5.3 Industrial wastewater treatment*

Industrial wastewaters treated by AnMBR include effluent from food processing, pulp and paper, tannery, chemical, pharmaceutical, textile, petroleum and manufacturing industries. They are generally characterized by high organic strength with relatively high solids concentration, and particularly those from industries other than food processing tend to have extreme physico-chemical characteristics (e.g. pH, temperature, and salinity), synthetic and natural chemicals, and toxins. Effluent from food processing industries are readily biodegradable and non-toxic and they fit perfectly in the "high organic strength, highly particulate" category of wastewater, deemed by Liao, et al. (2006) as the most suited for treatment by AnMBRs. On average, COD removal efficiency in treating industrial wastewater was over 90%, with applied OLRs ranging from 2-15 kg COD/$m^3$/day. Because most of the AnMBRs used CSTR configuration, this OLR range may seem a little lower than what can be achieved with the high rate anaerobic reactors (UASB, EGSB); they are, however, higher than the conventional CSTR digesters. Wastewaters with extreme characteristics, chemical and toxic materials can also be treated with AnMBR, provided they have auxiliary or pre-treatment steps in place. Evaporator condensate (EC), an important type of wastewater produced from pulp and paper mill industry, was treated by several researchers using both mesophilic and thermophilic AnMBRs resulting in a COD removal of 93-99% and good biogas production under OLRs of 1-24 kg COD/$m^3$/day. Fischer-Tropsch process wastewater, a typical petrochemical wastewater with high strength and low pH that consists of short chain organic

acids was treated by AnMBR achieving effluent COD of less than 500 mg/L and an OLR up to 25 kg COD/$m^3$/day, while fixed media systems proved to be a failure (Lin, et al, 2013). Use of AnMBRs for other type of wastewaters, such as textile, pharmaceutical, oil refinery and coke plant wastewater is limited, and usually seen to be applied in combination of aerobic MBR. The combined system, in these cases achieved satisfactory contaminant removal as opposed to using a single system (Lin, et al., 2013). In recent years, AnMBR has also been successfully tested for the treatment of meat processing/slaughter house effluent, palm oil mill effluent and cheese whey (Stuckey, 2012).

#### *2.5.3.1 Anaerobic treatment of meat processing wastewater*

Meat processing industry is usually large, and exists in almost every country. It generates a considerable amount of wastewater containing polluting components, owing to the fact that a large amount of water is used in the processing, cleaning and sanitizing stages (Liu & Haynes, 2011). As a wastewater of high organic strength, effluent from meat processing plants and slaughterhouses is considered to be highly suited for treatment by anaerobic processes (Johns, 1995; Mittal, 2006; Nacheva, et al., 2011).

#### *2.5.3.2 Characteristics of meat processing wastewater*

Meat processing/slaughterhouse wastewater contains blood, animal fat, hair, particles of skin and meat, and excrements – contributing to the high levels of $BOD_5$, COD, N, P, and pathogenic microorganisms (Massé and Masse, 2000; Rajeshwari, et al., 2000; López-López, et al., 2010, Liu & Haynes, 2011). Residual blood is considered as the main source of organic matter in such wastewater (Louvet, et al., 2013). The soluble fraction in slaughterhouse wastewater is in the range of 40–60%. The suspended and colloidal components in the form of fats, proteins, and cellulose can have an adverse impact on the performance of anaerobic reactors, leading to deterioration of the microbial activity (Lettinga, et al., 1997; Núñez & Martínez, 1999). This may limit the operation to

OLRs of 4–6 kg COD/$m^3$/day (Torkian, et al, 2003); however, reactors have operated on much higher OLRs, as will be discussed in the next section. TCOD and TSS values in this type of wastewater are seen to be in the range of 1.5 – 20.4 g/L and 0.1 – 4.7 g/L respectively. Strength and characteristics of the wastewater have been seen to vary largely depending upon plant size, time of the year, operation process, and nature and extent of activity. Typical characteristics of wastewater from slaughterhouses and meat processing plants are given in Table 2.4.

**Table 2-4: Characteristics of meat processing wastewater**

| Feed type | pH | $BOD_5$ (mg/L) | TCOD (mg/L) | SCOD (mg/L) | TSS (g/L) | VSS (g/L) | Phosphorus, $PO_4^{3-}$ (mg/L) | Nitrogen (mg/L) | Alkalinity (mg/L as $CaCO_3$) | Reference |
|---|---|---|---|---|---|---|---|---|---|---|
| Slaughterhouse wastewater | 6.8-7.1 | 490-650 | 1500-2200 | 50-100 | 0.7-2.1 | - | 12-20 | 120-180 (Kjeldahl) | - | Sayed, et al., 1987 |
| Slaughterhouse wastewater | 6.7 | 3120 | 5050 | - | 0.1 | 0.07 | 30 | 310 (Kjeldahl) | 410 | Borja, et al., 1995 |
| Slaughterhouse wastewater | 6.8-7.8 | - | 5200-11400 | 12-33% of TCOD | 0.6-1.7 | - | 8-28 | 19-74 (Ammoniacal) | - | Ruiz, et al., 1997 |
| Slaughterhouse wastewater | 6.8 | 1400 | 2500 | 1500 | 0.53 | - | - | - | 740 | Núñez & Martínez, 1999 |
| Slaughterhouse wastewater | 5.3–6.8 | 2200–9800 | 5800–20150 | - | 2.4–4.7 | - | - | 102–323 (Ammoniacal) | - | Fuchs, et al., 2003 |
| Slaughterhouse wastewater | 6.8-7.8 | 910-1920 | 3270–14290 | 2260–4960 | - | - | 7–26 | 35–104 (Ammoniacal) | 1200-1700 | Torkian, et al., 2003 |
| Slaughterhouse waste | - | - | 300000-530000 | - | - | - | - | 19500 (Tot. organic) | - | Siegrist, et al., 2005 |
| Slaughterhouse wastewater | 7.5-7.7 | 3500-8030 | 7100-20400 | 5400-15500 | - | - | - | - | - | Saddoud & Sayadi, 2007 |
| Slaughterhouse wastewater | 7.2 | 2646 | 3437 | 2589 | 1.2 | 1.0 | 17 | 131 (Ammoniacal) | 658 | Nacheva, et al., 2011 |
| Synthetic slaughterhouse wastewater | 5.8-7.9 | 630-650 | - | - | - | - | - | 63-254 (Total N) | - | Bustillo-Lecompte, et al., 2013 |

- = Not reported

*2.5.3.3 Performance evaluation of meat processing wastewater treatment*

Slaughterhouse and meat processing wastewater has been treated by both aerobic and anaerobic treatment methods. A common anaerobic method applied in the early stages was the anaerobic lagoon; however, in addition to the requirement of large area, it had the disadvantage of odour generation from the ponds (Rajeshwari, et al., 2000), and high fat and suspended solids in the effluent (Martínez, et al., 1995), necessitating the development of alternate options. Anaerobic filter, anaerobic fixed bed reactor, anaerobic fluidized bed reactor and UASB are the other treatment methods that were tried under several other studies with variable results. Three examples of treatment of this type of wastewater were found, where membrane filtration was coupled with anaerobic treatment.

An anaerobic fluidized bed reactor tested in laboratory by Borja, et al. (1995) achieved more than 94% COD reduction for an OLR up to 27 kg COD/$m^3$/day. Although the volumetric methane production went up with the increase of OLR from 2.9 to 54 kg COD/$m^3$/day, the methane content in biogas reduced from 78 to 59%. This was attributed to the inhibition of methanogenic bacteria by the increase of VFAs due to the higher OLR. It is the same reason why they had to maintain a high alkalinity (2500 mg/L as $CaCO_3$) at OLRs over 30 kg COD/$m^3$/day. At higher HRTs, the reactor performance was independent of the feed COD concentration. Ruiz, et al. (1997) attained a COD removal up to 93% with a UASB reactor at an OLR of 2.2 kg COD/$m^3$/day, and with the increase of OLR to 6.5 kg COD/$m^3$/day, the removal efficiency declined to 59%. The performance was lower for an anaerobic filter (AF), with a maximum COD removal of 83% at an OLR of 2 kg COD/$m^3$/day. Both the reactors showed good performance for OLR below 5 kg COD/$m^3$/day, and for similar OLR the UASB showed higher removal efficiency than the AF. Núñez & Martínez (1999) attained a maximum COD removal of 80% with an EGSB; however, the average COD removal was around 70% for OLRs ranging from 3 to 15 kg COD/$m^3$/day, and HRTs ranging from

19 to 5.2 hours, indicating that the treatment of slaughterhouse wastewater by EGSB may be a feasible option, but not the best one. The COD removal efficiency in this experiment was dependent on HRT, and was not significantly affected by varying OLRs for a given HRT. Application of an external module AnMBR proved effective in treating acidified slaughterhouse wastewater with an average COD removal of 93% at OLRs of 4.4 to 13.3 kg COD/$m^3$/day (Saddoud & Sayadi, 2007). The performance was significantly hindered with further raise of the OLR due to VFA accumulation. This problem was greatly minimized by the integration of a pre-acidogenesis step using a fixed bed reactor (FBR).

Nacheva, et al. (2011) showed that UASB can achieve high COD removal (90%) and modest methane yield (0.27 L $CH_4$/g $COD_{rem}$) at a reasonably high OLR (15 kg COD/$m^3$/day) while operating at ambient temperature, though the removal of nutrients were not significant. A recent study treating synthetic wastewater by Bustillo-Lecompte, et al. (2013) proved that combination of an anaerobic (baffled) and an aerobic (activated sludge) reactor provides excellent results in terms of removing total organic carbon (TOC), total nitrogen (TN) and carbonaceous BOD ($CBOD_5$), and an advanced oxidation ($UV/H_2O_2$) process as a post treatment step assisted to further polish the effluent quality. The combined anaerobic-aerobic-AOP process achieved up to 99.9% TOC, 82.8% TN, and 99.6% $CBOD_5$ removals from an influent concentration of 1,005 mg TOC/L and 200 mg TN/L at the HRT of 4 days and a flow-rate of 5.9 mL/min. Increasing the HRT increased the removal of TOC and TN in this study. Table 2.5 summarizes the operational conditions and performance data of studies described above along with some other studies that treated slaughterhouse or meat processing wastewater. The operating conditions (e.g. HRT, OLR) are shown as either the range or the optimum value attained in a study, and the performance parameters (percent COD removal, $CH_4$ yield) are shown as either the range or the maximum value; these conditions and values may not necessarily correspond to each other.

**Table 2-5: Operating conditions and treatment results for meat processing wastewater**

| Parameter | Sayed, et al., 1987 | | Borja, et al., 1995 | Ruiz, et al., 1997 | | Núñez & Martínez, 1999 | Torkian, et al., 2003 | Fuchs, et al., 2003 | Siegrist, et al., 2005 | Saddoud & Sayadi, 2007 | Nacheva, et al., 2011 | Bustillo-Lecompte, et al., 2013 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Reactor type | UASB | | Anaerobic fluidized bed | UASB | Anaerobic filter | EGSB | UASB | AnMBR | An. digester with UF membrane & air stripping | AnMBR + FBR | UASB | Anaerobic baffled + Aerobic AS + UV/$H_2O_2$ |
| Working volume | 33 L | 33 L | 1 L | 2L | 2 L | 2.7 L | 1000 L | 7 L | 17 L | 50 L | 15 L | 33.7 L |
| pH | - | - | 6.8-7.2 | 7.5-8.0 | 7.5-8.0 | 7.7 | - | - | 8-8.2 | 7.8 | 7.5-7.7 | 6.2 (effluent) |
| Temp | 30 $^0$C | 20 $^0$C | 35 $^0$C | 37 $^0$C | 37 $^0$C | 35 $^0$C | 33 $^0$C | 30 $^0$C | 37 $^0$C | 37 $^0$C | 21-25 $^0$C | 26 $^0$C |
| SRT | - | | - | - | - | - | 60.3-3.3 d | - | 40-30 d | - | 166-100 d | - |
| HRT | 9-1.7 h | 10-5 h | 8-0.5 h | 6.5-1.2 d | 0.5-7.1 d | 0.2 d | 7.1-2.3 h | 1.2 d | 35 d | 3.3-1.3 d | 0.9-0.3 d | 4-3 d |
| OLR | 11 Kg COD/$m^3$/day | 7 Kg COD/$m^3$/day | 2.9-54.0 Kg COD/$m^3$/day | 1-6.5 Kg COD/$m^3$/day | 0.9-11.2 Kg COD/$m^3$/day | 15 Kg COD/$m^3$/day | 13-39.5 Kg COD/$m^3$/day | 1-8 Kg COD/$m^3$/day | 5-17 Kg COD/$m^3$/day | 4.4-13.3 Kg COD/$m^3$/day | 4-15 Kg COD/$m^3$/day | 0.03-1.01 Kg TOC/$m^3$/day |
| Max COD removal | 87% | 91% | 98.9% | 90% | 83% | 80% | 83% | 97% | >90% | 98.8% | 90% | 99.6% ($CBOD_5$) |
| $CH_4$ yield | 5.2 kg $CH_4$-COD/$m^3$/day | 3.2 kg $CH_4$-COD/$m^3$/day | 0.32 L $CH_4$/g $COD_{rem}$ | 1.3 $m^3/m^3$/d | 1.1 $m^3/m^3$/d | - | 0.28 L $CH_4$/g $SCOD_{rem}$ | 0.25 L CH4/g $COD_{rem}$ | - | 0.33 L $CH_4$/g $COD_{rem}$ | 0.27 L $CH_4$/g $COD_{rem}$ | - |

- = Not reported

The configuration and performance of membrane from the studies that used membrane filtration (AnMBR) are given in Table 2.6, which demonstrates the differences in membrane configuration (submerged vs. cross flow) and specifications (pore size and surface area) between this study and other studies. From the comparisons (Table 2-5 and 2-6) it can be seen that in each of the three studies where AnMBR was used, the reactor was operated under controlled mesophilic temperature (30 – 37 $^{o}$C) and the membrane was installed in an external cross-flow configuration.

**Table 2-6: Configuration and performance of membrane in membrane coupled reactors treating slaughterhouse/meat processing wastewater**

| Parameter | Fuchs, et al., 2003 | Siegriest, et al., 2005 | Saddoud & Sayadi, 2007 | This study |
|---|---|---|---|---|
| Membrane type | MF | UF | UF | UF |
| Configuration | Cross-flow | Cross-flow | Cross-flow | Submerged |
| Pore size (µm) | 0.2 | 0.06-3 | 100 (kDa) | 0.04 |
| Surface area ($m^2$) | 0.126 | - | 1 | 0.046 |
| Cross-flow velocity (m/s) | 2-3 | - | 3 | NA |
| Gas sparging rate (L/min) | - | - | - | 1.5 |
| TMP (kPa) | - | - | 100 | 1 |
| Flux ($L/m^2/h$) | 5-10 | 40-100 | 2-8 | 1.1-6.4 |

### *2.5.4 Treatment of other waste streams*

Other waste streams treated by AnMBRs include high solids waste (e.g. wastewater treatment plant sludge, municipal solid waste and animal manure) and leachates. As hydrolysis (solubilisation) in the anaerobic degradation of organic solids is slow, longer SRT (20-70.5 days) and HRT (1.5-11.8 days) than the municipal or food industry wastewater were applied in several studies with a reported maximum OLR of 10 kg COD/$m^3$/day and COD removal of more than 90%. While treating landfill leachate and municipal solid waste leachate, AnBMRs have achieved COD removal of around 90% with OLRs generally over 2.5 kg COD/$m^3$/day (Lin, et al., 2013).

### *2.6 Challenges and future potentials of AnMBR*

Although the AnMBR has demonstrated efficient performance in treating various types of wastewater and its commercialization has seen a boost in the last few decades, challenges still exist in its more widespread application as seen for the aerobic MBR, especially in the large scale industrial sector. At the same time, there are further potentials where use of AnMBR can be practical.

Two important obstacles in the adoption and commercialization of AnMBR in industrial sector can be mentioned as membrane fouling and membrane sensitivity to toxicity. Apparently, membrane fouling is more prevalent in AnMBR than it is in aerobic MBRs; hence the former is operated at lower membrane fluxes. As cake formation on the membrane surface was found to be the key parameter for membrane fouling and flux control, it is imperative that ways to slow down cake formation be investigated, as lower membrane fluxes will render AnMBR uneconomical. As membrane foulants have already been identified, techniques should be applied to minimize their growth in the mixed liquor. Avoiding toxic shocks, pH shocks, careful selection of SRT/HRT and temperature, proper choice of membrane material and application of moderate but feasible membrane flux should confer a stable and long term AnMBR performance (Skouteris, et al., 2012).

An important issue in the use of membrane is the capital and operational cost (attributed to the high biogas flows required for scouring in submerged AnMBRs and high cross-flow velocity in side-stream AnMBRs) associated with membrane. Since a high rate of COD removal may not always be required, membranes can be substituted by low-cost filters (e.g. non-woven membranes, meshes or filter cloths) as the latter can obtain high fluxes even at low pressure because of larger pore size. Although low-cost membranes, like the non-woven ones provided satisfactory results in pilot-scale and full-scale applications, they have their own limitations of having lower tensile strength, lower resistivity to microbiological attacks and severe fouling due to their rough surface and too large

pore size. However, these limitations can be overcome by pre-coating of the membrane surface and pores or by membrane material modification, which can also be applied to conventional MF or UF membranes (Meng, et al., 2009). Need of high energy for scouring/cross-flow can be minimized by using PAC that helps in scouring the membrane surface (Akram & Stuckey, 2008). Therefore, improving membrane performance and reduction of overall cost of membrane usage (membrane price reduction, optimized biogas sparging) should be some of the prime interests to ensure continuing expansion of full-scale AnMBR operation. Optimization of other membrane operational parameters (backwashing/cleaning frequency, use of chemicals) also needs to be addressed.

AnMBRs are more prone to inorganic fouling by the precipitates of calcium, phosphorus and sulphur (struvite is the dominant inorganic foulant reported so far) due to their presence in high concentration in industrial wastewaters. Inorganic species can also interact with SMP and enhance stability of the fouling layer in a reactor. Better understanding of inorganic fouling and their mitigation is therefore important; pre-treatment or modification of influent sludge can be further investigated in this regard. Fine and colloidal particles significantly affect filterability in membrane process. Effectiveness of additives/sorbents (e.g. PAC) and/or coagulant has been proven in several studies to help improve flux (Dereli, et al., 2012). Further research is needed to determine their optimum dosage, effects on improvement of filterability characteristics, long term fate and regeneration process (Stuckey, 2012).

More research should be pursued towards treatment of wastewater types that are mentioned to be difficult to treat by AnMBR, especially high-strength soluble wastewaters (Liao, et al., 2006; Lin, et al., 2013), though there are some studies that reported successful treatment of such wastewater. The fact that AnMBR offers complete solids retention poses good opportunity for treating high strength low solids wastewater (Stuckey, 2012). Slaughterhouse wastewater and landfill leachates are some of the other types of wastewater where past research and understanding of AnMBR treatment is

limited compared to some others, and more potentials exist for future studies. Membrane sludge digesters appear to have great potential, and research is needed to determine the appropriate membrane configuration and optimum reactor design for minimum contact of the solids to the membrane. Although treatment of low-strength wastewater (e.g. MWW) showed promising results, treating at low to moderate temperature and combining membrane with HRARs already suitable for dilute wastewaters require more work. Integration of membrane and HRARs is important for future research due to another interesting fact. Optimized configuration of the membrane with the biomass retaining reactor can ensure that the membrane does not conduct all of the solid/liquid separation and hence fouling can be reduced. For applications of external cross-flow membranes, the mystery of potentially lower biomass activity due to pumping shear stress needs to be investigated further. Biomass activity can be assessed using phylogenetic analytical techniques in addition to the traditional activity assays (Liao, et al., 2006; Lin, et al., 2013).

A recent finding is that influencing fundamental processes in catabolism, i.e., inhibiting quorum sensing (cell-cell signalling) may be a new way of reducing fouling, as this process is supposed to reduce SMP excretion. Another parameter influencing SMP production that has not been investigated much is the level of VSS in the reactor. Although conventional knowledge on anaerobic digestion says that it should be quite high (20-40 g/L), high loads have been treated with VSS concentration as low as 2-3 g/L. As higher VSS concentration leads to higher fouling and higher COD (SMP) in the effluent, option of treating with low VSS should be explored in more detail (Stuckey, 2012).

