ASSESSMENT OF ANAEROBIC BIOREACTORS WITH DIFFERENT SIZES OF POWDERED ACTIVATED CARBON AND HYBRID MEMBRANE 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

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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 "**ASSESSMENT OF ANAEROBIC BIOREACTORS WITH DIFFERENT SIZES OF POWDERED ACTIVATED CARBON AND HYBRID MEMBRANE IN TREATING PALM OIL MILL EFFLUENT**" was prepared by TAI CHEE YONG has met the required standard for submission in partial fulfillment of the requirements for the award of Bachelor of Engineering (Hons) Environment Engineering at Universiti Tunku Abdul Rahman.

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ABSTRACT

In this study, different sizes of powder activated carbon (PAC) was added into four identical anaerobic bioreactors (AnMBRs) to investigate their effects on chemical oxygen demand (COD) removal, natural organic matters (NOM) removal, mixed liquor suspended solid (MLSS), mixed liquor volatile suspended solid (MLVSS) and membrane fouling control. With decrease of PAC sizes, the MLSS and MLVSS in the AnMBRs had increased which led to higher COD removal rate (89.45 ± 2.48%), bigger floc size (62.815 ± 1.450µm), lower NOM content (1133 ± 552 mg/L for protein and 75 ± 3 mg/L for polysaccharide) and better membrane fouling control. In addition, to investigate the performance of polyethersulfone (PES) membrane in fouling control, different concentrations of PAC had been incorporated into it. Results shows that by incorporating 5 %wt of PAC into the PES membrane, it shows better in performance. The hybrid membranes show positive results in COD removal (95.77 ± 0.27%), membranes fouling control and could produce higher quality of permeate.

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LIST OF ABBREVIATIONS

AnMBR	Anaerobic Membrane Bioreactor
BAC	Biological Activated Carbon
BOD	Biological Oxygen Demand
CD	Cellulosic Derivatives
COD	Chemical Oxygen Demand
GAC	Granular Activated Carbon
HRT	Hydraulic Retention Time
MBR	Membrane Bioreactor
MLSS	Mixed Liquor Suspended Solid
MLVSS	Mixed Liquor Volatile Suspended Solid
NMP	1-Methyl-2-pyrrolidone
NOM	Natural Organic Matter
PAC	Powdered Activated Carbon
PAN	Polyacrylnitrile
PES	Polyethersulfone
POME	Palm Oil Mill Effluent
PP	Polypropylene
PSF	Polysulfone
PVDF	Polyvinyldenefluoride
SRT	Sludge Retention Time
STSNs	Sulfated TiO2 deposited on SiO2 nanotubes
TMP	Trans-membrane Pressure
WWTP	Wastewater Treatment Plant

CHAPTER 1

INTRODUCTION

1.1 Background

Palm Oil Mill production is one of the largest industry activities in Malaysia currently. Since 1960, palm oil plantation was increasing at a rapid pace in which the total planted area in 2011 had grown up to total of 4.917 million hectares as shown in Figure 1.1 (Malaysian Palm Oil Board, 2011).

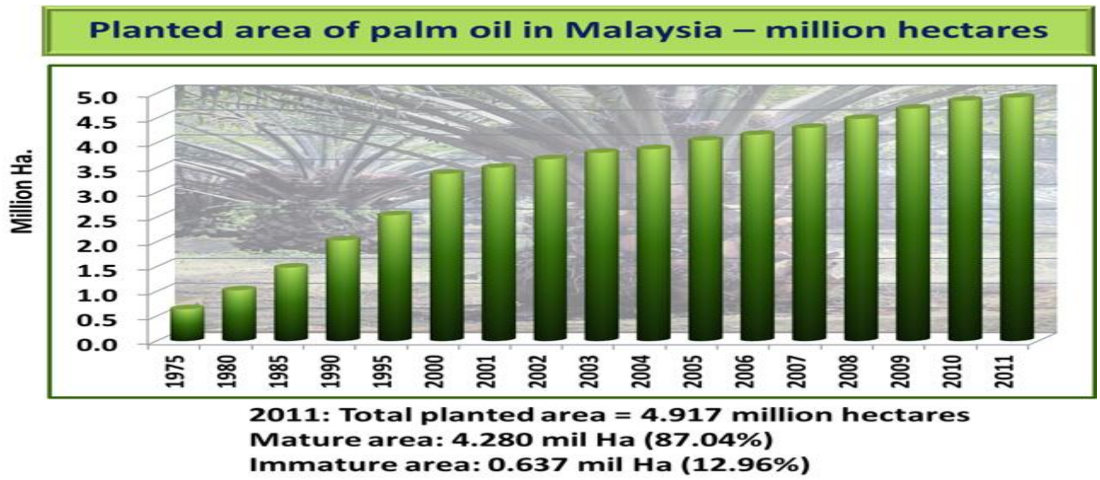


Figure 1.1 Planted area of palm oil in Malaysia (Malaysian Palm Oil Board, 2011)

Because of the rapid increase in palm oil production, the quality of Palm Oil Mill Effluent (POME) had become an issue that had to be taken into consideration in order to protect the environment from getting polluted by it.

There are different type of wastewater treatment technologies available in the market, based on the budget and type of wastewater to be treated, different technologies can be apply. Some of the most conventional method and widely used technologies are activated sludge system, sequencing batch reactor and trickling filter. Although there were many different technologies available in the market, there are still spaces for improvement in order to have an efficient and sustainable wastewater treatment system.

Among all the technologies use in treating wastewater, Membrane Bioreactor was found to be the most promising technologies so far due to its simple operation, higher separation efficiency, small footprint, low production of excess sludge, and high effluent water quality (Tan et al., 2013).

In addition, anaerobic membrane bioreactor (AnMBR) has advantages such as providing lower amount of biological waste and convert organic substances into valuable bio-gas compared with aerobic membrane bioreactor (Gouveia et al., 2015). Because of the advantages over aerobic membrane bioreactor, it had gained the interest from various fields such as municipal and industrial activities (Gouveia et al., 2015; Lin et al., 2013; Ozgun et al., 2013; Skouteris et al., 2012; Smith et al., 2012).

1.2 Problem Statement

The main problem that is commonly faced by a membrane used in wastewater treatment is membrane fouling. Fouling of membrane is a process in which particles suspended in the water deposited on the surface or inside the pore of the membrane as the water pass through it. This phenomenon often happened after a certain usage time, depends on the type of particulate it filters, whether it is highly concentrated or the other way around. This will determine the lifespan of the membrane. Membrane fouling must be controlled in order to avoid flux reduction, increase in trans-membrane pressure, resulting in high energy consumption which leads to increase in operation cost (Skouteris et al., 2015; Liu et al., 2007; Tian et al., 2010).

Membrane that had fouled can be cleaned by either physically, chemically, physico-chemically and biologically, based on the degree of fouling and type of foulants. Different methods, chemicals or micro-organisms can be used to enhance the cleaning efficiency (Zhao et-al., 1999).

Although membrane can be cleaned when it is being fouled, there is still certain lifespan on a membrane. In addition, frequent cleaning of membrane will also lead to higher maintenance cost, decrease in efficiency and also reduce in membrane lifespan. In order to increase the lifespan and efficiency of a membrane, fouling rate of membrane should be reduced.

In this study, PAC will be used to reduce the fouling rate of a membrane by adding it into anaerobic bioreactor and membrane.

1.3 Objectives

The objectives of this study are:

i. To evaluate the performance of Anaerobic Membrane Bioreactor with different sizes of Powdered Activated Carbon (PAC).
ii. To evaluate the performance of the hybrid membrane incorporated with different PAC concentrations in membrane fouling control and pollutants removal rate.

1.4 Project Scopes

The scopes of this study is to,

i) Fabrication of PES membrane using dry-wet phase inversion technique.

ii) Evaluate the performance of various anaerobic bioreactors added with different PAC sizes.

iii) Evaluate the performance of hybrid PES membrane incorporated with different PAC concentrations.

CHAPTER 2

LITERATURE REVIEW

2.1 Palm Oil Mill Effluent (POME)

During the processing of oil palm fresh fruit bunches, several by-products were produced such as POME, empty fruit bunches, mesocarp fibre and shell. Among all the by-products produce, POME was the one that may threatens the environment the most. At the early stage where palm oil industry was still a recently introduced industry in Malaysia at 1960's, POME was directly discharged into the environment without any treatment. This had resulted in major water pollution in the waterways during the 1970's (Malaysian Palm Oil Board, 2014).

