PERFORMANCE OF ANAEROBIC MEMBRANE BIOREACTORS (AnMBRs) WITH DIFFERENT DOSAGES OF POWDERED ACTIVATED CARBON (PAC) AT MESOPHILIC REGIME IN MEMBRANE FOULING CONTROL

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A project report submitted in partial fulfilment of the requirements for the award of Bachelor of Engineering (Hons) Environmental Engineering

Faculty of Engineering and Green Technology
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DECLARATION

I hereby declare that this project report is based on my original work except for citations and quotations which have been duly acknowledged. I also declare that it has not been previously and concurrently submitted for any other degree or award at UTAR or other institutions.

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APPROVAL FOR SUBMISSION

I certify that this project report entitled **"PERFORMANCE OF ANAEROBIC MEMBRANE BIOREACTORS (AnMBRs) WITH DIFFERENT DOSAGES OF POWDERED ACTIVATED CARBON (PAC) AT MESOPHILIC REGIME IN MEMBRANE FOULING CONTROL"** was prepared by **CONNIE CHONG TUNG** has met the required standard for submission in partial fulfilment of the requirements for the award of Bachelor of Engineering (Hons) Environment Engineering at Universiti Tunku Abdul Rahman.

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Signature : ________________, __________________

Supervisor : Dr Ng Choon Aun & Dr Mohammed J. K. Bashir

Date : ____________________

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ABSTRACT

Anaerobic membrane bioreactor (AnMBRs) has been widely employed in the municipal and industrial wastewater treatment due to its attractive advantages. By the comparison to the conventional aerobic membrane bioreactor, AnMBR has low yield anaerobic microbes which produce relatively lower sludge concentration, requires minimum energy that can produce biogas to be used as energy source. In this study, it was found that higher dosages of powdered activated carbon (PAC) had better membrane fouling control. To investigate the mechanisms involved in helping the fouling control, (i) particle size distribution; (ii) concentration of mixed liquor suspended solids (MLSS) and mixed liquor volatile suspended solids (MLVSS); (iii) chemical oxygen demand (COD); (iv) extracellular polymeric substances (EPS) concentration analysis as well as (v) biogas production were measured and analysed to support and explain the findings.

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AnMBR	Anaerobic membrane bioreactor
BAC	Biologically activated carbon sludge
BOD	Biochemical oxygen demand
COD	Chemical oxygen demand
EPS	Extracellular polymeric substance
F/M	Food to microorganism ratio
GAC	Granular activated carbon
HRT	Hydraulic retention time
MBR	Membrane bioreactor
MLSS	Mixed liquor suspended solid
MLVSS	Mixed liquor volatile suspended solid
O&G	Oil and Grease
PAC	Powdered activated carbon
POME	Palm Oil Mill Effluent
PSD	Particle Size Distribution
SAnMBR	Submerged anaerobic membrane bioreactor
SMP	Soluble microbial product
SRT	Sludge retention time
TMP	Trans-membrane pressure
TS	Total Solids
TSS	Total suspended solid
VFA	Volatile fatty acids
VSS	Volatile suspended solid

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CHAPTER 1

INTRODUCTION

1.1 Background

In the late 1960s, Membrane Bioreactor (MBR) is the combination of common biodegradation process by activated sludge and a direct separation of treated wastewater from anaerobic microorganisms by membrane filtration has allowed the MBR system to achieve an astonishing market value in conventional wastewater treatment (Le-clech et al., 2006). MBR provides advantages such as smaller footprint, higher quality of effluent, better disinfection capability, higher volumetric loading and low production of excess sludge (Judd, 2011).

In comparison to conventional activated sludge (CAS) process, higher operational costs is required in MBR as the use of aggressive chemicals for membrane cleaning has created an environmental burden (Brepols et al., 2008). However, the critical issue faced in MBR system are still major problem in membrane fouling, mainly due to irreversible fouling caused by cake accumulation (Ognier et al., 2002) onto membrane surface and pore blocking (Choi et al., 2005; Liu et al., 2005). The mitigation controls of membrane fouling include (i) backwashing (Le-clech et al., 2006); (ii) periodic filtration (Chua et al., 2002); (iii) sustainable flux; (iv) chemical cleaning (Lim & Bai, 2003) and (iv) specific hydrodynamic design.

Previous reported findings from many researchers mentioned that performance of MBR can be further enhanced by addition of powdered activated carbon (PAC) as an adsorbent to improve fouling control by modifying characteristics of mixed liquor suspension (Li et al., 2005; Seo et al., 2004; Ng et al., 2006; Ma & Yu, 2012). One of the remarkable effects brought by addition of PAC is the concurrent processes of adsorption and degradation effects where the formation of biofilm on PAC as known as biologically activated carbon (BAC) to undergo biodegradation process of pollutants that previously adsorbed by PAC. In addition, scouring effect of PAC on membrane surface has proved to perform well for the membrane flux (Ng et al., 2010; Yang et al., 2006).

Despite PAC addition enhances MBR performance, the operating condition to indirectly control the membrane fouling is important as well. The optimum sludge retention time (SRT) control can determine the stability of MBR sludge where the floc stability is reflected by the MBR fouling rate which indicated by changes in transmembrane pressure (TMP) (Yu & Su, 2012). Temperature is influenced by the viscosity of digester sludge in terms of mesophilic and thermophilic conditions. The correlation between SRT and temperature can be considered as the maximum SRT was determined by concentration of mixed liquor suspended solids (MLSS) and the viscosity of sludge (Meabe et al., 2013).

1.2 Problem statement

In MBR, many advantages are found when it comes to comparison between MBR and conventional activated sludge (CAS). However, the major drawback of using MBR as a wastewater treatment process is still limited by the membrane fouling issue which led to high operational and maintenance (O&M) cost. Besides, high energy consumption due to excess energy needed to heat the reactor in order to cultivate the anaerobic activated sludge at mesophilic and thermophilic conditions. (Lew et al., 2009; Lettinga et al., 2001).

Besides, many researches have studied the effect of PAC on membrane fouling control in AnMBR and mostly treatment are sewage or industrial wastewater as basis. Anyhow, the feasible studies on different dosage of PAC at mesophilic and thermophilic regimes on membrane fouling control are still limited especially on treating the Palm Oil Mill Effluent (POME). Therefore, in my studies, investigation and comparison of the effect at different low PAC dosages on membrane fouling control are concerned under mesophilic regimes in terms of cost saving prospective along with the POME as wastewater to be treated.

1.3 Objectives

i. To investigate the effects of hybrid AnMBRs added with different PAC dosage concentrations in treating POME.
ii. To study the mechanisms involved in controlling fouling of AnMBRs at different PAC concentrations at mesophilic regime.

1.4 Outline of reports

There are five chapters included in my studies. The first chapter, introduction chapter covers the background of studies, problem statement and objectives. Secondly, chapter two is literature review which highlights all the relevant information including the operating conditions, membrane fouling problem and effect of PAC on membrane fouling control which related to AnMBR in this project studies. Third chapter reviews the research methodology which includes experimental set-up, materials and analytical methods meanwhile the forth chapter reports the results and discussions based on the fact-finding throughout the experimental analysis. Last but not least, the conclusions and recommendations are proposed to improve the studies.

CHAPTER 2

LITERATURE REVIEW

In this chapter, there are four sections to be discussed which are related to summarization of the relevant information needed for this study. The first section is the introduction to anaerobic membrane bioreactor system (AnMBR). The second section is the operation conditions including sludge retention time (SRT) and temperature. The third section reviews the membrane fouling studies, for instances, the fundamentals, classification, factors and the mitigation control of membrane fouling. For the last section is the studies of the introduction, mechanisms and optimum dosage of the additives, powdered activated carbon in the MBR system.

2.1 Introduction to anaerobic membrane bioreactor system (AnMBRs)

Recently, anaerobic membrane bioreactor system (AnMBR) is gaining attention due to its ability to treat a wider range of strength of wastewaters. The ability to convert biochemical oxygen demand (BOD) of wastewater to usable biogas with minimum energy consumption (Chang, 2014) and AnMBR has become popular. In this study, high strength wastewater, Palm Oil Mill Effluent (POME) is treated using AnMBRs. POME pollute our environment if not properly treated (Rupani et al., 2010). The characteristics of raw POME and the regulatory discharge limits in Malaysia are as shown in FIG 2.1.

