BIOMETHANATION OF PALM OIL MILL EFFLUENT (POME) BY ULTRASONIC MEMBRANE ANAEROBIC SYSTEM (UMAS) USING POME AS SUBSTRATE

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NURI 'ADILAH NASHRULMILLAH BT IDRIS

Thesis submitted in partial fulfilment of the requirements
for the award of the degree of
Bachelor of Chemical Engineering (Technology Gas)
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JANUARY 2014

SUPERVISOR'S DECLARATION

We hereby declare that we have checked this thesis and in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Bachelor of Chemical Engineering (Technology Gas).

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I hereby declare that the work in this thesis entitled "Biomethanation of Palm Oil Mill Effluent (POME) by Ultrasonic Membrane Anaerobic System (UMAS) using POME as substrate" is my own except for quotations and summaries which have been duly acknowledged. The thesis has not been accepted for any degree and is not concurrently submitted for award of other degree.

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Dedication

Special Dedication to my supervisor, my family members, my friends, my fellow colleague and all faculty members for all your care, support and believe in me.

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ABSTRACT

The direct discharge of the Palm Oil Mill Effluent (POME) wastewater causes serious environmental pollution due to its high chemical oxygen demand (COD), total suspended solid (TSS) and biological oxygen demand (BOD). The conventional ways for POME wastewater treatment have both economical and environmental disadvantages. In this study, the potential of ultrasonic-assisted membrane anaerobic system (UMAS) was evaluated as alternative and cost effective method for treating POME wastewater to avoid fouling. The UMAS treatment efficiency was greatly improved by UMAS introduction. The membrane fouling and polarization at the membrane surface was significantly reduced. This research had proved that UMAS is the effective way to treat POME. The removal efficiency of COD was 95% with HRT of 6 days. The BOD removal efficiency was 71.59% while TSS removal rate was from 91 to 99.5%.The methane gas production efficiency was 94.14%.The UMAS treatment efficiency was greatly improved by UMAS introduction. The membrane fouling and polarization at the membrane surface was significantly reduced. This research had achieved the objectives by resolve the problem statement.

Key words: UMAS, Anaerobic, POME, COD, membrane, ultrasonic

ABSTRAK

Pelepasan air pemprosesan kelapa sawit (POME) tanpa rawatan akn menyebabkan pencemaran kerana ia mengandungi keperluan oksigen kimia (COD), keperluan oksigen biologi (BOD) dan jumlah pejal (TSS) yang tinggi. Rawatan konventional bukan sahaja memerlukan kos yang tinggi juga menyebabkan pencemaran. Dalam kajian ini, potensi kaedah rawatan dengan sistem membran anaerobik berultrasonik (UMAS) dikaji supaya dijadikan pilihan alternative dan kaedah kos efektif untuk rawatan air pemprosesan kelapa sawit dan mengelakkan masalah membrane tersumbat. Kecekapan sistem rawatan UMAS ditingkat dengan ultrasonik yang dipasang. Masalah membrane tersumbat dan polarasi didapati berkurangan. Kajian ini telah membuktikan bahawa UMAS adalah cara yang berkesan untuk merawat POME. Kecekapan penyingkiran COD adalah 95% dengan HRT 6 hari. Kecekapan penyingkiran BOD adalah 71,59% manakala TSS kadar penyingkiran adalah 91-99,5%. Kecekapan pengeluaran gas methana adalah 94,14%. UMAS menunjukkan keberkesanannya rawatan telah bertambah baik dengan pengenalan UMAS. Ini kejadian membran tersumbat dan polarisasi di permukaan membran telah dikurangkan dengan ketara. Kajian ini telah mencapai objektifnya berdasarkan menyelesaikan pernyataan masalah.

Kata kunci : UMAS, Anaerobik, POME, COD, membran, ultrasonik

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LIST OF SYMBOLS

<	less than
=	equal
%	Percentage
˚C	Degree Celsius
P	Pressure
T	Temperature

LIST OF ABBREVIATIONS

BOD	Biological Oxygen Demand
CDM	Certified emission reduction
CER	Clean development mechanism
CO_2	Carbon dioxide
COD	Chemical Oxygen Demand
CUF	Crossflow Ultrafiltration
Da	Dalton
GHG	Green House Gases
GFTS	Green Technology Financing Scheme
HRT	Hydraulic Retention Time
IR	Infrared
NaOH	Sodium hydroxide
MAS	Membrane Anaerobic system
MLSS	Mixed liquor suspended solid
MPOB	Malaysian Palm Oil Board
MWCO	Molecular cut off
OLR	Organic Loading Rate
POME	Palm Oil Mill Effluent
PVC	Polyvinyl chloride
RO	Reverse Osmosis
SRT	Solid Retention Time
SS	Suspended Solid
TPAD	Temperature phase anaerobic digester
TSS	Total Suspended Solid
UF	Ultrafiltration
UMAS	Ultrasonicated Membrane Anaerobic System

US	Ultrasonic
VFA	Volatile fatty acid
VSS	Volatile Suspended Solid
VS	Volatile Solid
W/W	Weight to weight

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CHAPTER 1

INTRODUCTION

1.1. BACKGROUND OF STUDY

The growth of the palm industry in Malaysia has been phenomenal. Indonesia and Malaysia are the two largest oil palm producing countries (Table 1) and is rich with numerous endemic, forest-dwelling species. Malaysia has a tropical climate and is prosperous in natural resources. Oil palm currently occupies the largest acreage of farmed land in Malaysia (Arif, 2011). While the oil palm industry has been recognized for its contribution towards economic growth and rapid development, it has also contributed to environmental pollution due to the production of huge quantified of by-product from the oil extraction process. The waste products from oil palm processing consist of oil palm trunks (OPT), oil palm fronds (OPF), empty fruit bunches (EFB), palm pressed fibers (PPF) and liquid discharge palm oil mill effluent (POME) (Yusoff, 2007).

Table 1: World Major Producers of Palm Oil 2002-2011 ('000 Tonnes)

Country	2002	2003	2004	2005	2006	2007	2008	2009	2010	2011
Indonesia	9,370	10,600	12,380	14,100	16,070	17,420	19,400	21,000	22,100	23,900
Malaysia	11,909	13,355	13,976	14,962	15,881	15,824	17,734	17,565	16,994	18,911
Thailand	600	690	735	700	860	1,050	1,300	1,310	1,380	1,530
Nigeria	775	785	790	800	815	825	840	870	885	900
Colombia	528	527	632	673	714	733	778	802	753	965
Ecuador	238	262	279	319	352	396	410	429	380	460
Papua New Guinea	316	326	345	310	365	382	465	478	500	525
Cote d'Ivoire	265	240	270	290	281	289	285	345	330	310
Honduras	126	158	170	237	258	265	278	280	275	278
Brazil	118	129	142	160	170	190	210	240	250	270
Costa Rica	128	155	180	181	189	200	202	220	230	250
Guatemala	86	85	87	92	125	130	185	180	182	193
Venezuela	55	41	61	63	66	70	89	84	75	77
Others	895	906	1,131	1,065	1,113	1,057	1,092	1,308	1,524	1,611
TOTAL	**25,409**	**28,259**	**31,178**	**33,952**	**37,259**	**38,831**	**43,268**	**45,111**	**45,858**	**50,180**

Source: Oil World Annual (2007-2011) & Oil World Weekly (9 December, 2011)

Palm Oil processing gives rise to highly polluting waste-water, known as Palm Oil Mill Effluent (POME), which is often discarded in disposal ponds, resulting in the leaching of contaminants that pollute the groundwater and soil, and in the release of methane gas into the atmosphere. POME is an oily wastewater generated by palm oil processing mills and consists of various suspended components. This liquid waste combined with the wastes from steriliser condensate and cooling water is called palm oil mill effluent (POME). Moreover, POME has a very high Biochemical Oxygen Demand (BOD) and Chemical Oxygen Demand (COD), which is 100 times more than the municipal sewage. POME is a non-toxic waste, as no chemical is added during the oil extraction process, but will pose environmental issues due to large oxygen depleting capability in aquatic system due to organic and nutrient contents (Zafar, 2013). Malaysian experiences in effluent control in the palm oil industry demonstrate that a set of well designed environmental policies can be very effective in controlling industrial pollution in a developing country. The Environmental Quality (prescribed Premises)(Crude Palm Oil) Regulation 1977, promulgated under the enabling powers of

Section 51 of the EQA, are the governing regulations and contain the effluent discharge standards. Other regulatory requirements are to be imposed on individual palm oil mills through condition of license according to Environmental Quality Act 1974 (Pierzynski, 2005). In order to reach the requirement of standard discharge limit, POME treatment can never to be dismissed. It incurs high non-profitable cost in an industry to resolve this problem either the waste water have to be reduced or treatment have to be enhanced in cost effective way.

Instead of treatment system such the conventional ponding system, the membrane anaerobic system (MAS) will be proposed to be utilized. The system consists of two technologies which are anaerobic digestion and membrane technology. Anaerobic digestion is widely adopted in the industry as a primary treatment for POME. Biogas is produced in the process in the amount of 20 m^3 per ton FFB. This effluent could be used for biogas production through anaerobic digestion. At many Palm-oil mills this process is already in place to meet water quality standards for industrial effluent. (Zafar, 2013). With the addition of application of membrane filtration in the system, the efficient of wastewater treatment is elevated that is capable of retaining biomass concentration within the reactor and produce high quality effluent. It is proven to be an effective way in separating biomass solid from digester suspension and recycle them to the digester.

