# PERFORMANCE OF ANAEROBIC HYBRID MEMBRANE BIOREACTORS INCORPORATED WITH DIFFERENT SIZES OF POWDERED ACTIVATED CARBON AND BIOGAS PRODUCTION IN TREATING PALM OIL MILL EFFLUENT

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

**Faculty of Engineering and Green Technology**

**Universiti Tunku Abdul Rahman**

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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 HYBRID MEMBRANE BIOREACTORS INCORPORATED WITH DIFFERENT SIZES OF POWDERED ACTIVATED CARBON AND BIOGAS PRODUCTION IN TREATING PALM OIL MILL EFFLUENT"** was prepared by **CHAI HUEY YEE** has met the required standard for submission on partial fulfillment of the requirements for the award of Bachelor of Engineering (Hons.) Environmental Engineering at Universiti Tunku Abdul Rahman.

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## ABSTRACT

Anaerobic membrane bioreactors (AnMBRs) have been widely used to treat wastewater and powdered activated carbon (PAC) is found as a good material with its adsorption ability. In this project, performance of four external 1 L hybrid AnMBRs which incorporated with different sizes of PAC (sizes in 471.005 $\pm$ 0.868 µm 226.824 $\pm$ 1.14 µm and 163.884 $\pm$ 1.31 µm) were analysed in terms of chemical oxygen demand (COD) removal efficiency, natural organic matter (NOM) removal rate, mixed liquor suspended solid (MLSS), mixed liquor volatile suspended solid (MLVSS), bioflocs formation, flux rate, membrane fouling control and biogas production. It was found that larger surface area by decreasing particle size of PAC could help to enhance microbial growth rate and relatively increase overall anaerobic digestion (AD) rate resulted higher removal efficiencies of organic matter and higher volume of biogas production. In addition, smaller particle sizes of PAC incorporated into polyethersulfone (PES) membrane has resulted highest performance of membrane fouling control by reducing transmembrane pressure (TMP) and produce better quality of effluent compared to membrane without addition of PAC. The best performance of the AnMBRs in COD, protein and polysaccharide removal efficiencies was 90.55 $\pm$ 0.21 %, 89.24 $\pm$ 1.59 % and 84.96 $\pm$ 0.16 % respectively.

# TABLE OF CONTENTS

## LIST OF TABLES

# LIST OF FIGURES

## LIST OF SYMBOLS/ ABBREVIATIONS

| | |
|---|---|
| AC | Activated carbon |
| AD | Anaerobic digestion |
| AnMBRs | Anaerobic membrane bioreactors |
| BAC | Biological activated carbon |
| BOD | Biological oxygen demand |
| CAD | Conventional anaerobic digestion |
| CAS | Conventional activated sludge |
| COD | Chemical oxygen demand |
| EPS | Extracellular polymeric substances |
| GAC | Granular activated carbon |
| HRT | Hydraulic retention time |
| MBR | Membrane bioreactor |
| MF | Microfiltration |
| MLSS | Mixed liquor suspended solids |
| MLVSS | Mixed liquor volatile suspended solids |
| NF | Nanofiltration |
| NMP | 1-Methyl-2-pyrrolidone |
| NOM | Natural organic matter |

| | |
|---|---|
| PAC | Powdered activated carbon |
| PES | Polyethersulfone |
| POME | Palm oil mill effluent |
| RO | Reverse osmosis |
| SRT | Sludge retention time |
| SS | Suspended solids |
| TMP | Transmembrane Pressure |
| UF | Ultrafiltration |

# LIST OF APPENDICES

# CHAPTER 1

# INTRODUCTION

## 1.1 Background

Malaysia is the second largest palm oil producer in the world contributing large portion to the edible oil globally and increasing the economy growth of oil market nowadays (Ding et al., 2016). The increasing popularity of palm oil in Malaysia is due to its wide applications in many areas such as food manufacturing and fuel for cars. As in February 2016, palm oil mill industries in Malaysia had produced 2,168,798 tonnes of palm oil and a total of 1,085,254 tonnes was exported in that month (Malaysian Palm Oil Board, 2016). However, such large amount of palm oil production has generated relatively large amount of wastewater which is known as palm oil mill effluent (POME). Basically, POME consists high concentration of COD, biological oxygen demand (BOD), and suspended solids (SS) that would lead to pollution of natural water resources if it is not treated properly before being discharged (Ahmed et al., 2015). Examples of some conventional methods designed to treat POME are adsorption, coagulation, membrane technologies, aerobic and anaerobic biodegradation (Tabassum, Zhang and Zhang, 2015).

Membrane bioreactor (MBR) is widely used in municipal and industrial wastewater treatment due to its combined processes of biological degradation and membrane filtration process. There are cases where chemicals are added to further improve its performance such as ion exchange resins and/or silica into MBR (Mutanim et al., 2013; Chaiprapat et al., 2016). MBR is an alternative solution used to replace conventional activated sludge (CAS) treatment system by including membrane filtration and utilizing suspended growth of biomass to remove contaminants without using clarifier (Mutanim et al., 2012; Woo et al., 2016). A MBR system provides various advantages such as (i) minimise excess sludge production, (ii) remove high rate of organic matter, (iii) reduce aeration cost for energy saving, (iv) produce smaller footprint and (v) generate superior effluent quality to achieve a more economical wastewater treatment system (Patsios and Karabelas, 2011; Basset et al., 2016).

Membrane filtration applies both separation and purification process in treating contaminated water, which is able to retain unwanted materials on it by controlling permeation rate effectively (Hong et al., 2015). However, membrane fouling is one of the major problems faced by filtration process, and mitigation to reduce fouling is still an on-going research (Mutanim et al., 2013). Researches had stated that fouling is usually caused by accumulation of macromolecules onto surface of membrane or blocking membrane's pores completely that subsequently prevent membrane from functioning properly (Trzcinski and Stuckey, 2016). Therefore, in order to solve this problem, solutions such as (i) gas sparging (Hong et al., 2002), (ii) backwashing, (iii) membrane brushing, (iv) chemical cleaning, (v) membrane configuration modification (Mutanim et al., 2012), (vi) new membrane materials development (Woo et al., 2016), and (vii) hybrid MBRs with porous and flexible suspended carriers (Cho and Fane, 2002) were implemented to reduce fouling rate of a membrane.

Based on previous research, it was found that activated carbon (AC) can be added onto membrane as a bio-fouling reducer to prolong membrane lifespan (Mutanim et al., 2012). Besides, by adding PAC into membrane bioreactor, it can enhance reduction of organic matter through simultaneously processes of adsorption and biodegradation, and unwanted particles will be retained on membrane surface (Shao et al., 2015). Incorporation of PAC into bioreactor allows biofloc formation attach on it and become biological activated carbon (BAC) which helps biomass to carry out biodegradation process easily (Ng et al., 2013).

## 1.2 Problem Statement

Although membrane bioreactor provides quite a number of benefits in treating wastewater compared to CAS treatment system, membrane fouling is still an on-going problem that would result in increasing operation cost due to higher energy consumption in membrane cleaning and maintenance, reduction in performance, and high membrane replacement cost if membrane is constantly foul and replacement is required (Woo et al., 2016). Even though the membrane can be physically or chemically cleaned when fouling occurred, its total resistance will be decreased and membrane service lifetime is being reduced as well (Meng et al., 2009). By comparison of aerobic MBR and AnMBRs system, reporters found that extracellular polymeric substances (EPS) in supernatant of AnMBRs was five times higher than aerobic system which subsequently causing higher chances to membrane fouling (Martin-Garcia et al., 2011). Thus, membrane fouling control in AnMBRs is a serious issue to be solved.

Through some researches done previously, addition of PAC into AnMBRs can enhance the biodegradation process and mitigate membrane fouling. However, different particle sizes of PAC incorporated into hybrid membrane is yet to be studied. Therefore, in this study different PAC sizes are added into several anaerobic bioreactors to investigate their performance based on contaminants removal and biogas production under controlled temperature and various particle sizes of PAC incorporated into PES hybrid membrane are carried out.

## 1.3 Objectives

The objectives in this study include:

i. To investigate efficiency of anaerobic bioreactor added with different sizes of PAC in treating POME and biogas production

ii. To fabricate and evaluate performance of hybrid membrane incorporated with different sizes of PAC in treating POME wastewater.

## 1.4 Scopes of Study

In this research, scopes of study are shown as following:

i. To evaluate suitable PAC sizes added into anaerobic bioreactor to treat POME and biogas production.

ii. To fabricate PES hybrid membrane by using dry-wet phase technique.

iii. To evaluate performance of different particle sizes of PAC incorporated into PES hybrid membrane in terms of membrane fouling.

## 1.5 Outline of Thesis

In this study, there are five chapters included in the report. Firstly, studies background, problem statement, objectives and scopes of study are included in introduction chapter. Next, literature review such as POME characteristics, introduction to membrane and AnMBRs, anaerobic digestion processes, MBR operating conditions, membrane fouling problem, addition of PAC affect to bioreactors, biogas production and those relevant important information on AnMBRs are included in this chapter meanwhile third chapter consists of research methodology such as instruments used, materials preparation and analytical methods. Forth chapter shows the results and discussions based on other researchers finding throughout the experimental analysis. Lastly, conclusion and recommendations are included in chapter five in order to improve the study.

# CHAPTER 2

# LITERATURE REVIEW

## 2.1 Introduction to Palm Oil Mill Effluent (POME)

POME is a thick brownish liquid mixture, non-toxic and possesses high strength wastewater which is generated from palm oil mill industries (Hassan, Kee and Hussain, 2013). According to Parveen et al. (2010), 1 tonne of crude palm oil production requires 5-7.5 tonnes of water involved in the production process. In the end, more than fifty percent of the water used is become POME. Due to its polluting properties to the natural environment, POME must be treated properly before it is being discharged into ecosystem. Screening, sedimentation and oil removal are found to be physical pre-treatment used for POME before it undergoes biological treatment processes (Parveen et al., 2010).

Characterization of POME is essential to be identified for designing its treatment method selection, process design and equipment sizing of wastewater treatment plant for palm oil mill industries (Poh, Yong and Chong, 2010). The characteristics usually depend on quality of raw materials include fresh fruit bunches in the palm oil production processes (Parveen et al., 2010). Table 2.1 shows general characteristics of raw POME.

**Table 2.1 General Characteristics of Raw POME (Malaysian Palm Oil Board, 2014)**

| **Parameters** | **Value (Average)** | **Range** |
|---|---|---|
| pH | 4.2 | 3.4-5.2 |
| BOD | 25,000 | 10,250-43,750 |
| COD | 51,000 | 15,000-100,000 |
| Total solids | 40,000 | 11,500-79,000 |
| SS | 18,000 | 5,000-54,000 |
| Volatile solids | 34,000 | 9,000-72,000 |
| Oil and grease | 6000 | 130-18000 |
| Ammonical Nitrogen ($NH_3$-N) | 35 | 4-80 |
| Total nitrogen | 750 | 180-1400 |

* All values are in mg/L except pH

Due to the contaminants found in raw POME, Environmental Quality Regulations has set a standard discharge limits to restrict it polluting watercourses (Malaysian Palm Oil Board, 2014). Table 2.2 listed latest data of POME standard discharge limit of different parameters.

**Table 2.2 POME Standard Discharge Limit (Malaysian Palm Oil Board, 2014)**

| **Parameters** | **Discharge Limit (1984 afterward)** |
|---|---|
| pH | 5-9 |
| BOD | 100 |
| COD | 1000 |
| Total Solids | 1500 |
| SS | 400 |
| Oil and Grease | 50 |
| Ammoniacal Nitrogen | 100 |
| Total Nitrogen | - |
| Temperature (℃) | 45 |

* All values are in mg/L except pH and temperature

## 2.2 Membrane

Over the years, membrane technology has become one of the main contributors in treating water resources. Based on different pore sizes distributions and physical properties of membrane, it provides a lot of advantages to remove contaminants effectively in watercourses compared to those conventional water treatment processes such as clarification and filtration (Peinemann and Nunes, 2010). Basically, membrane can be categorized based on its types, configuration and materials made. Further details are discussed as follows.