Table 2.1: Characteristics of raw POME and the regulatory discharge limits

Parameters	Value[a]	Regulatory discharge limits[b]
Temperature	80-90°C	45°C
pH	4.7	5.0-9.0
Biological Oxygen Demand (BOD); 3 days at 30°C	25,000 mg/L	100(50) mg/L
Chemical Oxygen Demand, COD	50,000 mg/L	-
Total Solids (TS)	40,500 mg/L	-
Total Suspended Solids (TSS)	18,000 mg/L	400
Total Volatile Solids (TVS)	34,000 mg/L	-
Oil and Grease (O&G)	4,000 mg/L	50

Source: (Ma, 2000)[a] , (Ahmad et al., 2003)[b]

Higher strength wastewaters consists of higher organic matter which induces a great energy which can be harvested in the anaerobic treatment (Visvanathan & Abeynayaka, 2012). The AnMBR is an integrated system of the anaerobic biological wastewater treatment process and the low pressure ultrafiltration or microfiltration membrane filtration which allows separation of treated wastewater from anaerobic biomass as well as concentrate the biomass in AnMBR. (Chang, 2014). There are several benefits of using AnMBR compared to common aerobic treatment such as (i) lower sludge production due to low yield of anaerobic microorganisms; (ii) lower energy consumption as no aeration needed; and (iii) potential resource recovery because energy (from biogas production) and nutrients (NH_4^+ and PO_4^{3-}) can be obtained from the anaerobic degradation process (Ferrera et al., 2015). However, this system has one main drawback which is membrane fouling (Liao et al., 2006).

2.2 Operating conditions

There are three major operating conditions such as sludge retention time (SRT), temperature and pH were fixed in this study.

2.2.1 Sludge retention time (SRT)

SRT is one of the critical operating paramaters which is used to manipulate the characteristics of biomass suspension and its fouling propensity (Grelier et al., 2006). Some researchers reported that longer SRTs can achieve higher effiency in treating wastewater by forming a more acclimatised biomass (Xing et al., 2000; Rosenberger & Kraume, 2002; Shin & Kang, 2001). Longer SRT (>15days) can relatively (i) reduce fouling propensity on the membrane (Bouhabila et al., 2001; Innocenti, et al., 2002) and with the high SRT, (ii) the concentration of MLSS and MLVSS would increase theoretically followed by (iii) decrease the net sludge production due to the resulting low (F/M) ratio and (iv) enhance the development of nitrifying bacteria to improve the nitrification capability (Xing et al., 2000; Bouhabila, et al., 2001; Rosenberger & Kraume, 2002; Huang & Qian, 2001; Han, et al., 2005). However, according to Meng et al. (2009), a too long SRT (>40 days) would result in membrane fouling while a too short SRT (<15 days) might detrimental to membrane performance. According to Tian & Su (2012), MBR sludge at lower SRT inhibits poorer stability and loose structure and might cause serious membrane fouling. Ng et al. (2013) also reported MBR with SRT (30 days) shows a better filtration performance compared to MBR with SRT 10 days. Prolonged SRT might (i) encourages higher MLSS and viscosity (Rosenberger & Kraume, 2002; Han et al., 2005), (ii) decrease the permeate quality with a prolonged SRT of more than 30 days (Innocenti et al., 2002) and (iii) accumulate the inorganic compounds at the bottom of the bioreactor (Rosenberger et al., 2002). Thus, the moderate SRT 30 days are fixed as one of the operating condition in my project.

2.2.2 Temperature

Temperature plays as one of the important operating conditions in MBR system due to its influence on permeate fluid viscosity (Mulder, 2000). Temperature was recently found to have effects on permeability including the sludge viscosity, shear stress/forces close to the membrane surface and solubilisation of organic matter (Lyko et al., 2008). There are two temperature conditions to be studied in this project which are at the mesophilic (35 ℃) and thermophilic (55 ℃) conditions. In mesophilic temperature regime, AnMBR operated well in anaerobic digestion compared to the thermophilic regime due to its weak stability (Meabe et al., 2013). In terms of COD level in the permeate, higher soluble COD (brown in colour) are found in thermophilic conditions compared to mesophilic condition (light yellowish in colour). It is because the increasing hydrolysis at higher temperature followed by the higher volatile fatty acids (VFA) concentration (Meabe et al., 2013). In addition, filtration performance shows a better permeability at mesophilic conditions as due to the size of particle pass through the membrane. The smaller particles in thermophilic sludge are deposited onto the membrane pore which enchance the pore-blocking mechanisms, thus, lead to low permeabilities (Meabe et al., 2013). It concluded that mesophilic regime is preferable in membrane filtration performance after the permeate flux is determined by the physical properties of sludge (Jeison & Lier, 2008).

However, thermophilic regimes can increase the destruction rate of organic solids and eliminate pathogens (Kim et al., 2002). Since the characteristics of sludge is significantly different when under the studied of both temperature, the smaller floc size and poorer supernatant quality at higher temperatures led to an increase in filtration resistance (Lin et al., 2009).

2.2.3 pH

pH would impact the microbial metabolism and chemical activity within the bioreactor. It can alter the energy yielding, efficiency of substrate degradation, membrane morphology, performance of methanogenic bacteria and the release of metabolic products (Baily & Ollis, 1986). The optimum pH for anaerobic bioreactor is within 6-8 (Ward et al., 2008). According to Gao et al. (2010), membrane fouling rate would increase significantly when pH 8 is increased to pH 9.1 and fouling rate is the highest at pH 10. Moreover, in terms of sludge properties, pH 8 shows no significant influence on MLSS concentration meanwhile pH 9.1 would retarde sludge production followed by decrease in MLSS concentration with a pH 10 (Gao et al., 2010).

2.3 Membrane fouling

Membrane fouling is the accumulation of undesirable materials onto the surface or inside the pores of a membrane and eventually lead to loss in permeability or membrane damage (Leonard et al., 2014).

2.3.1 Fundamentals of membrane fouling

According to Meng et al. (2009), factors which cause membrane fouling are mainly due the mechanisms such as (1) the adsorption and accumulation of colloids and sludge on the membrane surface; (2) separation of foulants due to shear forces; (3) the structural and physical changes of the foulant configuration during the long-term operation. TMP is defined as the characterization of the membrane fouling intensity as well as an indicator for the filtration performance in the MBRs (Ng et al., 2010). An occurrence of TMP jump, recently found in an investigation, was the sudden rise in the concentration of extracellular polymeric substances (EPS) at the bottom of cake layer and probably result the fatality of bacteria between cake layers (Hwang et al., 2008). Figure 2.1 shows the schematic illustration of the occurrence of TMP jump in three stages process: stage 1- conditioning fouling; stage 2-slow/steady fouling; stage 3-TMP jump. However, the relationship of membrane fouling rate is confused due to the fouling rate investigated in lab-scale is hardly used to justify in the comparison of long term full-scale operation (Kraume et al., 2009).

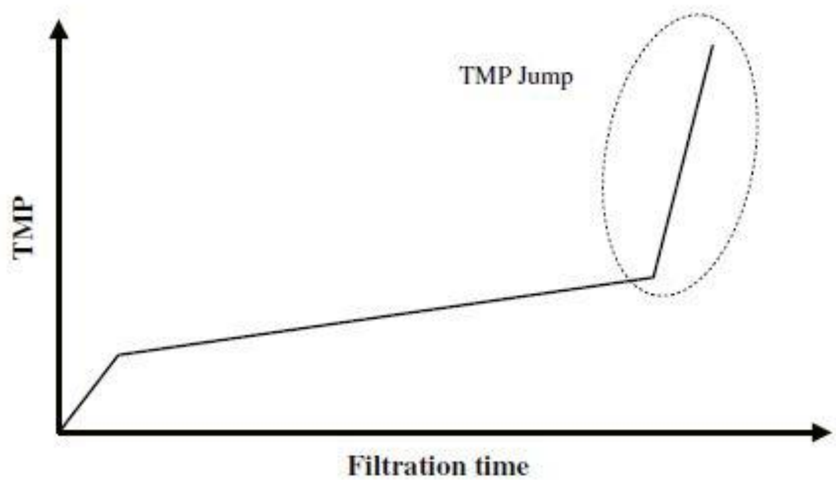

Figure 2.1: Schematic illustration of the occurrence of TMP jump

Source: Judd, 2011

2.3.2 Classification of membrane fouling

According to Lee at al. (2001), membrane fouling is affected by three predominant fouling components such as sludge particles, colloids and solutes as shown in Figure 2.2.