Membrane technology is one of the possible technology solutions to treat the high organic content effluent. Membrane treatment is a physical process alternative that is capable of providing a highly efficient treatment, requires minimal energy and does not introduce any additives to the waste system. Among the various membrane processing techniques, ultra filtration presents an attractive option for wastewater treatment. It is a low pressure-driven membrane process retaining most effectively macromolecules sized within 0.001 – 0.02 µm. Membrane ultrafiltration is capable of producing a higher quality effluent that can successfully meet the increasingly stringent effluent discharge standards set out in the Environmental Quality Act, 1974.

However, in this membrane anaerobic system has to be monitored properly as the processes rely solely on the micro-organism to break down the pollutants. The

micro-organism is very sensitive to changes in the environment for the micro organism. Besides there will be problem arises in the membrane system due to the characteristic of POME as it has a high suspended solid effluents. The membrane will be suffered from fouling and degradation during use. The process by which a variety of species present in the water increase the membrane resistance, by adsorbing or deposition onto its surface, adsorption onto the pore surface within the bulk membrane material (pore restriction) or by complete pore blocking. (Leonard Lim Lik Pueh, 2004)

1.2 PROBLEM STATEMENT

The main problem statement is POME contains highly organic content of wastewater. Since these compounds are harmful to the environment, it becomes necessary that effluents water should be treated or purified before discharge into the environment. This POME would normally have resulted in significant environmental pollution if discharge directly without efficient treatment being implemented. Coming to the context of water and air pollution, POME is one of the agriculture waste blame on.

Greenhouse gasses emitted from Palm Oil Mill Effluent anaerobic treatment pond such as methane and carbon dioxide exerted greenhouse effect to the earth. The capturing of methane gas will save the environment. Besides, the treatment of POME often incurs high non-profitable cost is an industries that reduces the company profit. In addition, the cost of fossil fuel increases with the increasing demand and the depleting resource making it even valuable. The concept of transforming waste to energy makes waste treatment seem more appealing and cost-effective.

Besides, membrane technology that applied in the efficient treatment of POME to be monitored properly because there will be problem arises in the membrane system due to the characteristic of POME as it has a high suspended solid effluents. The membrane will be suffered from fouling and degradation during use it continuously in industry scale. The study should be conducted to overcome all the problems arise in treatment of POME.

1.3 OBJECTIVES OF THE STUDY

The research aims to solve the problem statement by accomplishing the following specific objectives:

1. To enhance the treatability of high concentrated POME by Ultrasonic membrane anaerobic system (UMAS)
2. To study the membrane fouling control and increment of methane gas by application of Ultrasonic membrane anaerobic system (UMAS)
3. To evaluate the overall performance of Ultrasonic membrane an aerobic system (UMAS) in treating Palm Oil Mill Effluent (POME)

1.4 SCOPE OF RESEARCH

This study is focused on enhance the treatability of using UMAS for treatment of highly concentrated of POME. The system performance were evaluated with significant parameter such as COD, BOD, TSS, and VSS for the raw material, material in the reactor and the treated permeate to observe the efficiency of the system. In order to optimize the production of methane and overcome membrane fouling problem, a 150 L bioreactor system and cross flow membrane module is attached with ultrasonic device. The parameters such as pH and temperature are controlled and maintain in optimum operating condition.

1.5 RATIONALE AND SIGNIFICANT

As the palm oil industry require tackling the challenges in meeting the growing worldwide demand for palm oil as food, while at the same time has to demonstrate the sustainability of its products and operations. The issue of environmental pollution should be concern to maintain the sustainability of palm oil industry. Realizing the danger of the possible directly discharge of POME, the study will provide some efficient way in treating the POME by reducing the organic content of POME and reducing the emission of green houses gasses to the environment. The study can protect the environment from pollution effect of POME.

Besides, with the increasing awareness on the environmental issues and the rising oil prices, all governments across the world are forced to looking for alternative energy, the same phenomenon happen in Malaysia as well. The Renewable energy has been recognized as the country's fifth fuel under the 8^{th} and 9^{th} Malaysia Plan. So, this study can suggest by providing an alternative renewable energy that can be apply in the industry in return overcome the dependency on fossil fuel which is incurs high cost. Eventual, the cost for POME treatment can be reduced and provide alternative energy, it will support Malaysia Plan in the same time it will attract foreign investor without realized.

CHAPTER 2

LITERATURE REVIEW

2.1 PALM OIL MILL EFFLUENT (POME)

POME is generated as a result of sterilization of fresh palm oil fruit bunches, clarification of palm oil and effluent from hydro cyclone operation. (Borja et al, 1996) POME is a high strength agro-industrial polluter due to high value of COD and BOD.POME is in a form of highly viscous dark brown colloidal with fine suspended solid. POME colloidal suspension of 95-96% water, 0.6-0.7% oil and 4.5% total solids (Ma, 1993). The characteristic of POME are shown in **Table 2.1**. In 1980, Malaysian mills discharged 6 million tonnes of effluent which contain equivalent BOD as load generated by population of 7.3 million. However it's highly amendable by anaerobic digestion.

Table 2.1: Characteristic of untreated POME

Parameter	Concentration
pH	4.7
Temperarture	80-90
BOD 3-day, 30˚C	25,000
COD	50,000
Total soilids	40,500
Suspended solids	18,000
Total volatile Solids	34,000
Ammoniacal-Nitrogen	35
Total Nitrogen	750

*All parameter in mg/l except pH and temperature (˚C)

Source: (A.L Ahmad, 2003)

2.2 METHANE GAS FOR ELECTRICITY GENERATION

The generation of electricity from methane is possible, in all cases the steps that must be gone through are twofold, chemical energy to mechanical energy, and then from mechanical energy to electrical energy. For these conversion processes to be achieved, suitable engine is needed, and in principle there are two types of engine which have been used for biogas digester electricity generation that is gas engine and steam turbine.

According to the Malaysia Palm Oil Board (MPOB), 0.65 m3 POME is generated from every processed ton of Fresh Fruit Bunch. Based on a study of the potential for electricity generation from POME that have done by MPOB, if there was 38,870,000 m^3 of POME produced for every 59,800,000 tons of Fresh Fruit Bunches process annually. The annual energy content of the generated methane gas can be calculated to 7.07E+09 kWh. Based on a conversion efficiency of 38 percent (gas engine), the potential annual electrical power generation would be 2.69E+09 kWh. Thus, Palm Oil Mill Effluent has a huge potential for power generation (N.A Ludin et al, 2006).

2.3 POME TREATMENT

2.3.1 Ponding System/Lagoon system/Open Digester tank

Ponding system is the most common system employed in Malaysia which counted for 85% of the total treatment plant in Malaysia. In a ponding system it is basically divided into de-oiling pond tank, acidification ponds, anaerobic ponds and facultative pond or aerobic ponds. The discharge after the facultative or aerobic require further reduce of BOD to comply with the discharge standards. The typical size of the ponding system is equivalent to half a soccer field which is able to sustain the processing capacity of 54 tons per hour. This method is favored due to it can achieve reasonable degree of treatment with low construction and operating cost and is easily maintained as the technology employed is relatively unsophisticated. However, a large

land space is required. Direct emission of gasses generated in the treatment process will impose green house effect to the environment. Besides, the effectiveness in meeting the stringent standard is unsatisfactory. (Poh P.E et al, 2009)

Open digester tank are used for POME treatment when limited land area is available for ponding system. Apart from that, in the investigation by Yacobs et al (2006), he proved that anaerobic system emitted higher amount of methane compare to the open digester tank with an average methane composition of 54.4% compare to open digester tank. (Poh P.E et al, 2009)

2.3.2 Anaerobic Digestion

A biochemical process is organic matter is decomposed by bacteria in the absence of oxygen, producing methane and other by products. It's much depends on the bacterial consortia for degradation process, thus a longer time is require. The condition is also required to be always in the optimum condition for the bacterial to survive, as the bacterial are sensitive. However, anaerobic digestion is widely used to treat waste as it require low energy, high organic removal rate, low sludge production and production of methane as valuable by product. (Poh, P.E. et al, 2009)

The degrading process of POME consists of four stages that is hydrolysis and acidogenesis, fermentation, acetogenesis, methanogenesis (Poh, P.E et al,2009). In the first stage of hydrolysis, the polymeric organic materials are hydrolysed to its constituent such as glucose, fatty acids and amino acids by hydrolytic bacteria. The hydrolysis process is of significant importance in high organic waste and may become rate limiting. Solubilisation involves hydrolysis process where the complex organic matter is hydrolysed into soluble monomers. Fats are hydrolysed into fatty acids or glycerol; proteins are hydrolysed into amino acids or peptides while carbohydrates are hydrolysed into monosaccharides and disaccharides.

In fermentation stage, the hydrolysed products are converted to volatile fatty acids, alcohols, aldehydes, ketones, ammonia, carbon dioxide, water and hydrogen by the acid-forming bacteria. The organic acids formed are acetic acid, propionic acid,

butyric acid and valeric acid. Volatile fatty acids with more than four-carbon chain could not be used directly by methanogens (Wang et al., 1999).

The following stage is acedogenesis, where organic acids are further oxidised to acetic acid and hydrogen and carbon dioxide which are used in the subsequent process. Acetogenesis also includes acetate production from hydrogen and carbon dioxide by acetogens and homoacetogens.The transition of the substrate causes the pH of the system to drop which beneficial to acidogenic and acetagenic.(K.M Ostrem et al,2004)

Finally the reaction come across the the stage of methanogenesis. One is conversion of acetate to carbon dioxide and methane by acetotrophic organisms and another is reduction of carbon dioxide with hydrogen by hydrogenotrophic organisms. (Ling,L.Y, 2007).