### 2.2.1 Membrane Filtration Processes

Membrane can be classified based on the membrane pore sizes, driving force, pollutants removed and its applied pressure. There are 4 types of membrane filtration processes: microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO) (Peinemann and Nunes, 2010). In this study, the pore range between MF and UF are required to be used for fabricating hybrid PES membrane in order to retain particles in treated effluent from anaerobic bioreactor during filtration process. Table 2.3 shows general characteristics of membranes filtration processes.

**Table 2.3 Different Types of Membrane Processes with Its Characteristics (Water Environment Federation, 2006; Peinemann and Nunes, 2010)**

| Membrane operation | Pore size range (μm) | Driving force | Pollutants removed | Operating pressure (psi) |
|---|---|---|---|---|
| MF | 0.01-1 | Pressure/ vacuum | Clay, bacteria, viruses, suspended solids | 1-30 |
| UF | 0.001-0.01 | Pressure | Proteins, starch, viruses, organics | 3-80 |
| NF | 0.0001-0.001 | Pressure | Starch, pesticides, BOD, COD | 70-220 |
| RO | <0.0001 | Pressure | Metal ions, acids, sugars, amino acid | 800-1200 |

### 2.2.2 Mode of Membrane Filtration

There are two types of membrane filtration modes which are cross flow filtration and dead end filtration mode.

#### 2.2.2.1 Cross Flow Filtration Mode

According to Wang and Zhou (2013), cross flow filtration mode is feed stream moving parallel to membrane and partial of it passing through the membrane vertically as permeate. Remainder of the feed stream is considered as retentate and it will continue for processing or recirculate back to feed tank. Tangential feed stream keep continuously moving across the surface of membrane can help to prevent those particles accumulation and steady permeate flux with low TMP can be maintained. Figure 2.1 shows process flow of cross flow filtration.

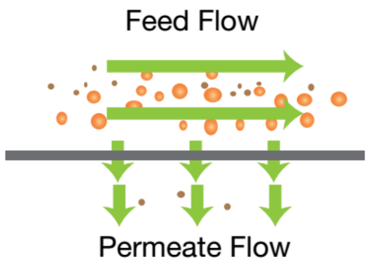


**Figure 2.1 Cross Flow Filtration Mode (Wang and Zhou, 2013)**

#### 2.2.2.2 Dead End Filtration Mode

Dead end filtration mode is feed stream moving and passing through membrane vertically as permeate. Those unwanted materials retain on membrane surface will form a filter cake that consequently cause to reduction in filtrate flux and increase in TMP over time. Its flowing mode is shown in Figure 2.2.

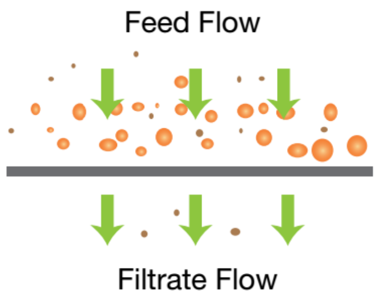


**Figure 2.2 Dead End Filtration Mode (Wang and Zhou, 2013)**

### 2.2.3 Membrane Configuration

In the market, various configuration of membrane are preferably used in MBR system can be tubular, flat sheet and hollow fiber membrane. Tubular membrane is cast inside of a tube as a finely porous surface layer; Flat sheet membrane is made up of paper-like backing material with a membrane cast on plate surface; whereas hollow fiber membrane is many fibers packed into bundles and potted into tubes

(Peinemann and Nunes, 2010). Different types of membrane modules are determined before it is used to treat different kind of contaminants present in wastewater. In this study, flat sheet membrane is used due to its arrangement can be used in external MBR system and it is easier to fabricate compared to other types. Table 2.4 shows the membrane modules with its significant properties. Advantages and disadvantages of these membrane configurations are summarized in Table 2.5.

**Table 2.4 Types of Membrane Modules Used in MBR System with Its Characteristics (Peinemann and Nunes, 2010)**

| Properties | Tubular membrane | Flat sheet membrane | Hollow fiber membrane |
|---|---|---|---|
| **Arrangement** | External- recycling | External/ submerged | External/ submerged |
| **Packaging density** | Low | Moderate | High |
| **Energy demand** | High (turbulent flow) | Low- moderate (laminar flow) | Low |
| **Cleaning** | Efficient + physical cleaning possible | Moderate | Backwashing possible |
| **Replacement** | Tubes / element | Sheets | Element |

**Table 2.5 Advantages and Disadvantages of Different Membrane Types (Peinemann and Nunes, 2010)**

| Membrane module | Advantages | Disadvantages |
|---|---|---|
| **Tubular membrane** | • Long membrane life<br>• High flux rate<br>• Easier mechanically cleaning process<br>• Can tolerate high solids<br>• No need membrane tank | • Expensive membrane replacement cost<br>• High capital cost |
| **Flat sheet membrane** | • Cost effective<br>• Easier cleaning as it can be removed<br>• Simple preparation method | • Cannot be backwashed |
| **Hollow fiber membrane** | • Can be backwashed<br>• Compact design<br>• Better footprint compared to flat sheet membrane<br>• Larger surface per unit volume<br>• Flexible (filtration process in “inside-out” or “outside-in”) | • Cannot withstand pressure shock<br>• Membrane damaged easily |

### 2.2.4 Membrane Based Materials

According to Peinemann and Nunes (2010), there are two major groups of membrane based materials which are inorganic membrane and polymeric membrane. Inorganic membrane is usually made of metals or ceramics that currently considered expensive than polymeric membrane. To date, polymeric membrane is still the preferable type used to treat wastewater. Based on research of Meng et al. (2009), PES can withstand wide range of pH from 2 to 12. It is also good resistant to oil and grease which normally can be found in POME wastewater. Therefore, PES is used to cast hybrid membrane in this study. In addition, consumers should consider selected membrane materials based on raw water quality and operating conditions. Pros and cons of some membrane based materials are summarized in Table 2.6.

**Table 2.6 Advantages and Disadvantages of Membrane Based Materials (Peinemann and Nunes, 2010; Meng et al., 2009; Water Environment Federation, 2006)**

| Membrane based materials | Advantages | Disadvantages |
|---|---|---|
| **Ceramic** | • Good performance in filtration due to high chemical resistance<br>• easy to clean | • Expensive to fabricate<br>• Fragile |
| **Polymeric**<br>• Cellulose acetate | • Solvent cast and easy to fabricate<br>• Inexpensive | • Poor thermal tolerant (only can be used at temperature below 30℃)<br>• Poor chemical tolerance (pH range of 3-6) |
| • Polyethersulfone (PES) | • Highly oxidant tolerant (can withstand >25000ppm/h)<br>• Withstand wide pH range of 2-12<br>• Good resistance to oil and grease<br>• Good fouling resistance (highly hydrophilic) | • Organic solvents like benzene can break material easily |

**Table 2.6 Advantages and Disadvantages of Membrane Based Materials (Peinemann and Nunes, 2010; Meng et al., 2009; Water Environment Federation, 2006) (continue)**

| Membrane based materials | Advantages | Disadvantages |
|---|---|---|
| • Polyamide | • Good thermal tolerant (Can be used at temperature >50℃ ) | • Sensitive to chlorine |
| • Polypropylene | • Withstand wide pH range of 2-14<br>• Good chemical resistance<br>• Can withstand 35 psig TMP | • Sensitive to chlorine<br>• Not oxidant tolerant ($<10^2$ ppm/ h) |
| • Polysulfone | • Withstand wide pH range of 1-13<br>• Good thermal tolerant (Can be used at temperature of 75℃)<br>• Good resistance to chlorine | • Poor chemical resistance to aromatic hydrocarbons |
| • Poly-vinyldenefluoride | • Highly oxidant tolerant (up to 5000 ppm chlorine)<br>• Withstand pH range of 2-10.5<br>• Can withstand 36 psig TMP | • Only can be applied in microfiltration and ultrafiltration pore sizes |
| • Poly-tetrafluoroethylene | • Good thermal tolerant (Can be used at temperature between -100 ℃ and 260 ℃) | • Expensive<br>• Only can be applied in microfiltration pore sizes |
| • Polyacrylnitrile | • Withstand pH range of 2-10<br>• Can withstand 44 psig TMP | |

## 2.3 Introduction to Anaerobic Membrane Bioreactors (AnMBRs)

AnMBRs is one of the treatment systems that combine anaerobic biological wastewater treatment process and membrane filtration process to provide a solid and liquid separation (Basile, Cassano and Rastogi, 2015). An anaerobic bioreactor generally consists of three phases; there are (i) gas phase (biogas), (ii) liquid phase (wastewater) and (iii) solid phase (sludge) (Abdelgadir et al., 2014). Gas phase is referred to produced biogas from biodegradation process by microbes; liquid phase is refer to treating wastewater above sludge bed whereas solid phase is bottom part of bioreactor, which includes sludge granules and microbes (Abdelgadir et al., 2014).

Although AnMBR is similar to aerobic MBR system, it can be functioned (i) to produce biogas (due to anaerobic digestion), (ii) without air injection (due to anaerobic process), (iii) sludge yield reduction, and consequently (iv) reduce operational costs if compared to aerobic one. Besides, as compared to CAS, AnMBRs serve to (i) enhance treatment quality (due to stringent requirement in effluent and mostly removal of present solids), (ii) reduce capital costs (as membrane is used to replace clarifier), (iii) lower footprint (AnMBR plant can be 50% smaller than conventional design) and (iv) reduce operational problems (due to reduction of floating sludge occurrence) (Peinemann and Nunes, 2010).

Configuration of AnMBR system can be categorized into submerged AnMBR and external AnMBR. Submerged AnMBR is system that installing membrane frame which composed of membrane series into process tank whereas external AnMBR installing the membrane module outside of bioreactor (Water Environment Federation, 2006). According to Peinemann and Nunes (2010), operation cost of external AnMBRs is relatively higher than submerged AnMBR due to external pumping system requirement from bioreactor to filtration tank and normally it requires high velocity to transfer treating water.

According to Basile, Cassano and Rastogi (2015), application of membrane filtration allow AnMBRs to have complete biomass retention and provides sufficient sludge retention time (SRT) for methanogens when compare to conventional anaerobic digestion (CAD). Due to poor biomass settling properties of CAD, it consequently resulted in present of biomass in treated effluent (Lin et al., 2013). Therefore, application of AnMBRs has become high rate wastewater treatment system in recent years (Chang, 2014).

## 2.4 Membrane Fouling

Membrane fouling is a physicochemical interaction occurred between membrane and biofluid (Mutanim et al., 2012). It is an action to reduce active area on membrane that consequently causes flux reduction during filtration process (Peinemann and Nunes, 2010; Jhaveri and Murthy, 2015). This phenomenon can be observed through rising rate of TMP which is pressure gradient across the membrane (Wang et al., 2016; Peinemann and Nunes, 2010; Meng et al., 2009). Figure 2.3 illustrates three stages of membrane fouling, which included (i) stage 1: initial adsorption, (ii) stage 2: slow TMP rise and (iii) stage 3: sudden TMP jump which means the membrane is fouled (Yoon, 2016).