The three most commonly used technology at that time were ponding system, open tank digester, extended aeration system, closed anaerobic digester and land application systems (Malaysian Palm Oil Board, 2014). The characteristic of a POME was shown in Table 2.1.

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

Parameter	Mean	Range
pH	4.2	3.4 - 5.2
Biological Oxygen Demands (BOD)	25000	10250 - 43750
Chemical Oxygen Demands (COD)	51000	15000 - 100000
Total Solids	40000	11500 - 79000
Suspended Solids	18000	5000 - 54000
Volatile Solids	34000	9000 - 72000
Oil & Grease	6000	130 - 18000
Ammoniacal Nitrogen	35	4 - 80
Total Nitrogen	750	180 - 1400

*Units in mg/l except pH

By the year of 1978, the enactment of Environment Quality Regulations list out the discharge standards for POME effluent, the required standards increases every year since the day of enactment until 1984 and thereafter (Malaysian Palm Oil Board, 2014). The discharge standards control the quality of the effluent, preventing palm oil industry from further polluting the environment. The discharge standards was illustrated in Table 2.2.

Table 2.2 Palm oil mill effluent discharge standards (Malaysian Palm Oil Board, 2014)

Parameter	Limits According to Periods of Discharge					
	1/7/78 - 30/6/79	**1/7/79 - 30/6/80**	**1/7/80 - 30/6/81**	**1/7/81 - 30/6/82**	**1/7/82 - 31/12/83**	**1/1/84 & thereafter**
pH	5 - 9	5 - 9	5 - 9	5 - 9	5 - 9	5 - 9
Biological Oxygen Demand	5000	2000	1000	500	250	100
Chemical Oxygen Demand	10000	4000	2000	1000	-	-
Total Solids	4000	2500	2000	1500	-	-
Suspended Solids	1200	800	600	400	400	400
Oil and Grease	150	100	75	50	50	50
Ammoniacal Nitrogen	25	15	15	10	150	100
Total Nitrogen	200	100	75	50	-	-
Temperature (°C)	45	45	45	45	45	45

*Units in mg/l except pH and temperature

2.2 Membrane Bioreactor (MBR)

Membrane Bioreactor (MBR) is a hybrid process which is a combination of of membrane filtration and microbiologic treatments. This hybrid process had increased in popularity in treating wastewater nowadays due to it surpasses other conventional treatment processes such as activated sludge system (Klaus and Suzana, 2010). The main advantage of an MBR system is it produces effluent that were clarified and slightly disinfected with smaller environmental footprint for a waswater treatment plant (WWTP) (Robles et al., 2013).

In addition, MBR is more compacted in terms of system because it requires less space and tank compared to conventional activated sludge process. By putting membrane inside the MBR can exclude the additional secondary sedimentation tank and sand filtration.

Compared to activated sludge system, MBR can be operated under higher mixed liquor suspended solid (MLSS), up to 15 g/L (Klaus and Suzana, 2010). Higher MLSS means that there are more organic materials and suspended solids present in the wastewater. In biological treatment, MLSS plays an important role as it serves as source of food for the microorganisms. MBR can operate under higher MLSS level, hence, more microorganisms can work to remove unwanted materials from the wastewater.

In addition, MBR can run with long sludge ages (>20 days) (Klaus and Suzana, 2010). This factor can benefit those slow growing microorganism so that there is enough time for the microorganisms to reach the stable level. Compared to conventional activated sludge process which cannot run under long sludge ages because of uneasy settle sludge, MBR has more advantages.

MBR can be separated into external loop or submerged configuration process. Both of the processes differ in where the membrane was place in the system, for external loop the membrane was placed away from the aeration tank; for submerged configuration the membrane was placed inside the aeration tank. For external loop process, transport of water to the membrane needs to be done in high velocity so that the fouling rate can be reduced in order to enhance the performance (Klaus and Suzana, 2010). This will increase in operation cost compared to submerged configuration which does not need extra energy to pump the water.

Anaerobic MBR system has shown promising results in terms of efficiency and sustainable compared with aerobic MBR. Due to its advantages such as lower sludge production, lower energy usage and generation of biogas as a source of energy (Robles et al., 2013).

2.3 Operating Conditions of MBR

This part of literature review discuss the operating conditions of a MBR system. Primarily focus on Sludge Retention Time (SRT), Hydraulic Retention Time (SRT) and temperature.

2.3.1 Sludge Retention Time (SRT)

Sludge retention time is one of the crucial condition while operating a MBR system, by controlling the SRT effectively the efficiency of pollutant removal can be enhanced and excess of sludge in the system can be prevented (Chen et al., 2011; Choi et al., 2008). MBR tends to operate at longer SRT which will help to promote the growth of microorganisms and further enhance the biodegradation of organic pollutants (Chen et al., 2011).

Although higher SRT promotes microbial growth, but the efficiency of the system is not directly proportional to the SRT. Due to longer SRT, changes in properties of mixed liquor, such as viscosity, amount and composition of microbial product and cell surface properties could reduce the biological capability, reducing the performance of the microorganisms (Chen et al., 2011; Shin and Kang, 2003; Cho et al., 2005; Massé et al., 2006). In addition, higher SRT could also lead to deposition of sludge particles on the surface of the membrane, resulting in higher probability in membrane fouling (Chen et al., 2011; Katayon et al., 2004; Ferreira et al., 2010).

It was found out that by fixing SRT of 20 to 40 days, the performance of a MBR was improved (Chen et al., 2011). Besides that, another research shows that higher SRT (30 days) had better membrane fouling control compared to lower SRT (15 days) due to more stronger and stable sludge flocs formation (Tian and Su, 2012).

2.3.2 Hydraulic Retention Time

Besides SRT, hydraulic retention time (HRT) was also an important parameter for the operation of MBR (Fallah et al., 2010; Clech et al., 2006; Meng et al., 2009). Report had shown that by having lower HRT, the performance of MBR will drop significantly due to higher organic loading rate (Fallah et al., 2010).

Based on the previous research done, by having HRT of 13 to 19 hours in a submerged MBR for the treatment of petrochemical wastewater, the treated effluent was found to be in the accepted range (Qin et al., 2007). Besides that, research also found that HRT of 12 to 30 hours have no effect on the removal performance of a MBR system (Chang et al., 2006). Which indicates that MBR system required at least 12 to 13 hours of HRT to prevent deficient in removal performance.

2.3.3 Temperature

Anaerobic digestion can take place at psychrophilic temperatures below 20ºC (Bouallagui et al., 2003) but most reactors operate at either mesophilic temperatures or thermophilic temperatures with temperature at 35ºC and 55º respectively (Ward et al., 2008).

Depend on the type of wastewater treated, different temperature may give rise to different results. Better removal of pollutants in olive mill and abattoir waste water (Gannoun et al. 2007) and vegetable waste and wood chips (Hegde and Pullammanappallil, 2007) was observed under thermophilic condition compared to mesophilic condition. Where else, better performance was observed in mesophilic condition while treating potato waste (Parawira et al. (2007). Figure 2.1 shows the optimum temperature for the growth of some methanogenic bacteria.

Table 2.3 Optimal growth temperatures for some methanogenic bacteria (Gerardi, 2003)

Temperature range	Genus	Optimal temperature (°C)
Mesophilic	*Methanobacterium*	37–45
	Methanobrevibacter	37–40
	Methanosphaera	35–40
	Methanolobus	35–40
	Methanococcus	35–40
	Methanosarcina	30–40
	Methanocorpusculum	30–40
	Methanoculleus	35–40
	Methanogenium	20–40
	Methanoplanus	30–40
	Methanospirillum	35–40
	Methanococcoides	30–35
	Methanolobus	35–40
	Methanohalophilus	35–45
Thermophilic	*Methanohalobium*	50–55
	Methanosarcina	50–55

2.4 Anaerobic Digestion

Anaerobic digestion is a multiple stages process which include biodegradation of organic substances by bacteria in an environment without the present of oxygen. Biogas was produced at the end of this process as a by-product which mainly consist of methane (CH_4), carbon dioxide (CO_2) and small amount of hydrogen sulphide (H_2S) and other gases (Dioha et al., 2013; Ryckebosch et al, 2011 & Garba et al, 1998).

Anaerobic digestion mainly consist of four key biological and chemical processes, which are hydrolysis, acidogenesis, acetogenesis and methanogenesis. Anaerobic digestion starts with hydrolysis in which large organic polymer were broken down into smaller constituent parts like simple sugars, amino acid and fatty acids which can be further degraded by other bacteria (Dioha et al., 2013).