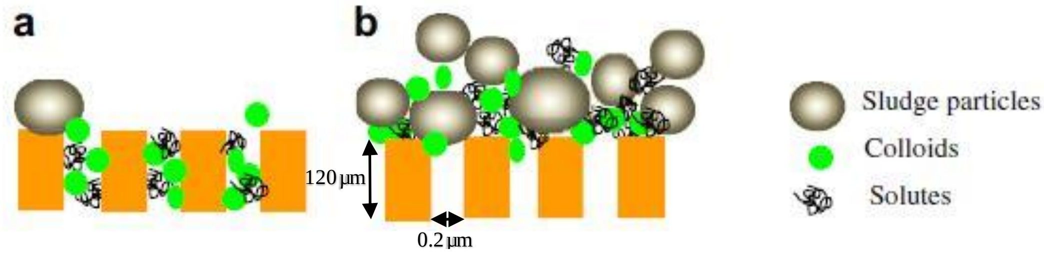

Figure 2.2: Membrane fouling process in MBRs: (a) pore blocking and (b) cake layer
Source: Lee et al., 2011

If the size of foulants are equivalent with the membrane pores size (i.e., colloids) or smaller than the membrane pores size (i.e., solutes), pore blocking and adsorption might happen (Meng et al., 2009). On the other hand, if the size of foulants (i.e., sludge flocs and colloids) are bigger the pore size of membrane, sludge cake deposition tends to form on the membrane surface (Meng et al., 2009).

To date, the perceptions on fouling classifications are mystifying due to different definitions proposed in publications. Table 2.1 shows the typical ranges of two fouling rates occurring at full scale (Kraume et al., 2009; Guglielmi et al., 2007; Pollice et al., 2005). Generally, the term reversible fouling is defined as fouling which can be removed by physical methods (i.e. backwashing or relaxation under crossflow conditions) and reversible fouling takes place due to loosely attached foulants (Drews, 2010). According to Choi et al. (2005), irreversible fouling occurs due to the formation of pore blocking followed by the strongly attached foulants during filtration. Chemical cleaning is needed to eliminate the foulants accumulated between the membrane layers.

Table 2.2: Typical ranges of two fouling rates occurring at full scale

Category	Fouling rate in mbar/min	Time frame
Reversible fouling	0.1-1	10 minutes
Irreversible fouling	0.001-0.01	6-12 months

Source: Kraume et al., 2009; Guglielmi et al., 2007; Pollice et al., 2005

2.3.3 Membrane fouling factors

The main factors that cause membrane fouling are related to the biomass features, type of feedstock and operating conditions (SRT, hydraulic retention time (HRT) and food to microorganism ratio (F/M)) (Le-Clech et al., 2006). In MBRs operation, the sludge characteristics and hydrodynamic conditions are used to govern the fouling behaviour. Table 2.2 presents the relationship between various fouling factors and membrane fouling based on the source from Meng et al. (2009).

Table 2.3: Relationship between various fouling factors and membrane fouling

Sludge Condition	Effect on membrane fouling	Reference
Sludge condition		
MLSS	MLSS ↑ → normalized permeability ↓	Trussell et al., 2007
	MLSS ↑ → fouling potential ↑	Psoch & Schiewer, 2006
	MLSS ↑ → cake resistance ↑, specific cake resistance ↓	Chang & Kim, 2005
EPS	Polysaccharide ↑ → fouling rate ↑	Drews et al., 2006
	Bound EPS influences on specific cake resistance	Cho et al., 2005
	Polysaccharide ↑ → fouling rate ↑	Lesjean et al., 2005
	Bound EPS ↑ → membrane resistance ↑	Chae et al., 2006
Operating condition		
SRT	SRT decrease from 100 to 20 days → TMP ↑	Ahmed et al., 2007
	SRT decrease from 30 to 10 days → fouling ↑	Zhang et al., 2006
	SRTs ↑ → fouling potentials of SMP ↑	Liang et al., 2007
	SRT decrease from 5 to 3 days → fouling ↑	Ng et al., 2006
HRT	HRT ↓ → membrane fouling ↑	Meng et al., 2007
Permeate	Sub-critical flux mitigates irremovable fouling	Lebegue et al., 2008
flux	Sub-critical flux mitigates fouling	Guo et al., 2007

Source: Meng et al., 2009

2.3.3.1 Extracellular polymeric substances (EPS)

According to Geesey (1982), EPS is termed as Extracellular Polymeric Substances which is the formation of microbial aggregates. Generally, EPS can be in either bound or soluble form. The soluble form of EPS is known as soluble microbial products (SMP) in MBRs. The major constistuents found in EPS are polysaccharides and proteins followed by other components such as humic acids, nucleic acids, lipids, uronic acids (Liu & Fang, 2003; Yu, 2008; Frolund et al., 1996). EPS created a significant barrier to permeate flow in

MBRs that results in membrane fouling. Numerous reports show that the effects of EPS in membrane fouling was through the extraction of EPS from the sludge floc (Le-clech et al., 2006). The techniques of extraction, for instances, cation exchange resin heating methods (Slang et al., 2005; Gorner et al., 2003; Frolund et al., 1996), heating methods (Morgan et al., 1990), centrifugation with formaldehyde (Zhang et al., 1999). Among all these extraction, formaldehyde centrifugation is the most effective method with the largest concentration of extracted EPS (eEPS).

2.3.3.2 Soluble microbial product (SMP)

Soluble microbial product (SMP) can exist in different amount with no fixed composition as well as its characteristics on determining the impact on membrane permeability (Drews, 2010). However, most of the publication reported that only SMP has the significant effect on membrane fouling when compared to bound EPS (Tardieu, et al., 1999; Rosenberger & Kraume, 2002; Rosenberger et al., 2006; Yamato, et al., 2006; Fan, et al., 2006).

SMP is termed as the matrix of soluble organic compounds that released during cell lysis followed by diffusion into cell membrane, is lost during synthesis or are excreted for certain purpose (Laspidou & Rittmann, 2002; Li, et al., 2005). Rosenberger et al. (2005) reported that SMP are would form gel-like structure on the membrane surface, blocked the membrane pores where they provide nutrient for biofilm formation and a hydraulic resistance to permeate flow during filtration process. This is because both soluble carbohydrate (polysaccharide) and humic substances are two key colloidal total organic carbon (TOC) components that result in membrane fouling (Fan, et al., 2006; Meng, et al., 2006) . In addition, the colloidal TOC acts as an indicator to predict the sludge fouling tendency. There are three techniques to separate the water phase from biomass in order to isolate SMP (Evenblij & van der Graaf, 2004). The most effective way of isolation when compared to centrifugation or sedimentation are the simple filtration through filter paper with pore size of 12 μm as it is most preferable due to its removal efficiency of colloidal material (Evenblij & van der Graaf, 2004).

2.3.4 Mitigation of MBR fouling

There are three major methods can be used to prevent MBR fouling. The elaboration of the methods are as follows.

2.3.4.1 Physical cleaning

The two standard operating strategies to control membrane fouling in physical ways are membrane backwashing and membrane relaxation (Le-clech et al., 2006). Most of the reversible fouling results from pore blocking can be eliminated by backwashing where the permeate pumped in reversible direction and detached sludge cake from membrane surface (Bouhabila et al., 2001; Psoch & Schiewer, 2005; Psoch & Schiewer, 2006). The optimization of backwashing is required based on some important key parameters such as its frequency, duration and the ratio between those two parameters, intensity, energy and permeate consumptions (Bouhabila et al., 2001; Psoch & Schiewer, 2005; Psoch & Schiewer, 2006). On the other hand, membrane productivity can be well improved by membrane relaxation (or non-continuous operation of the membrane) (Le-clech et al., 2006). During the membrane relaxation, reversible attached foulants can be removed from membrane surface under the concentration gradient and thus enhancing the back transport of foulants (Hong et al., 2002). If an air scouring effect is added during relaxation, the removal effiency of it might increase (Chua et al., 2002).

2.3.4.2 Chemical cleaning

There are three types of chemical cleaning according to different time basis. For instances, chemically enhanced backwash (on a daily basis), maintenance cleaning with higher chemical concentration (weekly) and intensive (or recovery) chemical cleaning (once or twice a year) (Le-clech et al., 2006). Chemical cleaning is a more robust way to remove EPS and foulants. The commonly used chemical cleaning agents include sodium hypochlorite (NaClO), sodium hydroxide (NaOH), hydrochloric acid (HCl) and citric acid in MBR (Brepols et al., 2008; Wei, et al., 2011; MJ. Kim, 2011). NaClO and NaOH have the highest cleaning removal effiency among these chemical agents due to their oxidizability and alkaline hydrolysis effect (Brepols et al., 2008). Despite of using chemical agents, sonification chemical process, one of the techniques to eliminate cake formation by breaking down the fouling cake into smaller fragments (Fang & Shi, 2005). However, sonification method is not effective on all types of fouling because the pore blocking might worsen this type of fouling. And thus, by combining sonification, backwashing and chemical agent cleaning can reach an optimization removal effect (Fang & Shi, 2005).