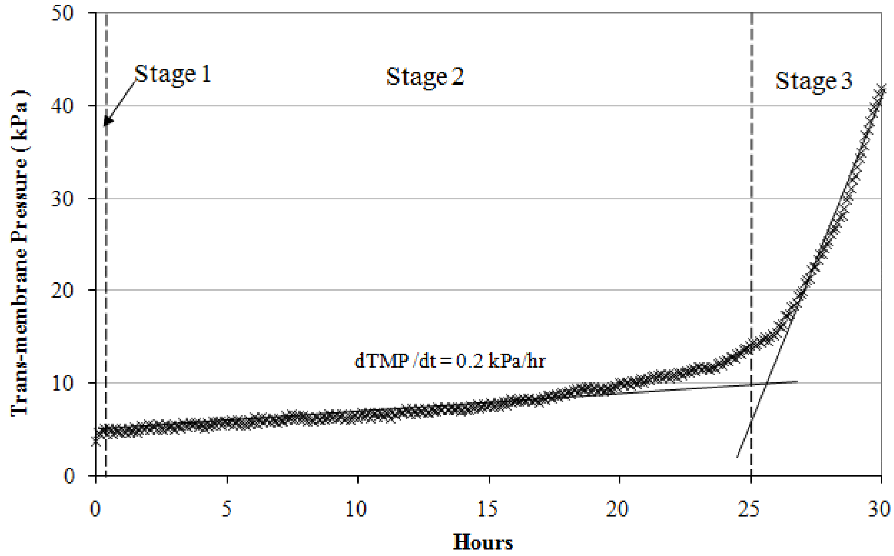


**Figure 2.3 TMP Rising Pattern during Membrane Fouling Occurrence (Yoon, 2016)**

### 2.4.1 Fouling Classification

Fouling can be categorized based on capability of backwash, which is related to attachment strength of contaminants to surface of membrane. For backwash-able fouling, contaminants can be removed by injecting water from opposite direction of permeate flow whereas non-backwash-able fouling only can be solved by application of chemical cleaning (Abdelrasoul, Doan and Lohi, 2013; Jhaveri and Murthy, 2016). However, it is hard to recover original flux due to chemisorb of foulants onto membrane surface (Jhaveri and Murthy, 2016).

Fouling can be further classified into different mode; there are organic fouling, inorganic fouling, colloidal fouling and biological fouling (Abdelrasoul, Doan and Lohi, 2013). Table 2.7 shows a summarization the fouling types and related fouling mode by foulants.

**Table 2.7 Types of Fouling Occurrence and Its Description (Abdelrasoul, Doan and Lohi, 2013; Peinemann and Nunes, 2010; Franken, 2009)**

| Fouling types | Explanation |
|---|---|
| **Organic fouling** | Natural organic matter (include protein & polysaccharide) and oil & grease that accumulate on membrane surface |
| **Inorganic fouling** | Contaminants precipitate when concentration of inorganic foulants (Calcium and Magnesium) increased |
| **Colloidal fouling** | Fouling layer formed by SS (ferric hydroxide, iron and colloidal silica) |
| **Biological fouling** | Bacteria grow on membrane surface and excretion of EPS |

### 2.4.2 Fouling Mechanisms

Fouling is usually caused by (i) adsorption or deposition of macromolecules onto membrane surface; (ii) adsorption of foulants onto pore surface; and (iii) completely pore-blocking (Trzcinski and Stuckey, 2016). Its mechanisms can be classified into four types; there are complete blocking, standard blocking, intermediate blocking and cake filtration (Mutanim et al., 2012; Peinemann and Nunes, 2010). Figure 2.4 illustrates schematic diagram of fouling mechanisms and explanation is summarized in Table 2.8.

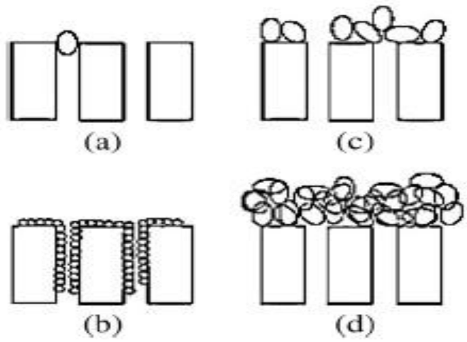


**Figure 2.4 Diagram of (a) Complete Blocking (b) Standard Blocking (c) Intermediate Blocking (d) Cake Filtration (Mutanim et al., 2012)**

**Table 2.8 Fouling Mechanisms with Its Characteristics (Peinemann and Nunes, 2010)**

| Fouling mechanisms | Characteristics |
|---|---|
| **(a) Complete blocking** | Large contaminants totally block the pore and reduce active membrane area |
| **(b) Standard blocking** | Small contaminants adsorb or deposit onto pore walls and restrict pathway |
| **(c) Intermediate blocking** | Contaminants retain on membrane surface partially or completely block the pores |
| **(d) Cake filtration** | Contaminants unable to pass through the pores build-up layer of cake formation on membrane surface and eventually cause fouling |

### 2.4.3 Foulants Types

Foulants are referring to different kind of compounds such as suspended solids, microbes and minerals. It can be divided into two groups which are substances found in feed water and substances generated during process. NOM in treating water is found to be major foulants in MF/UF treatment process whereas EPS is the metabolic product generated by microbes, which is primarily cause of membrane fouling (Shao et al., 2014).

### 2.4.4 Fouling control strategies

There are various types of membrane fouling control strategies such as membrane pre-treatment, hydraulic control, chemical control and biological control (Mutanim et al., 2012; Peinemann and Nunes, 2010; Meng et al., 2009).

Based on research of Peinemann and Nunes (2010), pre-treatment such as coagulation and sedimentation can be applied in membrane feed stream to reduce contaminants which includes organic matter and solid loading. This helps to reduce operating costs and increase in membrane lifetime due to reduction in TMP.

According to Meng et al. (2009), hydraulic control such as periodically backwashing can contribute to flux increment, longer membrane operation period but decrease the total resistance and lifetime of membrane.

Upon chemical control, PAC is suggested to reduce EPS and irremovable fouling in treatment process by its adsorption ability, which acts as bio-fouling reducer (Mutanim et al., 2012). Membrane fouling reducer is a cationic polymer that used to carry out flocculation process of activated sludge, which help to increase porous biofilm on membrane surface and improve permeation rate of membrane (Lee et al., 2007). Therefore, PAC is used in this study to control membrane fouling.

For biological control, increasing SRT can subsequently reduce bound EPS (such as protein, polysaccharide and lipids). It allows microbes to have sufficient time for regeneration of bio-growth and biodegrade those contaminants effectively (Meng et al., 2009).

## 2.5 Anaerobic Digestion Processes

There is a series of bacteria events occurred in anaerobic digestion processes which biodegrade those organic matter into carbon dioxide ($CO_2$) and methane ($CH_4$). These gases also known as biogas which generated from wastewater treatment and used as energy recovery purposes under absence of oxygen condition (Cavinato, 2011; Abdelgadir et al., 2014 and Parajuli, 2011). A flow of anaerobic digestion processes is summarized in Figure 2.5.

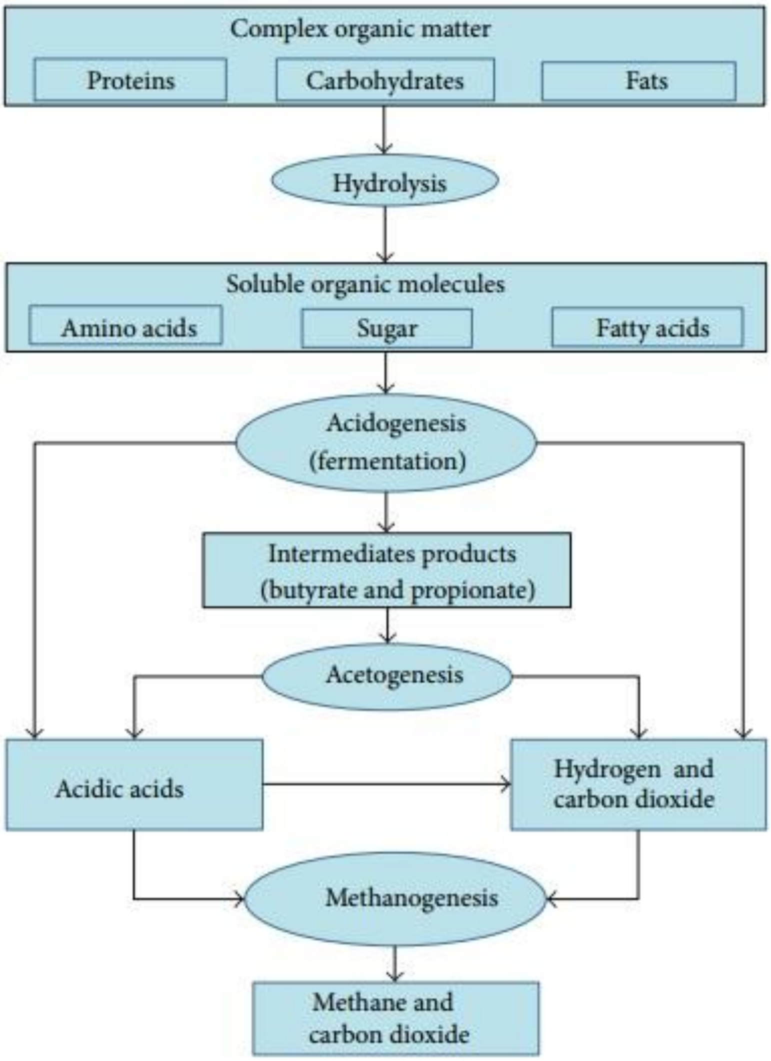


**Figure 2.5 Different Stages at Anaerobic Digestion Processes (Abdelgadir et al., 2014)**

Basically, anaerobic digestion processes mainly consist of four stages, which are hydrolysis, acidogenesis (fermentation), acetogenesis and methanogenesis (Chen et al., 2016; Zhang, Hu and Lee, 2016 and Abdelgadir et al., 2014). The first stage of anaerobic digestion processes, hydrolysis plays an important role as fermentative microbes unable to degrade complex polymer directly. Therefore, hydrolytic enzymes are responsible to convert insoluble organic polymers include lipids, protein and polysaccharide into fatty acids, amino acids and monosaccharide respectively. Next, fermentative bacteria will convert those monomers in first stage into ammonia, organic acids, hydrogen and $CO_2$. Then, acetogenic microbes will further degrade those organic acids to become $CO_2$, hydrogen and acetic acid. In last stage, methanogen bacteria (such as *Methanobacillus* and *Methanobacterium*) will digest all generated intermediate products previously and convert them into $CO_2$, $CH_4$ and $H_2O$ (Chen et al., 2016; Abdelgadir et al., 2014 and Parajuli, 2011).

## 2.6 Operating Conditions of MBR

### 2.6.1 Temperature

In AnMBRs, the operating condition of temperature for anaerobic digestion is usually controlled at either 35℃ which is optimum mesophilic temperature for lower bacteria bio-growth rate or 55℃ which is optimum thermophilic temperature for higher reaction rate (Stuckey, 2012). According to Ferrer et al. (2015), anaerobic microbes' growth rate will decrease significantly if controlled temperature of anaerobic process is too low. By comparing both temperature regimes mentioned previously, thermophilic condition will result better productivity due to higher bearing capacity of organic load than mesophilic condition (Mao et al., 2015). However, microbes with better process stability are found in mesophilic regime (Mao et al., 2015). Basile, Cassano and Rastogi (2015) also stated that effluent from mesophilic bioreactor is found comparatively better than thermophilic condition. This is because higher temperature with higher microbes decaying rate relatively increase amount of EPS in treating effluent (Lin et al., 2009). Furthermore, Jeison and van Lier (2007) also stated that permeate flux from mesophilic bioreactor can be two to three times higher than thermophilic bioreactor. Therefore, mesophilic condition is preferably for membrane filtration system (Basile, Cassano and Rastogi, 2015; Abdelgadir et al., 2014).

### 2.6.2 Sludge Retention Time (SRT)

SRT is a critical parameter used to manage anaerobic processes in MBR system by application of membrane filtration process which allows biomass retention completely (Basile, Cassano and Rastogi, 2015). Most of the MBR system takes SRT from 20 to 70 days (Water Environment Federation, 2006) and Ng et al. (2013) stated that 30 days SRT has better treating performance in MBR compared to 10 days SRT.

According to Mao et al. (2015), wastewater treatment under mesophilic condition requires SRT of 15-30 days to fully biodegrade organic matter. Jadhao and Dawande (2013) also stated that longer SRT can prevent microbes being washed out from bioreactor. Greater amount of biogas production also can be obtained if there is sufficient SRT for anaerobic digestion processes (Chen et al., 2016).