Typical reaction of anaerobic digestion:

$$C_6H_{12}O_6 \longrightarrow 2C_2H_5OH+CO_2 \quad [1]$$

$$C_2H_5OH+CO_2 \longrightarrow CH_4+ 2CHOOH \quad [2]$$

$$CH_3COOH \longrightarrow CH_4 + CO_2 \quad [3]$$

$$CO_2 + 4H_2 \longrightarrow CH_4 + 2H_2O \quad [4]$$

The advantages of adopting anaerobic system are low energy requirement as no aeration needed. Methane is produced as a valuable end product and generates sludge that could be used for land application. There are several anaerobic treatment method that have been widely used such as Anaerobic filtration, fluidized bed reactor, up-flow anaerobic sludge blanket reactor (UASB), Up flow anaerobic sludge fixed-film reactor (UASFF), continuos stirred tank reactor and Anaerobic contact process. Although these high rate or hybrid reactors are successfully shortened the retention time and efficiency (as shown in table) but all these biological treatment systems need proper maintenance and monitoring as the processes solely rely on micro-organisms to degrade the pollutants. How to ensure the stability of the system deserves most urgent concern. (Y.J Zhang et al, 2007) .The summary of comparisons of all other method are shown in table 2.2.

Table 2.2: Comparisons of various treatment methods on POME treatment

Table 6
:e of various anaerobic treatment methods on POME treatment

	OLR (kg COD/m^3day)	Hydraulic retention time (days)	Methane composition (%)	COD removal efficiency (%)	Reference
Anaerobic pond	1.4	40	54.4	97.8	Yacob et al. (2006a)
Anaerobic digester	2.16	20	36	80.7	Yacob et al. (2005)
Anaerobic filtration	4.5	15	63	94	Borja and Banks (1994b)
Fluidized bed	40.0	0.25	N/A	78	Borja and Banks (1995b)
UASB	10.63	4	54.2	98.4	Borja and Banks (1994c)
UASFF	11.58	3	71.9	97	Najafpour et al. (2006)
CSTR	3.33	18	62.5	80	Tong and Jaafar (2006)
Anaerobic contact process[a]	3.44	4.7	63	93.3	Ibrahim et al. (1984)

N/A: data unavailable.
[a] In terms of BOD.

Source: (Poh P.E et al, 2009)

2.3.3 Membrane Separation Technology

Membrane Separation technology is always employed in waste treatment as it's able to produce consistent and good water quality after treatment plants as well as it's able to disinfect the treated water. There have been inspiring performances by using membrane separation technology. For instances, A.L Ahmad et al (2003) have shown that the combination of UF & RO is able to achieve COD removal of 98.8%,BOD removal of 99.4%, Turbidity of 100% and pH 7 as a result. Another group of researcher have incorporated Hollow fiber membrane in their three phase decanter system to give 89.9% COD removal, 99.4% of TSS elimination, 97.9% Turbidity reduction and 92.9% for color removal (S.S Raja et al, 2005).However, short membrane life, membrane fouling and expensive cost are major constraint of this technique. In order to prolong the membrane life span and produce crystal clear effluent as well as methane as the end product, the integration of anaerobic system and membrane separation technology in a bio reactor is investigated by some researchers.

2.3.4 Membrane Anaerobic System

The idea of integration of the anaerobic digestion system and membrane separation technology is to enable the biomass to be retained in the reactor which improves methane gas emission as well as producing constant high quality effluent. According to Y.J Zhang et al in 2007 she has incorporating Expanded Granulated Sludge Blanket (EGSB) with UF & RO. As a result, COD Removal of 93%, biogas conversion rate of 43% is achieved. As we compared the result to the previous table, the biogas generation appears to improve drastically. In the later years, H.N Abdurahman et al (2011) have shown another more inspiring result by his Membrane Anaerobic System which a design of anaerobic bioreactor equipped with UF module membrane where COD Removal efficiency 96.6%-98.4% and biogas conversion rate up to 73% as a final result.

However, although the membrane fouling problem may relief compared to the case without anaerobic digestion as pretreatment but the membrane fouling problems still an issues and the idea of back flushing membrane which require an operation break is not feasible to the industrial application. Hence, as a solution application of ultrasonic technology in solving the membrane fouling problem is going to be investigated in this research work.

2.4 METHANOGENS

Methanogen are specialized group of Archae that utilized a limited number of substrates, principally acetate, carbon dioxide and hydrogen for methane production or methanogenesis. These substrate resulted from the degradation from more complex substrate. Methane-forming bacteria have many shapes (bacillus, coccus, and spirillum), sizes (0.1 to 15μm), and growth patterns (individual cells, filamentous chains, cubes, and sarcina). Methane-forming bacteria are oxygen-sensitive anaerobes and are found in habitats that are rich in degradable organic compounds. In these habitats oxygen is rapidly removed by bacterial degradation of the organic compounds.

Methane-forming bacteria are active within the pH range of 6.8 to 7.2. Methane forming bacteria are sensitive to pH values <6.8 and >7.2. With decreasing pH, methane-forming bacteria become less active, while fermentative bacteria remain active and continue to produce fatty acids. These acids destroy alkalinity and depress pH resulting in inhibition of methane-forming bacteria. Also, with decreasing pH, increases in the quantities of hydrogen sulphide (H2S) and hydrogen cyanide (HCN) occur. These two inorganic compounds are highly toxic to methane-forming bacteria. With increasing pH, an increase in the quantity of ammonia (NH3) occurs. Ammonia also is toxic to methane-forming bacteria. Therefore, anaerobic digesters should be operated at a near neutral pH value and should be monitored as needed to ensure an acceptable pH value and alkalinity residual.

Sufficient alkalinity is necessary for proper pH control. Alkalinity serves as a buffer that prevents rapid change in pH. Enzymatic activity of methane-forming bacteria is adversely affected by pH values <6.8 and >7.2. Adequate alkalinity in an anaerobic digester can be maintained by providing an acceptable volatile acid-to alkalinity ratio. The range of acceptable volatile acid-to-alkalinity ratios is 0.1 to 0.2.

Because methane-forming bacteria reproduce very slowly (generation times of 3–30 days) and produce very few offspring (sludge) from the degradation of substrates (approximately 0.02 pounds of sludge per pound of substrate degraded), methane-forming bacteria require smaller quantities of most nutrients. However, there are a few nutrients that are required by methane-forming bacteria in quantities two to five times greater than most other bacteria. These nutrients are cobalt, iron, nickel, and sulphur.

Methanogenesis occurs through three basic biochemical reactions that are mediated by three different groups of methane-forming bacteria (acteoclastic methanogens, hydrotrophic methanogens, and methyltrophic methanogens). Acetoclastic methanogens produce methane by “splitting’ acetate as shown in reaction equation 5. Hydrogenotrophic methanogens produce methane by combining hydrogen and carbon dioxide [6] while methyltrophic methanogens produce methane by removing methyl ($-CH_3$) groups from simple substrates. In anaerobic digesters, acetoclastic methane-forming bacteria produce most of the methane, while hydrotrophic methane-

forming bacteria produce approximately 30% of all methane. Methyltrophic methane-forming bacteria produce a relatively small quantity of methane in anaerobic digesters (Michael H. Geradi, 2006).

$$CH_3COOH \text{----------} > CH_4 + CO_2 \quad [5]$$

$$CO_2 + 4H_2 \text{----------} > CH_4 + 2H_20 \quad [6]$$

2.5 ANAEROBIC DIGESTION OPERATION

2.5.1 pH

pH is the crucial factor that determine whether the Membrane anaerobic system is working. The microbial community in anaerobic digester is sensitive to pH change. The pH affects the process in 2 ways that are affecting the enzymatic activity by changing their proteic structure which may occur drastically as a result of changes in the pH and affecting the toxicity of a number of compounds indirectly eg sulphide toxicity. The optimum pH for methane producing microorganism to achieve optimum growth range between 6.6 and 7.4 (V.S Marcos et al,2005). Methane producing bacteria require a neutral to slightly alkaline environment (pH 6.8 to 8.5) in order to produce methane (D.A Burke et al, 2001). Acid forming bacteria grow much faster than methane forming bacteria. If acid-producing bacteria grow too fast, they may produce more acid than the methane forming bacteria can consume. Excess acid builds up in the system. The pH drops, and the system may become unbalanced, inhibiting the activity of methane forming bacteria. Methane production may stop entirely.

Besides, the methanogenesis is strongly affected by pH and will be inhibited by the acid condition. The optimum pH for the methanogenesis stage is pH between 7.2-8.2 .If the pH fall below the pH of 6, anaerobic degradation rate will decrease and the lipids are not degraded (Ling,L.Y., 2007).The Acetic and butyric acids are favourable substrate for methanogens which form under neutral and acidic condition.

In addition, sudden pH change (pH shock) can adversely affect the process, and recover depend on series of factors, related to the type of damage caused to the

microorganism (either permanent or temporary). The buffer capacity used must be understood to avoid changes in pH (V.S Marcos et al, 2005).

2.5.2 Mechanical Mixing

Mixing will provides good contact between substrate and microbes ensure the temperature is uniform, reduce resistance to mass transfer, minimized build up of inhibitory intermediate and stabilizes environment conditions (N.H Abdurahman et al, 2010). The same theory is proposed by Leslie Grady et al (1999) as well where mixing able to bring bacteria consortia into contact with food. The agitation of the mixing will also reduce the particle size which promotes the release of biogas from mixing (Karim et al, 2005).