2.3.4.3 Sustainable flux

The sustainable flux in MBR is conceptualized as chemical cleaning is not necessary if the TMP increases gradually at an acceptable rate (Ng et al., 2005). A reasonable flux rate without significant fouling should be recognized as the most economical way to control membrane fouling (Le-clech et al., 2006). Most MBR systems operate at low fluxes to limit rapid and severe membrane since the permeate rate and fouling decrease simultaneously (Le-clech et al., 2006). As mentioned in 2.2 and Figure 2.1, the sustainable flux is occurring in slow and steady fouling stage (stage 2). Meanwhile for the critical flux is noticeable higher than sustainable operating flux since the critical flux happens in between stage 2 and stage 3 as shown in Figure 2.1 (Wang et al., 2006). Critical flux was examined during short term experiments while the sustainable flux have to be assessed for longer period of time (Le-clech et al., 2006). Thus, flux value is important to be determined and well managed in MBR system as it presents as one of the membrane fouling factors.

2.4 Adsorbent agent- Powdered Activated Carbon (PAC)

Adsorption refers as the attachment of a substance at the interface between liquid and solids in a physical and chemical process. Activated carbon is commonly used as adsorbent due to its highly porous material and a large surface area provided for the adsorption and biodegradation process (Brady & Moran, 2012). Activated carbon is available in two forms which are powdered activated carbon (PAC) and granular activated carbon (GAC).

2.4.1 Introduction to PAC

Addition of PAC into MBR system has been widely used as a method of membrane fouling control (Le-clech et al., 2006). PAC addition modified the sludge characteristics by increasing the removal of low molecular weight organics by adsorption; it also acts as a supporting medium for attached bacterial growth, influences the bacterial population and affects the EPS concentration (Kim et al., 1998). In addition, PAC additions could enhance membrane flux, increase the porosity of sludge cake layer, decrease in sludge production and increase the resistance to toxic substances (Kim et al., 1998; Li et al., 2005; Aquino et al., 2006; Lesage et al., 2007).

2.4.2 Mechanisms of PAC in fouling mitigation

Three fouling control mechanisms of using PAC in MBR identified. First, the behavior of PAC acts as an absorbent of the methanogenic bacteria, second, PAC with scouring effect to limit the foulant accumulation, and third, the combination of PAC to form biologically activated carbon (BAC) lead to concurrent adsorption and degradation effects (Ng et al., 2010). The most significant effect among the mechanisms is the combined adsorption and biodegradation effect rather than the biological or adsorption process alone (Pirbazari, et al., 1996; Liu, et al., 2005; Seo, et al., 2004). The function of this concurrent process is the adsorption of pollutants on PAC is allowed to be biodegraded by the bacteria in the biofilm of BAC where the PAC could act as a foundation for the formation of biofilm that consists of immobilized bacteria (Walker & Weatherley, 1999; Lin et al., 2000). Therefore, the bioregeneration of saturated BAC could be enhanced in the formation of a biofilm on the PAC (Li et al., 2005; Ng, et al., 2010). There are two different research findings comment upon the scouring effect of PAC which due to (i) neutralization and removal of fine foulants that deposited on membrane surface followed by enhancement of fluid turbulence with bubling effect (Li et al., 2005; Dosoretz & Boddeker, 2004) and (ii) formation of BAC with high porosity and low compressibility or formation of a permeable “precoat” BAC layer to allow cake deposition (Liu et al., 2005; Kim et al., 1998). Membrane fouling could be adversed with PAC addition if the PAC replenishment is not steady or the size of PAC is not within an optimum range (Ng et al., 2013). As a result, it is important to study and optimize the effect of PAC dosage and size in order to mitigate membrane fouling.

2.5 Biogas production

Biogas produced from AnMBR with POME as feedstock is able to be captured and used as a clean and renewable resource because of the discharge is less harmful to the environment compared to burning of fossil fuel (Lim & Low, 2013). The estimated potential energy to be harvested in biogas production can be used as fuel to generate electricity on the assumption of demonstration scale. The compounds in the biogas are methane (CH_4), carbon dioxide (CO_2), hydrogen sulphide (H_2S), moisture and other trace gas compound whereby methane concentration is in the range of 50-70% of biogas volume (Zhao et al., 2010).

CHAPTER 3

METHODOLOGY

This chapter consists of four sections which include experimental set-up, materials and analytical methods.

3.1 Experimental setup

Three anaerobic bioreactors (AnMBRs) with one litre capacity were setup at bench-scale in this study. The microbial seed and feedstock, Palm Oil Mill Effluent (POME) were required to cultivate the anaerobic sludge in AnMBRs and they were supplied by the local wastewater treatment plant, Tian Xiang Group in Perak, Malaysia. In this experiment, different PAC dosage at 1g/L, 3g/L and 5g/L were adopted. The operating condition for these AnMBRs such as SRT was fixed at 30 days and the temperature was set at 35 ℃. Replenishment of aged biologically activated carbon (BAC) and POME was practiced to maintain the good performance of AnMBRs. The POME was required to be filtered through 53 µm sieve to remove the large particle including dirt, sediment and grease before feed into AnMBRs. Nitrogen airbag was connected to the gas probe of AnMBRs for desludge and feed session to prevent oxygen intake in order to achieve an anaerobic condition. Supernatent extracted from the AnMBRs were undergoing dead-end filtration, an indicator of short term test in order to predict the long term performance of membrane fouling control. Last but not least, every single AnMBR was equipped with gas collector to collect the biogas. The schematic diagram of the lab scale AnMBR is shown in Figure 3.1.

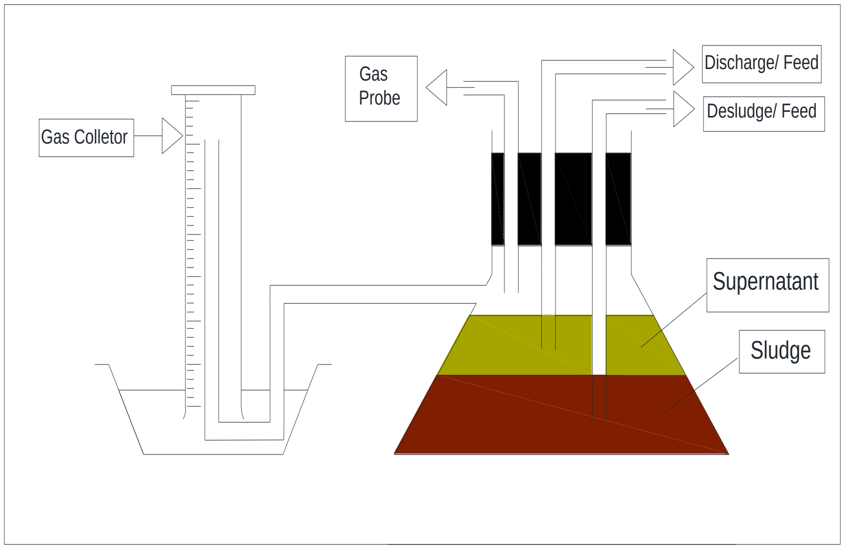

Figure 3.1: The schematic diagram of lab scale AnMBR

3.2 Materials

3.2.1 Powdered Activated Carbon (PAC)

The PAC adopt in this studies is extra pure Charcoal Powdered Activated Carbon from GENE Chem. The specification of PAC is listed in Table 3.1.

Table 3.1: The specification for PAC used in MBR

Composition	Value
pH	4.5-7.5
Soluble matter in ethanol	0.20 %
Soluble matter in hydrochloric acid	0.20 %
Chloride (Cl)	0.10 %
Sulfur compound (SO_4)	0.15 %
Iron (Fe)	0.10 %
Zinc (Zn)	0.10 %
Heavy metal (Pb)	0.01 %

3.2.2 Palm Oil Mill Effluent (POME)

The POME is supplied by a palm oil processing factory named Tian Xiang Group in Perak. The POME used is considered as high strength industrial wastewater with COD of 80000 mg/L. The feedstock of POME is required to be filtered by using filtered sieve plate with the mesh size of No. 270 (0.053mm).

3.2.3 Anaerobic sludge

The microbial seed used to cultivate the anaerobic sludge is supplied by Tian Xiang Group in Perak, Malaysia. Prior to use, the sludge was filtered by a 53 μm sieve to remove large particles such as sediments and dirt.

3.2.4 Membrane

Cellulose Acetate Membrane Filter with the combination of higher flow rate and thermal stability with very low adsorption characteristics and applicable to be used in pressure filtration devices. The characteristics are as shown in Table 3.2.