Acidogenesis will further break down the remaining components by acidogenic bacteria which will produce volatile fatty acids, ammonia, carbon dioxide and hydrogen sulphide as by products (Dioha et al., 2013).

After that during acetogenesis, acetogens will further digeste the simple molecules from acidogenesis to produce acetic acid, carbon dioxide and hydrogen (Dioha et al., 2013; Ferry, 1997). The final stage of anaerobic digestion is methanogenesis whereby all the intermediate products from the previous stages are converted into methane, carbon dioxide and water (Dioha et al., 2013).

Methanogenic bacteria are pH sensitive, they can only survive and function well between the pH of 6.5 to 8 (Dioha et al., 2013; Martin, 2007). The overall process flow of anaerobic digestion is illustrated in Figure 2.2.

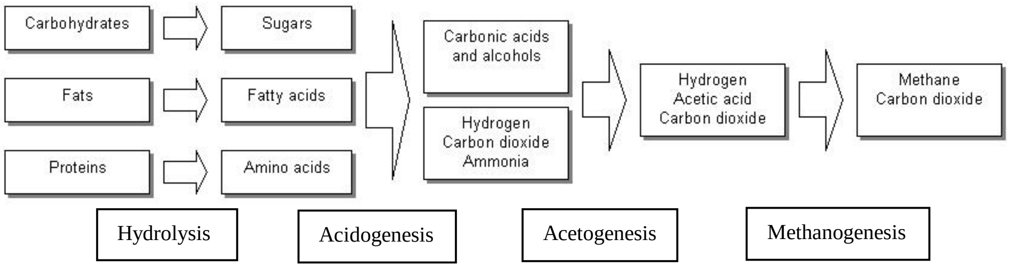


Figure 2.1 Process flow of anaerobic digestion

2.5 Polymeric Membrane

Polymeric membrane is a membrane constructed from a long chain of monomers to forma polymer.

2.5.1 Packaging of membrane

Packaging of membrane can be divided into four main types, which are spiral-wound membrane, plate and frame membrane, tubular membrane and hollow fiber membrane. Depends on the type of wastewater treated and economical consideration, different type of membrane can be used. The properties of each membrane structure was summarized in the table below.

Table 2.4 Types of membrane structure (Klaus and Suzana, 2010)

Membrane structure	Characteristics
Spiral-wound membrane	-Multiple layer of membrane rolled up around a perforated tube -Maximum area of membrane was utilise with minimum space required
Plant and frame membrane	-Membranes are place in a support plate, multiple plate was align
Tubular membrane	-Place inside a micro-porous material tube -Small size membrane surface, blockage of membrane unlikely to occur
Hollow fiber membrane	-High chance in blockage of membrane -Suitable for low suspended solid water treatment

2.5.2 Membrane material

Membrane can be made out from wide variety of materials. Depends on the type of wastewater treated and its characteristic such as pH range, VOC tolerance and oxidant tolerance. Some of the common materials used to produce membrane are Polypropylene (PP), Polyethersulfone (PES), Polysulfone, Polyvinyldenefluoride (PVDF), Cellulosic derivatives (CD), Polyacrylnitrile (PAN). The material used will determine the characteristic of the membrane, based on the usage of the membrane, different material will be chosen. The table below summarize the advantage of each materials.

Table 2.5 Membrane base material and their characteristics (Klaus and Suzana, 2010)

Membrane base material	**Characteristics**	
Polypropylene (PP)	-Wide range of pH (2-14) -Good chemical resistant -Able to withstand up to 35 psig transmembrane pressure (good mechanical strength)	-Not oxidant tolerant (<10^2 ppm/hr)
Polyethersulfone (PES)	-Highly oxidant tolerance (>250000 ppm/hr for chlorine) -Wide range of pH (2-12) -Resistant to oil & grease	-Highly hydrophilic (more resistant to fouling) -Vulnerable to organic solvent such as benzene
Polysulfone (PSF)	-Wide range of pH (1-13) -High temperature tolerance (up to 75 °C) -Good oxidant resistance	
Polyvinyldenefluoride (PVDF)	-Highly oxidant tolerant (up to 5000ppm chlorine) -Wide pH range (3-10.5) -Moderate temperature limit (up to 40° C)	- Able to withstand up to 36 psig transmembrane pressure (good mechanical strength)
Cellulosic derivatives (CD)	-Lower pH range (4-8.5) -Lower temperature limit (<35° C)	-Moderate oxidant tolerance (>10^5 ppm/hr)
Polyacrylnitrile (PAN)	- Able to withstand up to 44 psig transmembrane pressure.	-Moderate temperature limit (40° C) -Moderate pH range (2-10)

2.5.3 Modification of membrane

Several distinct research had shown that by adding additives into a membrane will improve the performance of a membrane by certain level.

PES ultrafiltration membrane combine with Polyethylene glycol (PEG) and coated with cobalt (II) doped iron oxides shows improvements in Cu (II) rejection. The results obtained show that the rejection of Cu (II) was more than 90% for modified membrane compared to a normal membrane with only having around 20% of rejection. In addition, PEG was studied in membrane preparation due to its pore forming ability and hydrophilic characteristic which can improve the flux rates of membranes (Chan et al., 2015).

In addition, sulfated TiO_2 deposited on SiO_2 nanotubes (STSNs) doped into PSF membrane had shown increasing in hydrophilic, anti-fouling, anti-compaction and tensile strength properties of the membrane. Wastewater containing oil was used to pass through the membrane, modified membrane with STSNs shows slightly higher oil rejection but with higher flux rate compared with normal PSF membrane (Zhang and Liu, 2015).

Research also shows that by doping nano-silica in PES membrane will improve the hydrophilic properties of a membrane, reducing the fouling rate of a membrane. Due to high hydrophilicity of nano-silica, only a small amount of dosage is needed (0.3%, SiO_2/PES ratio) to reduce irreversible fouling by 70% (Lin et al., 2015).

2.6 Membrane Fouling

Fouling of a membrane can be defined as the modification of membrane resulted by the combination of physical and chemical interactions between the membrane and the suspended component that pass through the membrane, which causes the membrane to lose its permeability (Vera et al., 2015).

There were several mechanisms which could lead to fouling of a membrane such as formation of a layer of filter cake and clogging within the membrane pore could lead to membrane fouling (Zhao et al., 1999). Other than that, filter cake layer consolidation and osmotic pressure effect can also result in membrane fouling (Vera et al., 2015).

In addition, fouling of a membrane can be caused by parameters such as membrane pore size distribution, material of membrane and surface of membrane (Klaus and Suzana, 2010).

Besides that, control the variable such as pH in coagulation process will also affect the fouling of a membrane. By using $FeCl_3$ and PFC_{10} which work best in acidic condition as coagulant, it can be observed that the fouling of the membrane had reduced due to larger and looser flocs properties (Dong et al., 2015). Larger and looser flocs has lower tendency to clog the membrane pore compared to smaller and denser flocs. In addition, pH change will also lead to precipitation of certain salts or hydroxide which could lead to membrane fouling too (Klaus and Suzana, 2010).

There are four main types of fouling mechanism, (i) complete pore blocking, (ii) internal pore blocking, (iii) partial pore blocking and (iv) cake filtration. How each of the fouling mode occur is summarized in the table below.