The bioreactor with stirrer have been applied by a mill under Keck Seng (Malaysia) Berhad in Masai Johor since 1980s.The palm oil mill successfully achieved 83% COD removal and production of 62.5% methane production (Poh P.E et al, 2009). In the research of Kim.M et al (2002), Mesophilic non-mixed reactor failed earlier than the continuously stirred reactors even though it showed much better performance than the continuously fed reactors prior to reactor failure when organic loading rate added up until reactor failure. (Kim.M et al, 2002).Besides, mechanical mixing is also exhibit a positive results in producing methane gas in the research of Choorit W. et al where a Mesophilic continuous stirred tank reactor is being used. Another inspiring example is research done by Ugoji (1997), the experiments display a result of COD removal in between 93.6 to 97.7% (Poh P.E, 2009). However, the complete mixed system is more sensitive to temperature changes (Kim M.et al, 2002).

In the animal waste research of Karim et al (2005) suggested that mixing improved the performance of digesters treating waste with higher concentration while slurry recirculation showed better results compared to impeller and biogas recirculation mixing mode. Mixing also improved gas production as compared to unmixed digesters. (Poh P.E, 2009) Boe K. et al have adopted intermittent mixing in the research of biogas production from manure rather than vigorous mixing (Boe K. et al, 2009). Research of Kaparaju et al. (2008) is also agreed with the theory of intermittent mixing

advantageous over vigorous mixing. However, mixing during start up is not beneficial as the digester pH will be lowered resulting in performance instability as well as leading to a prolonged start-up period.(Poh P.E, 2009).However there are no systematic research on mixing in treatment of POME.

2.5.3 Organic Loading Rate

Organic Loading rate is a measure of the anaerobic digestion biological conversion capacity. Various studies have proven that Organic Loading Rate (OLR) will reduce COD removal efficiency. However, it give a positive impact on the gas production where increase of with OLR until a stage when methanogens could not work quick enough to convert acetic acid to methane which in return increased the hydrogen partial pressure concomitantly decreased the methane yield. (N.H Abdurahman et al, 2010), (H.Patel et al,2002).

2.5.4 Temperature

The temperature range for anaerobic digestion can be categorised into Psychrophilic (<25˚C), Mesophilic (25 to 40˚C) and thermophilic (<45˚C).Methane production have been documented in various range of temperature, but the most productive in either mesophilic conditions, at 30-35˚C or in the thermophilic range at 50-55˚C.Once the maximum specific growth rate of microbial population rises as the temperature increase. However, maintaining a uniform temperature in the reactor maybe more important, once the anaerobic process is considered very sensitive to abrupt temperature changes, which may cause unbalance between the two largest microbial population and consequently result in process failure (the usual limit is about 2 ˚C per day) (V.S Marcos et al, 2005).

In mesophilic temperature condition methane forming micro-organism range belong to the genera *Mathanobacterium*, *Methanobrevibacter* and *Methanospirillum*, which are hydrogen-using micro-organism and to the genera Methanosarcina and Methnosaeta which are organism that use acetate to form methane. The temperature affects the biological enzymatic reaction rate and influencing substrate diffusion rate.

(V.S Marcos et al, 2005).There are several research successfully produce methane in Mesophilic temperature such as K.M Ostrem et al proved that for the mesophilic digester to operate to the optimum, the temperature have to be maintained at 30-35˚C(K.M. Ostrem et al,2004). Besides, N.H Abdurahman et al conducted their experiment in the Mesophilic temperature range and shown positive result in the production of methane (N.H Abdurahman, 2010). In the research of Zhang Y.J et al has once again shown that Mesophilic temperature range favour the production of methane (Zhang Y.J et al, 2007).

As mentioned before, methane production is productive in thermophilic condition as well. However, for a thermophilic digester the start up period is much longer than mesophilic digester to allow mesophilic sludge to acclimatize with the substrate as well as temperature swift (Poh P.E et al, 2010). There are several attempts to overcome this problem such as by introducing seed sludge for cultivation of mixed culture but it takes a longer time and even more expertise (eg. Molecular biology to identify the microbes in mixed cultured) to get the digester works well. (Poh P.E et al, 2010) Hence, the operational experience in this temperature range not been satisfactory and still many pending question such as whether resulting benefits overcome disadvantage, including additional energy required which increase operational cost, the poor quality supernatant and instability of the process. Besides, the external effects of the temperature on bacterial cell are important. For example, the degree of dissociation of several compound depend strongly on temperature such as specific case of ammonia. The thermodynamic of several reactions are also affected such as the dependence of the hydrogen pressure in anaerobic digesters where fermentation occurs in appropriate manner (V.S Marcos et al, 2005). B.K. Ahring et al (1995) shown that the perturbation of temperature impose the greatest effect on the final product of the such as methane production. Methane production almost ceased after the increase of temperature and had not resumed even 10 days later indicating the importance of a stable temperature of the process.

In the later year, the temperature phase anaerobic digester (TPAD) is developed in with combination of mesophilic and thermophilic condition, the two stage digester show improvement in performance. More than 20 full scaled TPAD systems

have been set up in United State for wastewater treatment (S.Sung, 2003). Despite of the advantages of the system, some researchers would go for other options as there are disadvantages in separating the acidogenic and methanogenic reaction which in turn disrupt the synthrophic relationship between bacteria and methanogens in addition of the complicated control process (Boe K et al, 2009).

As a result, Mesophilic digester would be chosen as the digester in this experiment to produce methane in a steady performance with the minimum constraint.

2.5.5 Hydraulic Retention time

Hydraulic Retention Time (HRT) is the number of days the materials stays in the tank. The Hydraulic Retention Time equals the volume of the tank divided by the daily flow (HRT=V/Q). The hydraulic retention time is important since it establishes the quantity of time available for bacterial growth especially for the growth of hydrolytic acidogenic bacteria and subsequent conversion of the organic material to gas (D.ABurke., 2001) The HRT is closely related to the OLR and substrate concentration, thus a good balance have to be achieve for good digester operation. (N.H Abdurahman, 2010).

2.5.6 Solid Retention time

The Solids Retention Time (SRT) is the average time the activated-sludge solids are in the system. The SRT is an important design and operating parameter for the activated-sludge process and is usually expressed in days. (Lenntech, 2010) Although the calculation of the solids retention time is often improperly stated, it is the quantity of solids maintained in the digester divided by the quantity of solids wasted each day as shown in equation below:

$$SRT = \frac{(V)(Cd)}{(Qw)(Cw)} \qquad [7]$$

V = Digester Volume

Cd = Solid Concentration in the digester

Cw = Solid Concentration in the waste

Qw = Volume wasted each day

In a conventional completely mixed, or plug flow digester, the HRT equals the SRT. However, in a variety of retained biomass reactors the SRT exceeds the HRT. (D.A Burke, 2001) As a result, the retained biomass digesters can be much smaller while achieving the same solids conversion to gas. At a low SRT sufficient time is not available for the bacteria to grow and replace the bacteria lost in the effluent. If the rate of bacterial loss exceeds the rate of bacteria growth, "wash-out" occurs. The SRT at which “wash-out” begins to occur is the "critical SRT". (M. Clara et al, 2004).

2.5.7 Volatile Fatty Acid

Volatile Fatty acid had been use as the process balance indicator. Change in VFA level were shown to be a good parameter, under unstable operation, intermediate such as volatile acid and alcohol accumulates at different rate depending on the substrate and type of perturbation causing instability. The volatile fatty acid accumulation reflects a kinetics uncoupling between acid producers and consumers and is typical for stress situations. (B.K Ahring et al, 1995) Review back to the fermentation stage the acidogenic bacteria convert the less soluble organic compounds to organic acids such as acetic acid, propionic acid and butyric acid which known as volatile fatty acids, alcohol and other intermediates. (Husnul Azan T. et al, 2006) Hence, accumulation of VFA indicates that the further digestion into methanogenic stage is affected. Besides, the imbalance can be reflected by pH, volatile solid reduction and gas composition. However, these are often too slow for the optimal detection of sudden changes. The VFA concentration results in pH drop in turn causing toxicity to the system. pH changes are small in highly buffered systems as often seen in reactor with high ammonia loads even when the process is severely stressed. Hill et al (1987) suggested that acetate concentration higher than 13mM have been suggested to indicate imbalance. Hill (1982) proposed that the propionate/acetate ratio should be used as a process indicator and a stable process should be below 1.4. In the later year on 1988, Hill and Holmberg showed that isobutyrate or isovalerate below 0.06 indicate stable process however different system have their own normal level VFA. (B.K Ahring et

al,1995) Several studies shown that high concentration of VFA have no effect on the biogas process.

2.6 MEMBRANE TECHNOLOGY

Advance treatment process such as membrane separation shows accelerated market growth result by the stringent environmental legislation and water scarcity around the world. Application of membrane technology which commonly employed in waste water treatment can contribute to developing an efficient waste water treatment process to produce high quality effluent and retain the biomass concentration within the reactor at the same time.

In general there are 5 types of membrane filtration process that are conventional filtration, microfiltration, ultra filtration, nanofiltration and reverse osmosis. The selection type of membrane process depends on the particles size that requires separation. Table 2.3 shows the filtration processes with their properties and applications. On the other hand, table 2.4 shows the apparent dimension of some particles.

Table 2.3: Filtration process with their properties and applications

Filtration Process	Pore size	Separation capability	Pressure (bar)	Application examples
NF	1-10nm	Mw200-20,000	5-25	Purification of sugar and salts, water treatment
UF	5-100nm	Mw of 10K-500k	0.5-5	Pharmaceutical industry, waste water treatment
MF	50nm-5µm	Bacteria and colloids	0.5-3	Prefiltration in water treatment, sterile filtratio

(Source: Ramakrishna et al, 2011)

Table 2.4: Apparent Dimensions of various Particles

Particle	Dimension (μm)
Yeast's, Fungi	1-10
Bacteria	0.3-10
Viruses	0.03-0.3
Protein ($10^4 - 10^6$ molwt)	0.002-0.1
Enzymes	0.002-0.005
Antibiotics , Polypeptides	0.0006-0.0012
Sugars	0.0008-0.001
Water	0.0002

Source : (N.H Abdurahman et al, 2011)

Membrane characteristics are relied on the geometry, flow direction, the surface characteristics (normally denoted by pore size) and materials which determining its properties such as the surface charges, hydrophobicity and porosity.