Table 3.2: The characteristics of membrane used in MBR

Characteristics	Value
Pore size	0.2 µm
Diameter	47mm
Thickness	120 µm
Flow rate for water	24 ml/min/cm^2/bar

3.3 Analytical Methods

3.3.1 Total Suspended Solid (TSS) and Volatile Suspended Solid (VSS)

The TSS and VSS which are also known as mixed liquor suspended solid (MLSS) and mixed liquor volatile suspended solid (MLVSS) can be determined by using the standard given, Standard Method, 21st Edition. The filtration process was conducted by using micro-glass fiber filter AH-934 after the samples were removed from AnMBRs. Then, weight of the filtered samples were measured by using M-power Analytical Balance AZ214 (Sartorius weighing technology, Germany) as shown in Figure 3.2. The filtered sample was placed in oven at the temperature of 105 ℃ for 2 hours. Later it was transferred to a dessicator to be cooled down. Weight was recorded to determine the TSS. After the TSS was determined, the samples were transferred to a Muffle Furnace and ignited at the temperature of 550 ^{o}C for 15 minutes. The samples had to cool down before being weighted.

Figure 3.2: M-power Analytical Balance AZ214

3.3.2 Chemical Oxygen Demand (COD)

Methods to determine COD were based on the Standard Method, 21^{st} Edition. The supernatant was extracted from bioreactors followed by dilution using 100mL of volumetric flask in the factor of 250. Then the samples were measured by the HACH test kits with a range of 0-1500 mg L^{-1} in the COD digester block (DRB 200, Germany). The samples were then heated in COD reactor (Figure 3.3) for 2 hours at the temperature of 150 ℃ and tested by a spectrophotometer after it was cooled down (DRB 6000, Germany)

Figure 3.3: COD Reactor Hach DRB-200

3.3.3 Particle Size Distribution (PSD)

Particle size of PAC and biomass floc size are measured by using the Malvern Mastersizer 2000 Particle Size Analyser is as shown in Figure 3.4. This analyzer is able to detect particle sizes from the range of 0.02 µm to 2000 µm. The scattered light is detected by a detector that induce the signal to a size distribution on volume or number basis. Each sample was calibrated 3 times with a standard deviation of 0.1-4.5%.

Figure 3.4: Malvern Mastersizer 2000 Particle Size Analyser

3.3.4 pH Measurement

The pH of the mixed liquor was determined by using pH electrode meter (Hanna HI 2550, USA) for a constant period in order to ensure the pH is lied within the neutral range. The electrode was calibrated with buffer solution of pH 4, 7 and 10 before the measurement. The electrodes were then rinsed with distilled water and dried with a tissue before each measurement.

3.3.5 Transmembrane pressure (TMP)

Transmembrane pressure is the pressure used to force the fluid in the MBR to pass through the membrane. In this study, the transmembrane pressure was measured using transmembrane pressure transducers and the data was recorded by a digital pressure data logger (Logit, USA).

3.3.6 Extracellular Polymeric Substances (EPSs)

EPSs consist of the major components which include protein and polysaccharides. They both were determined using supernatent of the samples. Firstly, the sample was centrifuged using HERMLE Centrifuge at 3000rpm, 9 acceleration at the temperature of 25 ℃ for 30 minutes. Supernatent was extracted into a few test tubes to test for the polysaccharides and protein by using micropipette. The concentration of polysaccharides were determined with the steps as follows, (i) 14mL of phenol filled together with some of deionized water was prepared, (ii) each 1mL of sample was added with 1mL of phenol followed by 5mL of 1mol/L H2SO4 and wrapped the test tubes with aluminium foil wrapper, (iii) the samples are required to place in Vortex Shaker at 1500rpm for 15 seconds and (iv) samples were placed in a dark spot area for 15 minutes and the concentration of polysaccharides were determined later by using HACH UV/VIS spectrophotometer (Model DR 6000) as shown in Figure 3.4. The concentration of protein were determined with the steps as follows, (i) 1mL of sample was added with 10mL Bradford reagent with bovine serum albumin (BSA) as standard, (ii) the samples are required to place in the Vortex Shaker at 1500rpm for 15 seconds and (iii) samples were allowed to settle for 15 minutes and concentration of protein were determined later by using HACH UV/VIS spectrophotometer (Model DR 6000).

Figure 3.5: HACH UV/VIS spectrophotometer (Model DR 6000)

CHAPTER 4

RESULTS AND DISCUSSIONS

In this chapter, performance of the various AnMBRs were discussed and analysed. Membrane fouling is still the large hindrance to the good performance of MBR in wastewater treatment. However, with the addition of PAC in MBR, it was found that PAC would be transformed into biologically activated carbon (BAC). BAC is able to adsorb the foulants and provide the previously attached bacteria to biodegrade the foulants (Ng et al., 2010). In this study, SRT (30days), temperature (35 ℃) and pH (7-8) were constant throughout the entire project. Frequent replenishment of old aged BAC of the AnMBRs is required because the saturated BAC with foulants without refreshing could have adverse effect on the membrane fouling control (Remy et al., 2010).

In this study, different dosages of PAC (1, 3 and 5g/L) were added into three AnMBRs at temperature of 35 ℃ to investigate effects of membrane fouling control. The factors affected the performance of the AnMBRs were compared in terms of their sludge characteristics (MLSS, SMP/EPS, particle size distribution); membrane fouling rate; COD removal efficiency as well as biogas production rate.

4.1 Start-up of AnMBRs

Three bench-scale 1L AnMBRs were setup at the temperature of 35 ℃. The three AnMBRs had different PAC dosages of 1g/L, 3g/L and 5g/L respectively. The indication for stabilisation of the three AnMBRs were investigated via the concentration of MLSS and MLVSS analysis by observing the growth rate of microbes.

4.2 Concentration of MLSS and MLVSS in AnMBRs

The concentration of MLSS represents the concentration of suspended solids including the organic, inorganic or non-biological solids in the bioreactor. Meanwhile, the concentration of MLVSS is the concentration of volatile suspended solids and they consist of microorganisms and organic matter. Therefore, the concentration of MLVSS is approximately equivalent to the amount of microorganisms in bioreactor. In order to conduct experimental analysis, the MLSS and MLVSS were measured 2-3 times per week when the system has reached steady state after 3 months of cultivation.

4.2.1 Effect of different dosages of PAC on MLSS and MLVSS concentration

As per Figure 4.1, the MLSS concentration has a steady increment of 14.7% (from 16.4g/L to 19.2 g/L) and 12.8% (from 19.2 g/L to 22.1 g/L) respectively when the dosage of PAC added into AnMBRs increased from 1 to 3 g/L and 3 to 5 g/L. Meanwhile, the MLVSS concentration also showed a steady increment of 16.0% (from 12.9g/L to 15.4 g/L) and 16.2% (from 15.4 g/L to 18.4 g/L) respectively when the dosage of PAC added increased from 1 to 3 g/L and 3 to 5 g/L. This may be because adsorption effect of PAC could enrich microbial growth as it provide large surface area for microbial to attach, feed and grow.

Moreover, the PAC acts as a shelter for the microorganisms to inhibit the high shear situation.

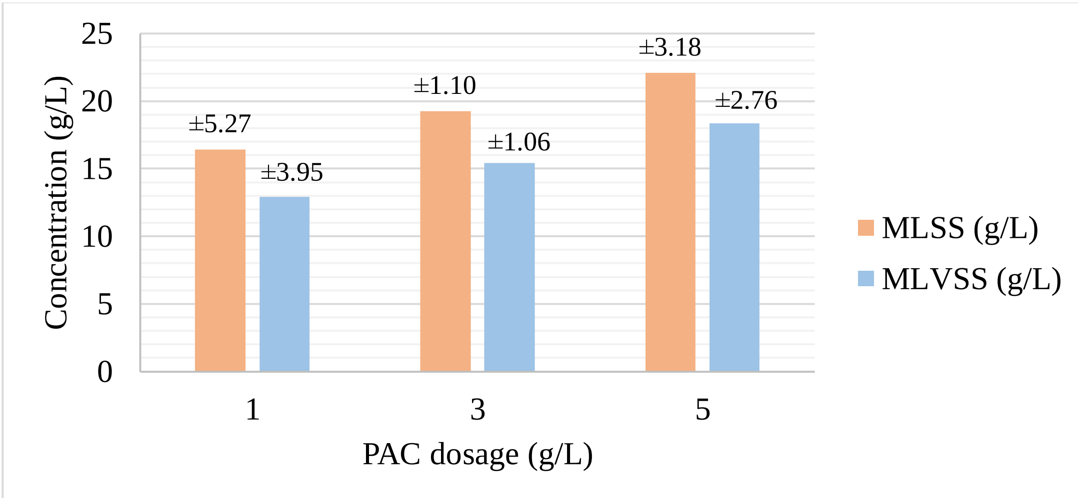

Figure 4.1: The concentration of MLSS and MLVSS at different dosage of PAC at mesophilic regime

4.2.2 Particle size distribution of BAC flocs in volume and number

In Figure 4.2 and Figure 4.3 show the floc size distribution of the AnMBRs at different dosages under mesophilic condition (35 ℃). Based on the volume distribution (D_{50}) curve, 3g/L and 5g/L of BAC were found to have larger floc size compared to 1g/L BAC. Bigger BAC size indicate it can accommodate more microorganisms and also good for membrane fouling control. Figure 4.3 also shows an increase in BAC floc size following an increase in PAC dosages. It could be explained that higher concentration of BAC can enhance the MLSS production and lead to larger floc size of BAC which is good for membrane fouling control.