In addition, based on studies of Mutanim et al. (2012), there is relationship between EPS formation and SRT. Longer SRT allow microbes to have sufficient time to carry out biodegradation process due to longer duration staying in bioreactor and consequently cause to EPS being reduced significantly. However, occurrence of membrane fouling become easily due to substance accumulation and higher viscosity of sludge if there is too long of SRT taken (Basile, Cassano and Rastogi, 2015). Besides, it will decrease permeate flux relatively (Smith et al., 2012).

### 2.6.3 Hydraulic Retention Time (HRT)

HRT is another control parameter for MBR, which defined as duration of soluble compound stay in bioreactor and normally range from 2 hours to 30 days (Chen at al., 2016). It can be calculated by using total volume ($m^3$) in bioreactor divided by influent flow rate ($m^3$/d) (Abdelgadir et al., 2014).

Mao et al. (2015) reported that prolong HRT will cause digester components unable to be utilized sufficiently whereas too short of HRT will cause accumulation of volatile fatty acids and result in membrane fouling (Chen at al., 2016). Besides, Chen at al. (2016) also stated that HRT decreased from 12 hours to 6 hours has reduced biogas production and increased COD accumulation in AnMBRs. Therefore, it indicated that AnMBRs system should be operated at HRT for at least 12 hours and longer HRT can result better efficiency in AnMBRs system (Isma et al., 2014).

### 2.6.4 pH

The changes in pH value can affect bacteria growing rate significantly based on their types. Mao et al. (2015) stated that optimum pH value which is pH 7 for methanogenesis bacteria and they can function effectively in pH range of 6.5-8.2. According to Abdelgadir et al. (2014), pH for methanogenesis is required to be maintained within its range as it will become toxic condition if under acidic environment. Also, if the pH value out of this range, methanogens microbes are unable to survive and cause apparently decreasing in their growth rate (Mao et al., 2015). In addition, Abbasi[a], Tauseef and Abbasi[b] (2012) reported that pH range will fall into the range of 7.2-8.2 if the productivity of $CH_4$ is stabilized. On the other hand, Franco et al. (2007) stated that operating pH of anaerobic bioreactor should fall in range of 6.7-7.4. However, most of AnMBRs systems are operating at pH range of 6.5-8.5 due to fermentation process requirement of methane (Chen et al., 2016).

## 2.7 Powdered Activated Carbon (PAC)

PAC is one of the chemical reagents used to allow particles attach on it by its good adsorption ability. It has been widely used in drinking water treatment and proved to be effectively water treatment with addition of PAC into membrane processes includes MF and UF (Shao et al., 2014; Peinemann and Nunes, 2010). Basically, AC can be found into two forms which are granular and powdered AC.

### 2.7.1 Function of PAC

According to Ng et al. (2013), PAC can be used as a protective layer on the membrane surface to prevent those particles blocking the membrane's pores. It can also be used to reduce chances of those fine foulants reaching membrane surface and generate scouring effect to prevent contaminants in water sources accumulate on membrane surface. Meanwhile, PAC also acts as a colony to allow succession bacteria gather to form biofilm ecosystem and improve recalcitrant biodegradation process (Ng et al., 2013). There is another research done by Satyawali and Balakrishnan (2009) had determined that effects of PAC addition into MBR system could help to (i) tolerate shock loading of inhibitory compounds; (ii) flux decline slowly and (iii) improve sludge dewater-ability (by change in particle sizes, floc and incompressible cake formation, and scouring effect). Synergistic effect is mechanism that happened in MBR consequently if PAC is applied. It includes those microbes and contaminants attach on PAC surface and form biofilm subsequently increasing microbial growth population and enzymatic activity. Therefore, it is a good way used to mitigate membrane fouling which widely applied in industrial MBR wastewater treatment (Satyawali and Balakrishnan, 2009).

### 2.7.2 Biological Activated Carbon (BAC)

BAC can be formed when the PAC is added into activated sludge. It enables two processes function simultaneously which includes adsorption and biodegradation process. Formation of BAC allows microbes to get a temporary colony meanwhile to biodegrade those NOM previously attached on PAC. Research found that BAC can help to remove inhibitory materials, microbes, and pollutants in industrial wastewater (Ng et al., 2013). There are also some BAC applications available in market nowadays such as enhancement on substrate removal and activated sludge filterability. However, research also found that aged BAC will relatively decrease performance of membrane filtration due to blocking pores by contaminants (Ng et al., 2013). Therefore, constant PAC replacement in bioreactors is required in treating wastewater.

## 2.8 Biogas

In anaerobic digestion processes of AnMBRs system, biogas is a worthy product that is produced from biodegradable materials in closed system. It mainly consists of 50~70 % methane ($CH_4$), 30~45 % carbon dioxide ($CO_2$), 5~10% moisture content ($H_2O$), and 0.005~2 % hydrogen sulphide ($H_2S$) (Yan et al., 2016). Latest data shows that there is an estimation of 60 tonne fresh fruit bunches per hour in production process can obtain 20,000 $m^3$ of biogas per day if the palm oil mill operating for 20 hours (Malaysian Palm Oil Board, 2014). A calorific value of 53, 000 kcal/$m^3$ can be obtained from the biogas. There were research showed that 1 g of COD removal could produce 0.36 L of $CH_4$ (Chen et al., 2016). Harvested biogas can be used to heat and generate electricity, which acts as renewable and recovery energy sources. This energy makes biogas to be usable biofuel instead of using conventional fossil energy sources such as coal, oil and natural gas (Chen et al., 2016). Meanwhile, biogas plant also provides advantages of saving costs which is an alternative way to purchase electricity from government.

According to Yan et al. (2016), presence of $CO_2$, incombustible component in biogas will relatively reduce the heat content and calorific value of biogas. This component is required to be removed to ensure methane concentration comply with efficient combustion standard with $CH_4$ concentration level > 90%. Common biogas upgrading technologies such as (i) chemical absorption, (ii) membrane separation, (iii) pressure swing adsorption and (iv) water scrubbing are the methods used which can be found nowadays (Yan et al., 2016).

# CHAPTER 3

# RESEARCH METHODOLOGY

This chapter includes experimental set-up, materials preparation and analytical methods of targeted parameters. An overall project flow in this research is illustrated and shown in Figure 3.1.

**Set-up of four 1 L anaerobic hybrid bioreactors at 45 ℃:**

(1) $B_{blank}$ (without PAC addition)

(2) $B_{coarse}$ (Raw PAC size: 471.005 μm)

(3) $B_{medium}$ (Raw PAC size: 226.824 μm)

(4) $B_{fine}$ (Raw PAC size: 163.884 μm)

**PES dope preparation added with different PAC sizes:**

(1) $M_{blank}$ (without PAC addition)

(2) $M_{coarse}$ (Raw PAC size: 471.005 μm)

(3) $M_{medium}$ (Raw PAC size: 226.824 μm)

(4) $M_{fine}$ (Raw PAC size: 100.337 μm)

**PES hybrid membrane casting by using dry-wet phase technique**

**Assessment of performance of anaerobic hybrid bioreactors in terms of:**

(1) pH

(2) COD

(3) EPS (Protein & Polysaccharide)

(4) MLSS & MLVSS

(5) Microbial floc sizes

(6) Biogas production & $CH_4$ content

**Assessment of performance of PES hybrid membrane in terms of:**

(1) TMP

(2) Flux rate

(3) Removal efficiencies of COD, protein & polysaccharide

**Figure 3.1 Project flow**

## 3.1 Experimental Set-Up

In this study, four batches of 1 L external AnMBRs were set-up. First anaerobic bioreactor (namely $B_{blank}$) was designed to treat POME without addition of PAC. The rest of three bioreactors (namely $B_{coarse}$, $B_{medium}$, and $B_{fine}$) were designed to be hybrid bioreactors with addition of different particle sizes of PAC with equal dosages of it. All AnMBRs were equipped with biogas probe, supernatant and sludge collector in order to collect samples respectively. A rubber pipe was connected between each bioreactor and measuring cylinder in water bath in order to determine volume of biogas production. The temperature of system was set at 45℃ (Stuckey, 2012; Ferrer et al., 2015). The SRT and HRT of these anaerobic bioreactors were fixed at 30 days and 6 days respectively. Concentration of PAC used in three bioreactors was 5g/L. 5 wt% PAC was used for preparation of hybrid membranes. These parameters used were based on previous researches in study of optimum operating conditions (Chong, 2015; Tai, 2016). The set up was shown in Figure A1 in appendices.

## 3.2 Materials

### 3.2.1 Palm Oil Mill Effluent (POME)

The POME, high strength industrial wastewater was taken from Tian Siang Oil Mill (Air Kuning) Sdn. Bhd. which is a palm oil mill industry located at Perak. It acts as a food source used to feed bacteria inside AnMBRs and allow them to carry out biodegradation processes. A filtered sieve with mesh size of 0.053 mm (No.270) is required to filter the POME feedstock before it is fed into AnMBRs (Chong, 2015).

### 3.2.2 Powdered Activated Carbon (PAC)

Grinding process is used to allow particles in granular sizes (in millimetre) being ground into powdered sizes (in micrometre). In this study, granular activated carbon (GAC) was ground into PAC in different sizes by using conventional Panasonic blender. Longer time for grinding process will contribute smaller sizes of AC. The GAC was ground with high speed for 10s, 20s, 30s, and 50s in order to make it become different particle sizes. The particle sizes of obtained PAC were determined through particle size analysis in terms of volume and number. The obtained PAC sizes (in $D_{50}$) after grinding process are shown in Figures 3.2 and 3.3 with results of 471.005 ± 0.868 μm, 226.824 ± 1.140 μm, 163.884 ± 1.310 μm, and 100.337 ± 1.340 μm (in terms of volume) and 2.024 ± 0.513 μm, 1.360 ± 0.578 μm, 1.390 ± 0.605 μm, and 1.257 ± 0.596 μm (in terms of number) respectively.

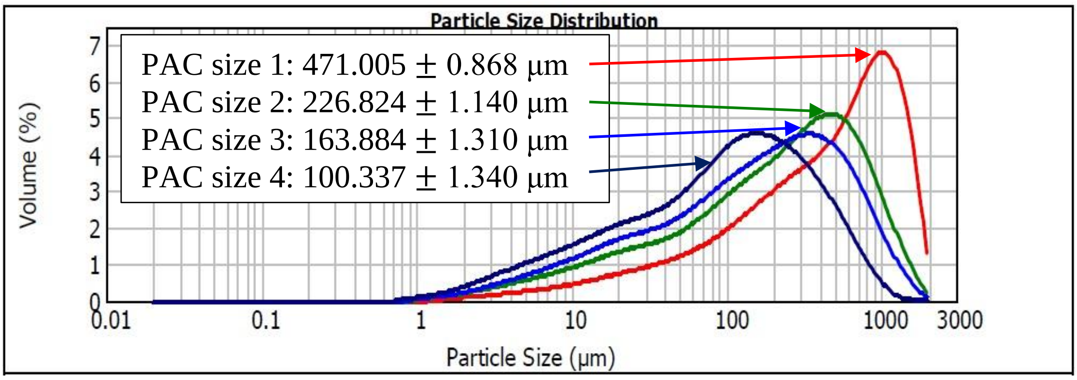


**Figure 3.2 Graph Distribution of Different PAC Sizes in Terms of Volume**

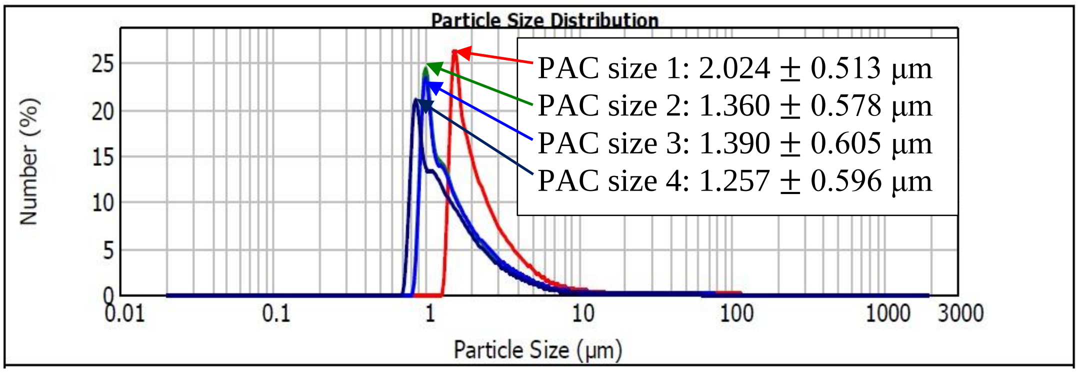


**Figure 3.3 Graph Distribution of Different PAC Sizes in Terms of Number**

### 3.2.3 Membrane

#### 3.2.3.1 Dope Preparation

PES was used to prepare membrane dope before membrane casting process. Amount of chemical usage such as PES and 1- Methyl-2-pyrrolidone (NMP, 99%) for different hybrid membrane dope preparation were calculated accordingly. Mass ratio used for chemical dosages calculation of NMP and PES was 87: 13. 100g of dope was required for every sample. Therefore, 87g of NMP and 13g of PES were used to produce the dope without addition of PAC. Concentration percentage of PAC used was 5% based on weight of PES. Calculated formula for dope preparation with different PAC particle sizes was tabulated in Table 3.1.