Table 2.6 Types of fouling mechanism and how it occurs (Klaus and Suzana, 2010)

Fouling mechanism	**Explanation**
Complete pore blocking	Larger particles completely block the pores
Internal pore blocking	Smaller particles adsorb or deposited inside the pores
Partial pore blocking	Particles either adhere on inactive area, partially blocking the pores or completely blocking the pores
Cake filtration	Formation of cake by particles which does not enter the pores

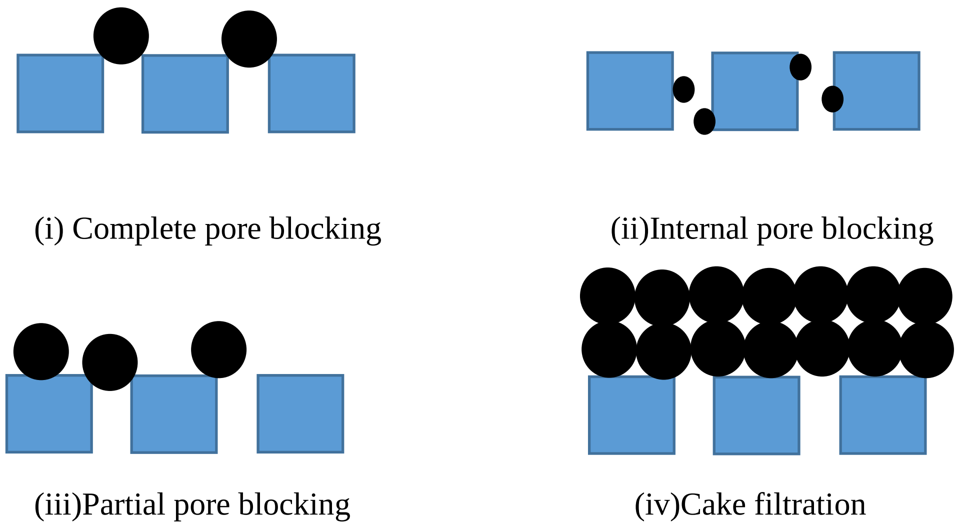


Figure 2.2 Different types of fouling mechanism

2.6.1 Foulants

Foulants are the main problems towards a membrane process, excessive accumulation of foulant will eventually reduce the performance of a membrane. Foulants can be classified into few categories which are organic precipitate (biological substance), inorganic precipitate (salts or hydroxides), particulate and colloids (Klaus and Suzana, 2010).

Among the types of foulants, bio-foulants are the major concern due to the presence of high level nutrients inside the wastewater which favour the rapid growth of biofilm. Fouling by organic and inorganic precipitate may occur simultaneously with the onset of biofilm formation (Klaus and Suzana, 2010).

Bio-foulants often adhere to surface of a membrane and colonize, the addition organic or inorganic precipitate may serve as nutrient for the biofilm, providing food source towards the microorganisms, stimulate the growth rate of the microorganisms resulting in biofilm formation.

2.6.2 Membrane fouling control

If fouling of membrane can be avoided or reduced, maintenance cost for membrane treatment can be reduced. There were several methods proposed by other that had shown promising results in fouling control.

By adding mixture of metal oxide adsorbents (crystalline silicotitanate (CST) and ferrihydrite (FH)) into the submerged membrane (0.3 g/L CST plus 0.1 g/L FH), the former metal oxide will enhance the metal ion removal rate and the later reduce membrane fouling. Fouling was reduced due to FH had higher sorption capacity for the main foulant, Tween 80 present in the system (Weerasekara and Choo, 2015).

In addition, introducing hydrophilic material into a hydrophobia membrane will achieve a suitable balance between hydrophobicity and hydrophilicity, which are related with low water solubility and high resistance to fouling respectively (Bai et al., 2015). By coating Multi-Walled Carbon Nanotube (MWCNTs) on the membrane, the surface of the membrane will not directly vulnerable towards fouling, membrane coated with MWCNTs will decrease the fouling rate of a membrane (Bai et al., 2015). In addition, reducing surface roughness of a membrane will decrease the fouling rate of a membrane (Zhang et al., 2013).

Other than that, cleaning of membrane will decrease the fouling rate of a membrane. The cleaning time interval will be determined based on the rate of fouling of that particular membrane. Cleaning of membrane should not be done too frequently as it will degrade the membrane and in the point of economic and environment it is not convenient (Klaus and Suzana, 2010).

Preventing membrane from fouling by pre-treatment in wastewater can also decrease the fouling rate of a membrane. Pre-treatment is the process that eliminates contaminant that can be easily remove such as suspended particles. Process such as coagulation and dissolved air floatation can be used to achieve less particle wastewater. (Klaus and Suzana, 2010).

2.7 Activated Carbon (AC)

Activated carbon come in all sizes such as in granular form or powder form. It can be produced mainly from by-product of agricultural industry, examples like coconut shells, pine tree, corn cobs and coffee husks. Due to easily available and low in cost, it is widely used in treatment of contaminated water (Tonucci et al., 2015).

Activated carbon was also been widely used in domestic drinking water treatment system for the removal of heavy metals ions and the organic matter due to low in cost (Chan et al., 2015).

Powder activated carbon had shown promising results in water treatment when added into membrane filtration process such as UF and MF (Klaus and Suzana, 2010). The added PAC function as a adsorbent in which contaminant such as dissolved organic can be adsorb on the surface of the PAC, in the same time membrane filtration reject the PAC and the adsorb contaminant, producing clean water from other side of the membrane. Furthermore, by adding PAC into an AnMBR, biological activated carbon (BAC) were formed and it could carry out two processes simultaneously namely as adsorption and biodegradation (Ng et al., 2013).

The adsorption capacity of a PAC vary with its particle sizes. As shown in one of the study conducted, the adsorption capacity of a PAC towards natural organic matters (NOM) increases as the particle size of PAC decreases (Matsui et al., 2015).