Pore size is the main physical properties determine its application for various feed solution characteristic. Ultra filtration membrane manufacturer frequently characterize their membranes using the "cut off" concept rather than pore size. The nominal molecular cut off weight defined as the lower limit of a solute molecular weight for which rejection is 95%-98%. As the molecular weight reduce the mean pore diameter for most UF is decreased. Hence, MWCO is a rough indication of the membrane ability to remove a given compound despite of other factor. (Norman N.Li, 2008) .

Besides, the materials of the membrane have great influence on performance. Synthetic polymer can be dividing into two classes that is hydrophobic and hydrophilic. Polysulfone ans polyethersulfone is hydrophilic and use for UF process. Hydrophobic membranes such as polytetraflouroethylene, polyvinylidene fluoride, polyethene are

commonly used for MF. The fouling potential for the hydrophobic membrane is highly due to the high binding affinity of the proteins and humic substances.

Besides, the surface charges implies different fouling tendency. Generally, membrane materials carry a negative charge because natural organic matter is negative charge at neutral pH due to phenolic and carboxylic functional groups. A negative charge of membrane therefore prevent deposition of foulant by charge repel.

2.6.1 Hollow fiber membrane

The hollow fiber configuration is the most common configuration for MF and UF membrane. The hollow fibers are 05-1.0mm (less than 5mm) in diameter and several thousand of hollow fibers are packed in a module. The most important merits is that no extensive pretreatment needed as the membrane can be backwashed. The excellent mass-transfer properties conferred by the hollow fibre configuration soon led to numerous commercial applications in various field. The hollow fibre membranes have two major advantages over flat sheet membranes. One is that hollow fibres have much larger ratio of membrane area to unit volume, and hence higher productivity per unit volume of membrane module. Another is that they are self-supporting which can be back-flashed for liquid separation. (Cheresources, 2010).

Hollow fibre membrane can be operated in two different flow modes which are shell side feed and bore side feed. The bore side feed has its advantages over shell side feed including minimal pressure drop inside the fibers. The diameter is usually larger than those of the fine fibres used in shell side feed system, it is important to ensure all fibres have identical fibres diameters and permeance to ensure module performance. Feed pressure is usually limited below 150 psig.

UF system are operate in two possible filtration modes which is cross flow configuration in which the feed water is pumped tangential to the membrane while the water that does not permeate is recirculation as concentrate and combine with feed. In dead end or direct filtration all the feed water passes through the membrane. Therefore recovery is 100% and small fraction is used periodically for back wash. Although dead

end filtration require lower energy but the cross flow filtration suit the system better where recirculation of retentate is encouraging.

2.7 MEMBRANE FOULING

A major obstacle for the application of Hollow fibre membrane in MAS is the rapid decline of the permeation flux as a result of membrane fouling (Cheresources, 2010). Fouling refers to blockage of membranes pores during filtration caused by the combination of sieving and adsorption particulates onto membrane surface and within the membrane pore. This blockage of the pores causes a flux decline over time when all other parameter kept constant. The predominant fouling mechanisms observed with ultrafiltration and micro filtration membranes are classified into three categories: the build-up of a cake layer on the membrane surface, blocking of membrane pores, and adsorption of fouling material on the membrane surface or in the pore walls (M.O.Laminen, 2004). To establish strategies for fouling control, understanding of the fouling mechanisms is indispensable. Sludge characteristics are significant parameters that affect membrane fouling in MAS.

Fouling can be broadly classified into backwashable and irreversible. Backwash able can be removed either by backwashing or chemical cleaning while the irreversible type neither of the method can recover the original flux.

Fouling can also be classified according to type of the fouling materials. Four categories of the membrane fouling are generally reognised. They are:

a) Inorganic fouling
b) Particle /colloidal fouling
c) Microbial fouling
d) Organic fouling

In organic fouling is caused by the deposition of inorganic materials such as metal hydroxides. Precipitates form when the concentration of such materials over its

saturation concentration. This type of fouling usually appears to be a problem for reverse osmosis and NF.

Particulates/ Colloidal fouling are due to algae, bacteria and some natural organic matter fall into the size range of particles and colloids. However they are different from other inert particles and colloids such as silt and clays. In most cases colloid and particles do not foul the membrane because it is largely reversible by hydraulic cleaning. Cross flow filtration can be used to control colloid fouling. (Norman N.Li, 2008)

Microbial Fouling is a result f formation of a biofilms on the membrane surfaces. Such films grow and release biopolymers as a result of microbial activity. Bacteria attached on the membrane and started to multiply and produce extracellular polymeric substances to form a viscous, slimy and hydrated gel. Severity of microbial fouling is greatly related to the characteristic of the feed.

Organic Fouling is an issue with lot of conflicting opinion, some researcher agree that proteins, amino acid sugars, polysaccharides and polyoxyaromatics as strong foulant while some partially agree showed organic colloidal fraction caused the most significant fouling. Some conclude that humic acid later fulvic acid. There is no definite answer, so further research on this subject is required. (Norman N.Li, 2008)

Membrane Fouling are sometimes related to the sludge settling problems where Sludge filamentous bulking and sludge deflocculating are the most common problems result in a deterioration of effluent quality. (F. Meng et al, 2007)

Deflocculation refers to a dysfunction of the activated sludge process characterized by the formation of a very small sludge floc, or the absence of floc formation. Deflocculation can be the result of operating conditions and environmental stresses such as shift in temperature, toxic compounds, metals, dissolved oxygen concentration, pH, substrate loading, and nutrient characteristics. (F. Meng et al., 2007)

Sludge bulking is a term used to describe the excessive growth of filamentous bacteria in activated sludge system, it is a condition in which sludge settling rates decrease and the thickening characteristics of settled sludge are poor. The existence of a small quantity of filamentous bacteria in sludge suspension can benefit the formation of strong flocs, which can be defined as normal sludge flocs. (F.Meng et al, 2007)

2.8 Methods reduce membrane fouling

Fouling rate can be slackened through different strategies as chemical cleaning or turbulent aeration.

2.8.1 Hydraulic Cleaning technique

One of the most helpful methods for fouling remediation is certainly represented by the sub-critical flux operation. (G. Andreottola et al, 2006)

From the Finding of Alves and Pinho and Schafer et al. on this phenomenon, it was have been proven that the cross flow velocity directly affecting the fouling rate where at higher cross flow velocity, the high shear tangential exerted to the membrane surface allowed the sweeping away of the deposited particles; therefore, the fouling layer on the surface of the membrane reduced. (A.L Ahmad et al, 2004) As a consequence, higher organic matter could pass through the membrane and percentage rejection become lower. (H.Mourad and M.Martine, 2002) also observed this in their study on the relationship between permeate flux and cross flow velocity. They tested for the highest cross flow velocity, and 88% of the mass carried by convection to the membrane surface was swept away by the tangential flow. The high shear tangential to the membrane surface swept deposited particles away (A.L Ahmad et al, 2004).

2.8.2 Backwashing/Chemical washing

For the conventional method, the UF were treated by chemical cleaning. The membranes were first circulated with clean water to flush out POME remaining in membranes, and then circulated with chemical solution mixed by 1% (*W*/*W*) NaOH and 0.6% (*W*/*W*) NaClO for 25 min. Finally, the membranes were rinsed again with clean water until a neutral pH was achieved.

Backwashing experiences degradation of flux between backwashes and requires a break in operation to be performed and problems incurred when chemical costs, waste disposal, and significant capital investments for equipment are needed.

2.8.3 Ultrasonic technology

Another remedy that is proven to be effective is by ultrasonic cleaning. Ultrasound is a sound wave travelling through a medium at a frequency above 18 kHz. Removal of particles on fouling surface can be accomplished with the right frequency, power intensities and duration. In comparison with other current membrane cleaning technologies include hydraulic, chemical, and mechanical methods, ultrasonic appear to be a better choice as common hydraulic cleaning technique.

2.8.3.1 Mechanism

In a liquid medium, ultrasound creates oscillating regions of high and low pressure. Cavitation bubbles are formed when the pressure amplitude exceeds the tensile strength of liquid during the rarefaction of sound waves. The cavitation bubble collapses during the compression cycle of sound waves. Localized hotspots are formed in aqueous solution reaching average bubble temperature of 4200K, peak core temperatures of 1700K and pressure of 500 atm at the bubble core. Acoustic streaming, micro streaming, micro streamers, micro jets and shock waves are generated as a result of ultrasound (D. Chen et al, 2006).

The high temperature and pressure resulting from cavitation collapse dissociate water into hydrogen atom and hydrogen radicals. More importantly with respect to membrane cleaning, cavitation collapse also produces a number of phenomena that results in high velocity fluid movement (M.O Laminen et al, 2004).

Several different mechanisms may lead to particles release from a particles-fouled surface as a result of ultrasound includes acoustic streaming. Acoustic streaming defined as the absorption of acoustic energy resulting in fluid flow. Acoustic streaming does not require the collapse of cavitation bubbles. This mechanism causes bulk water movement toward and away from the membrane cake layer that may scour the particle away. It is found to be an aid to cleaning the membranes but is likely not an important detachment mechanism. Acoustic streaming may remove detached particles from the vicinity of the membrane surface (M.O Laminen et al, 2004).