Past work has shown that it is possible to operate AnMBR at a low HRT of 3 hours and at a high SRT of several hundred days. Low HRT leads to a small footprint while high SRT is desirable for longer biomass retention leading to lower sludge yield and higher COD removal. However, longer SRT comes with the problem of higher biomass associated products- BAP or SMP which may be

hard to degrade, and hence increase in effluent COD. Therefore, the question of what will be the "optimum" SRT that minimizes sludge production and maximizes COD removal arises.

A perennial challenge for AnMBRs has been in treating nutrient rich wastewater. Thankfully, removal of nitrogen has seen satisfactory success under conventional and advanced anaerobic-aerobic processes (e.g. Anammox, autotrophic denitrification using hydrogen gas/elemental sulphur as electron donor, etc.). However, research needs to continue for further enhancement of their applicability and efficiency (Stuckey, 2012). Presence of EDCs in landfill leachate and MWW in recent years has been a great concern. Promising result was seen in an MBR system combined with post-treatment steps like nano-filtration and activated carbon adsorption compared to reverse osmosis alone (Yang, et al., 2006). It is believed that AnMBRs can also provide a suitable environment for EDC biodegradation due to complete biomass retention and maintenance of a more diverse microbial culture. There is also great opportunity for investigating post-treatment options for the type of wastewater that produce higher-than-acceptable quality of effluent. One of the major recent finds is the presence of dissolved methane, a greenhouse gas, in AnMBR permeates at high concentrations. As accumulation of dissolved methane is inevitable in AnMBRs, minimization of its escape in dissolved form by manipulating operating conditions and effective capture processes of the same from the permeate provide potential future research areas.

This study particularly aims at evaluating the performance of an AnMBR maintained at ambient temperature with a submerged hollow fibre membrane operated under low pressure. Assessments regarding impact on treatment performance, biogas production and membrane performance were conducted. An additional goal was to determine what could be the ideal start-up and operational parameters (viz. SRT, HRT, membrane flux) that can be replicated in a larger or pilot scale reactor treating similar wastewater.

# *Chapter 3*

# *Materials and Methods*

# 3 MATERIALS AND METHODS

## *3.1 AnMBR set-up*

This study used a lab-scale cylindrical AnMBR with a total volume of 5.75 L (inner diameter 10.3 cm and height 69.0 cm) and a working volume of 5 L. The reactor body was made of polyvinylchloride resting on a steel stand. It had several ports on the side for connection of a feed line, a bulk sludge recirculation line and required sensors. The four openings on the top were used for biogas recirculation (in and out), permeate production and a pressure gauge. A hollow-fibre ultrafiltration membrane module was immersed inside the anaerobic reactor to achieve the solid-liquid separation. Both the reactor and the membrane module were supplied by GE Water and Process Technologies, Canada. Characteristic features of the membrane used in this study are shown in Table 3.1.

**Table 3-1: Membrane characteristics**

| Parameter | Specification |
|---|---|
| Module name | ZeeWeed ® 500D |
| Material | PVDF |
| Pore size | 0.04 µm |
| Hydrophobicity | Hydrophilic |
| Surface area | 0.046 $m^2$ |
| Fibre diameter | 1.9 mm (outer) / 0.8 mm (inner) |
| Flow direction | Outside in |
| Fibre orientation | Vertical |
| Number of module | 01 (One) |
| Max operating temp. | 40 $^0$C |
| Max cleaning temp. | 40 $^0$C |
| Operating pH range | 5.0 – 9.5 |
| Cleaning pH range | 2.0 – 10.5 |

A pH probe and a temperature sensor were inserted in the reactor and were connected to a controller. Pressure gauges were installed to monitor pressure in the reactor and the permeate line in order to measure the transmembrane pressure (TMP). Data from the pressure devices were logged by a data acquisition system (DAQ, National Instruments, USA) using LabView 2012 software. Two digital peristaltic pumps with time control were set up for pumping feed and permeate, and another peristaltic pump for biogas and liquid circulation. The picture and schematic diagram of the reactor set-up are shown in Figure 3.1.

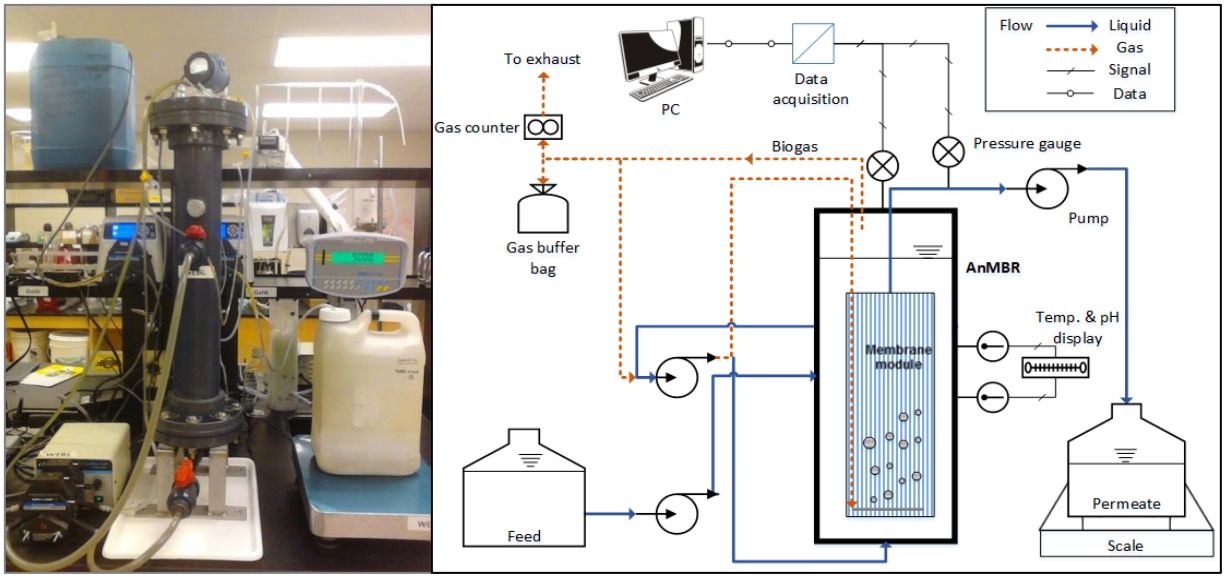


**Figure 3-1: Picture (left) and schematic diagram (right) of AnMBR set-up**

### *3.2 Feed and inoculum*

The reactor utilized wastewater from Conestoga Meat Packers, Woolwich, ON as the substrate. Sludge from an anaerobic digester (SRT=15 days) at Galt Wastewater Treatment Plant (WWTP), Cambridge, ON was used as the inoculum during the start-up. Table 3.2 shows the characteristics of the feed and the inoculum during the initiation. The average and standard deviation of the measurements were calculated from three samples.

**Table 3-2: Initial sludge and feed characteristics**

| Parameter | Seed sludge Avg±StD | Feed wastewater Avg±StD |
|---|---|---|
| TSS, mg/L | 15500±1080 | 1640±98 |
| VSS, mg/L | 11500±816 | 1460±59 |
| TCOD, mg/L | 31360±1134 | 4398±305 |
| SCOD, mg/L | 1686±116 | 651±29 |
| pH | 6.9 | 6.6 |

More characteristics of the feed wastewater are presented in Table 4.1. The feed wastewater was collected in pails with lids from the meat processing plant weekly and stored in a walk-in fridge ($4^0$C). The feed was naturally warmed up to room temperature and screened (1 mm metallic mesh) prior to refilling the feed container to remove larger suspended materials, especially the hairy particles which can be detrimental to the membrane.

### *3.3 Operating conditions*

The reactor was operated at ambient temperature and neutral pH. The SRT was kept constant at 50 days during all the three phases. Three different HRTs of 5, 2, and 1 day were evaluated which corresponded to average OLRs of 0.4, 1.3, and 3.1 kg COD/$m^3$/d, respectively. Both OLR and HRT were changed simultaneously by increasing feed inflow to the reactor. Table 3.3 summarizes the operating conditions of the three phases of operation.

Feed and permeate were pumped with two peristaltic pumps at synchronized operation mode. The pumps were operated at 7 minutes on (production) and 3 minutes off (relaxation) per cycle,

pumping at a rate of 0.9, 2.4 and 4.9 ml/minute to maintain the required permeate flux of 1.17, 3.13 and 6.4 LMH respectively. The intermittent permeation approach is part of the strategy to control membrane fouling by reducing stress on the membrane. The same approach is followed by the membrane manufacturer (i.e. GE) in their pilot and full scale systems. The biogas and the bulk liquid inside the reactor were circulated at a rate of 1 L/min for mixing and creating a shear flow to minimize cake formation on the membrane surface, thereby assisting in the reduction of membrane fouling.

**Table 3-3: Operating conditions of the AnMBR**

| Parameter | Unit | Phase I | Phase II | Phase III |
|---|---|---|---|---|
| Period of operation | d | 0-75 | 76-210 | 211-264 |
| HRT | d | 5 | 2 | 1 |
| SRT | d | 50 | 50 | 50 |
| Feed flow rate | L/d | 1.0 | 2.5 | 5.0 |
| Permeate flow rate | L/d | 0.9 | 2.4 | 4.9 |
| Sludge wastage | L/d | 0.1 | 0.1 | 0.1 |
| Flux | LMH | 1.17 | 3.13 | 6.4 |

### *3.4 Data measurement and analysis*

Data from daily operation were logged and results from routine analytical experiments were recorded during all three cycles of operation of the AnMBR. The temperature, pressure, pH, volume of permeate and biogas production were recorded daily. Feed wastewater, waste sludge, permeate, and biogas samples were routinely collected to perform a variety of analysis according to the plan given in Table 3.4.

**Table 3-4: Routine analysis and measurement plan**

| Measurement | Sample | Frequency |
|---|---|---|
| Temperature | Reactor | Daily |
| pH | Reactor | Daily |
| | Permeate | |
| Biogas production | Biogas | Daily |
| MLSS/MLVSS and TSS/VSS | Reactor | Twice per week during initial condition<br>Once per week during stable condition |
| | Feed | |
| TCOD/SCOD | Feed | Twice per week during initial condition<br>Once per week during stable condition |
| | Reactor | |
| | Permeate | |
| Biogas composition | Biogas | Twice per week during initial condition<br>Once per week during stable condition |
| Dissolved methane in permeate | Permeate | Once a month |
| Alkalinity and VFAs | Reactor | Once a month |
| Turbidity | Permeate | Twice a month<br>(or as and when required)* |

**Increased monitoring when particles sighted in permeate, and until permeate is clear again*

## *3.5 Analytical Methods*

The samples were measured in duplicates unless otherwise specified, and standards and blank samples were prepared for all chemical/biochemical tests.

### *3.5.1 Water quality analysis*

Temperature and pH of the reactor were measured by probes connected to a controller with a display and recorded daily. The pH of permeate was measured with a bench-top pH meter (710A, Orion, USA). The volume of permeate produced was measured using a digital scale (resolution 0.5g) (Adam GBK 35a, UK) and converting weight to volume. MLSS/MLVSS and TSS/VSS were measured following method 2540 D and 2540 E of the Standard Methods (APHA, AWWA, & WEF, 1992) using 10 mL of sample filtered with a 1.5 µm pore sized glass filter (Whatman™, 934-AH™, Glass Microfiber filters, GE Healthcare, UK).

Alkalinity was measured using method 2320 B of the Standard Methods (APHA, AWWA, & WEF, 1992) through potentiometric titration to a pH of 4.3. The TCOD and SCOD were measured colorimetrically following method 5220 D of the Standard Methods (APHA, AWWA, & WEF, 1992). Samples were digested in a preheated HACH COD reactor for 2 hours and absorbance measurements were taken using a UV spectrophotometer (DR/ 2000, HACH Company, USA) at a wavelength of 600 nm. A new calibration curve using at least 5 standards was made whenever new reagents had to be prepared. SCOD was measured after filtering the samples through 0.45 µm.

TCOD removal efficiency ($R_t$) and secondary COD removal efficiency ($R_s$) were calculated based on the influent and effluent, influent and supernatant of mixed liquor (SML) using Equations 3.1 and 3.2, respectively:

$$R_t = \frac{COD_{inf} - COD_{eff}}{COD_{inf}} \times 100\% \quad (3.1)$$

$$R_s = \frac{COD_{inf} - COD_{SML}}{COD_{inf}} \times 100\% \quad (3.2)$$

where $COD_{inf}$, $COD_{eff}$ and $COD_{SML}$ are the TCOD of the influent, TCOD of the effluent (membrane permeate) and filtered (0.45 µm) supernatant of mixed liquor (SML), respectively.

The volatile fatty acids (VFAs) were determined by titrimetric method with a three-point calibration after Kapp (1984) and using a modified Kapp equation (Buchauer, 1998). A pH meter and 0.1N $H_2SO_4$ solution was used to measure the total VFA following the steps summarized below.

- Before analysis the sample was filtered through a 0.45 µm syringe filter
- Filtered sample was put into a small beaker at a volume to guarantee that the tip of the pH electrode was always immersed below the liquid surface
- Initial pH was recorded

- The sample was titrated slowly with 0.1 N sulphuric acid until pH 5.0 was reached. The added volume of the titrant was recorded.
- More acid was slowly added until pH 4.3 was reached. The total volume of the added titrant was again recorded
- The latter step was repeated until pH 4.0 is reached, and the volume of added titrant recorded once more
- A constant mixing of sample and added titrant was ensured to minimise exchange of $CO_2$ with the atmosphere during titration using a magnetic stirrer.

Total VFAs was then calculated using the following formula:

$$VFA, mg/L = 131340 \times N \times \frac{V_A}{V_S} - 3.08 \times Alk - 25 \quad (3.3)$$

where, N= normality of acid; $V_A$= volume of acid consumed to titrate sample from pH 5.0 to 4.0 in mL; $V_S$= volume of sample in mL and Alk= alkalinity of sample in mmol/L.

### *3.5.2 Biogas analysis*

The daily biogas production was measured using a gas counter (resolution 3 ml) (MilliGascounter, Ritter Apparatebau, Germany). Biogas was sampled with a gas-tight syringe (Hamilton Gastight Syringe, 1.0 mL, USA) and its composition (methane, carbon dioxide and nitrogen) was analyzed by a gas chromatograph equipped with a thermal conductivity detector (GC-TCD) (SRI GC 310C, USA). Calibration curves for each of these gases were prepared using standard gases. The GC-TCD was installed with a packed column (PorapakQ, 6 ft x 1/8 inches, 80/100 mesh, Agilent Tech., USA) and helium (99.999 %, PraxAir, Canada) was used as the carrier gas with a flow rate of 10 ml/L under a pressure of 21 psi. The column oven temperature and detector temperature were $41^0$C and $200^0$C, respectively. Figure 3.2 shows the GC-TCD used for biogas composition analysis.

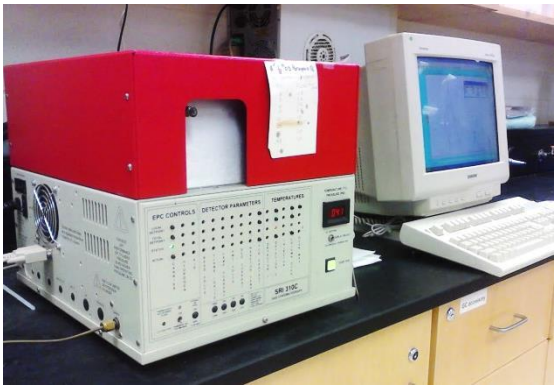

**Figure 3-2: GC-TCD used for the biogas composition analysis**

### *3.5.3 Dissolved methane calculation*

Dissolved methane in the permeate was measured using Henry's constant and following a methodology modified from Kampbell and Vandergrift (1998). Ten ml permeate was collected on-line using a three way valve and a syringe. The permeate was then injected into a 20 ml glass vial that was already sealed with a butyl rubber stopper and was purged with $CO_2$. The vial was then shaken by a vortex mixer for 6 to 10 minutes allowing the dissolved methane in the liquid phase to transfer to the gas phase and equalize. The test was performed at room temperature ($25^0$C), and atmospheric pressure was maintained while transferring permeate into the vial by releasing pressure through a separate line connected to a water filled jar. The gas in the headspace was then collected with a gas-tight syringe (Hamilton Gastight Syringe, 1.0 mL, USA) and analysed by GC-TCD (SRI GC 310C, USA). Dissolved methane was calculated using the following equation (Yeo & Lee, 2013):

$$CH_4(aq) = \left(C_{CH_4} \times P \times K_{CH_4} \times MW_{CH_4} \times \frac{1000mg}{1g}\right) + \left(C_{CH_4} \times V_{head} \times MW_{CH_4} \times \frac{1000mg}{1g} \times T_0\right) \times \frac{1}{V_{head} \times (22.4L/1mol) \times (1000mL/1L) \times T_1} \quad (3.4)$$

where $CH_4(aq)$ = concentration of dissolved methane in AnMBR permeate (mg/L), $C_{CH4}$ = methane percentage in headspace of vial, P = pressure (1 atm), $K_{CH4}$ = Henry's law constant at $25^0$C (0.0016

mol/L-atm), $MW_{CH4}$ = molecular weight of methane (16 g/mol), $V_{head}$ = volume of headspace in vial (10 ml), $T_0$ = 273.15K and $T_1$ = 298.15K.

The saturation concentrations of dissolved methane in the AnMBR during the three Phases were computed with the help of Eq. 3.5 and using Henry's constant:

$$CH_4(sat) = \left(C_{CH_4} \times P \times K_{CH_4} \times MW_{CH_4} \times \frac{1000mg}{1g}\right) \quad (3.5)$$

where $CH_4$(sat) = saturation concentration of dissolved methane in AnMBR permeate (mg/L), $C_{CH4}$ = methane percentage in headspace of reactor, P = pressure (atm), $K_{CH4}$ = Henry's law constant at $25^0$C (0.0016 mol/L-atm) and $MW_{CH4}$ = molecular weight of methane (16 g/mol). The pressure in the reactor was 1.010±0.003 atm throughout the operational period.

### *3.5.4 Extraction and characterization of EPS and SMP*

Concentrations of EPS and SMP in the microorganisms of the reactor were measured twice in each cycle to assess their variation with different organic loading rates. A separate experiment was conducted by the author to determine a suitable method to measure EPS and SMP.

SMP is usually extracted by centrifugation alone and quantified by further chemical analysis (Aquino & Stuckey, 2002). Here, Protein and carbohydrates were measured to quantify the SMP following the processes mentioned later in this section.

Out of several reported physical and chemical processes, EPS extraction was examined by one physical process (Ultrasonication) and two chemical processes (EDTA and NaOH+Formaldehyde) due to high extraction yields reported by these methods in the physical and chemical category of tests. Up to four iterations of extraction were applied to determine the protocol that provides optimal extraction yield without cell lysis, as opposed to the conventional practice which uses one

iteration only. Considering the EPS yield, complexity of test procedure and risk of cell rupture, EDTA method with two iterations was considered the most suitable method.

EPS and SMP were characterized by measuring protein and carbohydrate contents present, and expressed through normalization with the microbial content, i.e. VSS of the sludge (mg / g VSS). Proteins were measured using the Pierce BCA test kit (Pierce BCA Protein Assay, Thermo Scientific, USA) with bovine serum albumin as the standard, and carbohydrates were measured using phenol-sulphuric acid method (modified from Dubois et al., 1956) with glucose as the standard.

### *3.6 COD (Electron) balance in the AnMBR*

The calculations of COD balances were performed considering the total incoming and outgoing COD (electron) using Eq. 3.6; all the incoming and outgoing parameters contributing towards COD were calculated or converted to equivalent COD in mg of COD per day:

$$Total\ COD\ In, mg/d - Total\ COD\ Out, mg/d = \Delta COD, mg/d \quad (3.6)$$

where, Total COD In= COD in the feed, Total COD Out= COD in effluent liquid (permeate+wasted sludge) + equivalent COD in methane dissolved in liquid (permeate+wasted sludge) + equivalent COD in methane gas produced + COD used up for suspended biomass growth, and ΔCOD= the difference of COD concentration between incoming and outgoing CODs.