CHAPTER 3

RESEARCH METHODOLOGY

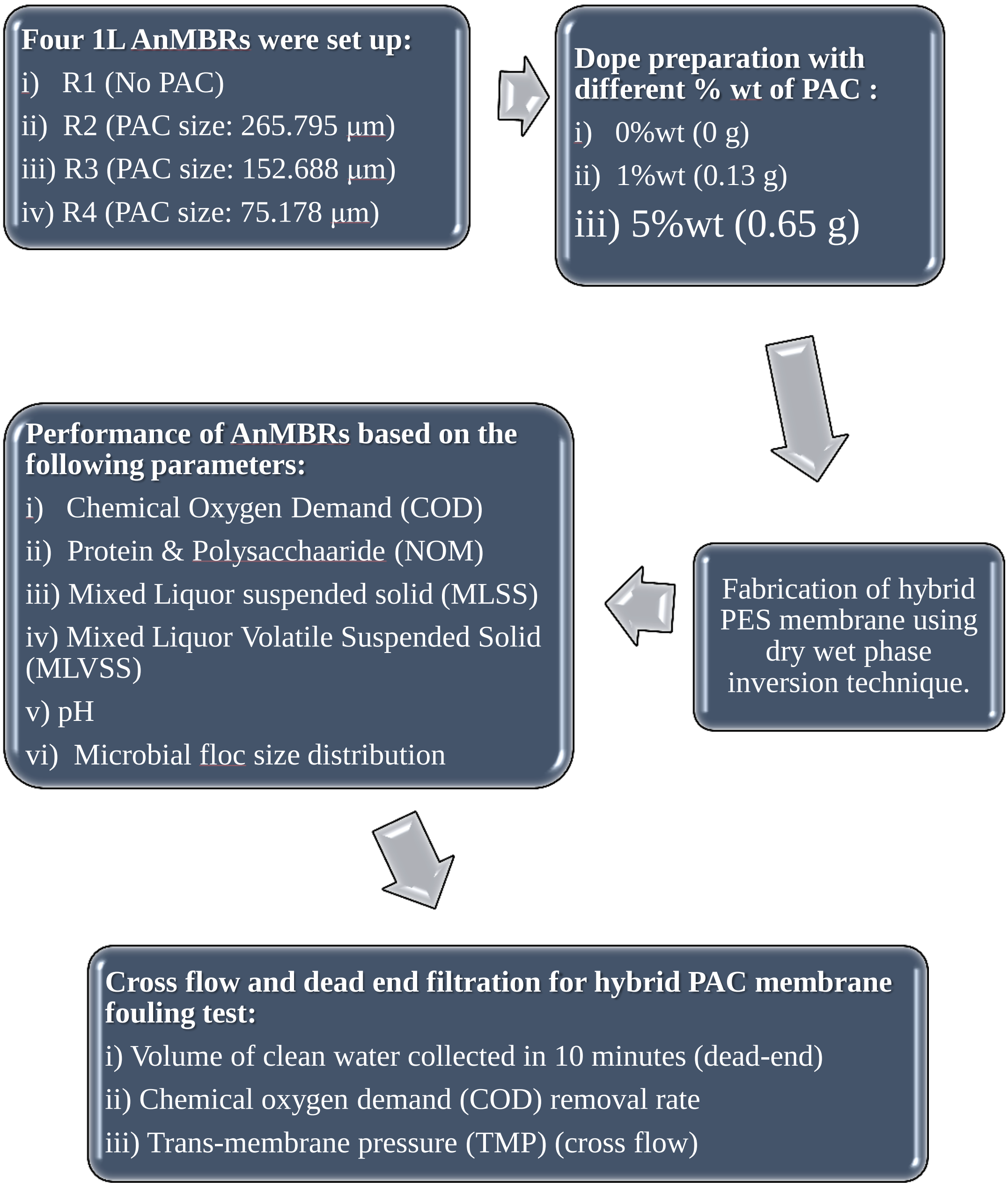


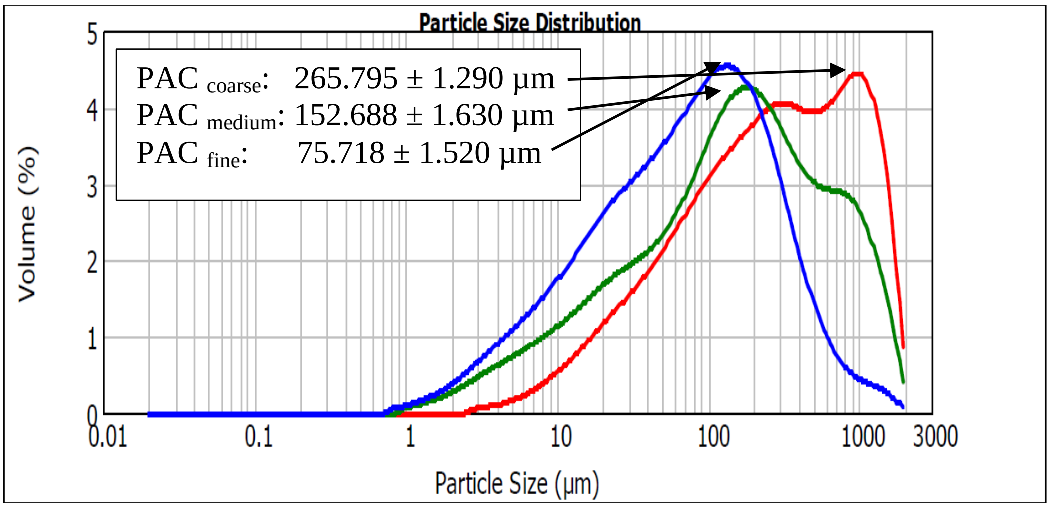
Figure 3.1 Project flowchart

3.1 Experiments Setup

Four 1L AnMBRs were kept in water bath with temperature of 45°C namely R1 (without PAC), R2 (with average PAC size of 265.795 µm), R3 (with average PAC size of 152.688 µm), and R4 (with average PAC size of 75.178 µm). All the bioreactors were added with 5g/L of PAC except for R1. The SRT and HRT of the four AnMBRs were fixed at 30 days and 12.5 days respectively. The parameters used was based on the study done by previous student, Connie in finding the optimum operating conditions.

3.2 Materials Used

Granular activated carbon (GAC) supplied by Bendosen company was used in this study and it was grounded by using a conventional Panasonic blender. Depends on the duration of the grinding process, different particle size can be obtained. The particle size was determine by using the particle size analyser. As shown in Fig 3.1, the D50 for all three sizes of PAC in terms of volume and number is 265.795 ± 1.290 µm and 3.470 ± 0.657 µm (coarse), 152.688 ± 1.630 µm and 2.148 ± 0.464 µm (medium), 75.718 ± 1.520 µm and 2.187 ± 0.480 µm (fine) respectively.



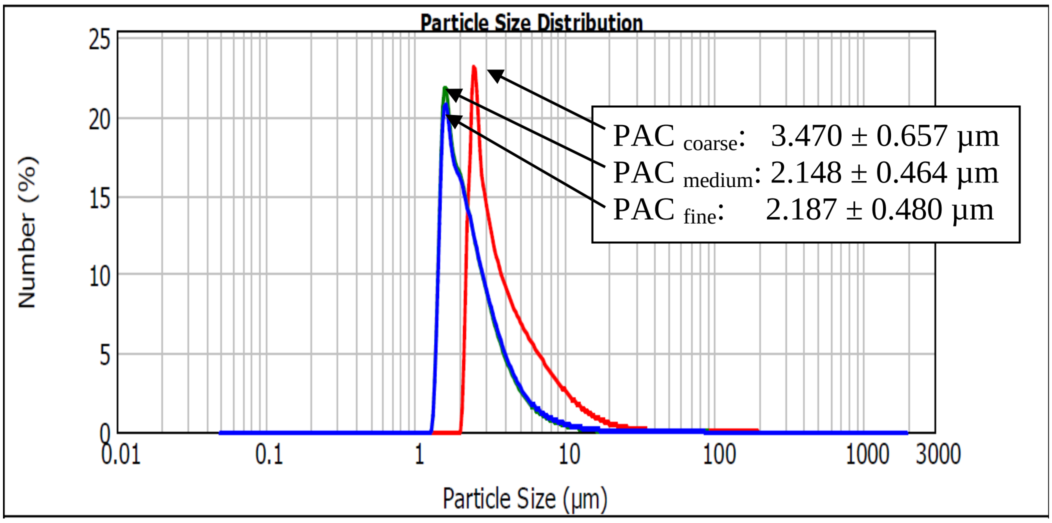


Figure 3.2 Particle size distribution of PAC

The powdered activated carbon (PAC) used to incorporate into the PES membrane is supply by GENE Chem Company. The anaerobic sludge and palm oil mill effluent (POME) were obtained from a POME treatment plant owned by Tian Siang Group located in Perak, Malaysia. The dope used to cast membrane consists of 1-Methyl-2-pyrrolidone (NMP) supplied by Friendemann Schmidt Chemical and Polyethersulfone (PES).

3.3 Dope Preparation

Before preparing the dope, PES and three head round-bottomed flask was left in the oven for 24 hour with temperature of 60^{o}C to remove excess of moisture.

First and foremost, the apparatus was set up accordingly and NMP was poured into the three head round-bottomed flask. The solvent will be heated until it reaches the temperature between 60^{o}C and 70^{o}C with slow stirring speed. After the temperature was maintained at the desired temperature for 10 minutes, PES was added spatula by spatula. The temperature was maintained between 60^{o}C and 70^{o}C with faster stirring speed until all the PES had dissolved (Ng, 2015).

After all the PES had been dissolved, heating mantle can be switch off and wait for the dope to cool down. The dope was pour into a scfott bottle afterward.

Before proceeding to membrane fabrication, the dope will be left in the sonicator bath for 8 hours to remove excess of air bubbles inside the dope and to ensure well mixing of the solution if PAC was added (Ng, 2015).

The dope was prepared with PES and NMP with a weight ratio of 13:87. 100g of dope was prepare in this experiment, hence, 13g of PES and 87g of NMP was used to prepare the dope without additive. Dope with additive 1 wt. % and 5 wt. of PAC was added base on the weight of PES. Formula for each dope was summarised in Table 3.1.

Table 3.1 Ingredients used in membrane dope preparation for polymer and hybrid membranes fabrication

Samples	Polyethersulfone (PES) gram	1-methyl-2-pyrrolidone (NMP) gram	Powdered Activated Carbon (PAC) gram
0% wt PAC	13.00	87.00	0.00
1% wt PAC	12.87	87.00	0.13
5% wt PAC	12.35	87.00	0.65

3.4 Fabrication of Polyethersulfone (PES) Membrane

The technique used in this study to fabricate the membrane was Dry-wet phase technique by using a semi-auto membrane casting machine. Knife gap with 15 micrometre thickness was used as the membrane thickness.

The dope was pour on top of the glass mounted on the machine, the machine will then spread out the dope evenly on the glass surface. After that, the glass was removed and immersed into the water bath. The moment the dope on top of the glass was in contact with the water it will form a layer of polymeric firm which will separate from the surface of the glass.

The casted membranes were left in a water bath for 24 hours, follow by immersing in methanol for 8 hours for post treatment purposes. After that, the membranes will be cut into a circle shape with diameter of 50mm and were left in water to prevent it from drying out.

3.5 Analytical Methods

3.5.1 Chemical Oxygen Demands (COD), Protein and Polysaccharide

COD were analysed by following the procedures from Standard Method, 21st Edition. Polysaccharide concentrations were measured with the methods of phenol-sulfuric acid (Dubois et al, 1956) and concentration of protein was measured by using Bradford reagent with bovine serum albumin (BSA) as standard (Bradford, 1967).

The samples were diluted to a ratio of 1:25 before mixing with the respective reagent. HACH UV/VIS spectrophotometer (Model DR 6000) was used to determine the concentration afterward.