Micro streaming is another mechanism which is time independent circulation of fluid occurring in the vicinity of bubbles set to motion by oscillating sound pressure. Oscillation of bubble cause rapid fluctuation in the magnitude and direction of the fluid movement and as a result significant shear forces occur. Micro streaming result in a dynamic velocity profile that will exert drag forces on the particles leading to removal. Micro streaming works in conjunction with other mechanism such as micro streamer to clean membrane surface (M.O Laminen et al, 2004).

Micro streamer is a mechanism where cavitation bubble form at nucleation sites within the liquid and are subsequently translated to a mutual location are called micro streamer. The bubbles travel in ribbon like structure along tortuous paths at velocities approximately an order of magnitude faster than the average velocity of the fluid. The antinodes located on the fouled surface may result in bubbles scouring away particles. It is likely to be the major mechanism for detaching particles from the membrane (M.O Laminen et al, 2004).

Micro jets are formed when a cavitation bubble collapse in the presence of an asymmetry. During collapse, the bubble wall accelerates more than one side opposite to a solid surface, resulting in the formation of strong jet of water estimated velocity of

100-200m/s. Micro jet although present, appear in isolated site and do not greatly removed particles from membrane surface (M.O Laminen et al, 2004).

2.8.3.2 Factor influencing effectiveness

Ultrasound aids cleaning may be affected by a number of factors, such as orientation and position of the ultrasonic field, ultrasonic power intensity and frequency, membrane material, membrane housing, operating pressure, and fouling material.

2.8.3.2.1 Power intensity and frequency

M. cai et al (2010) found that the performances of permeate flux were significantly enhanced by Ultrasonic frequencies of low frequency which is 28kHz and 45kHz but no obvious enhancement was observed for the Ultrasonic frequency at 100kHz.The resistances at the frequency of 28 and 45 kHz were decrease significantly resulting in an increase of permeate flux while at frequency of 100kHz the resistance were similar to that without Ultrasonic. The solution concentration may be decrease by acoustic stream and bubble cavitation effect (M. Cai et al,2010).

The same trend of result reported by M.O Lamminen et al (2004), increasing cleaned flux ratio with decreasing of frequency was found. This can be explained although there are more collapse but the collapse tend to be less violently producing lower temperature and pressure this suggested that violence of collapse at lower frequencies is more important than increased number of weaker collapse.

In the research of M.O Lamminen et al (2004), suggest that increasing power intensity result in greater cleaning of the membranes. Increase of power intensity to the system increases the number of cavitation bubbles formed and increase the size of the cavitating zone due to higher pressure amplitude of the sound wave with increased power intensity. The hydrodynamic turbulence induced increased with power intensity resulting from the implosion of bubbles that are collapse and increase absorption of acoustic energy by the medium. Complete recovery occurred on the increasing shorter time scale as frequency decreased or power intensity increased.

2.8.3.2.2 Particle concentration, hydrophobicity and size

Increasing the particle concentration may result in increase of attenuation of acoustic energy, enhanced nucleation of cavitation bubbles and increased viscosity of the solution. The effect of particle concentration is that particles induce additional cavitation bubbles within the solution in the zone of the cavitation close to ultrasonic probe. Particles act as nuclei within the liquid from which bubble can grow. Cavitation bubbles attenuate sound waves due to both scattering and absorption and thus impede the propagation of the sound waves, especially at its resonance size. The scattering and absorption results in a decrease of sound wave intensity compared to that in the absence of bubbles. Thus, the sound wave intensity decrease more rapidly with distance from source at high particle concentration compare to those with low particle concentration. The ability of the ultrasound to remove particles from the membrane surface as measure by lift force of particles is reduced (D.Chen et al,2006).

The sound wave intensity in the presence of hydrophilic silica particles was significantly higher than that for hydrophobic silica particles. This trend verify that sound waves intensity decreased more rapidly due to bubble shielding caused by hydrophobic particles inducing more cavitation bubbles near source than hydrophilic particles. The turbulence generated is less effective as the distance between the cavitation bubbles and the membrane surface is larger (D.Chen et al,2006).

On the other hand, the particle size influence the efficiency in the way of larger particles would be more effectively removed by ultrasonic turbulence due to greater drag and lift forces. However, particle size did not significantly affect sound wave intensity. (D.Chen et al, 2006).

2.8.3.2.3 Distance

When the membrane is outside the cavitations' region, the main mechanism of microjets, microstreaming, shock waves, microstreamers and acoustic streaming may directly contributed to the cleaning action of the ultrasound. However when the membrane is outside the cavitation region, the main mechanism of ultrasonic cleaning is

acoustic streaming and ultrasonically generated turbulence. A major difference between the fluid movement within or outside the cavitation region is the energy density, which is extremely high within the cavitation region

At closer distance between the ultrasonic probe and membrane surface, more ultrasonic energy and ultrasonically generated turbulence is focused on the membrane surface and therefore better permeate recovery was obtained. (D.Chen et al, 2006)..

2.8.3.2.4 Filtration Pressure

Permeate flux improvement decrease with increasing of filtration pressure. Higher filtration pressure cause higher drags force on the particle at the membrane surface. The permeate drag force is proportional to permeate velocity through the membrane. Thus, increase pressure because stronger permeation drags force lead to greater membrane fouling.

Another theory behind the filtration pressure is the increase of the compressive forces driving cavitation bubbles formed. The increased compressive force results in the increase of the velocity of the bubble wall during implosion. Consistent with an expected increase in violence of cavitation collapse. In addition, fewer bubbles present in solution at higher pressure may limit bubble shielding, in which bubbles attenuate sound waves due to both scattering and absorption also improved cleaning. Thus, stronger acoustic stream and ultrasonic generated turbulence form in return created higher velocity gradient produced more shearing stress cleaning membrane surface.

However, the increase of drag force is more significant compare to the turbulence increase (D. Chen et al, 2006).

2.8.3.2.5 Continuos/ pulse Operation

The permeate flux improvement decrease as the pulse interval increased. The loss of permeate flux improvement with increasing pulse interval was likely due to periodic losses in Ultrasonicated generated turbulence and subsequent deposition of

particles. However, pulsing affect bubble dynamics. During sonication, some bubbles grow by rectified diffusion to size greater than the resonance size are ineffective at producing cavitation effects and cause scattering and absorption of ultrasonic waves. Therefore, in continuous ultrasound, some bubbles are ineffective and wasted. (D.Chen et al, 2006) Pulse ultrasound did not result in damage of membrane but slightly less effective than continuous ultrasound. (M.O Lamminen et al, 2006).

2.8.3.3 Membrane integrity

Membrane damage was found when membrane located just within the ultrasonic cavitation region. Literature suggests microjets and .or shock waves are likely responsible for the surface damage of membrane. The velocity of microjets can be greater than 100m/s and the pressure amplitude of shock wave can be as high as 1GPa. (D.Chen et al, 2006). (M.O Laminen et al,2006) reported at higher applied power susceptible to cause membrane damage when operate at continuos mode.

CHAPTER 3

MATERIALS AND METHODS

3.1 INTRODUCTION

This chapter will discuss about material and methodology that going to be use. After analyse few methods and have high desire to achieve concept waste to energy successfully, this paper will be implement ultrasonic membrane anaerobic system (UMAS) to produce methane gas from palm oil mill effluent (POME). UMAS is integrated with the anaerobic digestion technique, membrane technology and ultrasonic technology at the same time. The raw materials (POME) will go through the fermentation process in where complex organic matter will be degrading within certain time and methane gas is produce. The further process is enhance by a membrane UF module which allow the removal suspended solid by ultra filtration to give a good quality effluent. Retain of biomass by the membrane will be recycle back to reactor while the permeate will be discharge. This paper also will implement the ultrasonic system that equipped with the membrane UF module housing to prevent the fouling of membrane. In this paper, the microorganism is leave in the bioreactor for the first few days to acclimatize with the bioreactor environment until the system is establish where the microorganism will self generate and retain the sufficient amount of microbial in the system. Then, this research will be conduct in mesophilic condition, the performance of this condition can be investigate by evaluate pH, VSS, TSS, COD and BOD. The process flow diagram of this research (refer **Figure 3.4**), methodology, and research instruments will be discus more detail in this chapter.

3.2 MATERIAL

3.2.1 Wastewater Preparation

POME was source from FELDA Lepar Hilir 2, Kuantan, Malaysia In order to prevent the wastewater from undergoing biodegradation due to microbial at action, it was preserved at a temperature lower than 4°C and higher than the freezing point and stored in PVC containers. Before the experiment start, the raw POME was sieved to remove unwanted suspended materials this is because to prevent the clogging and pump damage. Figure 3.0 and Figure 3.1 shows the anaerobic pond of raw POME.

Figure 3.0: Anaerobic pond

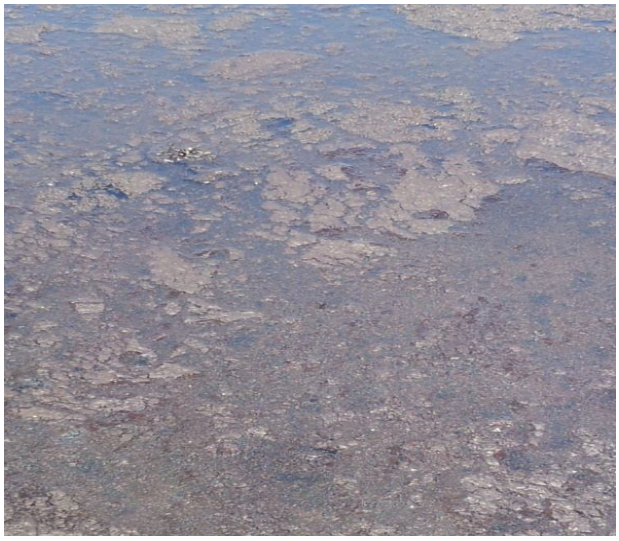

Figure 3.1: Anaerobic pond

3.2.2 Anaerobic reactor

The reactor is made up of clear PVC and its specification which is have an inner of 15 cm and a total height of 100 cm. The volume of the reactor is 100 liter, and connected with centrifugal pump. To prevent the wastewater sample in the reactor from any direct of sunlight, the reactor was covered with aluminum foil as shown in Figure 3.2

Figure 3.2: Anaerobic reactor

3.2.3 The pump

A 1 phase 0.63 kW, 4.6 Amps, 220/250 voltage pump model no 5130 1 R Motor type 353 was used to pump the digester content into cross ultrafiltration (CUF) and recycle the retentate back into the reactor.