The equivalent COD of methane gas produced was computed from the half reaction of methane to carbon di oxide (1 mol $CH_4$= 64 g COD) and using the daily production volume (1 mol $CH_4$ = 22.4 L $CH_4$), with necessary temperature correction. The dissolved methane concentration in mg/L was converted to mg COD/L (1 mol $CH_4$ = 64 g COD and 1 mol $CH_4$ = 16 g $CH_4$) and multiplied by the total liquid coming out (permeate and wasted sludge) in L/day. MLVSS is a common parameter used for the estimation of biomass concentration in a biological sludge. Taking the empirical

formula of microorganisms as $C_5H_7O_2N$ (Rittmann & McCarty, 2012), a relation can be calculated by means of the following oxidation reaction reflecting the endogenous respiration of biomass:

$$C_5H_7O_2N + 5O_2 \rightarrow 5CO_2 + NH_3 + 6H_2O \quad (3.7)$$

From Eq. 3.7 we have 113 g biomass MLVSS ($C_5H_7O_2N$ = 113 g) to be equivalent to 160 g COD ($5O_2$ = 160 g).

In a perfectly steady state condition where all the incoming COD is fully utilized and all the sinks of electrons are accounted for, the ΔCOD would be equal to zero. In practical scenario, however, a positive ΔCOD value can be expected. This may be the reflection of non-biodegradable or unutilized portion of incoming COD being retained in the reactor or COD being accumulated due to the growth of attached biomass in the reactor.

### *3.7 Membrane performance and membrane maintenance cleaning*

Performance of the membrane was monitored and calculated in terms of flux, TMP, permeability and fouling index using methods discussed in section 2.4.2. Period of steady flux without any cleaning was not determined in this study due to the risk of enhancing membrane fouling potential. Regular maintenance cleaning was performed during the study according to the manufacturer's practice. Over the operational period of membrane it is inevitable that the TMP will increase (or flux will decrease) at a specific pumping rate. Chemical cleaning of the membrane is commonly implemented to help restore the membrane towards its best practical TMP and flux performance, though "irremovable" fouling will build-up with time, and a recovery cleaning may be required if the TMP exceeds the manufacturer recommended limit of 30 kPa.

Maintenance cleaning of the membrane was performed once every week with citric acid solution (2000 mg/L) followed by sodium hypochlorite solution (200 mg/L). Each cleaning solution was pumped in reverse direction (back pulse) through the membrane for four cycles. Each cycle

comprised of pumping the solution at a flux of 30 LMH for 40 seconds and then relaxing for 3 minutes. Two cycles of freshwater was pumped at the same flux and timing to clear up the residue chemical in the tubing in between pumping of the two chemicals (to avoid reaction between them), and at the end of pumping the second chemical. So the sequence of pumping was citric acid then freshwater then sodium hypochlorite then freshwater. No noticeable adverse impact was observed on treatment performance and biogas production after the cleaning procedures.

# *Chapter 4*

## *Performance of the AnMBR Treating Meat Processing Wastewater*

# 4 PERFORMANCE OF THE ANMBR TREATING MEAT PROCESSING WASTEWATER

The overall efficiency of treatment by an AnMBR depends on many factors, including feed wastewater characteristics, operational environment, operating parameters and membrane behaviour. With the overall objective to ascertain whether an AnMBR with submerged, vacuum-driven membrane is applicable to treat high-strength, high-solids wastewater and to what extent, the study tried to focus on the following major issues from the experimental results:

- Determining the treatment performance of the AnMBR in terms of COD removal efficiency at different OLRs
- Determining the biogas and methane production
- Assessing the performance of the membrane
- Optimizing operational conditions

This chapter will discuss on the results of experiments and evaluate the performance of the system with regards to these specific areas. All the physical, chemical/biochemical and operational data, and a summary of the process performance parameters are presented in the Appendix.

## *4.1 Feed wastewater characteristics*

Real meat processing wastewater (Conestoga Meat Packers, Woolwich, ON) was chosen to represent an industrial food wastewater and to have better understanding of real-life scenario in terms of treatment and operational conditions. The raw wastewater was collected from the outlet of the processing plant just before entering the equalization tank of the plant's existing wastewater treatment facility. No pre-treatment was performed, except for screening the wastewater by 1 mm mesh before feeding into the AnMBR. Table 4.1 presents the characteristics of feed wastewater, with their average values, standard deviation and number of samples tested during the three phases.

**Table 4-1: Characteristics of the feed**

| Phase | pH | TCOD, mg/L | SCOD, mg/L | TSS, mg/L | VSS, mg/L | $PO_4^{3-}P$, mg/L | $NH_3$-N, mg/L | Alkalinity, mg/L as $CaCO_3$ |
|---|---|---|---|---|---|---|---|---|
| **I** | 6.8±0.1*(7)* | 2254±1074*(23)* | 681±297*(23)* | 869±463*(24)* | 783±414*(24)* | 87.3±12.6*(3)* | 51.0±18.9*(3)* | 760±225*(8)* |
| **II** | 6.7±0.1*(5)* | 2804±723*(16)* | 806±165*(16)* | 1405±505*(17)* | 1254±450*(17)* | 103.8±32.6*(3)* | 99.1±56.4*(3)* | 748±99*(3)* |
| **III** | 6.8±0.1*(5)* | 2986±971*(11)* | 1429±622*(11)* | 1237±500*(11)* | 1102±438*(11)* | 113.8±52.4*(3)* | 82.1±10.7*(3)* | 934±194*(3)* |

*Parentheses indicate the number of samples tested.*

As shown in Table 4.1, the wastewater characteristics were significantly fluctuated as expected. The average TCOD of the feed was 2,254 mg/L in phase I, which was slightly increased to 2,804 and 2,986 mg/L, respectively in Phase II and III. The SCOD fraction of the TCOD accounted for 29-48% of the TCOD, and particulate COD represented between 52 and 71% of the TCOD. The average TSS were 869, 1,405 and 1,237 mg/L for the three phases, and VSS contributed towards a large fraction of TSS in all phases with the average VSS to TSS ratio being 89-90%. Nutrients concentration was also fluctuated. The average ammonium concentration varied from 51 mg/L in phase I to 99 mg/L in phase II, while the average phosphate concentration ranged from 87 mg/L in phase I to 114 mg/L in phase III. The high standard variation in all the analysis emphasized the high variation in the feed wastewater characteristics. Such fluctuated wastewater is typical for industrial wastewater due to the changes in manufacturing and cleaning processes. For instance, slaughtering, processing, and cleaning would change the concentrations of COD, SS, N, and P in the wastewater.

### *4.2 Reactor solids*

The change in MLSS and MLVSS was not as pronounced as the feed wastewater, which suggests that anaerobic digestion of the wastewater was very stable during operation, mainly due to long SRT of 50 days (Fig. 4.1). During the start of the reactor, the MLSS concentration was 3.5 g/L. It gradually stabilized to 2-2.5 g/L, and towards the end of the experiment it went up to 2.6 g/L; the average ratios of MLVSS to MLSS were 0.85±.06, 0.80±0.02 and 0.80±0.02 in Phase I, II and III

respectively. Biomass concentration increased slightly with time (from Phase I to III), which well accords to the increase in TCOD. Stable concentrations of MLSS and MLVSS are one of significant benefits created by membrane separation in AnMBRs. In some examples of typical anaerobic systems, SRT was not controlled well due to complete-mixing conditions, and MLSS and MLVSS concentrations were relatively fluctuated (Luste & Luostarinen, 2010, Borja, et al., 1998). Such unstable biomass concentration in anaerobic digesters would cause variation in treatment efficiency and methane gas production. For this reason, the AnMBR would show steady treatment efficiency and methane generation, even if the feed wastewater is substantially fluctuated, which was seen in this study (Table 4. 1).

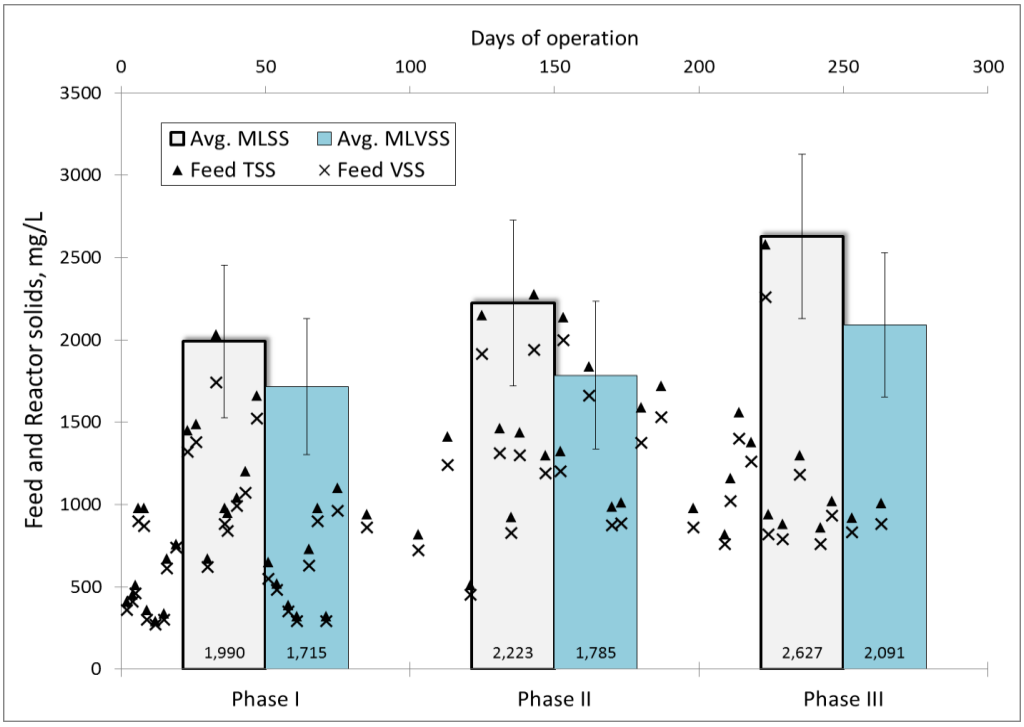


**Figure 4-1: Average MLSS/MLVSS in three Phases and variation of feed solids during the study**

### *4.3 Organics removal*

Influent TCOD and permeate COD concentrations are shown in Figure 4.2. The frequent and abrupt fluctuation of the feed TCOD can be observed (also evident in Table 4.1); this was due to the nature of the wastewater, which had different concentration of organic particles depending on the type of

operation in the plant at the particular time of sampling. Permeate (effluent) COD, however, was very stable against fluctuated TCOD in the feed. An immediate rise in the permeate COD at the beginning of each Phase was observed, which was due to the increase of OLR. The permeate COD stabilized gradually with the progress of each Phase. The average COD in permeate was 96±28, 170±36 and 373±76 mg/L, respectively, in Phase I, Phase II, and Phase III. The range of effluent COD concentrations obtained in this study were lower than those reported by Fuchs, et al. (2003), who had effluent concentrations in the range of 100-400 mg/L, using slaughterhouse wastewater as the feed in an AnMBR. Another study treating poultry slaughterhouse wastewater in an anaerobic biofilter (Debik & Coskun, 2009) reported effluent CODs as 80-460 mg/L against an influent COD of 1,600-9,100 mg/L. The TCOD removal efficiencies in this study were 95±3.1%, 94±2.3% and 88±4.6% for the three phases. These results are similar to the TCOD removal efficiency of 93.7% reported by Saddoud and Sayadi (2007), treating similar wastewater in an AnMBR.

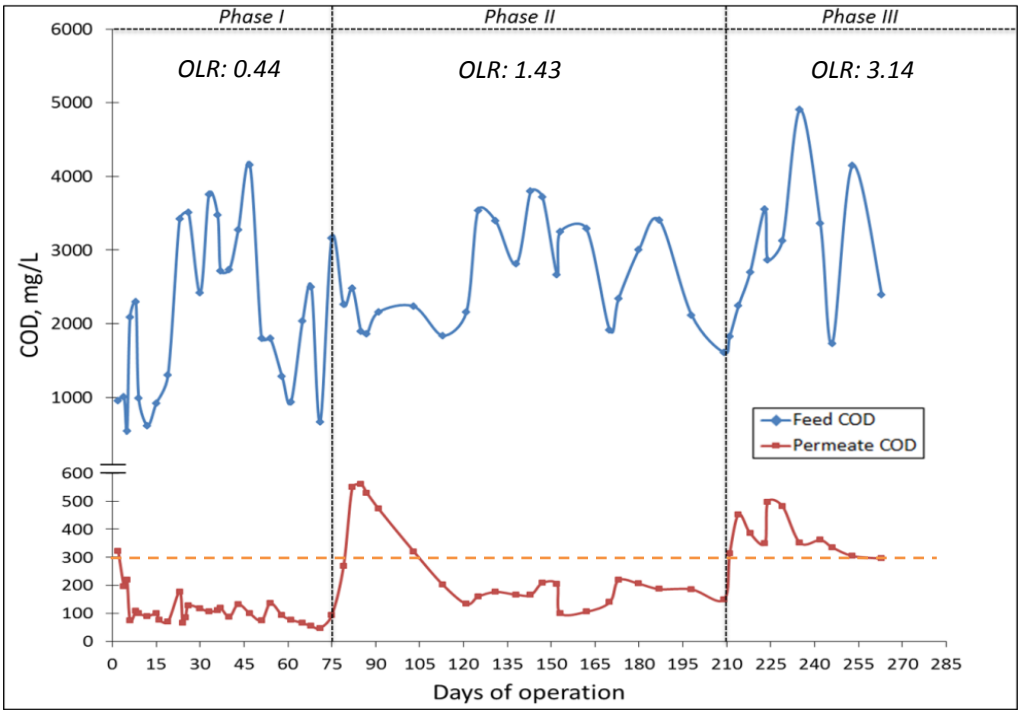


**Figure 4-2: Profile of influent and effluent COD**

Organic loading rates (OLR) of the AnMBR increased with the feed flow rates in Phase I, II and III, with average OLRs being 0.44±0.2, 1.43±0.4 and 3.14±1.1 kg COD/$m^3$/day, respectively. Studies by Fuchs, et al. (2003), and Saddoud and Sayadi (2007) utilizing slaughterhouse wastewater, however, achieved higher OLRs (6 – 16 Kg COD/$m^3$/day) than this study. This may have been due to the fact that their reactors were operated at higher temperatures (30-37 deg. C), which helped to attain higher rate of biodegradation. Fig. 4.3 presents the average OLRs with average TCOD removal efficiencies achieved in the three operational Phases of this study; the bars represent standard deviation. Corresponding to the rise in the effluent CODs, the TCOD removal efficiency dropped immediately after the increase of OLRs (at the beginning of Phase II and Phase III). The TCOD removal efficiencies improved as the system stabilized gradually in each Phase. At the beginning of Phase I, Phase II and Phase III the TCOD removal efficiencies were 60%, 71% and 80%, while the average TCOD removal efficiencies calculated at the end of each Phase were 94.5%, 93.5% and 87.5%, respectively. This showed that the microorganisms acclimated well with the increased feed loading and could gradually improve their efficiency in removal of organics as the Phases progressed.

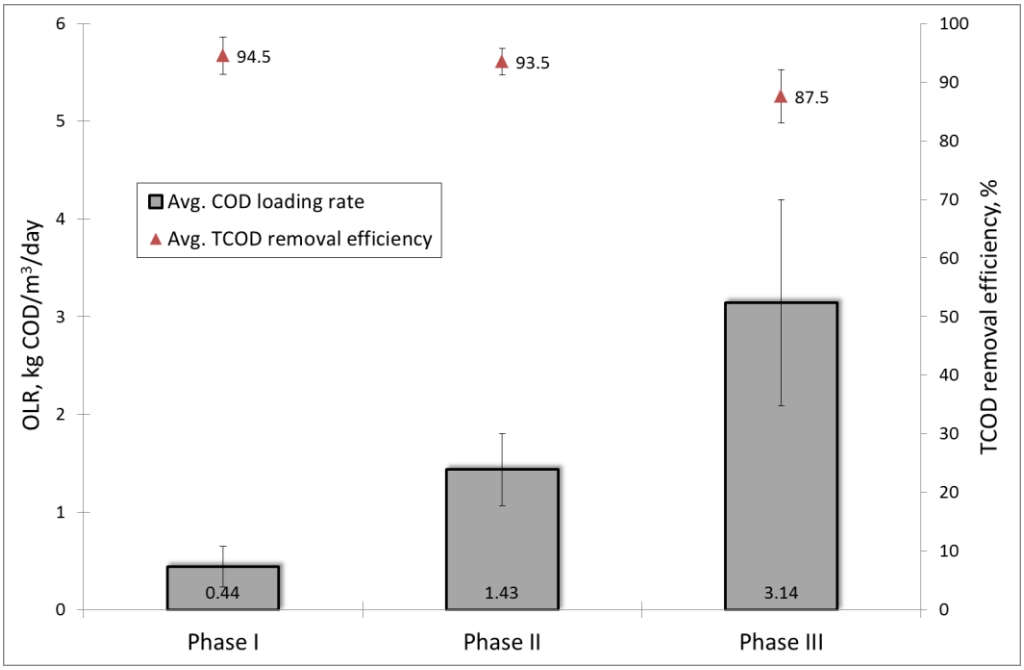


**Figure 4-3: Average OLRs and TCOD removal efficiency**

The average food to microorganism ratio (F/M) for Phase I, Phase II and Phase III were 0.30±0.19, 0.83±0.29 and 1.54±0.59 kg COD/kg MLVSS/day, respectively. The relatively lower F/M ratios in Phase I and Phase II is translated into better system performance than in Phase III in terms of COD removal efficiency. These results indicate that a F/M ratio of less than 1 is recommended for the AnMBRs treating meat-processing wastewater. In a study to evaluate the influence of F/M ratio on a batch anaerobic process, Montalvo, et al. (2012) ran experiments with F/M ratios ranging from 0.21 to 1.92 g COD/g VSS with a synthetic substrate; they reported higher COD removal and ammonia removal efficiencies (93% and 70%, respectively) at F/M ratio of 0.4 g COD/g VSS. Another study (Prashanth, et al., 2006) conducted with substrate containing complex compounds and high fraction of particulate COD found the optimum value of F/M to be in the range of 0.57 to 0.68 from a kinetic point of view.

### *4.4 Biogas and methane generation*

Biogas was generated at the average rate of 0.37±0.18, 1.05±0.24 and 2.82±0.62 L/day during Phase I to III, respectively. The percentage of $CH_4$ in the biogas was steady throughout the study

period, with an average of 72±4%. The other major components of the biogas were carbon dioxide and nitrogen. $CH_4$ production rate was 0.26±0.13, 0.79±0.17 and 2.18±0.29 L/day in the three phases. The daily $CH_4$ production along with the percentage of $CH_4$ in biogas is shown in Fig. 4.4. Daily methane production increased with increasing OLRs, as expected. The increase in methane production from Phase I to Phase II was 200% and from Phase II to Phase III was 176%, while the increase in OLR from Phase I to Phase II was 225% and from Phase II to Phase III was 119%.

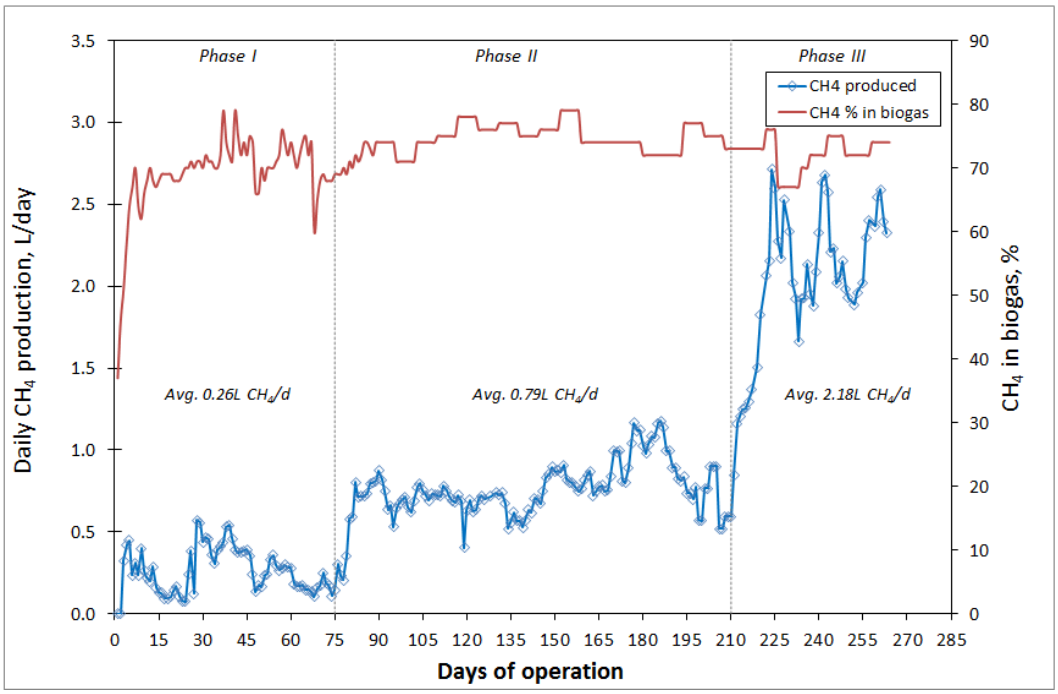


**Figure 4-4: Methane production rate and methane percentage**

The specific methane yield is a common parameter, used to denote the amount of methane produced by an anaerobic process by normalizing the methane production with - the unit mass (in COD equivalent) of waste stabilized (consumed), or a unit mass of the waste added, or a unit volume of the reactor. These three types of methane yields are usually expressed as "L $CH_4$/g COD removed", "L $CH_4$/g COD added" and "L $CH_4$/L of reactor/day", respectively.