3.5.2 Mixed Liquor Suspended Solid (MLSS) and Mixed Liquor Volatile Suspended Solid (MLVSS)

Mixed liquor suspended solid (MLSS) and mixed liquor volatile suspended solid (MLVSS) was analysed by following the procedures from Standard Method, 21st Edition. The weight of the samples was measured by using a M-power Analytical Balance AZ214 (Sartorius weighing technology, Germany).
The samples was filtrated using micro-glass fiber filter AH-934. After that, the filter was left inside the oven with 105ºC for 2 hours, the weight of the samples was measured as MLSS after the sample was left cooling down. The samples was further heated in a furnace for 550ºC for 15min, and the weight of the samples was measured as MLVSS after the sample was left cooling down.

3.5.3 pH Measurement

The pH of the samples was measured by using a pH meter (Hanna HI 2550, USA). Calibration was done before pH of the samples was measured, buffer solution with pH 4, 7 and 10 was used.

3.5.4 Particle Size Analysis

Particles size distributions of Powdered Activated Carbon (PAC) and microbial floc size was determined by using the particle size analyser (Malvern Mastersizer 2000, UK). The particle size of the samples was analysed in terms of volume and number.

3.5.5 Cross Flow and Dead End Filtration

Membrane with different concentration of PAC (0 wt. %, 1 wt. %, and 5 wt. %) was used to filter the supernatant from R4. Supernatant from R4 was diluted to a ratio of 1:10. Each of the hybrid membrane performance was tested by using both cross flow and dead end filtration system. Parameters tested during this stage was Chemical Oxygen Demand (COD) and Trans-membrane pressure (TMP). The trans-membrane data was recorded by a digital pressure data logger (Logit, USA).

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Performances of Anaerobic Bioreactors with Different PAC Sizes

The performance of the four AnMBRs are shown in Table 4.1. Throughout the study, pH of all four AnMBRs was maintained at the range of 7.8 to 7.9 and no big fluctuation of pH was observed. The stability in pH indicated that there is a balance in both acidogenic and methanogenic activities in the four AnMBRs. The COD removal efficiency for R1 is the lowest compared to other AnMBRs which is only 54.09 ± 15.24%. Other AnMBRs have relatively higher COD removal efficiency, R4 shows the best results among all the AnMBRs with COD removal efficiency of 89%. The addition of PAC could serve as a shelter for bacteria, transforming the PAC into Biological Activated Carbon (BAC) (Ng et al., 2013). Smaller PAC size could adsorb more COD due to its higher surface area and more COD could be biodegraded by the bacteria colonized on the surface of the PAC, resulting in higher COD removal efficiencies.

Table 4.1 Performance comparison of various Anaerobic Bioreactors added with different sizes of PAC

Parameter	**R1**	**R2**	**R3**	**R4**
Temperature, °C	45	45	45	45
pH	7.8 ± 0.1	7.8 ± 0.1	7.8 ± 0.1	7.8 ± 0.1
PAC dosage, g/L	NA	5	5	5
PAC particle size, D_{50} volume	NA	265.795 ± 1.290	152.688 ± 1.630	75.718 ± 1.520
PAC particle size, D_{50} number	NA	3.470 ± 0.657	2.148 ± 0.464	2.187 ± 0.480
SRT, d^{-1}	30	30	30	30
Feed in COD, mg/L	16449 ± 7840	16449 ± 7840	16449 ± 7840	16449 ± 7840
Feed in protein, mg/L	5599 ± 3511	5599 ± 3511	5599 ± 3511	5599 ± 3511
Feed in polysaccharide, mg/L	139 ± 2	139 ± 2	139 ± 2	139 ± 2
Supernatant COD, mg/L	7551 ± 363	2037 ± 270	1918 ± 320	1736 ± 302
COD removal efficiency, %	54.09 ± 15.24	87.62 ± 3.30	88.34 ± 2.80	89.45 ± 2.48
Protein, mg/L	1875 ± 551	1475 ± 615	1315 ± 592	1133 ± 552
Polysaccharide, mg/L	85 ± 2	75 ± 3	74 ± 3	75 ± 3

A test was conducted by adding 0.390g of PAC into a 80mL POME to study the COD removal rate of PAC at different sizes, the result was shown in Table 4.2. It can be clearly seen that relatively smaller particle size could remove more COD due to its higher surface area.

Table 4.2 Comparison of efficiency of PAC with different sizes in palm oil mill effluent (POME) treatment

PAC sizes (μm)	**COD before (mg/L)**	**COD after (mg/L)**	**Removal efficiency (%)**
265.795	31636	18215	42.4
152.688	31626	17942	43.3
75.718	31626	17298	45.3

Presence of natural organic matters (NOM) such as protein and polysaccharide in POME was the primary reason towards membrane fouling. As shown in Table 4.1, the removal rate of protein increase with decrease in PAC sizes. Identical to COD removal rate, smaller particle size of PAC provides larger surface area which could adsorb more protein.

4.2 Comparison of Biomass Concentration among Anaerobic Bioreactors

Mixed Liquor Suspended Solid (MLSS) and Mixed Liquor Volatile Suspended Solid (MLVSS) was measured on all four bioreactor. MLSS was used to determine the number of biomass and non-biodegradable substance inside the bioreactor, whereas MLVSS was used to solely determine the number of biomass available. Table 4.3 shows the results for MLSS and MLVSS in each bioreactor.

Table 4.3 Comparison of MLSS and MLVSS concentrations in various AnMBRs with different PAC sizes

Parameters	R1	R2	R3	R4
MLSS, mg/L^{-1}	13004 ± 3358	13362 ± 1192	13433 ± 1672	21420 ± 2604
MLVSS, mg/L^{-1}	9981 ± 2455	10741 ± 1192	10612 ± 1672	16452 ± 2604

The highest bacteria growth is in R4 with the concentration of 21420 mg/L. AnMBRs with no PAC and with bigger PAC size show rather lower value in MLSS (13004 mg/L, 13362 mg/L and 13433 mg/L respectively). The MLSS and MLVSS increased with decreased in particle sizes, this is due to smaller particle size contribute to larger surface area to promote attached growth of bacteria. Furthermore the increase of bacteria population would further enhance the removal rate of COD and NOM in POME, resulting in better supernatant quality.

4.3 Effect of Protein and Polysaccharide towards Membrane Fouling

Polymer membrane was used to carry out the filtration performance of the AnMBRs. Figure 4.4 shows the filtration performance of the AnMBRs. It can be observed that R4 with the lowest combine concentration of protein and polysaccharide had the best filtration result followed by R3 and R2. The worst performer is R1 which has no PAC inside its bioreactor.

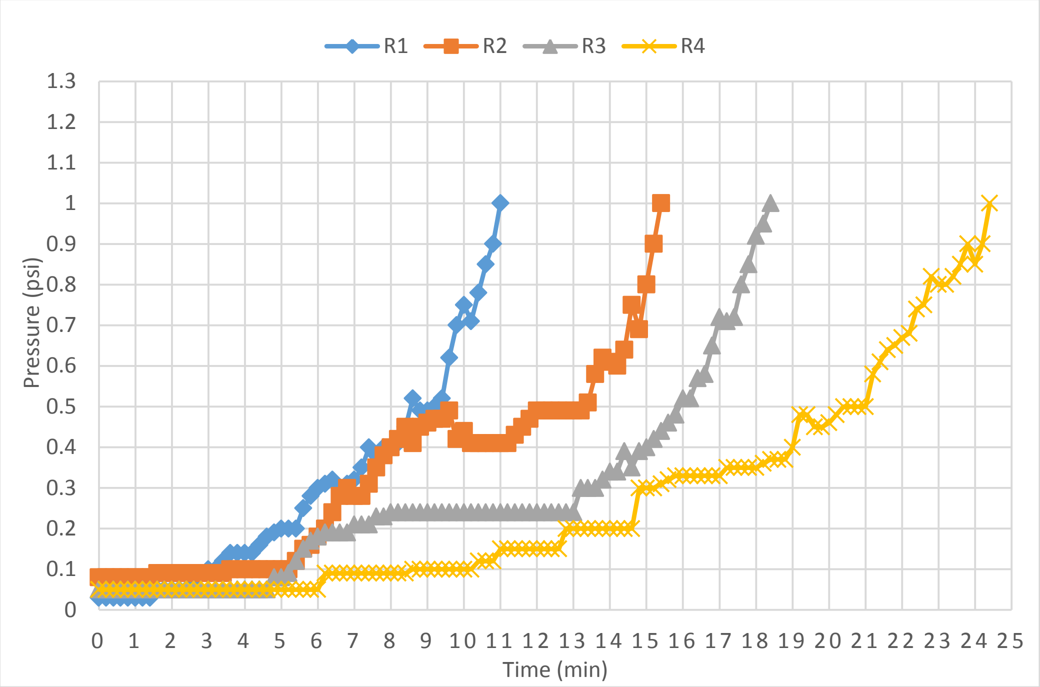


Figure 4.1 Performance of membrane fouling control of different anaerobic membrane bioreactors added with different sizes of powdered activated carbon

The present of NOM plays a significant roles in membrane fouling control (Huang et al., 2007). As the amount of NOM increases, it is more likely that a membrane would be fouled more easily. This is due to the adhesive and cohesive nature of protein and polysaccharide which stimulates the filter cake formation, resulting in higher membrane fouling rate.