3.2.4 UF Membrane

The UF membrane module has a molecular cut off weight of 200,000 and a tube diameter of 1.25cm and an average pore size of 0.1µm.The length of the tube is 30cm.The total effective area of the four membrane 0.048m^2 The ultrasonic frequency is 25 kHz, with 6 units of permanent transducers and bonded to the two sided of the

tank chamber and connected to one unit of 250 watts 25kHz Crest's Genesis Generator. The maximum operating pressure on membrane was 55 bars at 70˚C and it can be used in pH range from 2 to 12.The reactor was made up of clear PVC with a total height of 100cm.The operating pressure on the membrane was 1.5-2 bars by manipulating the gate valve of retentate line after the cross flow membrane ultra filtration unit.

3.2.5 Ultrasonic Technology

The ultrasonic frequency was 25 kHz, with 6 units of permanent transducers and bonded to the two sided of the tank chamber and connected to one unit of 250 watts 25kHz Crest's Genesis Generator. Transducer is a device that can convert electrical current to sound wave or vice versa with reversible transducer can convert in both direction, and is fabricated from material that poses piezoelectric or magnetostructive properties. The ultrasonic equipment is shown in Figure 3.3

Figure 3.3: Ultrasonic Equipment

3.3 METHODOLOGY

3.3.1 Operating procedure

In this study, a 50L volume laboratory scale Ultrasonic assisted Membrane Anaerobic System (UMAS) was used to treat raw POME. The experimental set-up is shown in **Figure 3.4.** Process units that are involved in the systems are anaerobic bioreactor, centrifugal pump, cross flow membrane module and ultrasonic device attached to membrane module.

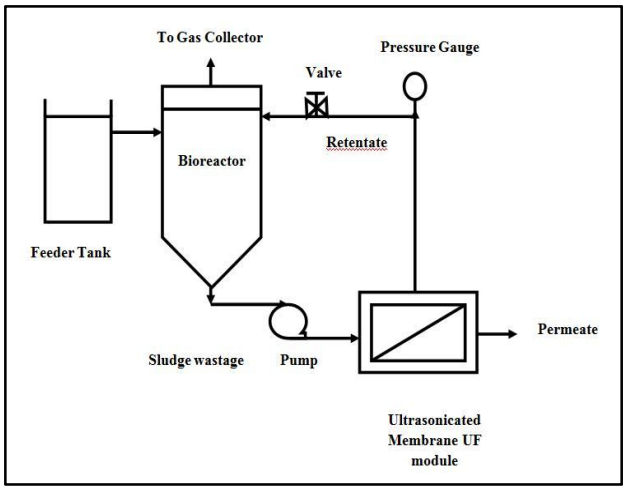

Figure 3.4: Experimental set-up

The sieved collected raw POME from site is left in the tank for 5 days for acclimation process which is the micro-organism acclimates with the reactor. Some of the POME from feeder tank was collected and analyse for the parameters such as pH, COD, BOD, TSS and VSS to obtain initial characteristic of the POME. In this experiment, the pH was controlled in the range of 6.8 to 7.4 while the optimum pressure was set to be 1.5 to 2.0 bar and the temperature was maintained within 25˚C to 37˚C. The value of pH is the most important process control parameter to avoid the accumulation of excess volatile acid. After acclimation period, the micro-organism community is stable then some of the POME in the feeder tank is collected to test and the reactor is left to operate (pump is switch on) for 5 hours, in this period the POME from the digester was pressurized into the UF membrane. After 5 hours, the permeate (filtrate) from the reactor were collected and tested for various parameters. The POME

in the digester that has gone through biological degradation was also collected for the COD, BOD, TSS and VSS test by using standard method and the percentage of removal for each parameter. The gas that produced was collected the designated syringe. The experiment was conducted for every of the subsequent days.

3.3.2 Methane gas measurement

For the methane gas component measurement a J-tube gas analyzer as shown in **Figure 3.5** employed. The assumption made on this method was the biogas produced consists of two gases which were CO_2 and CH_4.The potassium hydroxide(KOH)was first drawn into a syringe to absorb CO_2 where the remaining gas is CH_4.The device consist of a glass tube connected by flexible hose to a syringe. Initially, the syringe filled with 0.5M KOH solution was inserted into the gas line, where a column of biogas was drawn until certain mark and the end of the glass tube was immersed into water to prevent gas escape. By manipulating many times, the KOH absorb the carbon dioxide as evidence from reduction in the length of the biogas column and then measured the biogas column again.

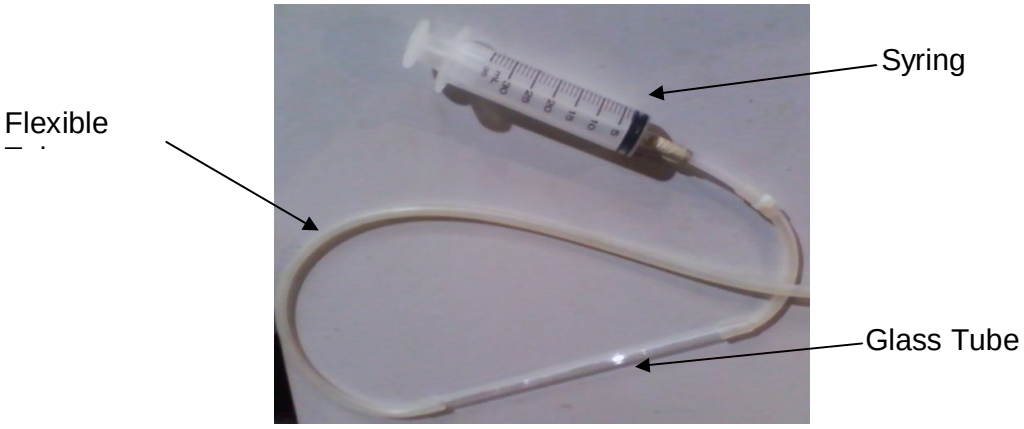

Figure 3.5: J-Tube gas analyser

The percentage of methane is calculated by using this formula:

$$\frac{\text{Final length of gas column}}{\text{Initial length of gas column}} \text{ x } 100\%$$

3.4 BIOREACTOR PARAMETERS

Main parameters monitored in this study and calculation methods are presented in **Table 3.1** where $COD_{out,an}$ is the COD concentration of anaerobically treated POME, COD_{in} is the influent COD concentration(mg/L), COD_{out} is the treated effluent COD concentration(mg/L) BOD_{in} is the influent BOD concentration (mg/L), BOD_{out} is the treated effluent BOD concentration (mg/L), TSS_{in} is the influent TSS concentration (mg/L), TSS_{out} is the treated effluent TSS concentration (mg/L), VSS_{in} is the influent VSS concentration (mg/L), VSS_{out} is the treated effluent VSS concentration (mg/L), Q_{CH4} is the methane production rate (L/day).

Table 3.1 : Main parameter monitored in the present study

No	Symbol	Unit	Description	Equation
1	%COD	%	Overall COD removal efficiency	$\frac{(COD_{in} - COD_{out})x100}{COD_{in}}$
2	%BOD	%	Overall BOD removal efficiency	$\frac{(BOD_{in} - BOD_{out})x100}{BOD_{in}}$
3	%VSS	%	Overall VSS removal efficiency	$\frac{(VSS_{in} - VSS_{out})x100}{VSS_{in}}$
4	%TSS	%	Overall TSS removal efficiency	$\frac{(TSS_{in} - TSS_{out})x100}{TSS_{in}}$
5	rCH_4	$I.CH_4/L$ day	Volumetric methane production rate	$\frac{Q_{CH4}}{V_{an}}$

3.5 ANALYTICAL METHOD

For anaerobic process, several monitoring parameters were evaluated during the entire operation including COD, BOD, TSS and VSS concentrations of the effluent, as well as pH and temperature.

3.5.1 Chemical Oxygen Demand (COD)

Chemical oxygen demand (COD) is used as a measure of oxygen requirement of a sample that is susceptible to oxidation by strong chemical oxidant. This chemical oxygen demand (COD) was measured by using Spectrophotometer HACH DR/2400 @ DR/2800(Figure 3.6) and COD Digester Reactor (Figure 3.7). A sample of 2 ml was placed in a vial with the oxidizing acid solution that was then held at 150°C for 2 h in COD Digestion Reactor. After cooling, the sample was then analysed in the HACH spectrophotometer. The colour of the samples varied from orange to dark green, indicating COD strength in the range of 0-15,000 mg/L.

Figure 3.6: Spectrophotometer HACH DR/2400

Figure 3.7: COD Digester Reactor

3.5.2 Biological Oxygen Demand (BOD)

Biochemical oxygen demand (BOD) test measures the ability of naturally occurring microorganisms to digest organic matter in 5 days incubation at 20°C by analyzing the depletion of oxygen as in Figure 3.8. The biological oxygen demand (BOD) of wastewater was measured using a Dissolved Oxygen Meter (Figure 3.9). 10mL sample was added into a 500 mL beaker and dilution water is added up to 300 mL into the same beaker. The pH value is adjusted around 6.5 to 7.5 by adding acid or alkali. The dissolved oxygen in the sample was measured prior to putting it into incubator for five days.