The theoretical methane yield as a function of COD removed is 0.38 L $CH_4$/g $COD_{removed}$ at 24 °C when all the organic strength (in COD) of the feed is converted to methane. In an AnMBR, the

methane production can be accounted towards the amount of methane in the biogas, and the amount of methane dissolved in the permeate. The specific methane yield considering methane gas production was 0.19±0.13, 0.14±0.04 and 0.19±0.08 L $CH_4$/g $COD_{removed}$ in Phase I, II and III respectively. High concentration of dissolved methane can account for low gaseous $CH_4$ yield. The dissolved methane concentrations measured in the permeate according to the procedure described in section 3.5.3 were 54.2±5.3, 34.1±15.5 and 25.0±5.0 mg/L in Phase I, II and III, respectively. Whereas, the saturation concentrations of dissolved methane in the AnMBR were computed at 18.2±0.9, 19.5±0.5 and 18.8±0.6 mg/L in the three Phases. Thus, dissolved methane was over-saturated in the AnMBR during all the Phases. Yeo and Lee (2013) also reported dissolved methane higher than thermodynamic equilibrium concentration in a completely mixed AnMBR (8 measurements out of a total of 20) operated at ambient temperature with complete mixing at a SRT of 20 days. The authors suggested the slower transfer rate of methane from aqueous to gaseous form than the rate of formation (of dissolved methane), and the dynamic behaviour of dissolved methane under vigorous mixing to be the two reasons behind this. In an earlier study, Pauss et al. (1990) found dissolved methane at a level 10-12 times higher than the thermodynamic equilibrium concentration in a completely mixed anaerobic digester.

The dissolved methane (in mg/L) measured in this study was converted to equivalent $CH_4$ in mL/d applying the temperature correction and using the molecular weight of methane (1 mol $CH_4$=16 g $CH_4$), molar volume of methane (1 mol $CH_4$ = 22.4 L $CH_4$) and the volume of daily permeate produced. Combining the dissolved methane with the gaseous methane produced, the specific methane yields were calculated to be 0.24±0.16, 0.16±0.05 and 0.20±0.09 L $CH_4$/g $COD_{removed}$ in Phase I, II and III respectively. Thus, the methane yield for the AnMBR was 42-63% of the theoretical yield. Fig. 4.5 presents the average methane yields along with the average daily methane production in the three Phases; the bars represent standard deviation.

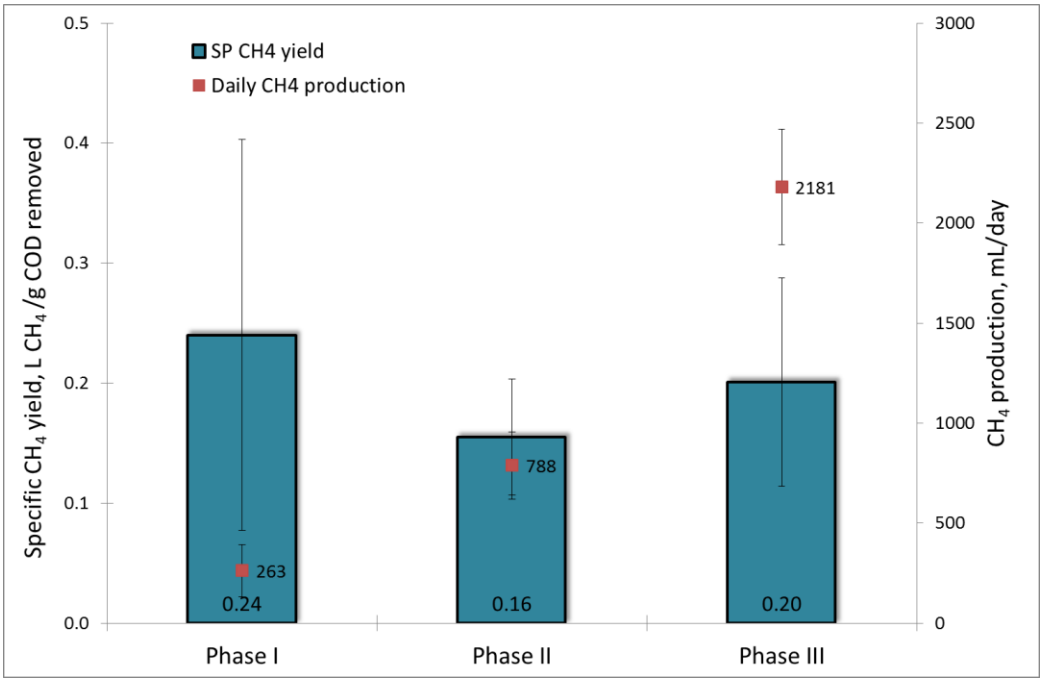


**Figure 4-5: Average specific methane yield and daily methane production**

The methane yields observed in this study are lower than the theoretical yield, and slightly lower than those of some previous studies conducted with similar wastewater, that showed methane yields of 0.2 to 0.3 L $CH_4$/g $COD_{removed}$ (Fuchs et al., 2003; Saddoud & Sayadi, 2007). This comparison indicates that the meat processing wastewater in this study contained complex and non-biodegradable organic compounds that could not be utilized fully. Factors relating to characteristics of the feed and the reactor in the mentioned studies also have a role for better methane yield. The feed COD in the study by Fuchs et al. (2003) were 5800-20,150 mg/L and the suspended organic matters in the feed were fully degraded. Saddoud and Sayadi (2007) similarly had a high feed COD of 7148-20400 mg/L, and a healthy biomass concentration of over 5 g/L (VSS). The high feed COD values meant abundant food for higher substrate utilization, and high VSS values helped to perform adequate biodegradation. The lower temperature used in this study could be another reason for lower methane production. Debik and Coskun (2009) also mentioned this reason for the lower methane production in their study.

The average specific methane yields in Phase I, II and III in terms of per gram COD of feed added were 0.22±0.14, 0.14±0.04 and 0.17±0.07 L $CH_4$ /g $COD_{added}$, and the average specific methane yield in Phase I, II and III in terms of the unit volume of the reactor were 67.2±26.1, 182.7±33.5 and 471.4±57.7 mL $CH_4$/L of reactor/day.

### *4.5 COD mass balance*

The COD mass (electron) balance calculated according to the process in section 3.6 is shown in Fig. 4.6. The average total incoming CODs were 2,029, 6,729 and 14,633 mg COD/day in Phase I, II and III, corresponding to HRTs of 5, 2 and 1 day respectively.

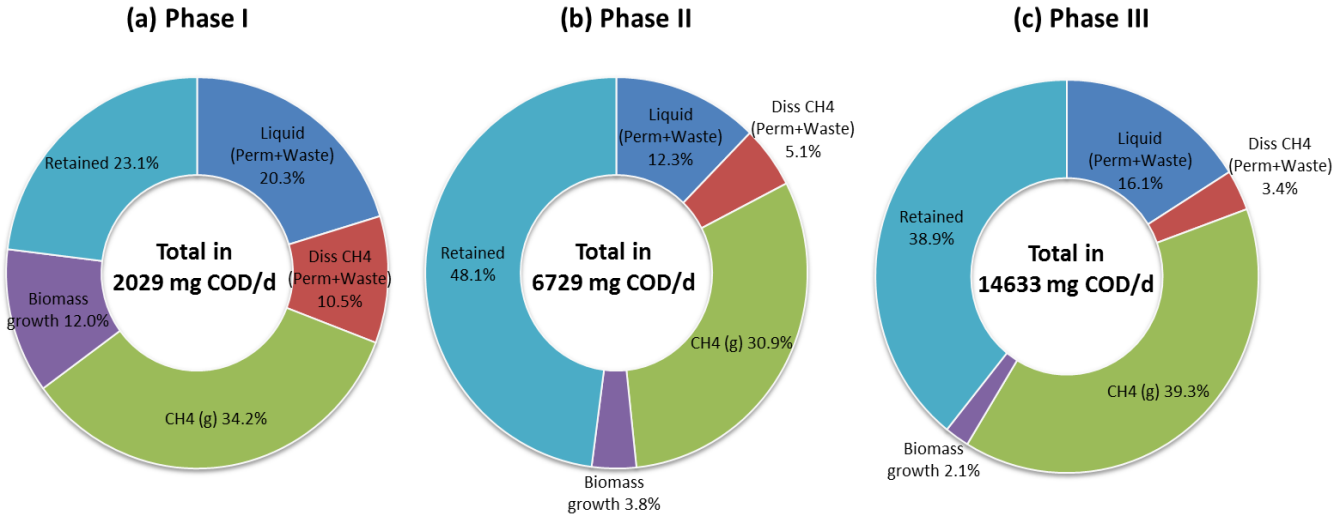


**Figure 4-6: COD balances in the AnMBR for the three Phases**

Among the known utilized CODs, the largest electron sink was methane gas with 693, 2,077 and 5,748 mg COD/d (34.2%, 30.9% and 39.3% of the input COD) in Phase I, II and III respectively. The second largest sink was COD in outgoing liquid (AnMBR permeate and wasted sludge), amounting to 412, 830 and 2,354 mg COD/d (20.3%, 12.3% and 16.1% of the input COD) in the three Phases. Dissolved methane (permeate and wasted sludge) was found to be as high as 10.5% of the input COD in Phase I, though it reduced to 5.1% in Phase II and to 3.4% in Phase III. The suspended biomass growth (calculated from MLVSS in the wasted sludge) accounted for 12.0% of

the incoming COD in the first Phase, while it was 3.8% and 2.1% in the subsequent two Phases. The remaining COD (ΔCOD) after all known sinks were accounted for amounted to 23.1%, 47.9% and 39.2% of the incoming COD in Phase I, II and III respectively. These can be attributed towards the COD retained in the reactor as un-hydrolyzed organic/inorganic particles that could not be degraded, and COD utilized for the accumulation/increase of biomass, both suspended and attached to the reactor wall and the membrane surface.

### *4.6 Reactor stability*

For routine monitoring of reactor stability, total VFAs and alkalinity concentrations in the permeate were regularly monitored. Table 4.2 shows the measured total VFAs and alkalinity in the reactor during the three phases (Average ± Standard deviation *(n)*), along with the VFA/Alkalinity ratio.

**Table 4-2: VFAs and Alkalinity measurements**

| Phase | Total VFAs, mg/L | Alkalinity, mg/L as $CaCO_3$ | VFA/Alkalinity |
|---|---|---|---|
| I | 172.8±22.3 *(3)* | 1220.5±94.3 *(11)* | 0.14±0.02 *(3)* |
| II | 286.1±82.2 *(3)* | 1323.7±37.4 *(3)* | 0.18±+0.03 *(3)* |
| III | 221.5±63.5 *(5)* | 1379.7±155.9 *(5)* | 0.17±0.07 *(5)* |

VFAs and alkalinity ratios were found to be 0.14±0.02, 0.18±0.03 and 0.17±0.07 during Phase I, Phase II and Phase III respectively. The VFA/Alkalinity values of less than 0.2 throughout the operational period suggests that there was no accumulation of excess volatile acids, indicating favourable condition for the methanogens in the reactor and the stability of the system; whereas a high concentration of VFAs would have indicated a stressful condition in the system (Aquino & Stuckey, 2002). The pH in the permeate (initial pH of the titration process) was consistently close to neutral pH (6.9±0.07) during all experiments.

### *4.7 Membrane performance*

Membrane maintenance cleaning was performed once a week during Phase I and II, and twice a week during Phase III (due to signs of membrane fouling); the production (permeation) was stopped for approximately 1.5 hours during each cleaning. This study clearly showed that a reasonable flux could be maintained with minimal to moderate cleaning. Baek and Pagilla (2006) performed weekly cleaning to maintain flux, whereas their feed was domestic wastewater with a TSS of around 150 mg/L. Zhang, et al. (2007) performed both weekly and monthly chemical cleaning on two separate membranes where the feed was swine manure, and opined that monthly cleaning could be more beneficial than weekly cleaning to avoid a slow increase of the membrane resistance. The frequency of chemical membrane cleaning should be kept to a minimum as cleaning will cause disruption in production cycle, introduce the microbes to harsh chemicals and can shorten the lifespan of the membrane material.

Permeate fluxes of 1.14±0.02, 3.15±0.04 and 6.15±0.37 LMH were observed during the three phases. The observed fluxes were consistent with the set fluxes (Table 3.3) throughout the first two phases and the first 15 days of the third phase, indicating that there was none or trivial membrane fouling. Scouring of membrane surface with biogas, application of intermittent pumping strategy (7 min on, 3 min off) and periodic membrane maintenance cleaning helped to keep stable membrane flux and restricted membrane fouling. The flux during Phase II and III are comparable to other AnMBR studies where the observed fluxes were in the range of 2 to 10 LMH (Fuchs, et al., 2003; Saddoud & Sayadi, 2007; Zhang, et al., 2007). However, with the progress of Phase III (at 1d HRT and flux of 6.4 LMH), and owing to the gradual development of irremovable fouling over time, the membrane became more fouled, probably due to pores clogging and cake formation, and rapid declines in flux were experienced. This prompted to increase the membrane maintenance cleaning frequency from once a week to twice a week. After 263 days of operation, the permeate flux

declined sharply to 3.98 LMH, and the reactor operation was stopped (Fig 4.7). At that stage, the membrane required a recovery cleaning before it can be operational again.

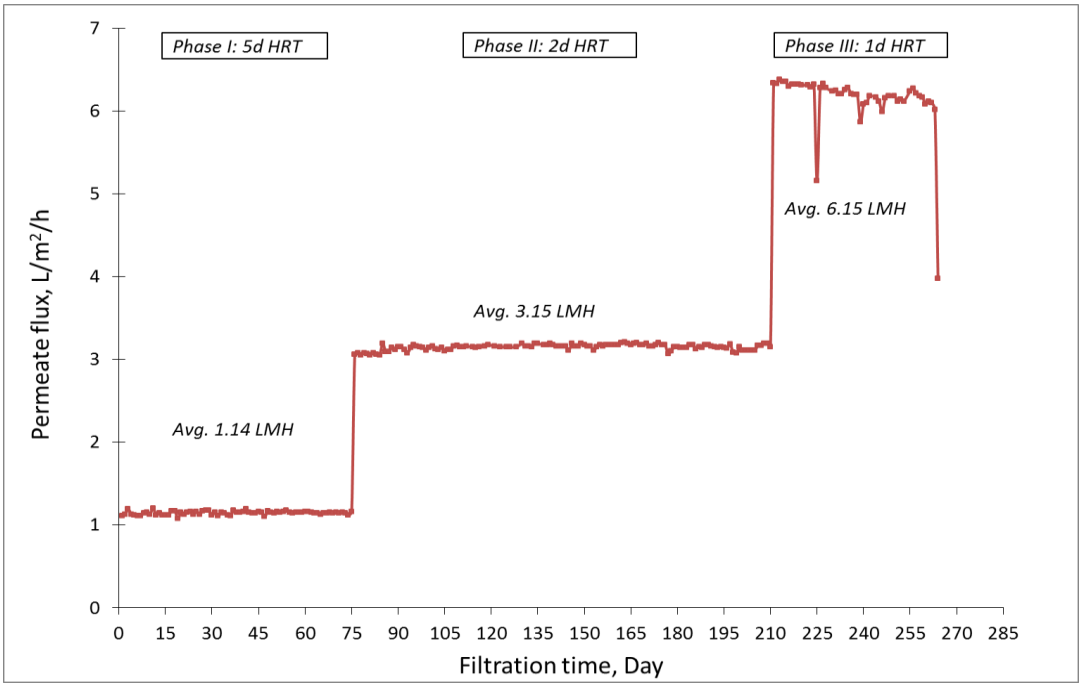


**Figure 4-7: Profile of permeate flux during AnMBR operation**

The TMP and permeability during a single production cycle for the three phases are shown in Fig. 4.8. At the end of Phase I, Phase II and at the beginning of Phase III the TMP remained below 2 kPa. However, at the end of Phase III when the permeate flux declined to 3.98 LMH, TMP reached higher than 40 kPa (over the recommended limit of 30 kPa by the manufacturer) at the end of a single production cycle of 7 minutes. For the same production cycles in concern (Fig 4.8), the increase of TMP over time (dTMP/dT), termed as Fouling Index, were 0.16, 0.18 and 0.21 kPa/min at the end of Phase I, end of Phase II and start of Phase III. The Fouling Index in a production cycle at the end of Phase III reached as high as 6.0 kPa/min, indicating the membrane was severely fouled. According to the manufacturer specifications, a TMP increase of 2 kPa/min would suggest severe membrane fouling. The membrane permeability (Flux/TMP) increased with the increase in flux rate as the operating phases proceeded, and remained fairly consistent throughout the

production cycle at the end of Phases I and II, and the beginning of Phase III. However, a sharp drop in permeability was observed from 3.8 LMH/kPa to 0.09 LMH/kPa.

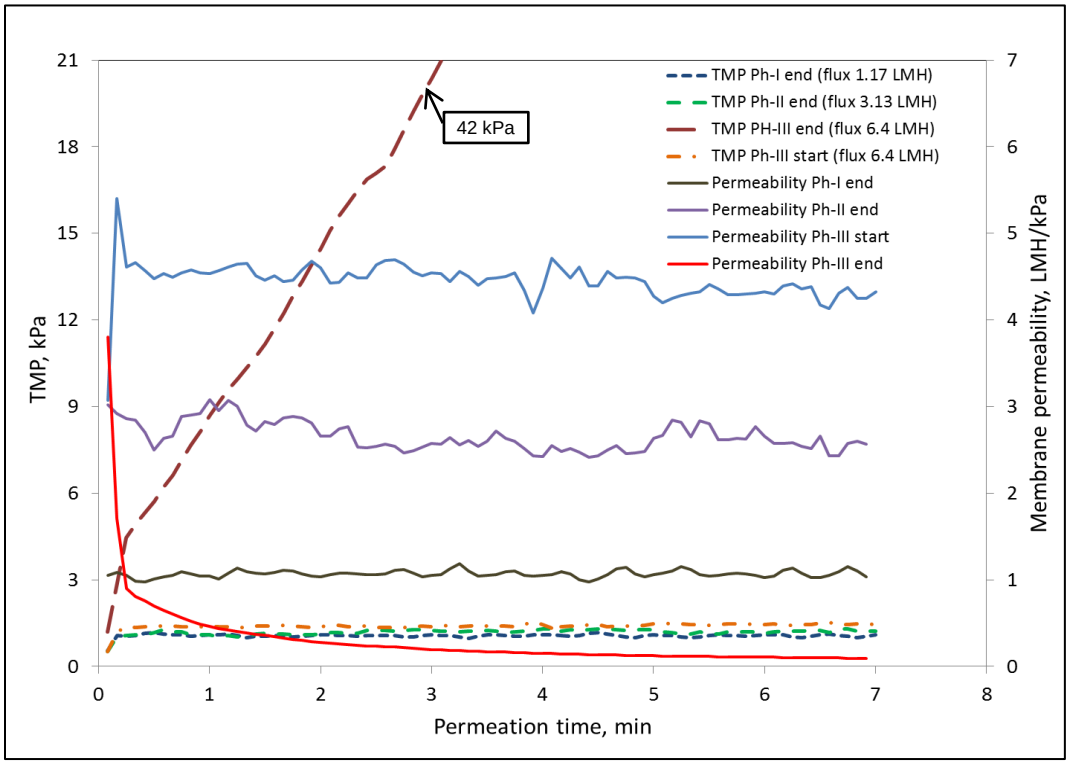


**Figure 4-8: TMP and membrane permeability during a single permeation cycle at the end of Phase I, Phase II and Phase III, and at the start of Phase III**

It has to be kept in mind that membrane fouling is not completely avoidable even by periodic chemical cleaning, and design and operation strategy of membrane bioreactors should be based upon factors like – minimization of fouling rate, use of chemical or physical cleaning, and accepting a slight reduction in flux resulting from fouling.