4.4 Effect of Floc Size towards Membrane Fouling

In addition to NOM which is being one of the contributors towards membrane fouling, the floc size also plays an important role in fouling controlled. As shown in Figure 4.5, the particle size distribution for activated sludge in R1 was smaller compared to other AnMBRs with PAC. This results show that by adding PAC into activated sludge, bigger particle size were produced.

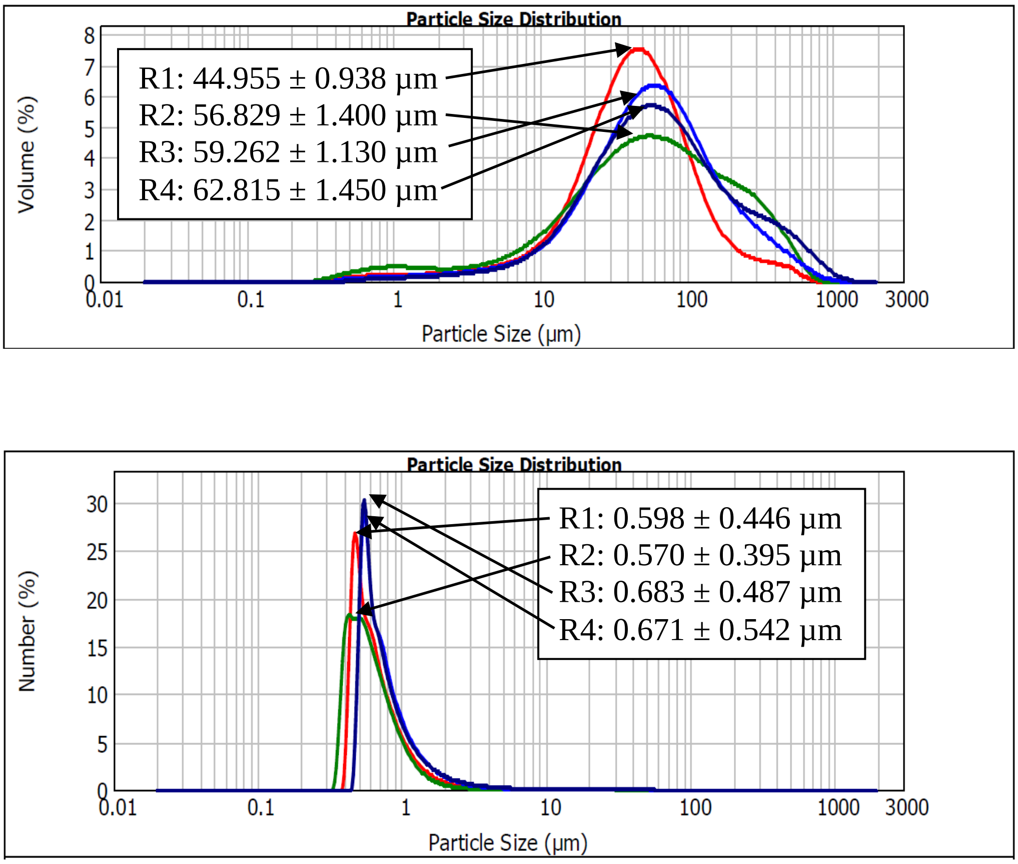


Figure 4.2 Microbial flocs size distribution of the different anaerobic membrane bioreactors added with different sizes of PAC

This indicates that PAC could act as an adsorbent which could attract bacteria to attach on its surface to transform the PAC to become biological activated carbon (BAC). As the size of PAC decreases, the floc size distribution show higher value, and this phenomenon may be caused by the PAC with smaller size are more porous compared to PAC with relatively larger size which results in forming larger floc more effectively (Yoshihiko et al., 2009).

Research show that larger floc size would produce greater porosity and permeability filter cake which could reduce the trans-membrane pressure (TMP) (Yoshihiko et al., 2009). As shown in Figure 2 and Figure 3, floc size in R4 is 62.185 ± 1.45 μm and it had the best membrane fouling control performance which had indicated the filter cake formed was more porous compared to other AnMBRs. R1 performed worst and it has the smallest floc size among the AnMBRs.

4.5 Performance of the Hybrid Membranes Incorporated with Different Concentrations of PAC

Supernatant from R4 was used to study the performance of hybrid membrane with PAC. The performance of the hybrid membranes incorporated with different PAC content of 0% wt, 1% wt and 5%wt was carried out. As shown in Figure 4.6, the performance of hybrid membrane for R4 increases as the PAC content increases, by incorporated 1% wt of PAC into the polymer membrane would only show little improvement of membrane fouling control. However, when the concentration of PAC increased from 1 to 5%wt, the hybrid membrane had much better membrane fouling control as per Figure 4.6.

There were studies shows that by coating the membrane with particles in micrometre range with high adsorption rate for NOM such as PAC or heated aluminium oxides can enhance the retention of organic substance in the filter cake, which could enhance the fouling resistance of a membrane towards NOM (Schulz et al., 2016; Kim et al., 2008; Cai et al., 2013; Ellerie et al., 2013).

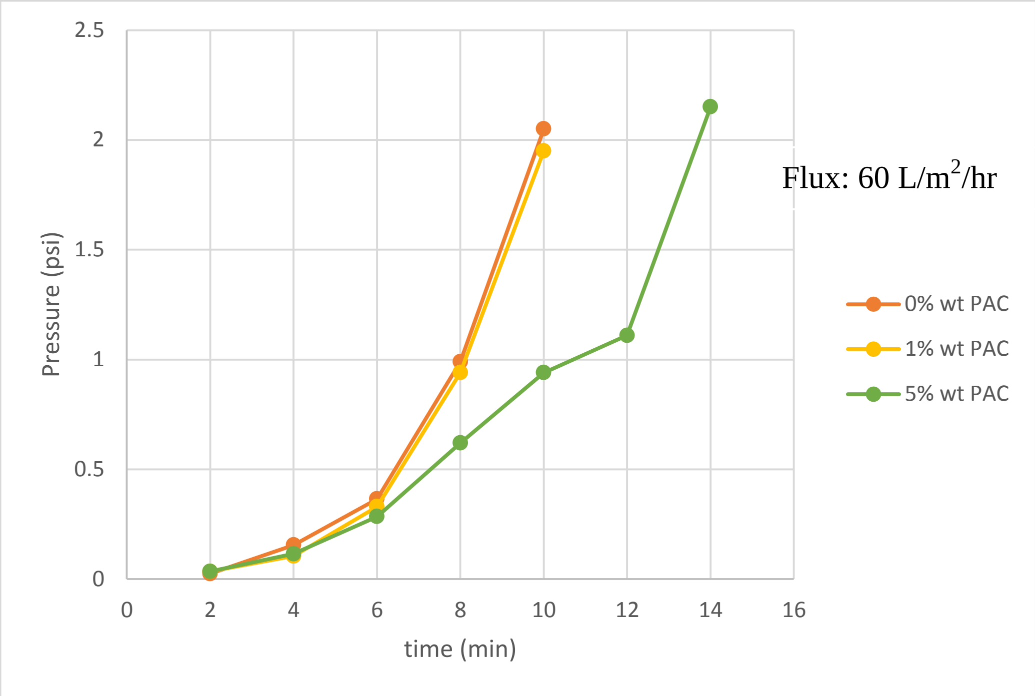


Figure 4.3 Comparison in pressure build up with respect to different PAC dosages in a hybrid membrane

A dead end filtration test was also being carried out to verify the above cross-flow filtration results and the same trend was observed. As shown in Table 4.4, the volume of permeate collected increases as the PAC content in hybrid membrane increases. This indicates that by incorporating the PAC into the polymer membrane would help to increase the performance of the membrane fouling control and produce better permeate quality as per Table 5. However, it was noticed that all the membrane and hybrid membranes used for R4 had good performance in pollutants removal as they could remove COD from the POME more than 90% as per Table 4.4.