**Table 3.1 Formula for Dope Preparation**

| Samples | NMP (g) | PES (g) | PAC (g) | Particle sizes of PAC used (µm) |
|---|---|---|---|---|
| $M_{blank}$ | 87.00 | 13.000 | - | - |
| $M_{coarse}$ | 87.00 | 12.350 | 0.650 | 471.005 |
| $M_{medium}$ | 87.00 | 12.350 | 0.650 | 226.824 |
| $M_{fine}$ | 87.00 | 12.350 | 0.650 | 100.337 |

Instrument and material such as three head round-bottomed flask and PES were placed in oven at 60 ℃ for 24 hours in order to remove moisture content. Heating mantle was set up and magnetic stirrer was placed inside the flask to help in homogenous mixing. Then, NMP was poured into three head round-bottomed flask. Solvent (NMP) was heated and controlled at temperature between 60 $^{o}$C and 70 $^{o}$C with slow stirring speed.  When the temperature was maintained at desired range for 10 minutes, PES was added by using spatula and stirring speed was increased to higher rate until PES fully dissolved together with solvent. Heating mantle was switched off and the dope was allowed to cool down. The dope was then poured into schott bottle and required to be placed into sonicator bath for 8 hours before membrane casting process. This process is used to remove excess air bubbles remained inside the dope and also ensure the dope is homogenous mixing (Tai, 2016).

#### 3.2.3.2 Hybrid Membrane Casting

Dry-wet phase technique was used to fabricate membrane by using semi-auto membrane casting machine. Knife gap with thickness of 15 μm was selected to be measured for the membrane thickness. Firstly, prepared dope was poured on the top of glass mounted on the machine and spread out automatically and evenly on the glass surface. Then, the glass was removed and slowly immersed into water bath. A layer of polymeric firm was formed by the dope on the glass and separated automatically from glass surface. This casted membrane was required to be left in water bath for 24 hours followed by immersing in methanol for 8 hours as a post-treatment purpose. After that, the membrane will be kept in water for storage to prevent it from drying out. Casted membrane was required to be cut into circle shape with diameter of 50 mm which is suitable to be placed in filtration testing system (Tai, 2016).

## 3.3 Analytical Parameters Methods

### 3.3.1 Mixed Liquor Suspended Solid (MLSS) and Mixed Liquor Volatile Suspended Solid (MLVSS)

MLSS and MLVSS was measured based on Standard Methods for the Examination of Water and Wastewater. Firstly, filtering crucibles with filter paper were placed into muffle furnace at 550 ℃ for 15 minutes and cooled down in desiccator for 5 minutes. Mass of filtering crucible with filter paper was measured and recorded by using M-power Analytical Balance AZ214. 1mL of sludge sample extracted from bioreactors was then required to be filtered through filtering crucible by using vacuum suction pump. Then, the filtered samples in crucible were placed into oven at

105 ℃ for 2 hours and cooled down in desiccator. The dried samples were measured and recorded to determine MLSS. After being weighed, dried samples were required to be placed into Muffle Furnace at 550 ℃ for 15 minutes and cooled down in desiccator. The ending dried samples were measured and recorded to determine MLVSS.

### 3.3.2 Chemical Oxygen Demand (COD)

COD amount contained in feedstock, supernatant in bioreactor and filtrated samples were measured based on Closed Reflux Colorimetric Standard Method stated in Standard Methods for the Examination of Water and Wastewater. Extracted samples were undergone dilution and injected into HACH COD test kit with range of 20 to 1500 mg/L (high range) or 200 to 15000 mg/L (high range plus) which depend on sample’s concentration. The test kits were then placed and heated in COD reactor block for 2 hours at 150℃. After heating, the test kits were taken out and allowed to cool down. The COD of samples were then measured by using HACH UV/VIS spectrophotometer (Model DR 6000).

### 3.3.3 Protein

Protein contained in supernatant in bioreactor and filtrated samples were measured in this study. Extracted samples were required to undergo dilution with ratio of 1:25. 0.5 mL of each diluted sample with 5mL Bradford Reagent with bovine serum albumin (BSA) as standard was injected into test tube by using pipette. The samples were required to be placed in the Vortex Shaker at 1500rpm for 15 seconds. The samples were then allowed to settle for 15 minutes and concentration of protein was measured by using HACH UV/VIS spectrophotometer (Model DR 6000) (Tai, 2016).

### 3.3.4 Polysaccharide

Polysaccharide concentration in treated supernatant in bioreactor and filtrated samples were measured by using phenol-sulfuric acid method (Tai, 2016). Extracted samples were required to undergo dilution with ratio of 1:25. 1mL of each diluted sample was added with 1mL of phenol followed by 5 mL of 1 mol/L $H_2SO_4$. The solution was required to be wrapped with aluminium foil wrapper due to light sensitive characteristic of phenol. The samples were placed in Vortex Shaker at 1500 rpm for 15 seconds. The samples were then allowed to settle for 15 minutes at dark spot area and concentration of polysaccharide was measured by using HACH UV/VIS spectrophotometer (Model DR 6000).

### 3.3.5 Transmembrane Pressure (TMP)

TMP is the pressure used to force fluid to pass through the membrane. In this study, TMP was measured by using TMP transducers and the data was recorded by a digital pressure data logger.

### 3.3.6 Biogas Production

Biogas production from bioreactors were measured by using water displacement method (Parajuli, 2011). One of the ends of rubber pipe was connected to bioreactor and another end was inserted up into the inverted measuring cylinder (250 mL). The inverted measuring cylinder was placed into water bath and filled with water until a certain level. The initial reading at the level was recorded. Biogas produced from the bioreactor would be transferred through the rubber pipe and the water level was gradually decreased. After 1 hour, the final reading was recorded and volume of the water being displaced was considered volume of biogas produced.

### 3.3.7 Biogas Content

Biogas content was measured by using RASI 700 biogas analyser. The analyser can be used to measure amount of $CH_4$ content in collected biogas.

### 3.3.8 Particle Size Distribution

Particle size of biomass floc and PAC were measured by using Malvern Mastersizer 2000 particle size analyser. This analyser is able to differentiate particle sizes from range of 0.02 to 2000 μm.

### 3.3.9 pH Measurement

pH meter (Hanna HI 2550, USA) was used to measure pH value of the samples. Buffer solution with pH of 4, 7, and 10 were used for calibration process before the pH measurement of samples in order to prevent error occurrence.

### 3.3.10 Performance of PES Hybrid Membranes

The supernatant of best treating efficiency out of four anaerobic hybrid bioreactors was used to be filtered through casted PES hybrid membrane with addition of different PAC particle sizes (PAC concentration in 5 wt%). Extracted supernatant was tested with different particle sizes of PAC PES hybrid membrane. This process was operated by using cross flow and dead end filtration system.

# CHAPTER 4

# RESULTS AND DISCUSSIONS

This chapter discusses about performance of (i) anaerobic hybrid bioreactors by adding different sizes of PAC in terms of removal efficiencies of COD, protein, polysaccharide, amount of MLSS and MLVSS, biogas production, and (ii) hybrid membranes incorporated with different sizes of PAC in terms of membrane fouling and flux rate determination.

## 4.1 Assessment of Performance on Anaerobic Hybrid Bioreactors

In this study, four anaerobic hybrid bioreactors were investigated and discussed respectively. The PAC sizes used in different hybrid bioreactors and overall treating performance were tabulated in Table 4.1. Firstly, pH value of each condition in bioreactors were determined to ensure it was suitable condition for bacteria growth. The condition were maintained and stabilized at approximately pH 7.8 throughout the study. This pH condition stated the anaerobic digestion processes in bioreactors were balanced and treated water had fulfilled standard discharge limit (Malaysian Palm Oil Board, 2014). The experiment showed that bioreactor without addition of PAC which namely $B_{blank}$ had the lowest treating efficiency compared to the rest of bioreactors which added with PAC of different particle sizes. Smaller PAC sizes applied in bioreactors had the best removal efficiency compared to bioreactors added with larger PAC sizes. By comparison of COD removal efficiencies, $B_{blank}$ had reached $64.90 \pm 1.46\%$ whereas $B_{fine}$ which applied the smallest PAC sizes into bioreactors had reached 78.53 $\pm$ 0.66%. This indicated B $_{fine}$ had the best performance to treat POME in this study.

PAC acts as a colony to allow microbes attached on it and they are synergistic and mutual in bioreactors (Satyawali and Balakrishnan, 2009). In anaerobic bioreactors, PAC was developed to BAC and carried out biodegradation process to decompose natural organic matter (NOM) in POME (Ng et al., 2013). Constant replenishment of PAC was done based on fixed SRT (30 days) in order to maintain the PAC concentration in bioreactors as aged BAC will relatively affect NOM removal efficiency (Ng et al., 2013). The presence of PAC in smaller sizes had contributed larger surface area that allowed more microbes and organic matter adsorbed on it, resulting an enriched environment for microbes' metabolism. Thus, increase in surface area of PAC due to its famous adsorption characteristics can relatively improve the COD removal efficiency and treating performance.

**Table 4.1 Performance of Anaerobic Hybrid Bioreactors Added with Different Sizes of PAC**

| **Parameter** | **B** $_{\text{blank}}$ | **B** $_{\text{coarse}}$ | **B** $_{\text{medium}}$ | **B** $_{\text{fine}}$ |
|---|---|---|---|---|
| Temperature, ℃ | 45 | 45 | 45 | 45 |
| SRT, days | 30 | 30 | 30 | 30 |
| HRT, days | 6 | 6 | 6 | 6 |
| Raw POME pH | 4.16 ± 0.01 | 4.16 ± 0.01 | 4.16 ± 0.01 | 4.16 ± 0.01 |
| Supernatant pH | 7.79 ± 0.01 | 7.81 ± 0.02 | 7.73 ± 0.07 | 7.78 ± 0.04 |
| PAC dosage, g/L | NA | 5 | 5 | 5 |
| Raw PAC size, $D_{50}$ (volume), μm | NA | 471.005 ± 0.868 | 226.824 ± 1.140 | 163.884 ± 1.310 |
| Raw PAC size, $D_{50}$ (number), μm | NA | 2.024 ± 0.513 | 1.360 ± 0.578 | 1.390 ± 0.605 |
| Raw POME COD, mg/L | 95729 ± 5115 | 95729 ± 5115 | 95729 ± 5115 | 95729 ± 5115 |
| Feed in COD, mg/L | 7658 ± 408 | 7658 ± 408 | 7658 ± 408 | 7658 ± 408 |
| COD of supernatant, mg/L | 2695 ± 337 | 2075 ± 305 | 1937 ± 322 | 1647 ± 175 |
| Protein of supernatant, mg/L | 2407 ± 230 | 1677 ± 124 | 1194 ± 122 | 887 ± 247 |
| Polysaccharide of supernatant, mg/L | 71 ± 1.827 | 66 ± 0.435 | 57 ± 1.481 | 52 ± 1.073 |
| Removal efficiency of COD, % | 64.90 ± 1.46 | 72.99 ± 1.47 | 74.80 ± 1.66 | 78.53 ± 0.66 |

## 4.2 Assessment of MLSS & MLVSS in Various Anaerobic Hybrid Bioreactors Added with Different Sizes of PAC

Ratio of MLVSS to MLSS is used to determine sludge activities as it will significantly affect to treating performance of bioreactors (Fan et al., 2015). MLSS is defined as total concentration of biological and non-soluble solids whereas MLVSS is total number of microbes' concentration. In this study, biomass concentration of four anaerobic hybrid bioreactors were measured and analysed which is shown in Table 4.2.