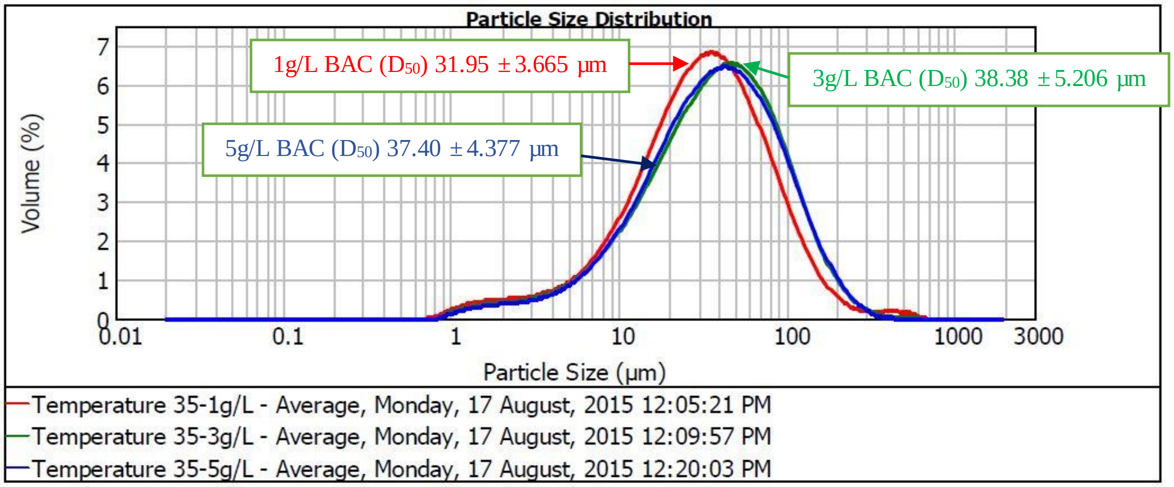

Figure 4.2: Particle size distribution of BAC in volume at different dosages under mesophilic condition

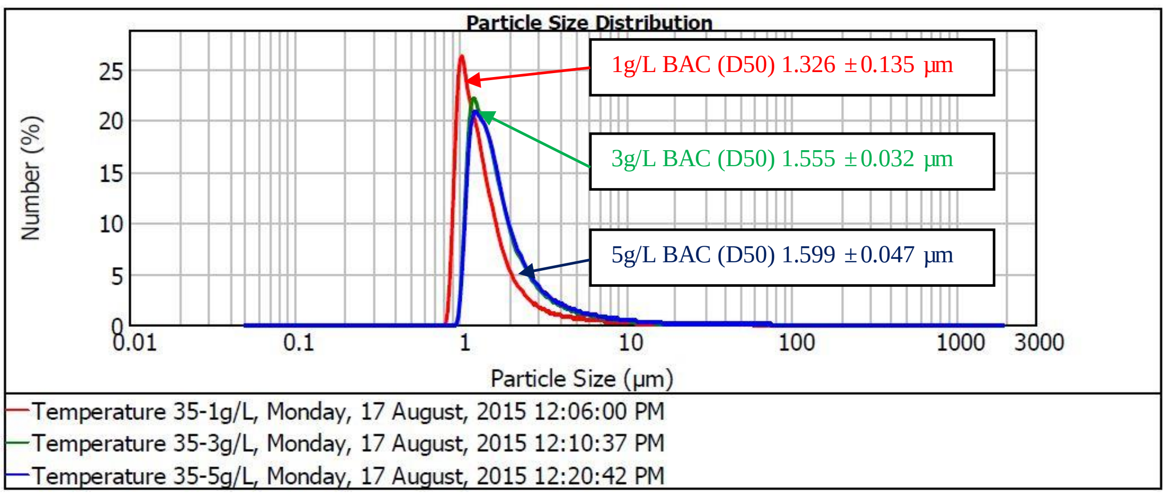

Figure 4.3: Particle size distribution of BAC in number at different dosages under mesophilic condition

4.3 Concentration of protein and polysaccharides in the AnMBRs added with different dosages of PAC at mesophilic regime (35 ℃)

Bound EPS as mentioned previously in chapter 2, it consists of proteins, polysaccharides, lipids, humic acids, nucleic acids, etc (Meng et al., 2006). Figure 4.6 shows an increment in PAC dosages resulted in lower concentration of proteins and polysaccharides. With an increase in PAC dosage, the polysaccharides concentration shows a decrease up to 6% meanwhile the protein concentration shows a decrease up to 9.3%. The PAC added were able to adsorb the organic or inorganic substrates in the AnMBRs and allowed the bacteria attached to surface of PAC to form BAC to biodegrade the extracellular of organics adsorbed. According to Ahmed et al. (2007), when the bound EPS rose, the accumulation of foulants were increased, consequently resulted in the rise of TMP and reduce the performance of membrane fouling control. Figure 4.5 shows the common zig-zag trend of protein concentration from day 1 to day 15 which induces the higher standard deviation as shown in Figure 4.4. This is because the molecular weight of proteins have a wider range from 67 to 200 kilodalton (kDa) and different results may be attributed to different sludge used (Gorner et al., 2003). In the principle of different sludge used, the concentration of protein from feedstock was varied due to the replenishment of feedstock every week.

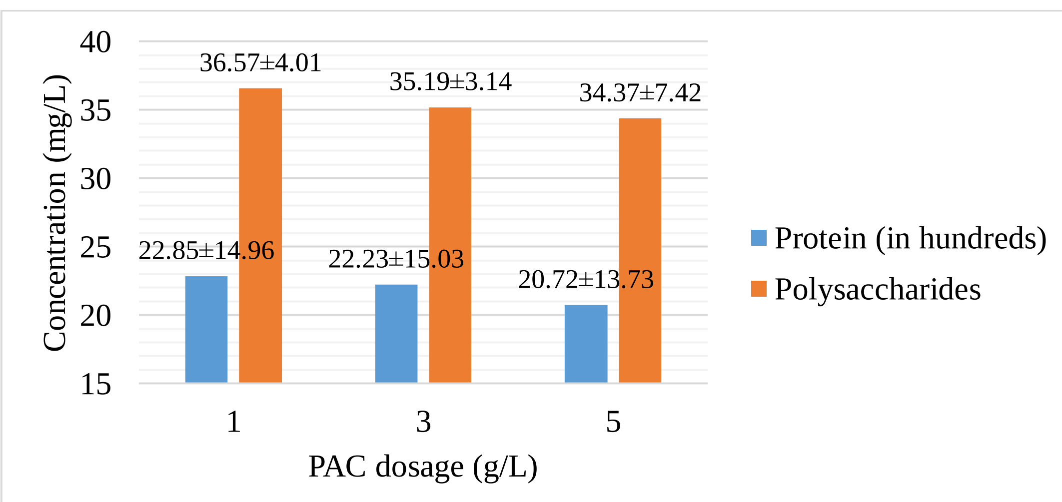

Figure 4.4: Concentration of proteins and polysaccharides with different dosages of PAC at mesophilic regime