Table 4.4 Performance of different hybrid membranes incorporated with different concentrations of PAC in palm oil mill effluent (POME) treatment

Parameter	**0% wt PAC**	**1% wt PAC**	**5% wt PAC**
Volume of effluent collected in 10 minutes, mL	10 ± 5	10 ± 6	21 ± 5
COD, mg/L^{-1}	1066 ± 33	740 ± 66	696 ± 274
COD removal efficiency, %	93.52 ± 2.27	95.50 ±1.35	95.77 ± 0.27

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

Addition of PAC in an AnMBR was proven to be effective in terms of COD and NOM removal. It was found that simultaneous processes of adsorption and biodegradation are the main elements helped to nhance the performance of AnMBR added with PAC. AnMBR with relatively smaller PAC size (75.178 μm) could perform better compared to the AnMBR with relatively bigger PAC sizes or with PAC.

In addition, reduction of NOM for the AnMBR added PAC also contributed to the better membrane fouling control. Bigger floc size was noticed for the AnMBR using smaller PAC size which helped to produce a more permeate filter cake and resulting in having better membrane fouling control.

It was found that by adding PAC into PES membrane to produce a hybrid membrane could improve the fouling resistance and enhanced fine pollutants removal rates. PES hybrid membrane perform best by having 5% wt of PAC incorporated into it. PAC could act as an adsorbent for contaminants, preventing contaminants from directly contact with membrane surface. Its effect would be better after being transformed into BAC which is equipped with the ability to do simultaneous adsorption and biodegradation processes.

5.2 Recommendations

There are few recommendations that can be made to further improve the performance of AnMBR which are:

I. Enable constant mixing in the anaerobic bioreactor in order to obtain a more efficient and stable results.
II. Optimum ratio of PES to PAC% wt should be study to obtain the optimum dosage of PAC required to incorporate into a PES membrane.
III. Biogas production based on different sizes of PAC.

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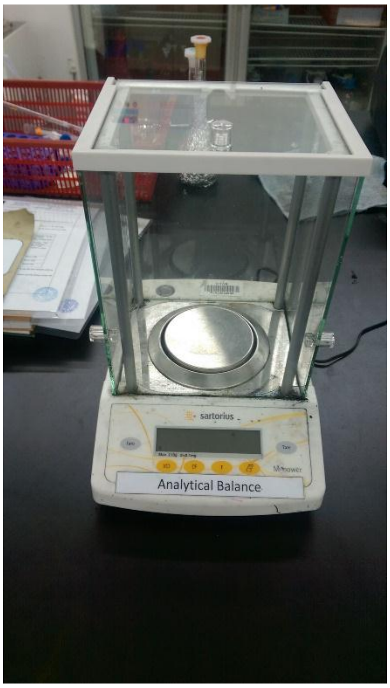
APPENDIX

Dope preparation equipment setup

AnMBRs setup

Dead End Filtration System

Cross Flow Filtration System



M-power Analytical Balance AZ214 (Sartorius weighing technology,

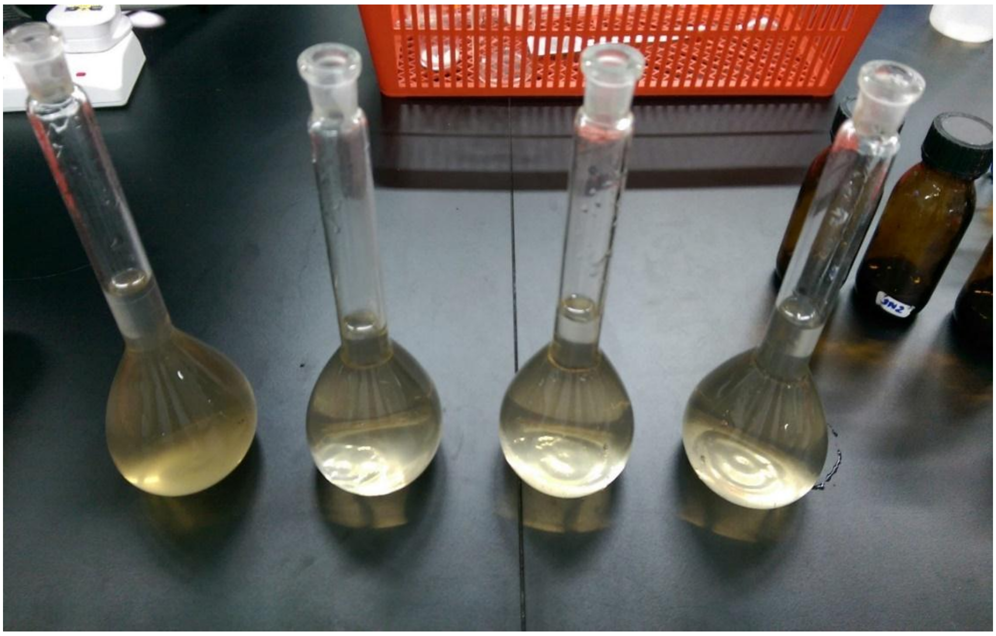
COD Reactor

Membrane auto casting machine

HACH UV/VIS spectrophotometer (Model DR 6000)

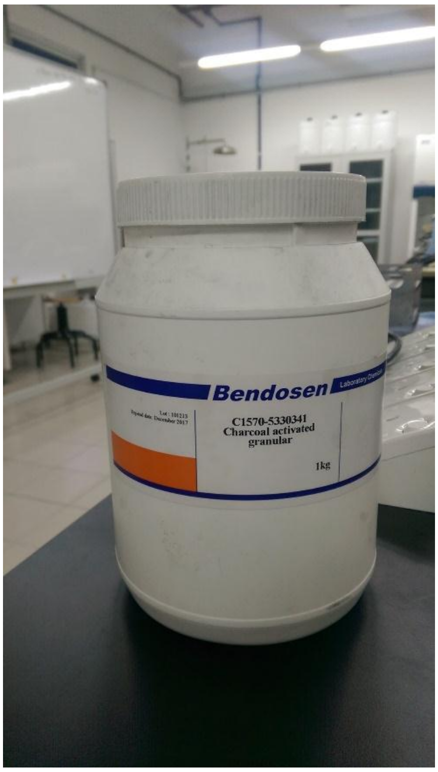
Sonicator bath

Particle size analyser (Malvern Mastersizer 2000, UK)

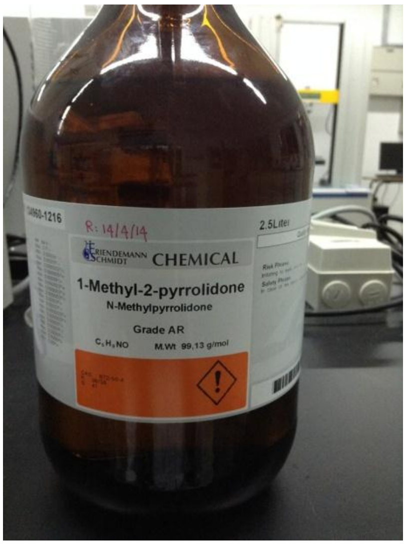


Dilution of samples (R1, R2, R3, R4)

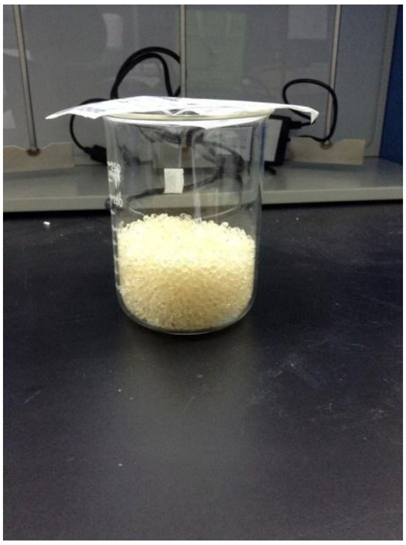
MLSS & MLVSS



Granular Activated Carbon



1_Methyl-2 pyrorolidone (NMP)



Polyethersulfone (PES)