Fig. 4.9 shows a comparative view of a fresh membrane and a fouled membrane. The cake sludge that built up on the membrane surface is clearly visible in Fig 4.9(b). As discussed in the following section, the high EPS concentration was a contributor towards this sludge build up. However, the cake sludge was not analysed under the scope of this study to determine the foulant components (organic and inorganic) and their characteristics, neither was the microbial community existent in

the bulk sludge or cake sludge categorized. Such analyses warrant a separate study, which can be undertaken in the future.

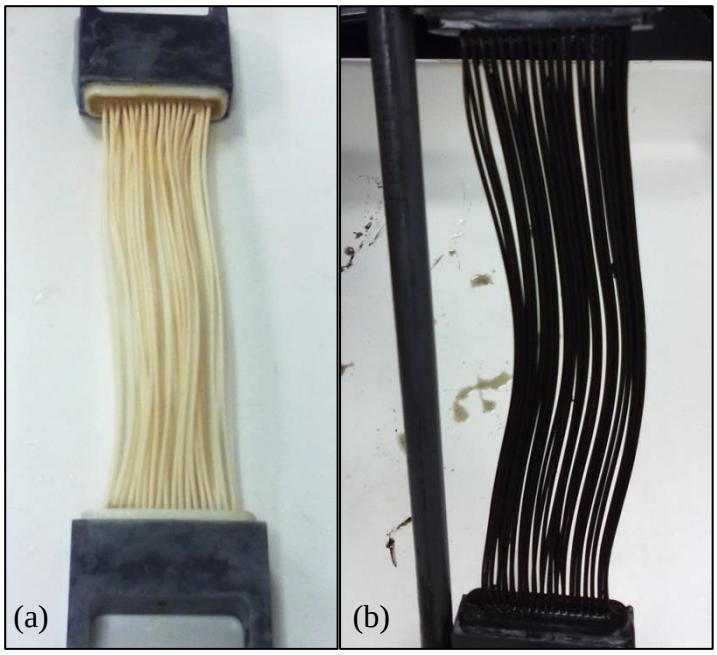


**Figure 4-9: Comparative view of a fresh/cleaned membrane (a) and a fouled membrane (b)**

### *4.8 EPS and SMP in the bulk sludge*

The EPS and SMP measured in the bulk sludge were characterized as protein and carbohydrate contents and presented in Table 4.3. The contents were normalized as mg per g of VSS. The protein contents in the EPS were measured to be 83.2±7.2, 86.2±6.3 and 91.2±3.5 mg/gVSS and the carbohydrates were found to be 13.7±1.4, 14.0±2.2 and 14.1±1.4 mg/gVSS, respectively in Phase I, II and III. The protein to carbohydrates (P/C) ratio varied slightly but did not show a significant variation, being in the range of 6.1 to 6.5. Lin et al. (2011a) had similar P/C values, where they obtained a P/C ratio of 6.2 to 6.8. However, their protein contents were much lower (21 mg/gVSS) than the current study. This is due to the fact that their feed wastewater originated from a pulp and paper mill, whereas this study used meat processing wastewater as feed (high protein content). D'Abzac, et al. (2010) obtained a P/C ratio of 2.5 while extracting EPS from granular sludge of an anaerobic digester treating vinasses of brandy by using EDTA; however, the ratio was 6.1 for the same sample when extraction was performed by Formaldehyde+NaOH. Sludge with higher P/C

ratio will have higher stickiness and favour the development of cake formation (Lin, et al., 2009). Therefore, higher P/C ratio in the EPS of the bulk sludge would contribute to increased membrane fouling in an AnMBR due to formation of cake sludge on the membrane surface.

The building block of protein is amino acid. Animal blood contains a large amount of amino acids and proteins. Additionally, de Lange, et al (2003) reported that protein is the major chemical constituent in a pig's body, while carbohydrate is present in very small amounts. These explain high protein concentration and high P/C ratio found in the EPS under this study. As the Phases progressed, the protein contents increased with the increase of OLR. However, while the protein contents increased by 9.6% from Phase I to Phase III, the carbohydrates increased by only 2.9%.

**Table 4-3: Protein and carbohydrate content and their ratio from EPS extracts**

| Phase | Protein, mg/L | Carbohydrate, mg/L | Protein, mg/gVSS | Carbohydrates, mg/gVSS | Prot./Carb. |
|---|---|---|---|---|---|
| I | 99.8±8.7 | 16.4±1.7 | 83.2±7.2 | 13.7±1.4 | 6.1±0.1 |
| II | 140.1±10.2 | 22.8±3.6 | 86.2±6.3 | 14.0±2.2 | 6.2±0.5 |
| III | 200.6±7.7 | 30.9±3.2 | 91.2±3.5 | 14.1±1.4 | 6.5±0.4 |

The total EPS content (protein + carbohydrate) was 96.9±8.6, 100.2±8.5 and 105.2±5.0 mg/gVSS in Phases I, II and III respectively (Fig. 4.10). The increase in total EPS from Phase I to Phase II was 3.5%, and from Phase II to Phase III was 5.0 %, with an overall increase of 8.6% from Phase I to III. This increase in the total EPS concentration can be related with increase in the OLR and feed TCOD in Phase II and Phase III.

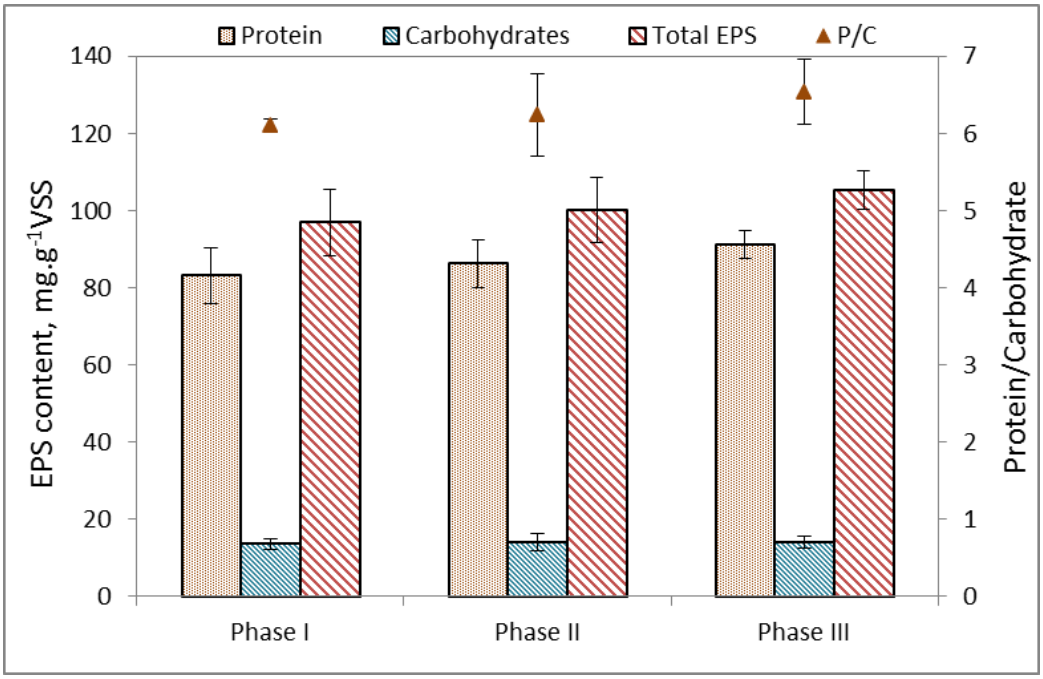


**Figure 4-10: EPS contents in bulk sludge**

Similar to EPS, the normalized production of SMP increased linearly with increasing OLR and feed COD. The combination of protein and carbohydrates concentration present in SMP was 26.7±3.5, 27.0±3.1 and 27.2±1.4 mg/gVSS in Phase I, II and III respectively (Table 4.4). This increase coincides with the increase seen in the SCOD values in the bulk sludge (272, 341 and 502 mg/L in the three Phases) and the COD values in the permeate (96, 170 and 373 mg/L in the three Phases), as SMP contributes towards a considerable fraction of the soluble COD (Aquino & Stuckey, 2002). Although the normalized protein content (mg/gVSS) was seen to rise by 8.9% from Phase I to Phase III, the carbohydrate contents did not increase during the latter two Phases.

**Table 4-4: Protein and carbohydrate content in SMP**

| Phase | Protein, mg/L | Carbohydrate, mg/L | Protein, mg/gVSS | Carbohydrates, mg/gVSS |
|---|---|---|---|---|
| I | 24.4±4.6 | 7.7±0.4 | 20.3±3.8 | 6.4±0.3 |
| II | 34.9±3.8 | 9.0±1.2 | 21.5±2.3 | 5.5±0.8 |
| III | 48.6±4.0 | 11.2±0.8 | 22.1±1.8 | 5.1±0.4 |

# *Chapter 5*

# *Conclusions*

# 5 CONCLUSIONS

In this study, a suspended growth completely mixed anaerobic reactor with a submerged UF membrane was operated to evaluate its feasibility for treating real meat processing wastewater. External heating was not applied to challenge its ability to perform in ambient temperature. Low to moderate OLR and membrane permeation flux were applied for the duration of the study (263 days). Based on the results of this study, the following conclusions can be drawn:

- The AnMBR was capable of successfully treating meat processing wastewater and achieved COD removal efficiencies of 88% to 95% with OLRs ranging from 0.4 to 3.1 Kg COD/$m^3$/day.
- A fairly stable MLVSS of 1.7 to 2.1 g/L was observed in the reactor.
- Effluent COD concentration in the range of 96 to 373 mg/L could be achieved.
- The daily biogas/methane production increased with the increase of OLR and decrease of HRT; the daily methane productions were 0.26±0.13, 0.79±0.17 and 2.18±0.29 L/day in Phase I, II and III respectively. The specific methane yields taking the dissolved methane into account were 0.24±0.16, 0.16±0.05 and 0.20±0.09 L $CH_4$/g $COD_{removed}$ in Phase I, II and III respectively. The relatively lower yield was due to the presence of complex and non-biodegradable organics, and lower operational temperature.
- The membrane showed better performance when the permeate flux was less than 6 LMH. For permeate flux higher than 6 LMH, maintenance cleaning twice per week is suggested. Scouring of membrane surface with biogas, intermittent mode of permeation and periodic membrane maintenance cleaning helped to keep stable membrane flux and restricted membrane fouling.

- Considerable amount of EPS were calculated from the bulk sludge (96.9±8.6, 100.2±8.5 and 105.2±5.0 mg EPS/gVSS in Phases I, II and III respectively). This contributed towards the formation of cake sludge on the membrane surface, and accelerated membrane fouling.
- The start-up and operational information from the successful performance of this lab-scale reactor can be used as a baseline for implementation of a larger or pilot scale AnMBR treating similar wastewater.

# *Chapter 6*

# *Recommendations*

# 6 RECOMMENDATIONS

The following studies are suggested for the future application of AnMBR in treatment of meat processing wastewater or other high-strength industrial wastewater.

- High levels of nutrients (N and P) were observed in the wastewater. Evaluating the performance of a submerged AnMBR in removing these nutrients, and determination of an appropriate post treatment process, if needed is a potential area of research.
- An important benefit of anaerobic process is the production of energy in the form of methane. Future works can be directed towards assessment of self-sustainability of AnMBR through economic analysis of the capital and maintenance cost, and the net energy production.
- Microbiological investigation can be performed to determine the dominant species of bacteria or methanogens (e.g. acetotrophic/hydrogenotrophic) in such an environment and their behaviour with changing conditions. Microbial population in the bulk sludge (suspended) and those attached to the membrane (cake sludge) can be characterized and differentiated.
- A future initiative can involve the membrane foulant analyses, in order to characterize the fouling components (organic and inorganic) both qualitatively and quantitatively, to visualize the distribution of foulant layers, and to understand the extent to which EPS plays a role in formation of the cake sludge.
- The current study implemented intermittent pumping; one filtration cycle consisted of production (permeation) for seven minutes and relaxation for three minutes for minimizing membrane fouling. Further study can emphasize on determining the optimum relaxation time and its impact on membrane fouling. Also, the flow rate for biogas sparging can be optimized with regards to energy requirement and fouling minimization.

- The minimum HRT applied during this study was 1 day and SRT was kept constant at 50 days. HRT can be lowered to obtain higher volumetric throughput and higher SRT can be advantageous by providing better organic degradation. However, both the parameters have direct impact on membrane fouling, dissolved methane concentration and effluent COD concentration. Therefore, future work can identify the minimum HRT and the optimum SRT that can deliver the desired treatment efficiency without adverse operational condition.

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# *Appendix*

## *Analytical and operational data*

# APPENDIX: ANALYTICAL AND OPERATIONAL DATA

## Table A-1: Feed and permeate analytical data

| | Feed - analytical | | | | | | | | Permeate - analytical | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| # of days system run | pH | TCOD | sCOD (0.45µm) | TSS (1.5µm) | VSS | Alkalinity (as CaCO3) | Total phosphate | $NH_3$ - N | TCOD | Turbidity | Dissolved methane | Saturation methane | Total phosphate | $NH_3$ - N |
| | | mg/L | mg/L | mg/L | mg/L | mg/L | mg/L | mg/L | mg/L | NTU | mg/L | mg/L | mg/L | mg/L |
| 1 | | | | | | | | | | | | 9.6 | | |
| 2 | | 956 | 331 | 410 | 360 | | | | 323 | | | 11.9 | | |
| 3 | | | | | | | | | | | | 13.2 | | |
| 4 | | 1003 | 325 | 450 | 410 | | 73.8 | | 196 | | | 15.0 | | |
| 5 | | 544 | 341 | 510 | 460 | | | | 219 | | | 16.5 | | |
| 6 | | 2087 | 163.576 | 980 | 900 | | | 58.7 | 75 | | | 17.3 | | |
| 7 | | | | | | | | | | | | 18.1 | | |
| 8 | | 2301 | 181.356 | 980 | 870 | | | | 109 | | | 16.5 | | |
| 9 | | 985 | 344.932 | 360 | 300 | | | | 100 | | | 16.0 | | |
| 10 | | | | | | | | | | | | 17.1 | | |
| 11 | | | | | | | | | | | | 17.6 | | |
| 12 | | 615 | 355.6 | 290 | 270 | | | | 91 | | | 18.1 | | |
| 13 | | | | | | | | | | | | 17.6 | | |
| 14 | | | | | | | | | | | | 17.3 | | |
| 15 | | 921 | 178 | 340 | 300 | | | | 100 | | | 17.6 | | |
| 16 | | | | 670 | 610 | | | | 76 | | 52.1 | 17.8 | | |
| 17 | | | | | | | | | | | | 17.8 | | |
| 18 | | | | | | | | | | | | 17.8 | | |
| 19 | | 1302 | 604 | 760 | 740 | | | | 71 | | | 17.8 | | |
| 20 | | | | | | | | | | | | 17.6 | | |
| 21 | | | | | | | | | | | | 17.6 | | |
| 22 | | | | | | | | | | | | 17.6 | | |
| 23 | | 3421 | 739 | 1,450 | 1,320 | | 89.5 | | 178 | | | 17.8 | | |
| 24 | | | | | | | | | 66 | | | 18.1 | | |
| 25 | | | | | | | | | 86 | | | 18.1 | | |
| 26 | | 3513 | 520 | 1,490 | 1,380 | | | | 128 | | | 18.4 | | |
| 27 | | | | | | | | | | | | 18.1 | | |
| 28 | | | | | | | | | | | | 18.4 | | |
| 29 | | | | | | | | | | | | 18.4 | | |

| | Feed - analytical | | | | | | | | Permeate - analytical | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| # of days system run | pH | TCOD | sCOD (0.45µm) | TSS (1.5µm) | VSS | Alkalinity (as CaCO3) | Total phosphate | $NH_3$ - N | TCOD | Turbidity | Dissolved methane | Saturation methane | Total phosphate | $NH_3$ - N |
| | | mg/L | **mg/L** | **mg/L** | **mg/L** | **mg/L** | **mg/L** | **mg/L** | **mg/L** | **NTU** | **mg/L** | **mg/L** | **mg/L** | **mg/L** |
| 30 | | 2418 | 823 | 670 | 620 | | | | 118 | | | 18.1 | | |
| 31 | | | | | | | | | | | | 18.6 | | |
| 32 | | | | | | | | | | | | 18.4 | | |
| 33 | | 3753 | 990 | 2,030 | 1,740 | | | | 106 | 54.2 | | 18.4 | | |
| 34 | | | | | | | | | | 124.7 | | 18.1 | | |
| 35 | | | | | | | | | | 136 | | 18.1 | | |
| 36 | | 3474 | 1,027 | 980 | 880 | | | | 112 | 170 | | 18.6 | | |
| 37 | 6.9 | 2717 | 1,013 | 950 | 840 | 400.4 | | | 120 | 44 | | 20.4 | | |
| 38 | | | | | | | | | | 58.1 | | 19.1 | | |
| 39 | | | | | | | | | | | | 18.6 | | |
| 40 | 6.9 | 2735 | 1,015 | 1,040 | 990 | 570.5 | | | 88 | 24.00 | | 18.4 | | |
| 41 | | | | | | | | | | | 50.3 | 20.4 | | |
| 42 | | | | | | | | | | | | 19.4 | | |
| 43 | 6.7 | 3275 | 994 | 1,200 | 1,070 | 860.8 | | | 133 | | | 18.6 | | |
| 44 | | | | | | | | 29.4 | | | | 19.1 | | |
| 45 | | | | | | | | | | | | 18.6 | | |
| 46 | | | | | | | 98.7 | | | | | 19.4 | | |
| 47 | | 4153 | 723 | 1,660 | 1,520 | | | | 100 | | | 19.1 | | |
| 48 | | | | | | | | | | | | 17.1 | | |
| 49 | | | | | | | | | | | | 17.1 | | |
| 50 | | | | | | | | | | | | 18.1 | | |
| 51 | 6.8 | 1799 | 940 | 650 | 550 | 620.5 | | | 75 | | | 17.6 | | |
| 52 | | | | | | | | | | | | 18.1 | | |
| 53 | | | | | | | | | | | | 18.1 | | |
| 54 | 6.7 | 1796 | 1024 | 520 | 480 | 670.6 | | | 137 | | | 18.1 | | |
| 55 | | | | | | | | | | | | 18.4 | | |
| 56 | | | | | | | | | | | | 18.6 | | |
| 57 | | | | | | | | | | | | 19.7 | | |
| 58 | 6.7 | 1284 | 908 | 390 | 350 | 980.9 | | | 93 | 78.6 | | 19.1 | | |
| 59 | | | | | | | | | | 9.8 | | 18.6 | | |
| 60 | | | | | | | | | | 21.8 | | 19.1 | | |
| 61 | 6.8 | 935 | 769 | 320 | 290 | 1003.0 | | | 78 | | | 18.6 | | |
| 62 | | | | | | | | | | | | 18.1 | | |