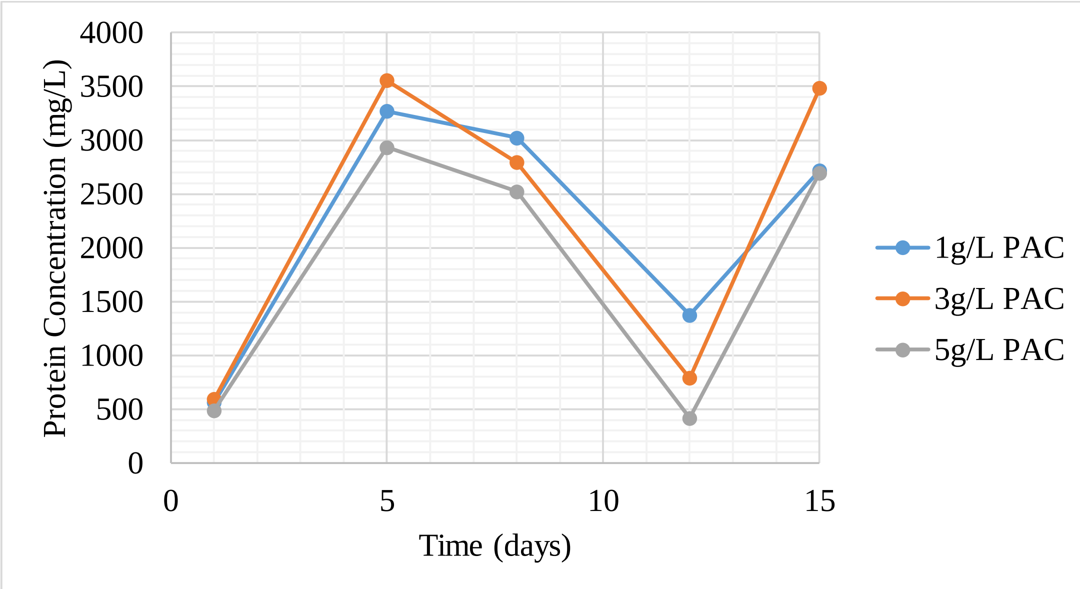

Figure 4.5: Variation of protein concentration during the experimental analysis

4.4 Performance of AnMBRs added with different PAC dosages in membrane fouling control

According to Park et al. (1999), by increasing PAC doses up to 5g/L could reduced the membrane fouling rate and cake layer formation on membrane surface. In this study, performance of three AnMBRs with addition of different PAC dosages were investigated and Figure 4.6 and Figure 4.7 shows that AnMBR added with 1g/L of PAC performed worst compared to the AnMBRs added with higher dosages. The AnMBR added with 5g/L of PAC had the best membrane fouling control. It is because less PAC in AnMBR resulted in less surface area provided for the adsorption of soluble organices and biopolymers, the attachment of microbial cells and fine particles, and higher deposition of inorganic precipitates on membrane surface and thus induce higher TMP and reduce the permeate flux. In addition, higher PAC concentration in the AnMBRs would have large floc size which is good for better membrane fouling control.

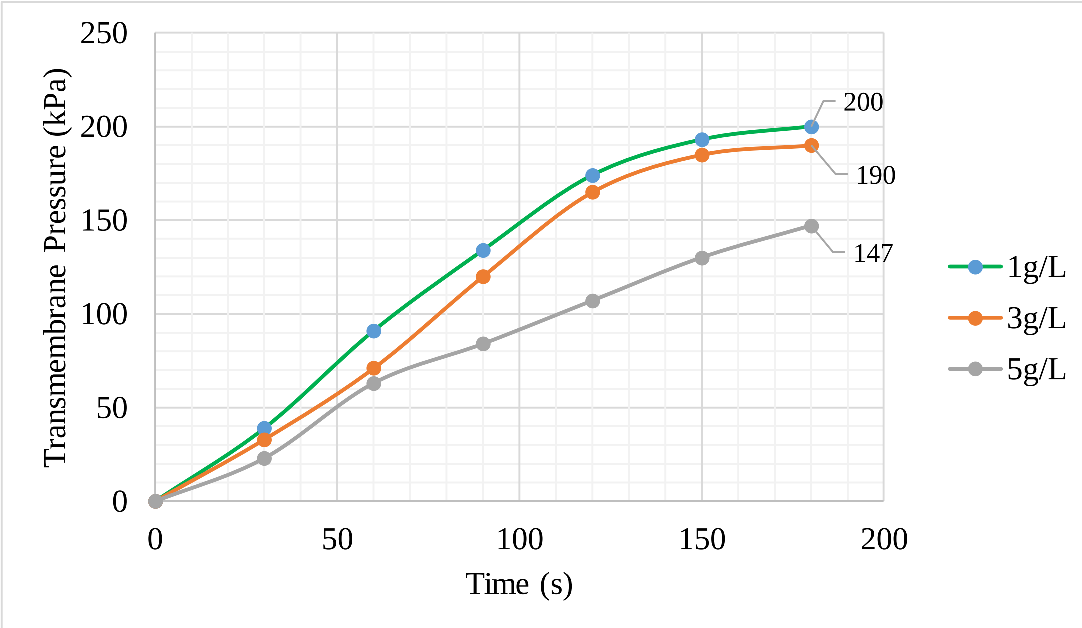

Figure 4.6: Transmembrane pressure of AnMBRs at different PAC dosages added at mesophilic regime

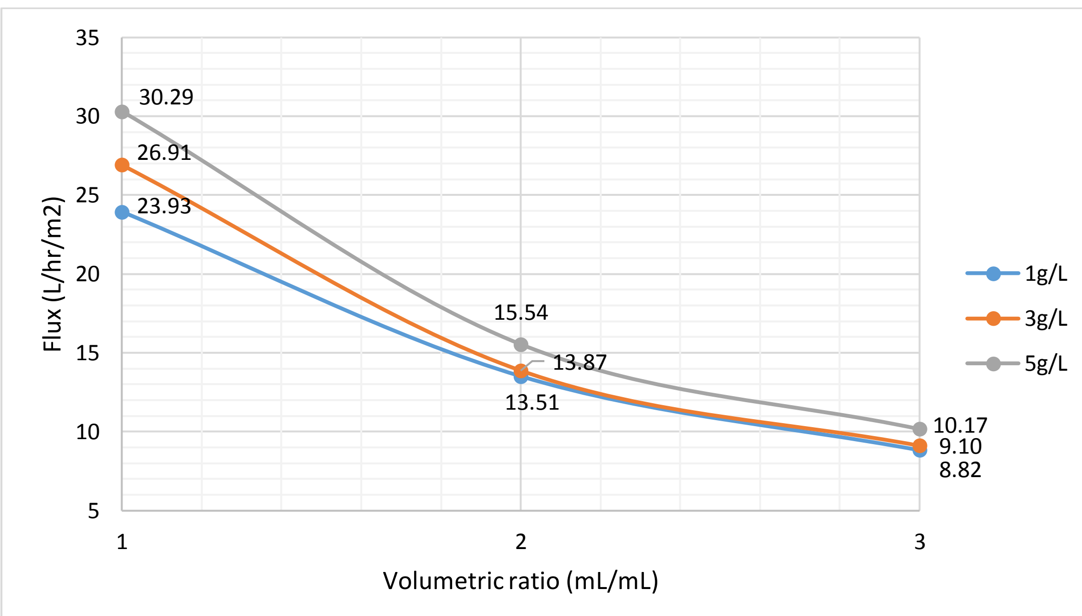

Figure 4.7: Flux decline profiles of AnMBRs at different PAC dosages added at mesophilic regime

4.5 Comparison of biogas production, total COD removal efficiency and reactor efficiency

4.5.1 Effect of different PAC dosages in biogas production

In general, biogas refers to gas produces from anaerobic digestion. This is addressing global energy needs and providing various environmental benefits. The source of biogas production is originated from the energy released from methanogenic activities and metabolism (Mao et al., 2015). Figure 4.8 shows that biogas production was enhanced with an increasing PAC dosages in bioreactor. This can suggest that higher dosage of PAC provides larger surface area as mentioned previously and thus promote relatively higher methanogenic activity (Borowski et al., 2014).

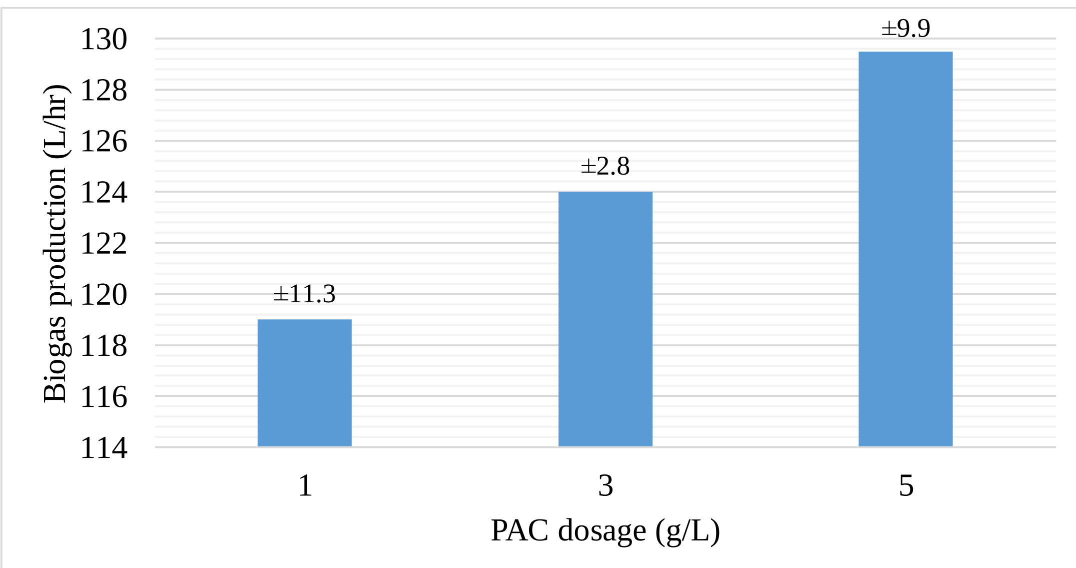

Figure 4.8: Biogas production at different PAC dosages added at mesophilic regime

4.5.2 Effect of different PAC dosages in total COD removal efficiency

Figure 4.9 shows the COD removal rate increased with an increasing of PAC dosages. This may be due to higher BAC concentration is able to remove more COD from the AnMBR through the processes of adsorption, biodegradation and regeneration.