**Table 4.2 Comparison of MLSS & MLVSS in Hybrid Bioreactors Added with Different Sizes of PAC**

| Parameter | $B_{blank}$ | $B_{coarse}$ | $B_{medium}$ | $B_{fine}$ |
|---|---|---|---|---|
| **MLSS, mg/L** | $10900 \pm 1200$ | $13800 \pm 100$ | $17800 \pm 3100$ | $18950 \pm 3850$ |
| **MLVSS, mg/L** | $9250 \pm 1250$ | $12700 \pm 700$ | $15450 \pm 2450$ | $16100 \pm 3200$ |
| **MLVSS/MLSS** | $0.85 \pm 0.01$ | $0.92 \pm 0.03$ | $0.87 \pm 0.01$ | $0.85 \pm 0.002$ |

Throughout the experiment, the ratio of MLVSS to MLSS in bioreactors were stabilized and maintained at approximately 0.85 which is allowable condition for bacteria survival (Fan et al., 2015). It stated that there was enough food for microbes' growth and able to degrade NOM sufficiently. Based on Table 4.2, $B_{fine}$ had the highest biomass with $16100 \pm 3200$ mg/L whereas $B_{blank}$ had only $9250 \pm 1250$ mg/L which was the lowest biomass obtained. This increment results indicated addition of PAC and more surface area on smaller PAC particle sizes could benefit biomass activities and growth rate in bioreactors. Higher suspended biomass rate can also help to remove NOM in POME subsequently increase contaminants removal efficiencies.

### 4.3 Assessment of Microbial Floc Size in Various Anaerobic Hybrid Bioreactors Added with Different Sizes of PAC

Sludge floc is one of the parameters causing membrane fouling and it can be controlled by application of PAC (Lee and Kim, 2013). Transformation of PAC to BAC in bioreactors can help to enhance membrane filtration performance by reducing and preventing NOM reaching membrane surface (Ng et al., 2013). Microbial floc sizes from different bioreactors were determined in terms of volume and number which are shown in Figures 4.1 and 4.2 respectively.

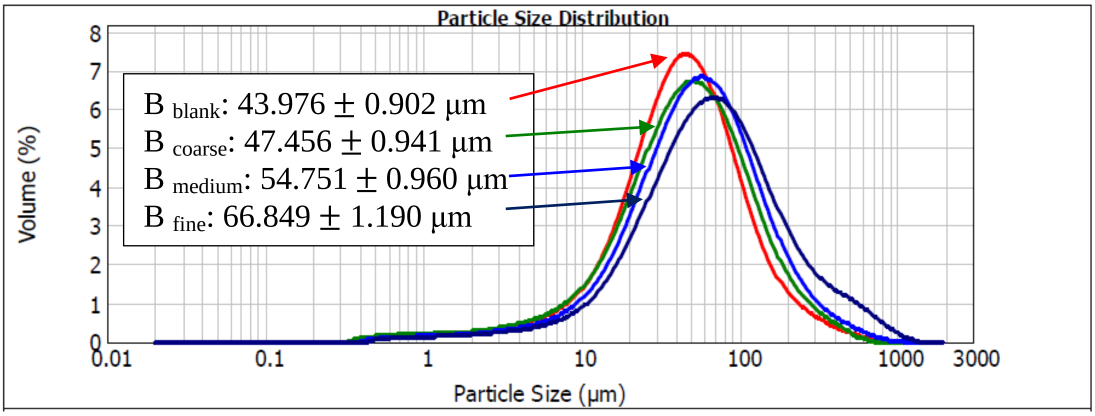


**Figure 4.1 Microbial Floc Sizes Distribution of Different Anaerobic Hybrid Bioreactors in Terms of Volume**

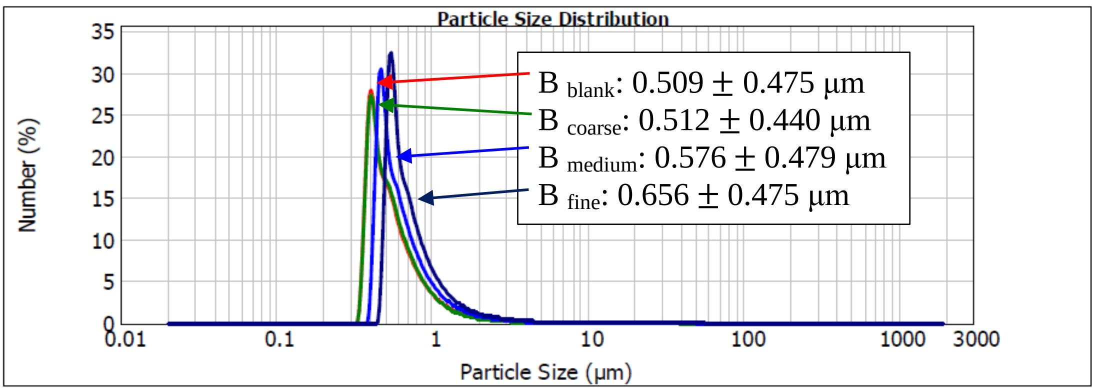


**Figure 4.2 Microbial Floc Sizes Distribution of Different Anaerobic Hybrid Bioreactors in Terms of Number**

B $_{blank}$ was found to be the smallest particle sizes in biofloc among others which was only 43.976 ± 0.902 μm whereas B $_{fine}$ was 66.849 ± 1.190 μm in terms of volume (in $D_{50}$). From Figures 4.1 and 4.2, it is proven that larger biofloc can be formed by adding PAC into bioreactors. There were increment results in biofloc sizes by added coarse PAC to fine PAC separately into bioreactors. B $_{fine}$ was observed to be the largest biofloc size due to the greatest surface area that allowed microbes' population gathered together and form larger biofloc relatively.

## 4.4 Membrane Fouling Control by Adding Different Sizes of PAC into Anaerobic Hybrid Bioreactors

In this study, cross-flow filtration test was carried out to determine overall efficiencies of AnMBRs by using polymer membrane. Permeate of each anaerobic bioreactors undergone filtration process were tested and results were tabulated in Table 4.3. It could be observed that B $_{blank}$ had relatively poor COD removal efficiency (which was only 72.03 ± 0.74 %) than B $_{coarse}$, B $_{medium}$, and B $_{fine}$ with results of 79.91 ± 0.16 %, 79.86 ± 1.12 %, and 80.36 ± 1.29 % respectively. There was same trend obtained by comparing removal efficiencies of protein and polysaccharide, which indicated that higher microbes' population could result better treating efficiency.

**Table 4.3 Performance of Anaerobic Hybrid Bioreactors towards Membrane Fouling Control**

| **Parameter** | **B $_{blank}$** | **B $_{coarse}$** | **B $_{medium}$** | **B $_{fine}$** |
|---|---|---|---|---|
| COD of permeate, mg/L | 1718 ± 82 | 1354 ± 27 | 1233 ± 55 | 948 ± 56 |
| Protein of permeate, mg/L | 1229 ± 41 | 654 ± 13 | 203 ± 20 | 108 ± 6 |
| Polysaccharide of permeate, mg/L | 19.98 ± 1.42 | 13.34 ± 0.27 | 11.43 ± 1.39 | 10.59 ± 1.59 |
| Removal efficiency of COD, % | 77.56 ± 0.07 | 82.32 ± 0.34 | 83.90 ± 0.08 | 87.62 ± 0.04 |
| Removal efficiency of protein, % | 48.76 ± 1.85 | 60.92 ± 1.23 | 83.03 ± 0.05 | 87.28 ± 1.74 |
| Removal efficiency of polysaccharide, % | 72.03 ± 0.74 | 79.91 ± 0.16 | 79.86 ± 1.12 | 80.36 ± 1.29 |

EPS such as protein and polysaccharide are major metabolic products of bacteria lead to membrane fouling (Shao et al., 2014). As shown in Figure 4.3, addition of PAC had performed better compared to AnMBRs without PAC. B $_{blank}$ was fouled easily in short time and B $_{fine}$ performed the best in membrane fouling control. This phenomenon was caused by untreated EPS and NOM from bioreactors reached membrane surface, blocked membrane's pores and promote cake formation that resulted increased in TMP shortly. For the best performer, B $_{fine}$, reduction in EPS in bioreactors had successfully reduced amount of EPS reaching membrane surface and relatively extended rising time of TMP.

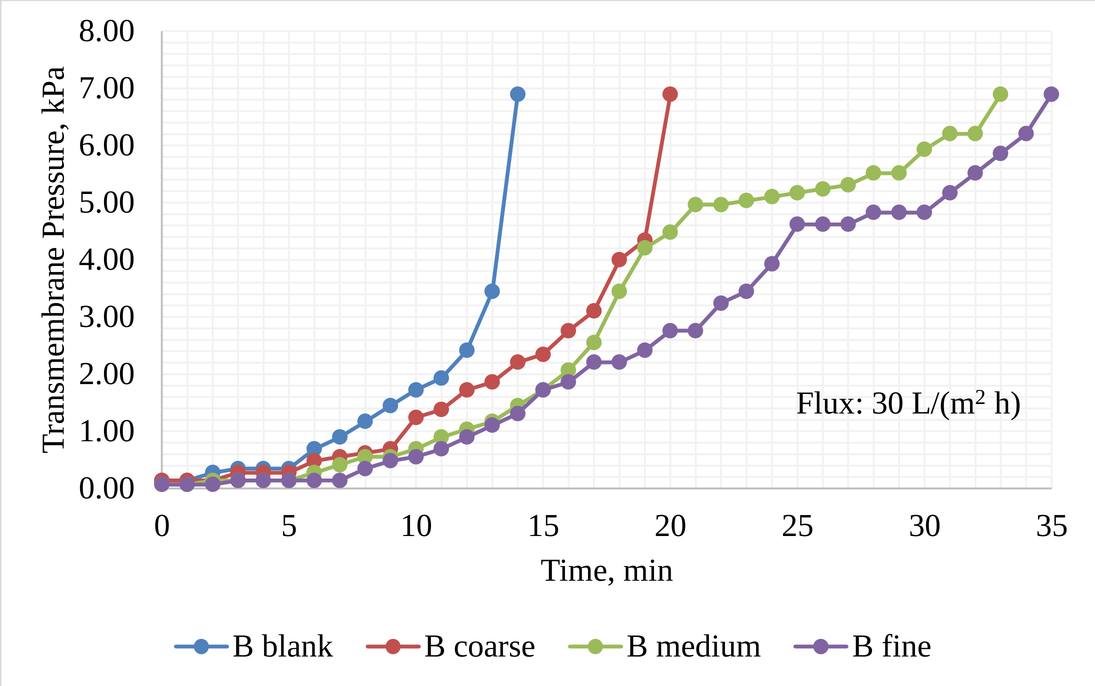


**Figure 4.3 Comparison of Different Anaerobic Hybrid Bioreactors towards Membrane Fouling Control**

### 4.5 Effects of Biogas Production in Various Anaerobic Hybrid Bioreactors Added with Different Sizes of PAC

Bacteria has ability to convert organic matter into biogas during anaerobic biodegradation process (Chen et al., 2016; Zhang, Hu and Lee, 2016). In this study, biogas production from hybrid bioreactors were measured and shown in Figure 4.4 and Table 4.4. There was an increment trend which stated that $B_{blank}$ had the lowest biogas production of 111 ± 5 mL/h whereas $B_{fine}$ had produced the highest volume of biogas of 142 ± 12 mL/h.