| | Feed - analytical | | | | | | | | Permeate - analytical | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| # of days system run | pH | TCOD | sCOD (0.45µm) | TSS (1.5µm) | VSS | Alkalinity (as CaCO3) | Total phosphate | $NH_3$ - N | TCOD | Turbidity | Dissolved methane | Saturation methane | Total phosphate | $NH_3$ - N |
| | | mg/L | **mg/L** | **mg/L** | **mg/L** | **mg/L** | **mg/L** | **mg/L** | **mg/L** | **NTU** | **mg/L** | **mg/L** | **mg/L** | **mg/L** |
| 63 | | | | | | | | | | | 60.2 | 18.6 | | |
| 64 | | | | | | | | | | | | 19.1 | | |
| 65 | 6.7 | 2038 | 436 | 730 | 630 | 970.9 | | | 66 | | | 19.4 | | |
| 66 | | | | | | | | | | | | 18.6 | | |
| 67 | | | | | | | | | | | | 19.1 | | |
| 68 | | 2496 | 795 | 980 | 900 | | | | 55 | | | 15.5 | | |
| 69 | | | | | | | | | | | | 16.8 | | |
| 70 | | | | | | | | | | | | 17.6 | | |
| 71 | | 665 | 629 | 320 | 290 | | | | 48 | | | 17.8 | | |
| 72 | | | | | | | | 64.8 | | | | 17.6 | | |
| 73 | | | | | | | | | | | | 17.6 | | |
| 74 | | | | | | | | | | | | 17.6 | | |
| 75 | | 3161 | 496 | 1,100 | 960 | | | | 94 | | | 17.8 | | |
| 76 | | | | | | | | | | | | 17.8 | | |
| 77 | | | | | | | | | | | | 17.8 | | |
| 78 | | | | | | | | | | | | 18.1 | | |
| 79 | | 2258 | 797 | | | | | | 269 | | | 17.8 | | |
| 80 | | | | | | | | | | | | 18.4 | | |
| 81 | 6.8 | | | | | 856.6 | | | | | | 18.1 | | |
| 82 | | 2476 | 933.00 | | | | | | 550 | | | 18.6 | | |
| 83 | | | | | | | | | | | | 18.4 | | |
| 84 | | | | | | | | | | | | 18.6 | | |
| 85 | | 1900 | | 940 | 860 | | | | 560 | | | 19.1 | | |
| 86 | | | | | | | | | | | | 19.1 | | |
| 87 | | 1864 | | | | | | | 530 | | | 18.9 | | |
| 88 | | | | | | | | | | | | 18.6 | | |
| 89 | | | | | | | | | | | | 19.1 | | |
| 90 | | | | | | | | | | | | 19.1 | | |
| 91 | | 2160 | | | | | | 96.3 | 473 | | | 19.1 | | |
| 92 | | | | | | | | | | | | 19.1 | | |
| 93 | | | | | | | | | | | | 19.1 | | |
| 94 | | | | | | | | | | | | 19.1 | | |
| 95 | | | | | | | | | | | | 19.1 | | |

| | Feed - analytical | | | | | | | | Permeate - analytical | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| # of days system run | pH | TCOD | sCOD (0.45µm) | TSS (1.5µm) | VSS | Alkalinity (as CaCO3) | Total phosphate | $NH_3$ - N | TCOD | Turbidity | Dissolved methane | Saturation methane | Total phosphate | $NH_3$ - N |
| | | mg/L | mg/L | mg/L | mg/L | mg/L | mg/L | mg/L | mg/L | NTU | mg/L | mg/L | mg/L | mg/L |
| 96 | | | | | | | | | | | | 18.4 | | |
| 97 | | | | | | | | | | 32.4 | | 18.4 | | |
| 98 | | | | | | | | | | 12.8 | | 18.4 | | |
| 99 | | | | | | | | | | | | 18.4 | | |
| 100 | | | | | | | | | | | | 18.4 | | |
| 101 | | | | | | | | | | | | 18.4 | | |
| 102 | | | | | | | | | | | | 18.4 | | |
| 103 | | 2236 | | 820 | 720 | | | | 321 | | | 19.1 | | |
| 104 | | | | | | | | | | | | 19.1 | | |
| 105 | | | | | | | | | | | | 19.1 | | |
| 106 | | | | | | | | | | | | 19.1 | | |
| 107 | | | | | | | | | | | 48.4 | 19.1 | | |
| 108 | | | | | | | | | | 9.3 | | 19.1 | | |
| 109 | | | | | | | | | | | | 19.1 | | |
| 110 | | | | | | | | | | | | 19.4 | | |
| 111 | | | | | | | | | | | | 19.4 | | |
| 112 | | | | | | | | | | | | 19.4 | | |
| 113 | | 1838 | 751 | 1,413 | 1,238 | | | | 202 | | | 19.4 | | |
| 114 | | | | | | | | | | | | 19.4 | | |
| 115 | | | | | | | | | | | | 19.4 | | |
| 116 | | | | | | | | | | | | 19.4 | | |
| 117 | | | | | | | | | | | | 20.2 | | |
| 118 | | | | | | | | | | | | 20.2 | | |
| 119 | | | | | | | | | | | | 20.2 | | |
| 120 | | | | | | | | | | | | 20.2 | | |
| 121 | | 2159 | 732 | 510 | 450 | | | | 134 | 50.2 | | 20.2 | | |
| 122 | | | | | | | | 156.9 | | | | 20.2 | | |
| 123 | 6.7 | | | | | 662.1 | | | | | | 20.2 | | |
| 124 | | | | | | | 99.1 | | | | | 19.7 | | |
| 125 | | 3540 | 972 | 2,150 | 1,912 | | | | 160 | 98.6 | | 19.7 | | |
| 126 | | | | | | | | | | | | 19.7 | | |
| 127 | | | | | | | | | | | | 19.7 | | |
| 128 | | | | | | | | | | | | 19.7 | | |

| | Feed - analytical | | | | | | | | Permeate - analytical | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| # of days system run | pH | TCOD | sCOD (0.45µm) | TSS (1.5µm) | VSS | Alkalinity (as CaCO3) | Total phosphate | $NH_3$ - N | TCOD | Turbidity | Dissolved methane | Saturation methane | Total phosphate | $NH_3$ - N |
| | | mg/L | **mg/L** | **mg/L** | **mg/L** | **mg/L** | **mg/L** | **mg/L** | **mg/L** | **NTU** | **mg/L** | **mg/L** | **mg/L** | **mg/L** |
| 129 | | | | | | | | | | | | 19.7 | | |
| 130 | | | | | | | | | | | | 19.7 | | |
| 131 | | 3396 | 743.00 | 1,463 | 1,312 | | | | 177 | 101 | | 19.9 | | |
| 132 | | | | | | | | | | 87.4 | | 19.9 | | |
| 133 | | | | | | | | | | | | 19.9 | | |
| 134 | | | | | | | | | | | | 19.9 | | |
| 135 | | | | 925 | 825 | | | | | | | 19.9 | | |
| 136 | | | | | | | | | | | | 19.9 | | |
| 137 | | | | | | | | | | | | 19.9 | | |
| 138 | | 2813 | 919 | 1,438 | 1,300 | | | | 167 | 78.2 | | 19.4 | | |
| 139 | | | | | | | | | | | | 19.4 | | |
| 140 | | | | | | | | | | | | 19.4 | | |
| 141 | | | | | | | | | | | | 19.4 | | |
| 142 | | | | | | | | | | | | 19.4 | | |
| 143 | | 3798 | 773 | 2,275 | 1,938 | | | | 166 | 87.2 | | 19.4 | | |
| 144 | | | | | | | | | | | | 19.4 | | |
| 145 | | | | | | | | | | | | 19.7 | | |
| 146 | | | | | | | | | | | | 19.7 | | |
| 147 | | 3722 | 1009 | 1,300 | 1,188 | | | | 209 | | | 19.7 | | |
| 148 | | | | | | | | | | | | 19.7 | | |
| 149 | | | | | | | | | | | | 19.7 | | |
| 150 | | | | | | | | | | | | 19.7 | | |
| 151 | | | | | | | | | | 7.4 | | 19.7 | | |
| 152 | | 2665 | 672 | 1,325 | 1,200 | | | | 204 | | | 20.4 | | |
| 153 | | 3248 | 820 | 2,137 | 2,000 | | | | 101 | 38.6 | | 20.4 | | |
| 154 | | | | | | | | | | | | 20.4 | | |
| 155 | | | | | | | | | | | | 20.4 | | |
| 156 | | | | | | | | | | | | 20.4 | | |
| 157 | | | | | | | | | | | | 20.4 | | |
| 158 | | | | | | | | | | | | 20.4 | | |
| 159 | | | | | | | | | | | | 19.1 | | |
| 160 | | | | | | | | | | | | 19.1 | | |
| 161 | | | | | | | | | | | | 19.1 | | |

| | Feed - analytical | | | | | | | | Permeate - analytical | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| # of days system run | pH | TCOD | sCOD (0.45µm) | TSS (1.5µm) | VSS | Alkalinity (as CaCO3) | Total phosphate | $NH_3$ - N | TCOD | Turbidity | Dissolved methane | Saturation methane | Total phosphate | $NH_3$ - N |
| | | mg/L | **mg/L** | **mg/L** | **mg/L** | **mg/L** | **mg/L** | **mg/L** | **mg/L** | **NTU** | **mg/L** | **mg/L** | **mg/L** | **mg/L** |
| 162 | | 3289 | 712 | 1,838 | 1,663 | | | | 106 | 52.4 | | 19.1 | | |
| 163 | | | | | | | | | | | | 19.1 | | |
| 164 | 6.7 | | | | | 725.6 | | | | | 36.0 | 19.1 | | |
| 165 | | | | | | | | | | | | 19.1 | | |
| 166 | | | | | | | | | | | | 19.1 | | |
| 167 | | | | | | | | 44.2 | | | | 19.1 | | |
| 168 | | | | | | | | | | | | 19.1 | | |
| 169 | | | | | | | | | | | | 19.1 | | |
| 170 | | 1916 | 587 | 988 | 875 | | 73.8 | | 140 | | | 19.1 | 55.2 | |
| 171 | | | | | | | | | | | | 19.1 | | |
| 172 | | | | | | | | | | | | 19.1 | | |
| 173 | | 2338 | 655 | 1,013 | 888 | | | | 219 | | | 19.1 | | |
| 174 | | | | | | | | | | 76.8 | | 19.1 | | |
| 175 | | | | | | | | | | | | 19.1 | | |
| 176 | | | | | | | | | | | | 19.1 | | |
| 177 | | | | | | | | | | | | 19.1 | | |
| 178 | | | | | | | | | | | | 19.1 | | |
| 179 | | | | | | | | | | | | 19.1 | | |
| 180 | | 3008 | 1055 | 1,587 | 1,375 | | | | 206 | 12.7 | | 18.6 | | |
| 181 | | | | | | | | | | | | 18.6 | | |
| 182 | | | | | | | | | | | | 18.6 | | |
| 183 | | | | | | | | | | | | 18.6 | | |
| 184 | | | | | | | | | | | | 18.6 | | |
| 185 | | | | | | | | | | | | 18.6 | | |
| 186 | | | | | | | | | | | | 18.6 | | |

| | Feed - analytical | | | | | | | | Permeate - analytical | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| # of days system run | pH | TCOD | sCOD (0.45µm) | TSS (1.5µm) | VSS | Alkalinity (as CaCO3) | Total phosphate | $NH_3$ - N | TCOD | Turbidity | Dissolved methane | Saturation methane | Total phosphate | $NH_3$ - N |
| | | mg/L | **mg/L** | **mg/L** | **mg/L** | **mg/L** | **mg/L** | **mg/L** | **mg/L** | **NTU** | **mg/L** | **mg/L** | **mg/L** | **mg/L** |
| 187 | 6.6 | 3402 | 1147 | 1,720 | 1,530 | | 138.4 | | 187 | | | 18.6 | 82.2 | |
| 188 | | | | | | | | | | | | 18.6 | | |
| 189 | | | | | | | | | | | | 18.6 | | |
| 190 | | | | | | | | | | | | 18.6 | | |
| 191 | | | | | | | | | | 48.3 | | 18.6 | | |
| 192 | | | | | | | | | | | | 18.6 | | |
| 193 | | | | | | | | | | | | 18.6 | | |
| 194 | | | | | | | | | | | | 19.9 | | |
| 195 | | | | | | | | | | | | 19.9 | | |
| 196 | | | | | | | | | | | | 19.9 | | |
| 197 | | | | | | | | | | | | 19.9 | | |
| 198 | | 2116 | 706 | 980 | 860 | | | | 186 | | | 19.9 | | |
| 199 | | | | | | | | | | | | 19.9 | | |
| 200 | | | | | | | | | | 30.8 | 17.7 | 19.9 | | |
| 201 | | | | | | | | | | | | 19.4 | | |
| 202 | | | | | | | | | | | | 19.4 | | |
| 203 | | | | | | | | | | | | 19.4 | | |
| 204 | | | | | | | | | | | | 19.4 | | |
| 205 | 6.8 | | | | | | | | | | | 19.4 | | |
| 206 | | | | | | | | | | | | 19.4 | | |
| 207 | | | | | | | | | | | | 19.4 | | |
| 208 | | | | | | | | | | | | 18.9 | | |
| 209 | | 1609 | 645 | 820 | 760 | | | | 149 | | | 18.9 | | |
| 210 | | | | | | | | | | | | 18.9 | | |
| 211 | | 1825 | 577 | 1,160 | 1,020 | | | | 313 | 46.2 | | 18.9 | | |
| 212 | | | | | | | | | | | | 18.9 | | |
| 213 | | | | | | | | | | | | 18.9 | | |
| 214 | 6.8 | 2242 | 669 | 1,560 | 1,400 | | | | 453 | 28.5 | | 18.9 | | |
| 215 | | | | | | | 67.8 | | | | | 18.9 | 51.8 | |

| | Feed - analytical | | | | | | | | Permeate - analytical | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| # of days system run | pH | TCOD | sCOD (0.45µm) | TSS (1.5µm) | VSS | Alkalinity (as CaCO3) | Total phosphate | $NH_3$ - N | TCOD | Turbidity | Dissolved methane | Saturation methane | Total phosphate | $NH_3$ - N |
| | | mg/L | **mg/L** | **mg/L** | **mg/L** | **mg/L** | **mg/L** | **mg/L** | **mg/L** | **NTU** | **mg/L** | **mg/L** | **mg/L** | **mg/L** |
| 216 | | | | | | | | | | | | 18.9 | | |
| 217 | | | | | | | | 74.1 | | | | 18.9 | | 110.35 |
| 218 | 6.7 | 2698 | 1007 | 1,380 | 1,260 | 720.1 | | | 386 | 4.8 | 26.5 | 18.9 | | |
| 219 | | | | | | | | | | | | 18.9 | | |
| 220 | | | | | | | | | | | | 18.9 | | |
| 221 | | | | | | | | | | | | 18.9 | | |
| 222 | | | | | | | | | | | | 19.7 | | |
| 223 | | 3556 | 1369 | 2,580 | 2,260 | | | | 349 | 12.7 | 26.3 | 19.7 | | |
| 224 | | 2864 | 1794 | 940 | 820 | | | | 498 | | | 19.7 | | |
| 225 | | | | | | | | | | | 30.6 | 19.7 | | |
| 226 | | | | | | | | | | | | 17.3 | | |
| 227 | | | | | | | | | | | | 17.3 | | |
| 228 | | | | | | | 114.3 | | | | | 17.3 | 71.6 | |
| 229 | | 3123 | 1,657 | 880 | 790 | | | | 483 | | | | | |
| 230 | | | | | | | | | | | | 17.3 | | |
| 231 | | | | | | | | | | | | 17.3 | | |
| 232 | | | | | | | | | | | | 17.3 | | |
| 233 | | | | | | | | | | | | 17.3 | | |
| 234 | 6.7 | | | | | | | | | 2.4 | | 18.1 | | |
| 235 | | 4904 | 2,114 | 1,300 | 1,180 | 982.8 | | | 352 | | | 18.1 | | |
| 236 | | | | | | | | | | | | 18.1 | | |
| 237 | | | | | | | | | | | | 18.6 | | |
| 238 | | | | | | | | | | | | 18.6 | | |
| 239 | | | | | | | | | | | | 18.6 | | |
| 240 | | | | | | | | | | | | 18.6 | | |
| 241 | | | | | | | | | | | | 18.6 | | |
| 242 | | 3361 | 1,983 | 860 | 760 | | | | 362 | 4.8 | | 18.6 | | |
| 243 | | | | | | | | | | | | 19.4 | | |
| 244 | | | | | | | | | | | | 19.4 | | |
| 245 | | | | | | | | | | | | 19.4 | | |
| 246 | 6.8 | 1733 | 916 | 1,020 | 930 | | 159.3 | 94.3 | 336 | | 17.0 | 19.4 | 59.4 | 156.5 |
| 247 | | | | | | | | | | | | 19.4 | | |
| 248 | | | | | | | | | | | | 19.4 | | |

| | Feed - analytical | | | | | | | | Permeate - analytical | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| # of days system run | pH | TCOD | sCOD (0.45µm) | TSS (1.5µm) | VSS | Alkalinity (as CaCO3) | Total phosphate | $NH_3$ - N | TCOD | Turbidity | Dissolved methane | Saturation methane | Total phosphate | $NH_3$ - N |
| | | mg/L | **mg/L** | **mg/L** | **mg/L** | **mg/L** | **mg/L** | **mg/L** | **mg/L** | **NTU** | **mg/L** | **mg/L** | **mg/L** | **mg/L** |
| 249 | | | | | | | | | | | | 18.6 | | |
| 250 | | | | | | 1098.5 | | | | | | 18.6 | | |
| 251 | | | | | | | | | | | | 18.6 | | |
| 252 | | | | | | | | | | | | 18.6 | | |
| 253 | | 4149 | 2,471 | 920 | 830 | | | | 305 | 19.6 | | 18.6 | | |
| 254 | | | | | | | | | | | 24.8 | 18.6 | | |
| 255 | | | | | | | | | | | | 18.6 | | |
| 256 | | | | | | | | | | | | 18.6 | | |
| 257 | | | | | | | | | | | | 18.6 | | |
| 258 | | | | | | | | | | | | 19.1 | | |
| 259 | | | | | | | | | | | | 19.1 | | |
| 260 | | | | | | | | | | | | 19.1 | | |
| 261 | | | | | | | | 78 | | | | 19.1 | | 103.5 |
| 262 | 6.9 | | | | | | | | | | | 19.1 | | |
| 263 | | 2395 | 1165 | 1010 | 880 | | | | 296 | | | 19.1 | | |
| 264 | | | | | | | | | | | | | | |

**Table A-2: Reactor analytical data and operational data**

| | Reactor - analytical | | | | | | | Operational data | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| # of days system run | TCOD | sCOD (0.45) | MLSS (1.5µm) | MLVSS | MLVSS/MLSS | Alkalinity (as CaCO3) | VFAs | Tank temp | pH, reactor | pH, permeate | Permeate pumped | Calculated flux | Biogas | $CH_4$ in biogas | $CH_4$ produced |
| | mg/L | mg/L | mg/L | mg/L | | mg/L | mg/L | deg C | | | L/d | LMH | mL | % | mL |
| 1 | | | | | | | | 24.0 | 6.9 | 7.3 | 0.86 | 1.11 | 0 | 37 | 0 |
| 2 | 9,317 | 661 | 3,500 | 2,800 | 0.80 | | | 24.0 | 6.9 | 7.2 | 0.87 | 1.13 | 0 | 46 | 0 |
| 3 | | | | | | | | 24.0 | 6.8 | 7.2 | 0.92 | 1.19 | 638 | 51 | 326 |
| 4 | 8,072 | 370 | 3,100 | 2,500 | 0.81 | | | 24.0 | 6.7 | 7.2 | 0.87 | 1.13 | 729 | 58 | 423 |
| 5 | 6,596 | 213 | 2,800 | 2,200 | 0.79 | | | 24.0 | 6.7 | 7.2 | 0.87 | 1.12 | 698 | 64 | 447 |
| 6 | | | 3,700 | 3,300 | 0.89 | | | 24.0 | 6.8 | 7.3 | 0.86 | 1.11 | 350 | 67 | 235 |
| 7 | | | | | | | | 24.0 | 6.8 | 7.2 | 0.86 | 1.11 | 444 | 70 | 311 |
| 8 | | | | | | | | 24.0 | 6.8 | 7.1 | 0.88 | 1.14 | 367 | 64 | 235 |
| 9 | | | 3,100 | 2,800 | 0.90 | | | 24.0 | 6.8 | 7.1 | 0.89 | 1.15 | 648 | 62 | 402 |
| 10 | | | | | | | | 24.0 | 6.7 | 7.0 | 0.88 | 1.13 | 396 | 66 | 261 |
| 11 | | | | | | | | 24.0 | 6.7 | 7.0 | 0.93 | 1.20 | 323 | 68 | 219 |
| 12 | | | 2,800 | 2,700 | 0.96 | | | 24.0 | 6.7 | 7.0 | 0.86 | 1.12 | 284 | 70 | 199 |
| 13 | | | | | | | | 24.0 | 6.6 | 6.9 | 0.88 | 1.14 | 423 | 68 | 288 |
| 14 | | | | | | | | | | | | 1.12 | 241 | 67 | 162 |
| 15 | | | | | | | | 23.0 | 6.9 | 7.2 | 0.86 | 1.12 | 194 | 68 | 132 |
| 16 | 4,881 | | 2,300 | 1,900 | 0.83 | | | 24.0 | 6.9 | 7.2 | 0.87 | 1.12 | 182 | 69 | 125 |
| 17 | | 171 | | | | | | 23.0 | 6.8 | 7.2 | 0.90 | 1.17 | 135 | 69 | 93 |
| 18 | | | | | | | | 22.0 | 6.7 | 7.1 | 0.90 | 1.17 | 133 | 69 | 92 |
| 19 | 4,374 | 86 | 3,100 | 2,500 | 0.81 | | | 23.0 | 7.0 | 7.4 | 0.83 | 1.08 | 152 | 69 | 105 |
| 20 | | | | | | | | 23.0 | 7.0 | 7.3 | 0.89 | 1.15 | 208 | 68 | 141 |
| 21 | | | | | | | | 23.0 | 7.0 | 7.3 | 0.87 | 1.13 | 246 | 68 | 167 |
| 22 | | | | | | | | 24.0 | 6.9 | 7.3 | 0.89 | 1.15 | 157 | 68 | 107 |
| 23 | 4,365 | 152 | 2,400 | 2,000 | 0.83 | | | 24.0 | 6.9 | 7.4 | 0.90 | 1.16 | 111 | 69 | 76 |
| 24 | | | | | | | | 23.0 | 7.0 | 7.4 | 0.88 | 1.13 | 106 | 70 | 74 |
| 25 | 3,787 | 177 | | | | | | 23.0 | 7.1 | 7.4 | 0.89 | 1.16 | 343 | 70 | 240 |
| 26 | 4,632 | 220 | | | | | | 23.0 | 7.0 | 7.4 | 0.87 | 1.13 | 542 | 71 | 385 |
| 27 | | | | | | | | 23.0 | 7.0 | 7.3 | 0.91 | 1.17 | 178 | 70 | 124 |
| 28 | | | | | | | | 24.0 | 6.9 | 7.3 | 0.91 | 1.18 | 804 | 71 | 571 |
| 29 | | | | | | | | 25.0 | 6.9 | 7.3 | 0.91 | 1.18 | 786 | 71 | 558 |
| 30 | 4,036 | 279 | | | | | | 26.0 | 6.9 | 7.2 | 0.87 | 1.12 | 623 | 70 | 436 |
| 31 | | | | | | | | 26.0 | 6.8 | 7.2 | 0.89 | 1.15 | 649 | 72 | 467 |