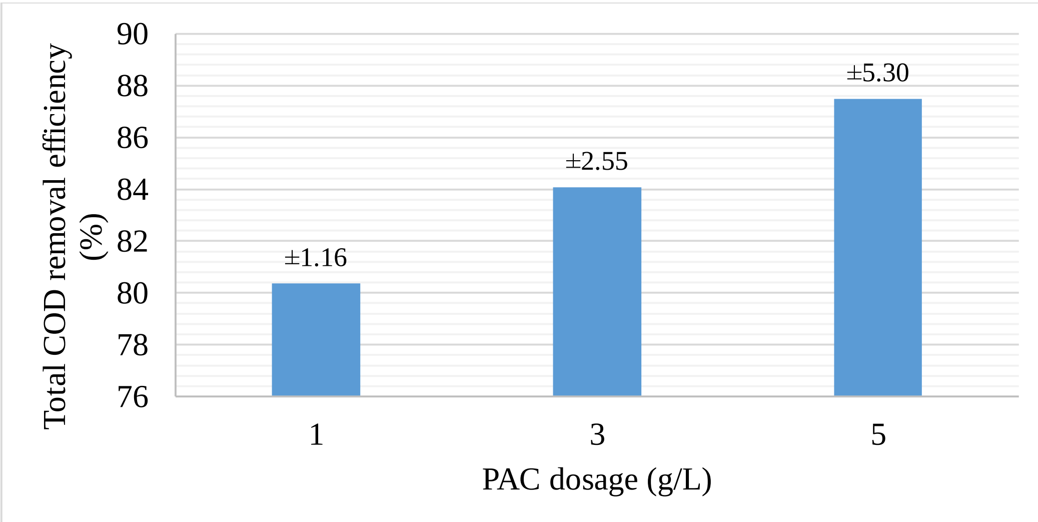

Figure 4.9: Total COD removal efficiency at different PAC dosages added at mesophilic regime

4.5.3 Relationship between biogas production, COD removal efficiency and reactor efficiency

COD represents the organic compounds and it can converted to biogas by bacteria under an anaerobic condition and thus biogas can refer as a direct indication for COD degradation (Mao et al., 2015). In this way, AnMBR operation costs can be reduced by harvesting the biogas and convert it to become energy to support the system. Reactor efficiency is computed as the conversion of the production of biogas for 1g of COD produced. In this study, POME is categorized as high strength wastewater which the conversion of 1g of COD produced 25.11mL/hr of biogas in PAC dosage of 1g/L. On the other hand, in 3g/L (PAC) produced 26.26mL/hr of biogas while 5g/L produced 27.32mL/hr of biogas in the conversion of 1g of COD.

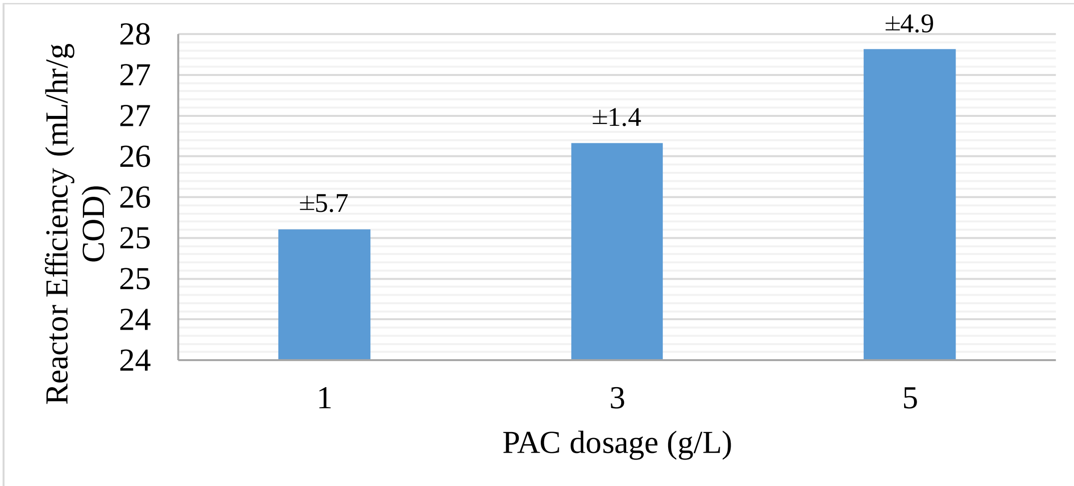

Figure 4.10: The reactor efficiency at different PAC dosages added at mesophilic regime

4.6 Treatment performance of three AnMBRs

The performance of the various AnMBR added with different PAC dosages are summarized as per Table 4.1. Table 4.1 shows that the best performance is the AnMBR added with 5g/L of PAC. This may be due to AnMBR with higher PAC dosages had lower EPS, larger floc sizes and higher MLSS and MLVSS.

Table 4.1: Treatment Performance of AnMBRs

Parameter	AnMBR 1	AnMBR 2	AnMBR 3
Temperature, ℃	**35**	**35**	**35**
SRT, days	**30**	**30**	**30**
HRT, days	**6**	**6**	**6**
PAC dosage, g/L	**1**	**3**	**5**
pH	**7.41 ±0.23**	**7.34 ± 0.25**	**7.38 ± 0.20**
Feed COD, g/L	**4.74 ±1**	**4.74 ± 1**	**4.74 ± 1**
Permeate COD, g/L	0.92 ±0.14	0.74 ±0.04	0.57 ±0.13
Total COD removal efficiency, %	80.36 ±1.16	84.08 ±2.55	87.49 ±5.30
MLSS, g/L	16.40 ±5.27	19.23 ±1.10	22.07 ±3.18
MLVSS, g/L	12.93 ±3.95	15.40 ±1.06	18.37 ±2.76
Protein concentration, mg/L	2284.98 ± 1495.71	2222.76 ± 1503.42	2072.44 ± 1373.24
Polysaccharides concentration, mg/L	36.57 ±4.01	35.19 ±3.14	34.37 ±7.42
Particle size D_{50} (volume), µm	31.95 ±3.67	38.38 ±5.21	37.40 ±4.38
Particle size D_{50} (number), µm	1.326 ±0.14	1.555 ±0.03	1.599 ±0.05
Biogas production, mL/day	119 ±11.3	124 ±2.8	130 ±9.9
Reactor Efficiency ($mLhr^{-1}$ biogas/g COD)	25.11 ±5.7	26.16 ±1.4	27.32 ±4.9

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

In the considerations of cost effective, low dosages of 1g/L, 3g/L and 5g/L of PAC were studied about their efficiency to enhance the performance of the AnMBRs at mesophilic regime instead of thermophilic regime. Several mechanisms has explained the positive effect of low PAC dosages on sludge characteristics, COD removal efficiency and biogas production were investigated. In this study, with an increasing PAC dosages in AnMBR; (i) concentration of activated sludge were increased; (ii) BAC floc sizes were bigger; (iii) EPS concentration (proteins and polysaccharides) were reduced; (iv) total COD removal efficiencies increased and (v) performance of membrane fouling control were enhanced.

The study has shown AnMBRs added with PAC dosage of 5g/L operated at SRT 30 days and mesophilic regime performed best in membrane fouling control. This may be due to the higher PAC dosage provided greater surface area to allow more microbes and organic or inorganic substances to attach on it. In addition, this would enhance the biogas released from the metabolism of microbial activity.

5.2 Recommendations

The study of optimisation on membrane fouling control using mathematical modelling could be carry out in terms of PAC dosages difference, PAC sizes and temperature ranges from ambient, mesophilic and thermophilic regimes. Besides, the mehanisms could be focus more on identification and characterisation of membrane fouling (i.e., biocake architecture, advanced analyses of individual components, two phase fluiddynamics and the role of specific microorganisms). Last but not least, enhancement of the performance of low-cost membrane should be determined by modifying their surface properties.

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APPENDICES

APPENDIX A: Experimental Set-up

Figure A.1: Three AnMBRs with different PAC dosages (1g/L, 3g/L and 5g/L)

Figure A.2: Three biogas collectors and water reservoir tank

APPENDIX B: Membrane Filtration Test

Figure B.1: Cross Flow Membrane Test Rig

Figure B.2: Dead End Membrane Test Rig