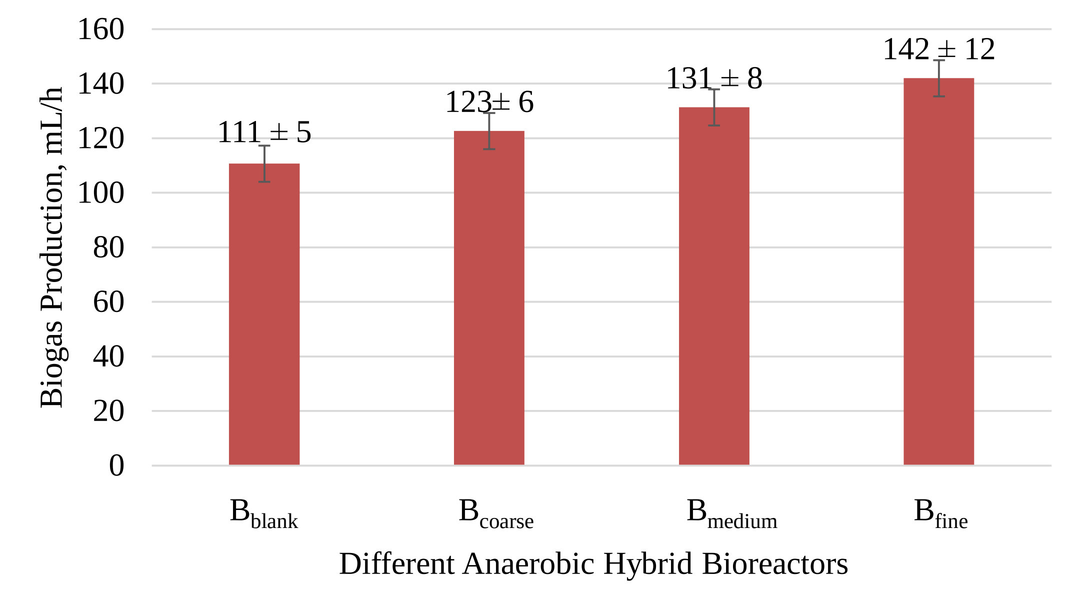


**Figure 4.4 Biogas Production in Various Anaerobic Hybrid Bioreactors Added With Different Sizes of PAC**

PAC transformed into BAC in anaerobic bioreactors could help to enhance anaerobic digestion processes in faster rate and promote methane-forming bacteria biodegrade NOM subsequently produced biogas effectively (Chen et al., 2016). Generated methane ($CH_4$) in biogas acts as a renewable energy sources that can be harvested and converted into electricity which is a way of saving operation cost and environmental friendly concept (Yan et al.,2016). Based on Table 4.4, $B_{fine}$ had reached the highest volume of methane produced with result of 81.77 ± 4.01 mL/h followed by $B_{medium}$ (75.15 ± 2.51 mL/h), $B_{fine}$ (70.56 ± 1.99 mL/h) and $B_{blank}$ (62.85 ± 1.60 mL/h). This phenomenon proved that $B_{fine}$ added with the smallest PAC size had the best methane yield due to biomass richness in anaerobic bioreactors throughout the study.

**Table 4.4 Produced Biogas from Various Anaerobic Hybrid Bioreactors**

| **Biogas Content** | **$B_{blank}$** | **$B_{coarse}$** | **$B_{medium}$** | **$B_{fine}$** |
|---|---|---|---|---|
| **Biogas Production, mL/h** | 111 ± 5 | 123 ± 6 | 131 ± 8 | 142 ± 12 |
| **$CH_4$, %** | 56.79 ± 0.33 | 57.52 ± 0.11 | 57.22 ± 0.71 | 57.59 ± 0.15 |
| **$CH_4$, mL/h** | 62.85 ± 1.60 | 70.56 ± 1.99 | 75.15 ± 2.51 | 81.77 ± 4.01 |

## 4.6 Performance of PES Hybrid Membrane Incorporated with Different Sizes of PAC towards Membrane Fouling Control

Hybrid membrane incorporated with PAC can help to improve filter performance and reduce membrane fouling due to function of PAC incorporated into membrane able to carry out adsorption and collision effects on membrane surface simultaneously (Schulz et al., 2016; Satyawali and Balakrishnan, 2009; Ng et al., 2013). In this study, B $_{fine}$ was identified as supernatant of the best efficiency out of four hybrid bioreactors. Thus, performance of various fabricated PES hybrid membranes were tested by using this supernatant. A comparison was made among them and results were plotted and shown in Figure 4.5. M $_{blank}$, which is PES membrane without addition of PAC had the lowest performance as it fouled shortly compared to others. The contaminants blocked the pores on the M $_{blank}$ and caused TMP rose in faster rate. Instead, PES hybrid membrane added with the smallest size of PAC, M $_{fine}$ had resulted the highest fouling resistance performance followed by M $_{medium}$ and M $_{coarse}$ at flux of 30 L/(m$^2$ h).

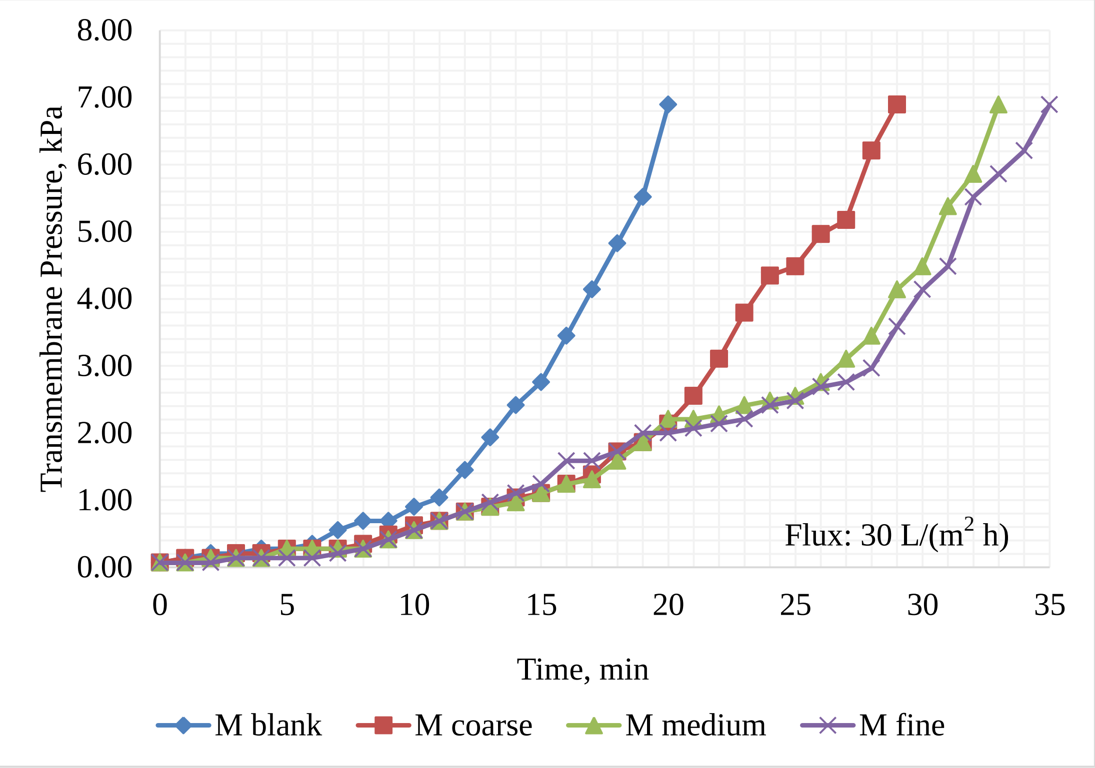


**Figure 4.5 Comparison of Various PES Hybrid Membrane Incorporated with Different Sizes of PAC towards Membrane Fouling Control**

PAC with larger surface area has relatively higher porosity which allow contaminants adsorb onto it before it blocking pores on membrane surface (Satyawali and Balakrishnan, 2009; Lee et al., 2007). It was noticed that the smallest PAC incorporated into PES membrane able to reduce TMP by forming permeate filter cake with the highest porosity. This phenomenon could help to enhance membrane fouling resistance and maintain service lifetime of membrane.

Another dead end filtration test was carried out to determine flux performance of hybrid PES membranes. Supernatant from $B_{fine}$ was extracted to test the PES membrane filtration performance and obtained results was shown in Tables 4.5 and 4.6. Based on Table 4.5, it was observed that $M_{fine}$ had obtained the highest flux rate at $32.09 \pm 1.25$ L/(m$^2$ h) compared to $M_{coarse}$ which had only reached flux rate at $23.68 \pm 0.62$ L/(m$^2$ h). The worst performer was membrane without addition of PAC which only obtained $16.81 \pm 1.25$ L/(m$^2$ h). This trend stated that smaller sizes of PAC with higher porosity could allow more permeate pass through membrane and reach higher productivity.

**Table 4.5 Flux Performance of Various PES Hybrid Membranes Incorporated with Different Sizes of PAC**

| **Hybrid membrane incorporated with different PAC sizes, µm** | **Flux, L/(m$^2$ h)** |
|---|---|
| Blank (Without PAC), $M_{blank}$ | $16.81 \pm 1.25$ |
| $471.005 \pm 0.868$, $M_{coarse}$ | $23.68 \pm 0.62$ |
| $226.824 \pm 1.14$, $M_{medium}$ | $28.27 \pm 0.62$ |
| $100.337 \pm 1.34$, $M_{fine}$ | $32.09 \pm 1.25$ |

According to Table 4.6, overall treating performance of PES hybrid AnMBRs were studied and the most effective result obtained was $M_{fine}$ with removal efficiency of COD ($90.55 \pm 0.21$ %), protein ($89.24 \pm 1.59$ %) and polysaccharides ($84.96 \pm 0.16$ %). It was observed that PAC incorporated into PES hybrid membrane had effectively retained contaminants on membrane surface due to good absorptivity of PAC and relatively produced higher quality effluent.

**Table 4.6 Overall Treating Performance by Using PES Hybrid Membranes Incorporated with Different Sizes of PAC**

| **Parameter** | **M** $_{\textbf{blank}}$ | **M** $_{\textbf{coarse}}$ | **M** $_{\textbf{medium}}$ | **M** $_{\textbf{fine}}$ |
|---|---|---|---|---|
| COD of permeate, mg/L | 1550 $\pm$ 35 | 1222 $\pm$ 32 | 1002 $\pm$ 31 | 723 $\pm$ 11 |
| Protein of permeate, mg/L | 707 $\pm$ 27 | 366 $\pm$ 15 | 162 $\pm$ 13 | 91 $\pm$ 3 |
| Polysaccharide of permeate, mg/L | 19.06 $\pm$ 0.33 | 12.33 $\pm$ 0.12 | 9.70 $\pm$ 0.09 | 7.89 $\pm$ 0.02 |
| Removal efficiency of COD, % | 79.73 $\pm$ 0.36 | 84.03 $\pm$ 0.25 | 86.91 $\pm$ 0.17 | 90.55 $\pm$ 0.21 |
| Removal efficiency of protein, % | 70.53 $\pm$ 0.97 | 78.12 $\pm$ 0.42 | 86.38 $\pm$ 0.20 | 89.24 $\pm$ 1.59 |
| Removal efficiency of polysaccharide, % | 73.30 $\pm$ 0.13 | 81.42 $\pm$ 0.03 | 82.88 $\pm$ 0.17 | 84.96 $\pm$ 0.16 |

# CHAPTER 5

# CONCLUSION AND RECOMMENDATIONS

## 5.1 Conclusion

Throughout the study, hybrid anaerobic bioreactors by adding PAC have been found able to enhance removal of organic matter in POME treatment and control membrane fouling problem effectively. It could be noticed that PAC with relatively smaller sizes (PAC size in $163.884 \pm 1.31$ μm) helped to increase microbial growth rate and biofloc formation. Transformation of PAC into BAC in anaerobic bioreactors obviously helped in reducing natural organic matter (NOM) amount by carrying out adsorption and biodegradation processes simultaneously. This could help to reduce membrane fouling rate and prolong its lifespan. Application of PAC in smaller sizes into bioreactors also contributed higher volume of biogas production as more methanogens bacteria functioned together in faster rate to convert NOM into biogas during biodegradation process. Besides, PES membrane coated with smaller sizes of PAC (PAC size in $100.337 \pm 1.34$ μm) has been proven that higher flux and more clean permeate can be obtained. This is due to larger biofloc formed with greater porosity can help to form permeate filter cake instead of blocking the pores on membrane surface by unwanted particles.