| | Reactor - analytical | | | | | | | Operational data | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| # of days system run | TCOD | sCOD (0.45) | MLSS (1.5µm) | MLVSS | MLVSS/MLSS | Alkalinity (as CaCO3) | VFAs | Tank temp | pH, reactor | pH, permeate | Permeate pumped | Calculated flux | Biogas | $CH_4$ in biogas | $CH_4$ produced |
| | mg/L | mg/L | mg/L | mg/L | | mg/L | mg/L | deg C | | | L/d | LMH | mL | % | mL |
| 32 | | | | | | | | 25.0 | 6.9 | 7.1 | 0.85 | 1.11 | 643 | 71 | 456 |
| 33 | 4,221 | 268 | 2,500 | 2,200 | 0.88 | | | 25.0 | 6.9 | 7.2 | 0.89 | 1.15 | 504 | 71 | 358 |
| 34 | | | | | | | | 25.0 | 6.9 | 7.2 | 0.88 | 1.14 | 436 | 70 | 305 |
| 35 | | | | | | | | 24.0 | 6.8 | 7.2 | | 1.12 | 558 | 70 | 391 |
| 36 | 2,943 | 196 | 2,000 | 1,800 | 0.90 | | | 24.0 | 6.8 | 7.2 | 0.86 | 1.11 | 571 | 72 | 411 |
| 37 | 2,854 | 334 | 2,100 | 1,900 | 0.90 | 1134.3 | | 24.0 | 6.9 | 7.2 | 0.91 | 1.18 | 554 | 79 | 438 |
| 38 | | | | | | | | 24.0 | 7.0 | 7.3 | 0.89 | 1.15 | 717 | 74 | 531 |
| 39 | | | | | | | | 23.0 | 7.0 | 7.2 | 0.89 | 1.15 | 752 | 72 | 541 |
| 40 | 3,254 | 212 | 1,800 | 1,600 | 0.89 | 1050.9 | | 22.0 | 6.9 | 7.1 | 0.89 | 1.16 | 645 | 71 | 458 |
| 41 | | | | | | | | 23.0 | 7.0 | 7.3 | 0.92 | 1.19 | 492 | 79 | 388 |
| 42 | | | | | | | | 23.0 | 6.9 | 7.2 | | 1.15 | 497 | 75 | 373 |
| 43 | 3,200 | 270 | 1,800 | 1,600 | 0.89 | 1151.0 | | 23.0 | 6.9 | 7.2 | 0.88 | 1.14 | 527 | 72 | 379 |
| 44 | | | | | | | | 23.0 | 6.9 | 7.3 | | 1.14 | 520 | 74 | 385 |
| 45 | | | | | | | | 23.0 | 6.9 | 7.3 | 0.90 | 1.16 | 543 | 72 | 391 |
| 46 | | | | | | | | 23.0 | 6.9 | 7.4 | 0.89 | 1.15 | 474 | 75 | 356 |
| 47 | 3,040 | 246 | 1,600 | 1,300 | 0.81 | | | 23.0 | 6.9 | 7.3 | 0.85 | 1.10 | 321 | 74 | 238 |
| 48 | | | | | | | | 23.0 | 6.9 | 7.4 | 0.90 | 1.17 | 205 | 66 | 135 |
| 49 | | | | | | | | 23.0 | 6.9 | 7.3 | | 1.15 | 265 | 66 | 175 |
| 50 | | | | | | | | 23.0 | 6.9 | 7.3 | 0.88 | 1.14 | 235 | 70 | 165 |
| 51 | 2,818 | 443 | 1,700 | 1,500 | 0.88 | 1209.4 | | 23.0 | 7.0 | 7.5 | 0.90 | 1.16 | 344 | 68 | 234 |
| 52 | | | | | | | | 23.0 | 7.0 | 7.4 | 0.89 | 1.15 | 348 | 70 | 244 |
| 53 | | | | | | | | | 7.0 | 7.4 | | 1.16 | 476 | 70 | 333 |
| 54 | 2,543 | 327 | 1,600 | 1,300 | 0.81 | 1267.8 | | 23.0 | 6.9 | 7.3 | 0.91 | 1.18 | 514 | 70 | 360 |
| 55 | | | | | | | | 23.0 | 7.0 | 7.3 | | 1.15 | 418 | 71 | 297 |
| 56 | | | | | | | | 23.0 | 7.0 | 7.3 | 0.88 | 1.14 | 371 | 72 | 267 |
| 57 | | | | | | | | 23.0 | 7.0 | 7.2 | 0.89 | 1.15 | 368 | 76 | 279 |
| 58 | 2,374 | 313 | 1200 | 1000 | 0.83 | 1411.2 | | 23.0 | 7.0 | 7.3 | 0.89 | 1.15 | 401 | 74 | 297 |
| 59 | | | | | | | | | 6.9 | 7.2 | | 1.15 | 395 | 72 | 285 |
| 60 | | | | | | | | 23.0 | 6.9 | 7.2 | 0.90 | 1.16 | 380 | 74 | 281 |
| 61 | 2,338 | 499 | 1,100 | 800 | 0.73 | 1311.2 | | 23.0 | 6.9 | 7.2 | 0.90 | 1.16 | 254 | 72 | 183 |
| 62 | | | | | | | | 23.0 | 6.9 | 7.3 | 0.89 | 1.15 | 238 | 70 | 166 |
| 63 | | | | | | | | | 6.9 | 7.3 | | 1.14 | 234 | 72 | 168 |
| 64 | | | | | | | | | 6.9 | 7.2 | | 1.14 | 236 | 74 | 174 |

| | Reactor - analytical | | | | | | | Operational data | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| # of days system run | TCOD | sCOD (0.45) | MLSS (1.5µm) | MLVSS | MLVSS/MLSS | Alkalinity (as CaCO3) | VFAs | Tank temp | pH, reactor | pH, permeate | Permeate pumped | Calculated flux | Biogas | $CH_4$ in biogas | $CH_4$ produced |
| | mg/L | mg/L | mg/L | mg/L | | mg/L | mg/L | deg C | | | L/d | LMH | mL | % | mL |
| 65 | 2,507 | 303 | 1,000 | 800 | 0.80 | 1210.2 | | 23.0 | 6.9 | 7.2 | 0.88 | 1.13 | 196 | 75 | 147 |
| 66 | | | | | | | | | 6.8 | 7.3 | | 1.14 | 200 | 72 | 144 |
| 67 | | | | | | 1241.1 | 187.6 | 23.0 | 6.9 | 7.3 | 0.88 | 1.14 | 182 | 74 | 135 |
| 68 | 2,951 | 356 | 1,300 | 1,100 | 0.85 | | | 23.0 | 6.9 | 7.3 | 0.89 | 1.15 | 176 | 60 | 106 |
| 69 | | | | | | | | | 6.8 | 7.2 | | 1.14 | 240 | 65 | 156 |
| 70 | | | | | | | | 23.0 | 6.9 | 7.3 | 0.89 | 1.15 | 254 | 68 | 173 |
| 71 | 2,054 | 276 | 1,600 | 1,400 | 0.88 | 1234.4 | 183.6 | 23.0 | 6.9 | 7.3 | 0.88 | 1.14 | 363 | 69 | 250 |
| 72 | | | | | | | | 22.0 | | | | 1.15 | 274 | 68 | 187 |
| 73 | | | | | | | | 22.0 | | | 0.88 | 1.14 | 259 | 68 | 176 |
| 74 | | | | | | | | 22.0 | 6.9 | 7.3 | 0.86 | 1.12 | 162 | 68 | 110 |
| 75 | 2,356 | 314 | 1100 | 800 | 0.73 | 1204.2 | 147.2 | 22.0 | 6.9 | 7.3 | 0.90 | 1.16 | 212 | 69 | 146 |
| 76 | | | | | | | | 22.0 | 6.8 | 7.2 | 2.37 | 3.06 | 438 | 69 | 303 |
| 77 | | | | | | | | 22.0 | 6.9 | 7.2 | 2.38 | 3.08 | 333 | 69 | 230 |
| 78 | | | | | | | | 22.0 | 6.9 | 7.3 | 2.36 | 3.05 | 291 | 70 | 203 |
| 79 | | | | | | | | 23.0 | 6.9 | 7.3 | 2.38 | 3.08 | 512 | 69 | 353 |
| 80 | | | | | | | | 23.0 | 6.8 | 7.2 | 2.37 | 3.07 | 818 | 71 | 581 |
| 81 | | | | | | 1338.7 | 374.2 | 23.0 | 6.9 | 7.2 | 2.36 | 3.05 | 849 | 70 | 595 |
| 82 | 3,718 | 334 | 1,000 | 900 | 0.90 | | | 23.0 | 6.9 | 7.2 | 2.38 | 3.08 | 1118 | 72 | 805 |
| 83 | | | | | | | | 23.0 | 6.9 | 7.2 | | 3.06 | 1005 | 71 | 714 |
| 84 | | | | | | | | 23.0 | 6.9 | 7.2 | 2.36 | 3.05 | 994 | 72 | 716 |
| 85 | | | 1,400 | 1,100 | 0.79 | | | 23.0 | 6.8 | 7.1 | 2.46 | 3.19 | 968 | 74 | 716 |
| 86 | | | | | | | | | 6.8 | 7.1 | | 3.09 | 993 | 74 | 735 |
| 87 | | | | | | | | 23.0 | 6.8 | 7.1 | 2.39 | 3.09 | 1090 | 73 | 796 |
| 88 | | | | | | | | 23.0 | 6.9 | 7.2 | | 3.14 | 1111 | 72 | 800 |
| 89 | | | | | | | | 23.0 | 6.9 | 7.2 | | 3.12 | 1097 | 74 | 812 |
| 90 | | | | | | | | 23.0 | 6.9 | 7.3 | 2.44 | 3.15 | 1181 | 74 | 874 |
| 91 | | | | | | | | | | | 2.42 | 3.15 | 1106 | 74 | 819 |
| 92 | | | | | | | | | | | | 3.12 | 1012 | 74 | 749 |
| 93 | | | | | | | | 23.0 | 6.9 | 7.2 | 2.38 | 3.08 | 859 | 74 | 635 |
| 94 | | | | | | | | | | | | 3.14 | 892 | 74 | 660 |
| 95 | | | | | | | | 23.0 | 6.9 | 7.2 | 2.46 | 3.18 | 718 | 74 | 531 |
| 96 | | | | | | | | | | | | 3.16 | 912 | 71 | 647 |
| 97 | | | | | | | | 23.0 | 6.9 | 7.1 | | 3.15 | 953 | 71 | 677 |

| | Reactor - analytical | | | | | | | Operational data | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| # of days system run | TCOD | sCOD (0.45) | MLSS (1.5µm) | MLVSS | MLVSS/MLSS | Alkalinity (as CaCO3) | VFAs | Tank temp | pH, reactor | pH, permeate | Permeate pumped | Calculated flux | Biogas | $CH_4$ in biogas | $CH_4$ produced |
| | mg/L | mg/L | mg/L | mg/L | | mg/L | mg/L | deg C | | | L/d | LMH | mL | % | mL |
| 98 | | | | | | | | | | | | 3.14 | 984 | 71 | 699 |
| 99 | | | | | | | | 23.0 | 6.9 | 7.2 | 2.40 | 3.11 | 1005 | 71 | 714 |
| 100 | | | | | | | | | | | | 3.14 | 916 | 71 | 650 |
| 101 | | | | | | | | 23.0 | 6.9 | 7.2 | 2.44 | 3.16 | 875 | 71 | 622 |
| 102 | | | | | | | | | | | | 3.13 | 976 | 71 | 693 |
| 103 | 3,490 | 306 | 1,625 | 1,250 | 0.77 | | | | | | 2.41 | 3.12 | 1047 | 74 | 775 |
| 104 | | | | | | | | 23.0 | 6.8 | 7.1 | 2.43 | 3.14 | 1074 | 74 | 794 |
| 105 | | | | | | | | 23.0 | 6.8 | 7.1 | 2.40 | 3.10 | 1019 | 74 | 754 |
| 106 | | | | | | | | | | | | 3.12 | 976 | 74 | 722 |
| 107 | | | | | | | | 23.0 | 6.8 | 7.1 | | 3.12 | 933 | 74 | 690 |
| 108 | | | | | | | | | | | | 3.16 | 990 | 74 | 733 |
| 109 | | | | | | | | | | | | 3.17 | 983 | 74 | 728 |
| 110 | | | | | | | | 23.0 | 6.8 | 7.1 | | 3.15 | 968 | 75.0 | 726 |
| 111 | | | | | | | | | | | | 3.15 | 956 | 75.0 | 717 |
| 112 | | | | | | | | 24.0 | 6.9 | 7.1 | | 3.16 | 1041 | 75.0 | 781 |
| 113 | 3,261 | 329 | 1,800 | 1,400 | 0.78 | | | | | | 2.44 | | 1003 | 75.0 | 752 |
| 114 | | | | | | | | 23.0 | 6.9 | 7.1 | | 3.15 | 951 | 75.0 | 714 |
| 115 | | | | | | | | | | | | 3.14 | 920 | 75.0 | 690 |
| 116 | | | | | | | | 23.0 | 6.8 | 7.0 | | 3.15 | 905 | 75.0 | 679 |
| 117 | | | | | | | | | | | | | 931 | 78.0 | 726 |
| 118 | | | | | | | | | | | | 3.16 | 879 | 78.0 | 686 |
| 119 | | | | | | | | 23.0 | 6.8 | 7.1 | 2.46 | 3.18 | 518 | 78.0 | 404 |
| 120 | | | | | | | | 23.0 | | | | | 808 | 78.0 | 630 |
| 121 | 4,209 | 393 | 2,200 | 1,700 | 0.77 | | | 24.0 | | | 2.43 | 3.16 | 891 | 78.0 | 695 |
| 122 | | | | | | | | 24.0 | | | | | 802 | 78.0 | 625 |
| 123 | | | | | | 1351.2 | 272.7 | 24.0 | 6.8 | 7.0 | | 3.15 | 814 | 78.0 | 635 |
| 124 | | | | | | | | 24.0 | | | | | 919 | 76.0 | 698 |
| 125 | 4,178 | 283 | 2,400 | 1,950 | 0.81 | | | 24.0 | | | 2.43 | 3.15 | 946 | 76.0 | 719 |
| 126 | | | | | | | | 24.0 | 6.8 | 7.1 | 2.43 | 3.15 | 918 | 76.0 | 697 |
| 127 | | | | | | | | | | | | | | 76.0 | |
| 128 | | | | | | | | 24.0 | 6.9 | 7.1 | | 3.15 | 943 | 76.0 | 717 |
| 129 | | | | | | | | | | | | | | 76.0 | |
| 130 | | | | | | | | 24.0 | 6.9 | 7.0 | 2.47 | 3.19 | 972 | 76.0 | 739 |

| | Reactor - analytical | | | | | | | Operational data | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| # of days system run | TCOD | sCOD (0.45) | MLSS (1.5µm) | MLVSS | MLVSS/MLSS | Alkalinity (as CaCO3) | VFAs | Tank temp | pH, reactor | pH, permeate | Permeate pumped | Calculated flux | Biogas | $CH_4$ in biogas | $CH_4$ produced |
| | mg/L | mg/L | mg/L | mg/L | | mg/L | mg/L | deg C | | | L/d | LMH | mL | % | mL |
| 131 | 3,702 | 241 | 1,950 | 1,550 | 0.79 | | | | | | 2.43 | 3.16 | 943 | 77.0 | 726 |
| 132 | | | | | | | | | | | | 3.16 | 961 | 77.0 | 740 |
| 133 | | | | | | | | 24.0 | 6.9 | 7.1 | 2.43 | 3.15 | 872 | 77.0 | 671 |
| 134 | | | | | | | | 24.0 | 6.9 | 7.1 | 2.47 | 3.19 | 674 | 77.0 | 519 |
| 135 | | | 1,950 | 1,550 | 0.79 | | | 23.0 | 6.9 | 7.1 | 2.46 | 3.19 | 722 | 77.0 | 556 |
| 136 | | | | | | | | | | | | | 807 | 77.0 | 621 |
| 137 | | | | | | | | 24.0 | 6.9 | 7.1 | 2.46 | 3.18 | 737 | 77.0 | 568 |
| 138 | 3,902 | 382 | 2,100 | 1,600 | 0.76 | | | | | | 2.44 | 3.18 | 759 | 75.0 | 570 |
| 139 | | | | | | | | 24.0 | 6.9 | 7.1 | 2.46 | 3.19 | 704 | 75.0 | 528 |
| 140 | | | | | | | | | | | | 3.18 | 761 | 75.0 | 570 |
| 141 | | | | | | | | 25.0 | 6.9 | 7.1 | | 3.16 | 848 | 75.0 | 636 |
| 142 | | | | | | | | 25.0 | | | | 3.16 | 822 | 75.0 | 617 |
| 143 | 3,631 | 312 | 2,400 | 2,000 | 0.83 | | | 25.0 | 6.9 | 7.0 | 2.43 | 3.16 | 939 | 75.0 | 704 |
| 144 | | | | | | | | 25.0 | | | | 3.16 | 931 | 75.0 | 698 |
| 145 | | | | | | | | 24.0 | 6.9 | 7.0 | 2.40 | 3.11 | 885 | 76.0 | 672 |
| 146 | | | | | | | | 23.0 | | | | 3.19 | 987 | 76.0 | 750 |
| 147 | 3,687 | 487 | 1,500 | 1,200 | 0.80 | | | 23.0 | 6.9 | 7.1 | 2.42 | 3.16 | 1095 | 76.0 | 832 |
| 148 | | | | | | | | | | | | 3.16 | 1123 | 76.0 | 854 |
| 149 | | | | | | | | 24.0 | 6.8 | 7.0 | 2.46 | 3.19 | 1178 | 76.0 | 895 |
| 150 | | | | | | | | | | | | 3.18 | 1140 | 76.0 | 866 |
| 151 | | | | | | | | 24.0 | 6.9 | 7.0 | | 3.16 | 1152 | 76.0 | 875 |
| 152 | 3,580 | 244 | 2,300 | 1,800 | 0.78 | | | | | | 2.43 | 3.16 | 1092 | 79.0 | 863 |
| 153 | 3,300 | 354 | 1,750 | 1,450 | 0.83 | | | 24.0 | 6.9 | 7.1 | 2.40 | 3.11 | 1148 | 79.0 | 907 |
| 154 | | | | | | | | | | | | 3.15 | 1038 | 79.0 | 820 |
| 155 | | | | | | | | 24.0 | 6.9 | 7.1 | 2.46 | 3.18 | 1019 | 79.0 | 805 |
| 156 | | | | | | | | | 6.9 | 7.1 | | 3.16 | 1014 | 79.0 | 801 |
| 157 | | | | | | | | | 6.9 | 7.1 | | 3.18 | 983 | 79.0 | 777 |
| 158 | | | | | | | | 25.0 | 6.9 | 7.1 | 2.46 | 3.18 | 946 | 79.0 | 748 |
| 159 | | | | | | | | | 6.9 | 7.1 | | 3.18 | 1031 | 74.0 | 763 |
| 160 | | | | | | | | 25.0 | 6.9 | 7.1 | | 3.18 | 1100 | 74.0 | 814 |
| 161 | | | | | | | | | 6.9 | 7.1 | | 3.18 | 1146 | 74.0 | 848 |
| 162 | 3,875 | 157 | 2,750 | 2,300 | 0.84 | | | 26.0 | 6.9 | 7.1 | 2.47 | 3.20 | 1177 | 74.0 | 871 |

| | Reactor - analytical | | | | | | | Operational data | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| # of days system run | TCOD | sCOD (0.45) | MLSS (1.5µm) | MLVSS | MLVSS/MLSS | Alkalinity (as CaCO3) | VFAs | Tank temp | pH, reactor | pH, permeate | Permeate pumped | Calculated flux | Biogas | $CH_4$ in biogas | $CH_4$ produced |
| | mg/L | mg/L | mg/L | mg/L | | mg/L | mg/L | deg C | | | L/d | LMH | mL | % | mL |
| 163 | | | | | | | | 25.0 | 6.9 | 7.1 | 2.48 | 3.21 | 974 | 74.0 | 721 |
| 164 | | | | | | 1281.1 | 211.4 | | | | | 3.19 | 1012 | 74.0 | 749 |
| 165 | | | | | | | | 25.0 | 6.9 | 7.1 | 2.45 | 3.18 | 1051 | 74.0 | 778 |
| 166 | | | | | | | | | 6.9 | 7.1 | | 3.19 | 1059 | 74.0 | 784 |
| 167 | | | | | | | | 25.0 | 6.9 | 7.1 | 2.47 | 3.20 | 1009 | 74.0 | 747 |
| 168 | | | | | | | | | 6.9 | 7.1 | | 3.18 | 1022 | 74.0 | 756 |
| 169 | | | | | | | | | 6.9 | 7.1 | | 3.18 | 1138 | 74.0 | 842 |
| 170 | 4,158 | 405 | 2,450 | 1,950 | 0.80 | | | 25.0 | 6.8 | 7.1 | 2.46 | 3.19 | 1349 | 74.0 | 998 |
| 171 | | | | | | | | | 6.8 | 7.0 | | 3.16 | 1340 | 74.0 | 992 |
| 172 | | | | | | | | 25.0 | 6.7 | 6.9 | 2.44 | 3.16 | 1346 | 74.0 | 996 |
| 173 | 4,402 | 362 | 2,350 | 1,950 | 0.83 | | | | 6.8 | 7.0 | | 3.18 | 1094 | 74.0 | 809 |
| 174 | | | | | | | | 25.0 | 6.8 | 7.0 | 2.47 | 3.20 | 1080 | 74.0 | 800 |
| 175 | | | | | | | | | | | | 3.18 | 1207 | 74.0 | 894 |
| 176 | | | | | | | | | | | | 3.18 | 1404 | 74.0 | 1039 |
| 177 | | | | | | | | 25.0 | 6.8 | 7.0 | 2.37 | 3.07 | 1574 | 74 | 1165 |
| 178 | | | | | | | | | | | | 3.10 | 1511 | 74 | 1118 |
| 179 | | | | | | | | 25.0 | 6.8 | 7.0 | 2.43 | 3.15 | 1514 | 74.0 | 1121 |
| 180 | 4,783 | 401 | 2,450 | 1,950 | 0.80 | | | | | | 2.41 | 3.15 | 1424 | 72.0 | 1026 |
| 181 | | | | | | | | 25.0 | 6.8 | 7.0 | 2.42 | 3.14 | 1361 | 72.0 | 980 |
| 182 | | | | | | | | | 6.8 | 6.9 | | 3.14 | 1438 | 72.0 | 1035 |
| 183 | | | | | | | | | 6.8 | 6.9 | | 3.14 | 1504 | 72.0 | 1083 |
| 184 | | | | | | | | 25.0 | 6.8 | 7.0 | 2.46 | 3.18 | 1494 | 72.0 | 1075 |
| 185 | | | | | | | | | 6.8 | 7.0 | | 3.18 | 1613 | 72.0 | 1162 |
| 186 | | | | | | | | 25.0 | 6.8 | 7.0 | 2.42 | 3.13 | 1634 | 72.0 | 1177 |
| 187 | 5,476 | 535 | 2,533 | 2,000 | 0.79 | | | 25.0 | 6.8 | 7.0 | 2.43 | 3.15 | 1584 | 72.0 | 1140 |

| | Reactor - analytical | | | | | | | Operational data | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| # of days system run | TCOD | sCOD (0.45) | MLSS (1.5µm) | MLVSS | MLVSS/MLSS | Alkalinity (as CaCO3) | VFAs | Tank temp | pH, reactor | pH, permeate | Permeate pumped | Calculated flux | Biogas | $CH_4$ in biogas | $CH_4$ produced |
| | mg/L | mg/L | mg/L | mg/L | | mg/L | mg/L | deg C | | | L/d | LMH | mL | % | mL |
| 188 | | | | | | | | 25.0 | 6.8 | 7.0 | | 3.14 | 1382 | 72.0 | 995 |
| 189 | | | | | | | | 25.0 | 6.8 | 6.9 | 2.46 | 3.18 | 1382 | 72.0 | 995 |
| 190 | | | | | | | | | 6.8 | 6.9 | | 3.18 | 1238 | 72.0 | 891 |
| 191 | | | | | | | | 25.0 | 6.8 | 7.0 | 2.44 | 3.16 | 1238 | 72.0 | 891 |
| 192 | | | | | | | | | 6.8 | 7.0 | | 3.15 | 1147 | 72.0 | 826 |
| 193 | | | | | | | | | 6.8 | 6.9 | 2.43 | 3.14 | 1124 | 72.0 | 809 |
| 194 | | | | | | | | | 6.8 | 7.0 | | 3.15 | 1087 | 77.0 | 837 |
| 195 | | | | | | | | 25.0 | 6.8 | 7.00 | | 3.14 | 956 | 77.0 | 736 |
| 196 | | | | | | | | 25.0 | 6.8 | 7.00 | 2.42 | 3.14 | 956 | 77.0 | 736 |
| 197 | | | | | | | | 25.0 | 6.8 | 7.00 | 2.46 | 3.18 | 907 | 77.0 | 699 |
| 198 | 5,200 | 212 | 2,800 | 2,300 | 0.82 | | | 26.0 | 6.8 | 7.00 | 2.38 | 3.08 | 1003 | 77.0 | 772 |
| 199 | | | | | | | | 26.0 | 6.8 | 7.00 | | 3.08 | 741 | 77.0 | 571 |
| 200 | | | | | | | | 26.0 | 6.9 | 7.00 | 2.43 | 3.15 | 738 | 77.0 | 568 |
| 201 | | | | | | | | 26.0 | 6.9 | 7.00 | | 3.11 | 1019 | 75.0 | 764 |
| 202 | | | | | | | | 26.0 | 6.9 | 7.10 | 2.40 | 3.11 | 1019 | 75.0 | 764 |
| 203 | | | | | | | | | 6.9 | 7.10 | | 3.11 | 1200 | 75.0 | 900 |
| 204 | | | | | | | | | 6.9 | 7.00 | | 3.11 | 1200 | 75.0 | 900 |
| 205 | | | | | | | | 25.0 | 6.9 | 7.00 | 2.40 | 3.11 | 1200 | 75.0 | 900 |
| 206 | | | | | | | | 26.0 | 6.9 | 7.10 | | 3.17 | 694 | 75.0 | 520 |
| 207 | | | | | | | | 26.0 | 6.9 | 7.00 | 2.45 | 3.17 | 694 | 75.0 | 520 |
| 208 | | | | | | | | 26.0 | 6.9 | 7.00 | | 3.19 | 812 | 73.0 | 593 |
| 209 | 5,507 | 354 | 2,100 | 1,700 | 0.81 | | | 25.0 | 6.8 | 7.00 | 2.47 | 3.19 | 812 | 73.0 | 593 |
| 210 | | | | | | | | 26.0 | 6.8 | 7.10 | 2.43 | 3.15 | 812 | 73.0 | 593 |
| 211 | 5,373 | 393 | 2,700 | 2,100 | 0.78 | | | 26.0 | 6.8 | 7.00 | 4.90 | 6.34 | 1163 | 73.0 | 849 |
| 212 | | | | | | | | 25.0 | 6.8 | 7.00 | 4.89 | 6.33 | 1594 | 73.0 | 1163 |
| 213 | | | | | | | | 25.0 | 6.8 | 7.00 | | 6.38 | 1654 | 73.0 | 1207 |
| 214 | 6,286 | 519 | 2,300 | 1,800 | 0.78 | 1159.4 | 297.7 | 25.0 | 6.8 | 7.00 | 4.91 | 6.36 | 1709 | 73.0 | 1248 |
| 215 | | | | | | | | 26.0 | 6.8 | 7.00 | | 6.36 | 1722 | 73.0 | 1257 |
| 216 | | | | | | | | 26.0 | 6.8 | 7.00 | 4.87 | 6.30 | 1776 | 73.0 | 1296 |

| | Reactor - analytical | | | | | | | Operational data | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| # of days system run | TCOD | sCOD (0.45) | MLSS (1.5µm) | MLVSS | MLVSS/MLSS | Alkalinity (as CaCO3) | VFAs | Tank temp | pH, reactor | pH, permeate | Permeate pumped | Calculated flux | Biogas | $CH_4$ in biogas | $CH_4$ produced |
| | mg/L | mg/L | mg/L | mg/L | | mg/L | mg/L | deg C | | | L/d | LMH | mL | % | mL |
| 217 | | | | | | | | 26.0 | 6.8 | 7.00 | 4.89 | 6.32 | 1873 | 73.0 | 1367 |
| 218 | 5,979 | 511 | 2,533 | 2,000 | 0.79 | 1443.8 | 214.5 | 25.0 | 6.8 | 7.00 | | 6.32 | | 73.0 | |
| 219 | | | | | | | | 25.0 | 6.8 | 7.00 | 4.89 | 6.32 | 2064 | 73.0 | 1507 |
| 220 | | | | | | | | 25.0 | 6.7 | 6.90 | 4.88 | 6.31 | 2502 | 73.0 | 1826 |
| 221 | | | | | | | | 25.0 | 6.7 | 6.90 | | | | 73.0 | |
| 222 | | | | | | | | 25.0 | 6.7 | 6.90 | 4.88 | 6.31 | 2718 | 76.0 | 2065 |
| 223 | 5,468 | 496 | 2,267 | 1,867 | 0.82 | | | 25.0 | 6.7 | 6.90 | 4.86 | 6.29 | 2835 | 76.0 | 2155 |
| 224 | 6,860 | 618 | 3,200 | 2,500 | 0.78 | | | 25.0 | 6.8 | 7.00 | 4.89 | 6.33 | 3570 | 76.0 | 2714 |
| 225 | | | | | | | | 25.0 | 6.8 | 7.00 | 3.99 | 5.16 | 3417 | 76.0 | 2597 |
| 226 | | | | | | | | 25.0 | 6.8 | | 4.86 | 6.28 | 3397 | 67.0 | 2276 |
| 227 | | | | | | | | 24.0 | 6.8 | 7.00 | 4.89 | 6.33 | 3241 | 67.0 | 2171 |
| 228 | | | | | | | | 25.0 | 6.8 | 7.00 | 4.85 | 6.28 | 3768 | 67.0 | 2525 |
| 229 | 5,877 | 559 | 2,933 | 2,267 | 0.77 | | | | | | | | | | |
| 230 | | | | | | | | 24.0 | 6.7 | 6.90 | 4.82 | 6.24 | 3482 | 67.0 | 2333 |
| 231 | | | | | | | | 24.0 | 6.8 | 7.00 | 4.83 | 6.25 | 3018 | 67.0 | 2022 |
| 232 | | | | | | | | 25.0 | 6.8 | 7.10 | 4.80 | 6.21 | 2870 | 67.0 | 1923 |
| 233 | | | | | | | | 25.0 | 6.9 | 7.00 | 4.81 | 6.21 | 2484 | 67.0 | 1665 |
| 234 | | | | | | 1276.1 | 268.6 | 25.0 | 6.9 | 7.10 | 4.84 | 6.26 | 2745 | 70.0 | 1921 |
| 235 | 5,108 | 522 | 2,667 | 2,133 | 0.80 | | | 25.0 | 6.8 | 7.00 | 4.86 | 6.28 | 2753 | 70.0 | 1927 |
| 236 | | | | | | | | 25.0 | 6.8 | 7.00 | 4.80 | 6.21 | 3047 | 70.0 | 2133 |
| 237 | | | | | | | | 25.0 | 6.8 | 7.10 | 4.79 | 6.20 | 2707 | 72.0 | 1949 |
| 238 | | | | | | | | 24.0 | 6.9 | 7.00 | 4.79 | 6.20 | 2611 | 72.0 | 1880 |
| 239 | | | | | | | | 24.0 | 6.8 | 7.00 | 4.54 | 5.87 | 2896 | 72.0 | 2085 |
| 240 | | | | | | | | 25.0 | 6.9 | 7.10 | 4.70 | 6.08 | 3234 | 72.0 | 2328 |
| 241 | | | | | | | | 25.0 | 6.9 | 7.10 | 4.71 | 6.10 | 3660 | 72.0 | 2635 |
| 242 | 5,452 | 408 | 2,933 | 2,400 | 0.82 | | | 25.0 | 6.9 | 7.00 | 4.78 | 6.18 | 3719 | 72.0 | 2678 |
| 243 | | | | | | | | | | | | | 3433 | 75.0 | 2575 |
| 244 | | | | | | | | 25.0 | 6.9 | 7.10 | 4.77 | 6.17 | 2946 | 75.0 | 2209 |
| 245 | | | | | | | | 24.0 | 6.8 | 7.00 | 4.73 | 6.12 | 2974 | 75.0 | 2231 |
| 246 | 5,380 | 540 | 2,500 | 2,000 | 0.80 | 1513.8 | 137.9 | 24.0 | 6.8 | 7.00 | 4.63 | 5.99 | 2693 | 75.0 | 2020 |
| 247 | | | | | | | | 25.0 | 6.9 | | 4.76 | 6.16 | 2745 | 75.0 | 2059 |
| 248 | | | | | | | | 25.0 | 6.9 | 7.00 | 4.78 | 6.18 | 2869 | 75.0 | 2152 |
| 249 | | | | | | | | | | | | | 2752 | 72.0 | 1982 |

| | Reactor - analytical | | | | | | | Operational data | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| # of days system run | TCOD | sCOD (0.45) | MLSS (1.5µm) | MLVSS | MLVSS/MLSS | Alkalinity (as CaCO3) | VFAs | Tank temp | pH, reactor | pH, permeate | Permeate pumped | Calculated flux | Biogas | $CH_4$ in biogas | $CH_4$ produced |
| | **mg/L** | **mg/L** | **mg/L** | **mg/L** | | **mg/L** | **mg/L** | **deg C** | | | **L/d** | **LMH** | **mL** | **%** | **mL** |
| 250 | | | | | | | | 24.0 | 6.9 | 7.10 | 4.78 | 6.18 | 2676 | 72.0 | 1927 |
| 251 | | | | | | | | 24.0 | 6.9 | 7.00 | 4.73 | 6.12 | | 72.0 | |
| 252 | | | | | | | | 24.0 | 6.9 | | 4.76 | 6.14 | 2620 | 72.0 | 1886 |
| 253 | 6,233 | 567 | 2,200 | 1,800 | 0.82 | | | 25.0 | 6.8 | 7.00 | 4.73 | 6.12 | 2725 | 72.0 | 1962 |
| 254 | | | | | | 1505.5 | 188.8 | 24.0 | 6.9 | 7.00 | | | | 72.0 | |
| 255 | | | | | | | | 25.0 | 6.9 | | 4.82 | 6.24 | 2808 | 72.0 | 2022 |
| 256 | | | | | | | | 25.0 | 6.9 | 7.10 | 4.85 | 6.27 | 3191 | 72.0 | 2297 |
| 257 | | | | | | | | 25.0 | 6.9 | | 4.80 | 6.22 | 3337 | 72.0 | 2402 |
| 258 | | | | | | | | 24.0 | 6.9 | 7.10 | 4.78 | 6.18 | | 74.0 | |
| 259 | | | | | | | | 24.0 | 6.9 | | 4.77 | 6.17 | 3195 | 74.0 | 2364 |
| 260 | | | | | | | | 24.0 | 6.8 | 7.00 | 4.70 | 6.08 | 3440 | 74.0 | 2546 |
| 261 | | | | | | | | 25.0 | 6.8 | | 4.73 | 6.12 | 3500 | 74.0 | 2590 |
| 262 | | | | | | | | 25.0 | 6.9 | 7.00 | 4.72 | 6.10 | 3231 | 74.0 | 2391 |
| 263 | 4200 | 386 | 2667 | 2133 | 0.80 | | | 24.0 | 6.8 | | 4.65 | 6.02 | 3139 | 74.0 | 2323 |
| 264 | | | | | | | | 24.0 | 6.8 | 7.0 | 3.07 | 3.98 | | | |

**Table A-3: Summary of process performance**

| Parameter | Unit | Phase I (HRT 5 days) | | | | | Phase II (HRT 2 days) | | | | | Phase III (HRT 1 day) | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Avg | StD | Max | Min | n | Avg | StD | Max | Min | n | Avg | StD | Max | Min | n |
| F/M ratio | kg COD/kg MLVSS/day | 0.31 | 0.19 | 0.79 | 0.04 | 19 | 0.83 | 0.29 | 1.56 | 0.46 | 15 | 1.54 | 0.59 | 2.28 | 0.82 | 7 |
| COD loading rate (OLR) | kg COD/ $m^3$/day | 0.44 | 0.21 | 0.79 | 0.12 | 23 | 1.43 | 0.37 | 1.92 | 0.83 | 15 | 3.14 | 1.05 | 4.77 | 1.60 | 7 |
| COD loading rate | kg COD/kg-MLVSS/day | 0.31 | 0.19 | 0.79 | 0.04 | 19 | 0.83 | 0.29 | 1.56 | 0.46 | 15 | 1.54 | 0.59 | 2.28 | 0.82 | 7 |
| Tot. COD removal rate | % | 94.5 | 3.1 | 97.8 | 85.3 | 23 | 93.5 | 2.3 | 96.9 | 89.0 | 16 | 87.5 | 4.6 | 92.8 | 80.6 | 8 |
| Secondary COD removal rate | % | 84.6 | 13.3 | 95.6 | 46.6 | 18 | 87.0 | 5.1 | 95.2 | 78.0 | 16 | 82.9 | 6.6 | 89.4 | 68.8 | 8 |
| Specific $CH_4$ yield | L $CH_4$/g COD removed | 0.24 | 0.16 | 0.61 | 0.05 | 23 | 0.16 | 0.05 | 0.26 | 0.09 | 14 | 0.20 | 0.09 | 0.34 | 0.10 | 7 |
| Specific $CH_4$ yield | mL $CH_4$/L of reactor/day | 67.2 | 26.1 | 128.9 | 29.5 | 70 | 182.7 | 33.5 | 260.4 | 105.9 | 96 | 471.4 | 57.7 | 577.9 | 336.5 | 40 |
| Specific $CH_4$ yield | L $CH_4$/g COD added | 0.22 | 0.14 | 0.55 | 0.05 | 23 | 0.14 | 0.04 | 0.24 | 0.09 | 15 | 0.17 | 0.07 | 0.27 | 0.09 | 7 |
| Specific Biogas yield | L gas/g COD added | 0.25 | 0.18 | 0.74 | 0.04 | 23 | 0.17 | 0.05 | 0.29 | 0.10 | 15 | 0.22 | 0.08 | 0.34 | 0.12 | 7 |
| VFA/Alkalinity | - | 0.14 | 0.02 | 0.15 | 0.12 | 3 | 0.18 | 0.03 | 0.20 | 0.16 | 3 | 0.17 | 0.07 | 0.26 | 0.15 | 5 |