## 5.2 Recommendations

Although optimum operating conditions from previous study were applied in this project, there are some recommendations can be made in order to enhance the research study which are shown as follows:

(i) Constant stirrer should be applied to ensure homogeneous mixing inside bioreactors and controlled at lowest speed to prevent over shaking to biofloc formation.

(ii) Optimum SRT should be determined to analyse microbial growth rate and removal efficiency of organic matter.

(iii) Optimum dosage of PAC should be determined to investigate biogas production.

(iv) Fresh fabricated PES membrane and membrane after filtration are suggested to undergo Scanning Electron Microscopy (SEM) to analyse structure of pores and cake formation.

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# APPENDICES

## Appendix A: Experimental Set-up

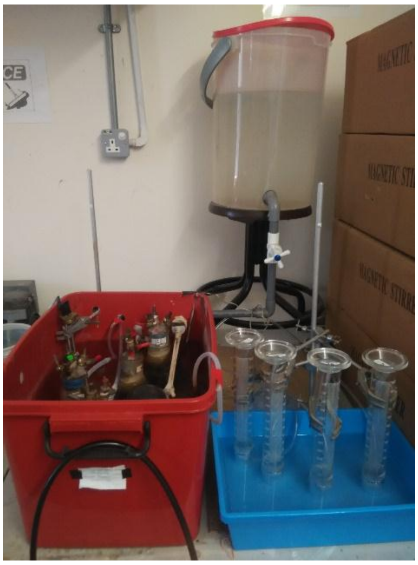

**Figure A1: Four 1 L Anaerobic Bioreactors Connected with Biogas Collectors & Water Tank**

**Figure A2: Dope Preparation Set-up**

# Appendix B: Analytical Laboratory Instruments


**Figure B1: Muffle Furnace**


**Figure B2: Oven**


**Figure B3: COD Reactor**


**Figure B4: UV-Vis Spectrophotometer (DR 6000)**


**Figure B5: Sonicator Bath**


**Figure B6: Cross Flow Filtration Test Rig**


**Figure B7: Dead End Filtration Test Rig**


**Figure B8: Biogas Analyser (RASI 700)**

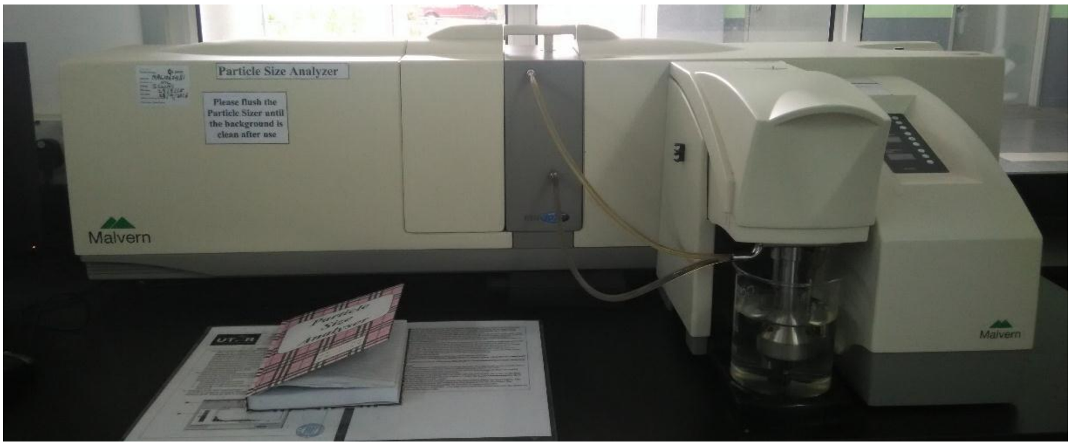


**Figure B9: Particle Size Analyser**

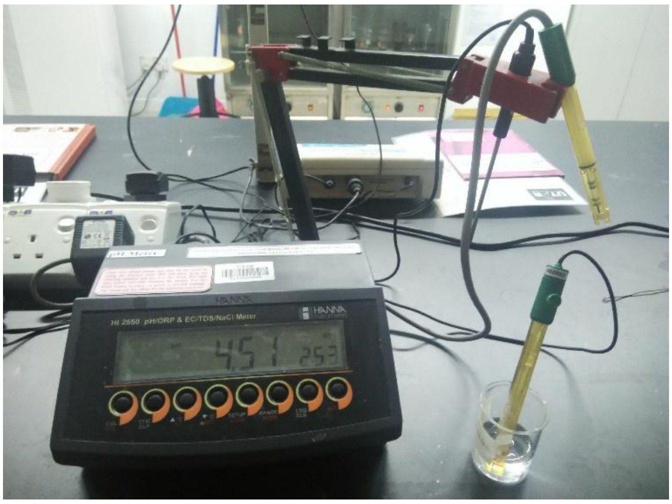

**Figure B10: pH Meter**

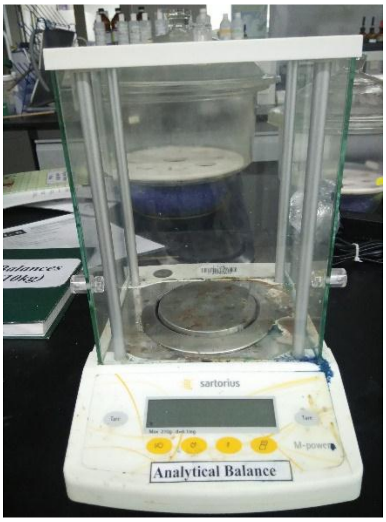


**Figure B11: Analytical Balance**


**Figure B12: Membrane Auto Casting Machine**

## Appendix C: Materials

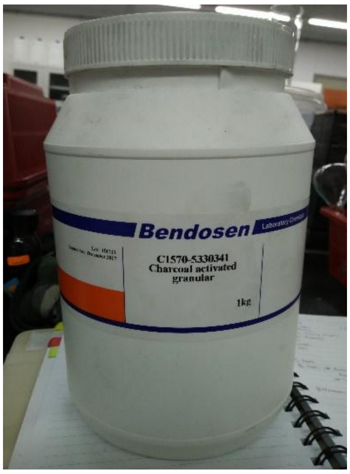


**Figure C1: GAC**


**Figure C2: Raw POME**


**Figure C3: Bradford's Reagent**

**Figure C4: PES**

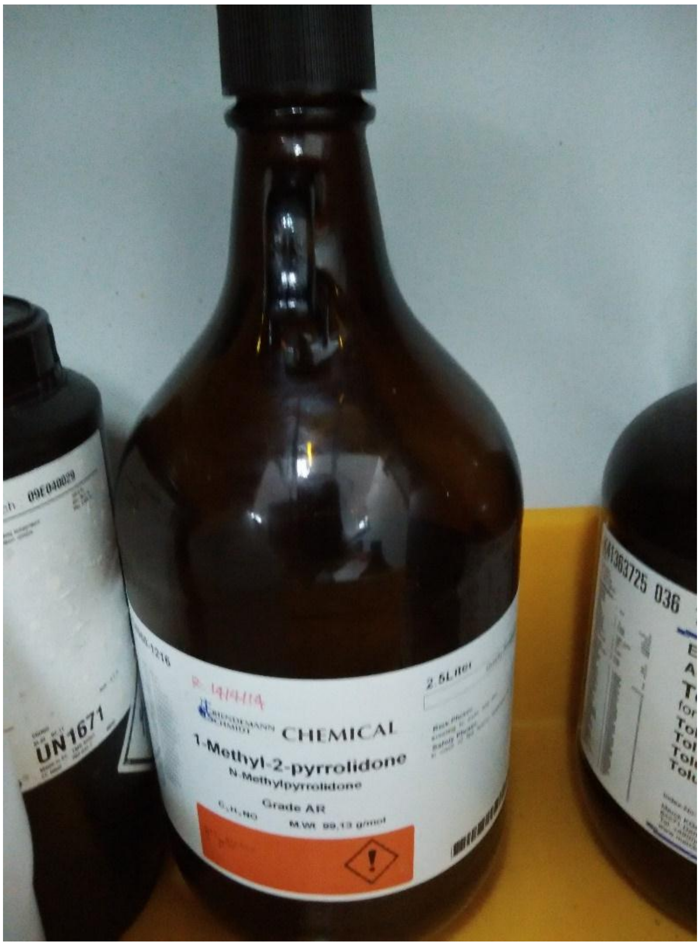


**Figure C5: NMP**

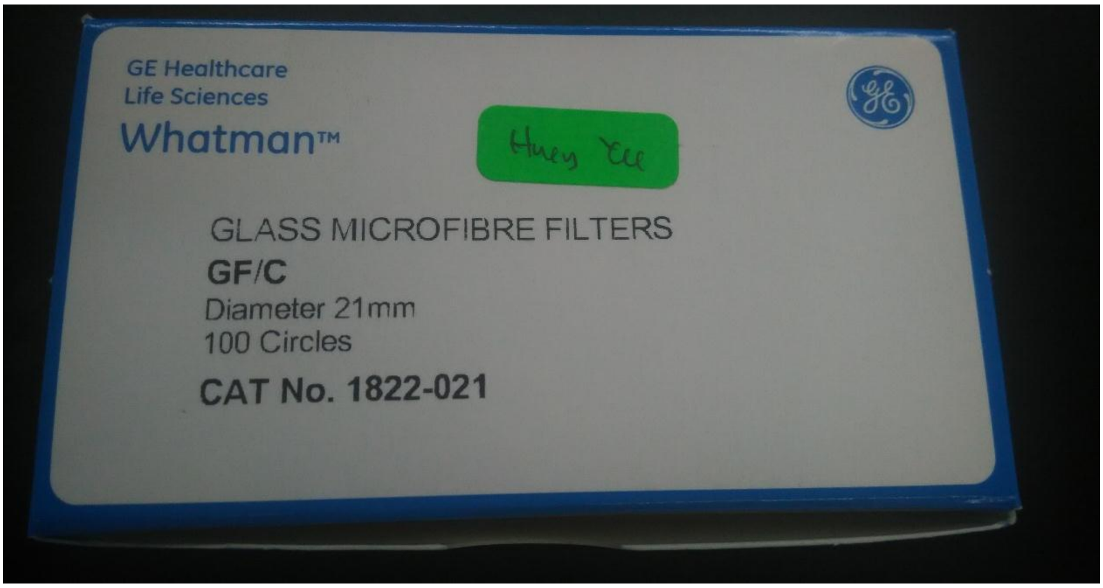


**Figure C6: Glass Microfibre Filters**

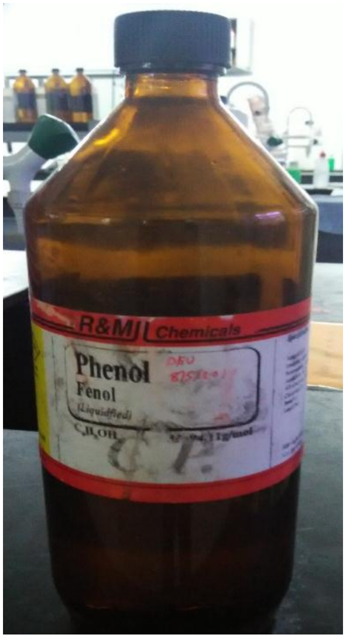


**Figure C7: Phenol**