Figure 3.8: Wastewater is left 5 days in incubation at 20°C

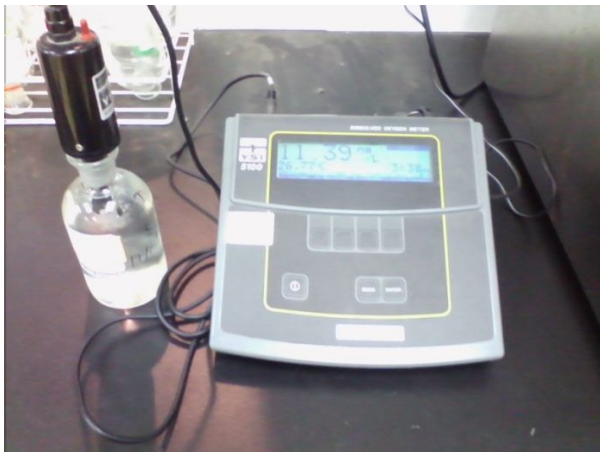

Figure 3.9: Dissolved Oxygen Meter

3.5.3 Total suspended solid

The total suspended solid (TSS) was measured to identify the amount of inorganic or organic particles or immiscible liquid that suspended in the sample. Firstly, the glass fibre filter disk was dried in the oven at 103˚C to 105˚C for 1 hour, and then it would be put in desiccators and weighed. The filtering apparatus will be assembled as shown in Figure 3.10 and filtration process will be started by begin suction. The filter was wetted with a small volume of distilled water to seat it. 50ml of the sample pipette onto the centre of filter disk in a Buchner flask by using gentle suction. Filter was washed by 3 successive 10ml volumes of distilled water and 3 min suction is continued to completion. The filter was transfered to aluminum weighing dish/crucible dish as a support. The filter was dried at least one hour at 103˚C to 105˚C for 1 hour in an oven, cool in desiccators to balance temperature and weigh. The cycle of drying, cooling, desiccating, and weighing until a constant weight is obtained.

Figure 3.10: Filtering apparatus

3.5.4 Volatile suspended solid

In order to measure the volatile suspended solid, the residues from end samples from TSS test was continued to dried and firing in furnace with a temperature of 550˚C for 30 minutes. The organic fraction or volatile substances was converted to carbon dioxide, water, vapor and other gasses and escaped. The remaining materials will represent the inorganic or fixed residue. Figure 3.11 show a muffle furnace.

Figure 3.11: Muffle Furnace

3.6 MEMBRANE CLEANING

To keep the sustainability of membrane, the membrane cleaning should be taken. The conventional method, the UF was treated by chemical cleaning. The membranes were first circulated with clean water to flush out POME remaining in membranes, and then circulated with chemical solution mixed by 1% (*W*/*W*) NaOH and 0.6% (*W*/*W*) NaClO for 25 min. Finally, the membranes were rinsed again with clean water until a neutral pH was achieved. The efficiency of the cleaning procedure was checked by comparing the clean water filtration flux to the initial flux. The second method used was to soak the membrane in 0.1 M NaOH for a day (24hours) rigorous brushing with water. In both methods membrane was taken out from membrane housing. However, this method has its limitation where a plant has to be shut down for the cleaning process or 2 membranes are installed and being used alternatively but this would incur more cost in a long run. Besides, constantly back flushing will degrade the membrane and hence shorten its life span. In order to overcome this problem, an ultrasonic is equipped on membrane housing where the ultrasonic send sound wave constantly to the membrane to detach the foulant and we use distilled water to backwash the membrane.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 INTRODUCTION

This chapter presents results obtained from experiment conducted according to the methodology in chapter 3. In order to achieve the objective of enhancing methane gas emission and treatability of POME the operational parameters such as pH and temperature was controlled strictly. The UMAS efficiency was evaluated for the parameters COD, BOD, VSS, TSS and Methane gas composition. These parameters were measured every day before and after membrane treatment for 5 hours. The raw data Table and the details calculations are enclosed in the appendix.

4.2 ANAEROBIC SYSTEM

4.2.1 Chemical Oxygen Demand (COD) and Biochemical Oxygen Demand (BOD) removal efficiency.

The organic matter can be classified into soluble fraction and a particulate fraction. Hence, COD and BOD served as the variables representing the soluble fraction of substrate concentration. **Figure** 4.1 and 4.3 illustrates COD and BOD profile respectively. In the beginning of the experiment, the COD increased from 25650mg/L to 32400mg/L while BOD increased from 878mg/L to 1240.5 mg/L on the 3rd day indicating that assimilation of complex organic compound into simple soluble compound. Somehow, COD obtained greater values compared to BOD because COD measure biodegradable and non biodegradable organic compound while the later did

not. The fluctuation occurs where some of the BOD and COD values such as value in 2nd day in the system were even greater than influent are due to the recycle of solid in the system making high particulate organic matter represented by the microbial population. This conclusion was supported by literature of Marcos et al (2005).

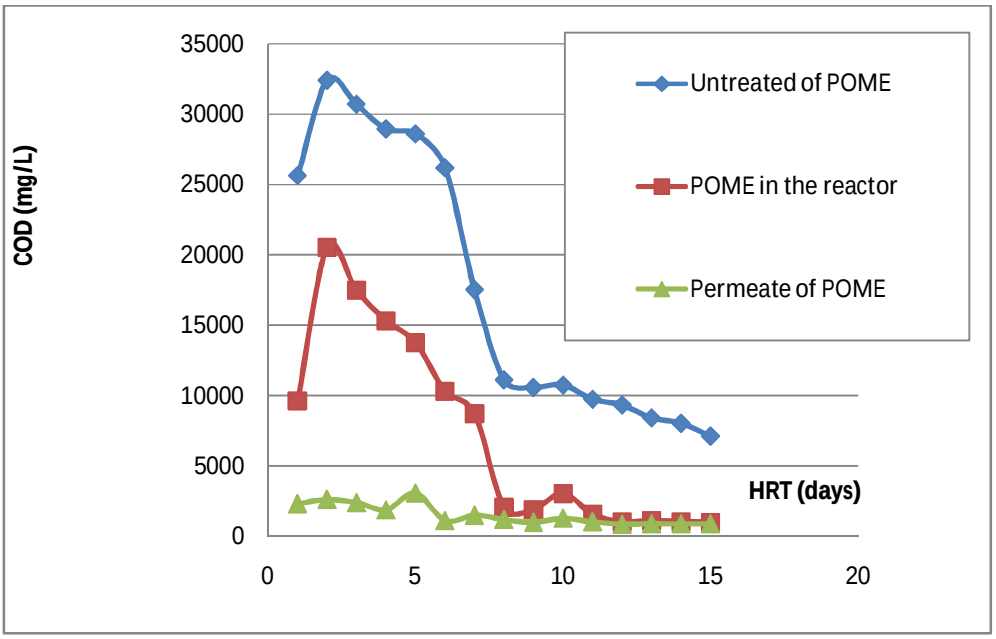

Figure 4.1: COD V.S HRT

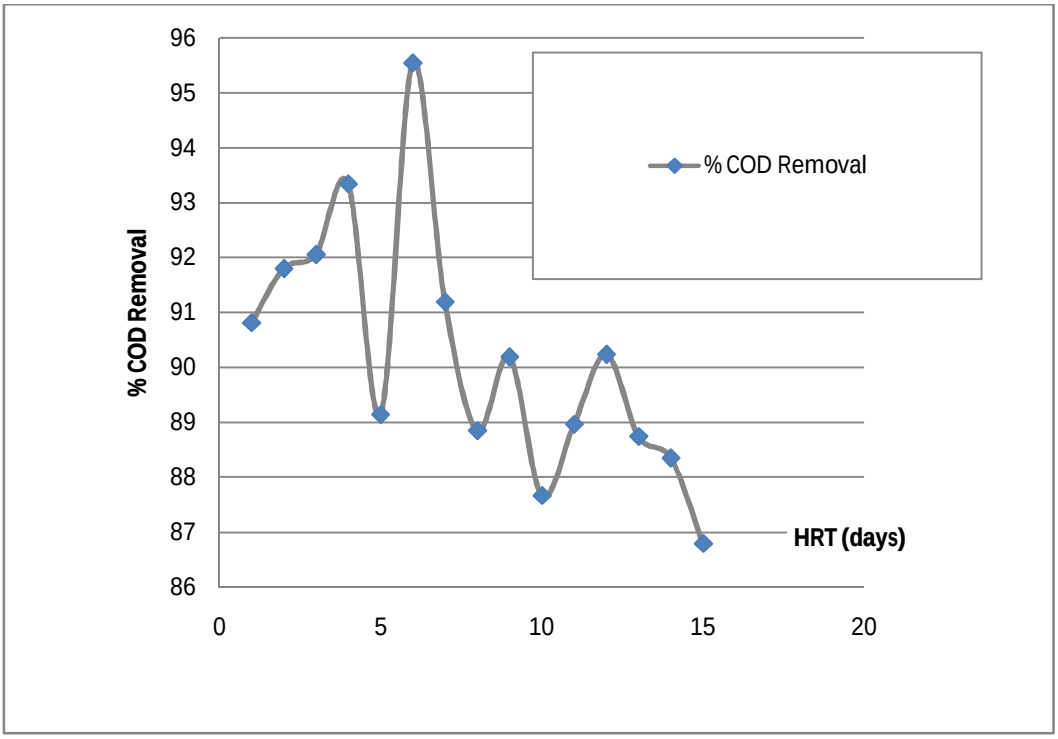

Figure 4.2: COD Removal Efficiency

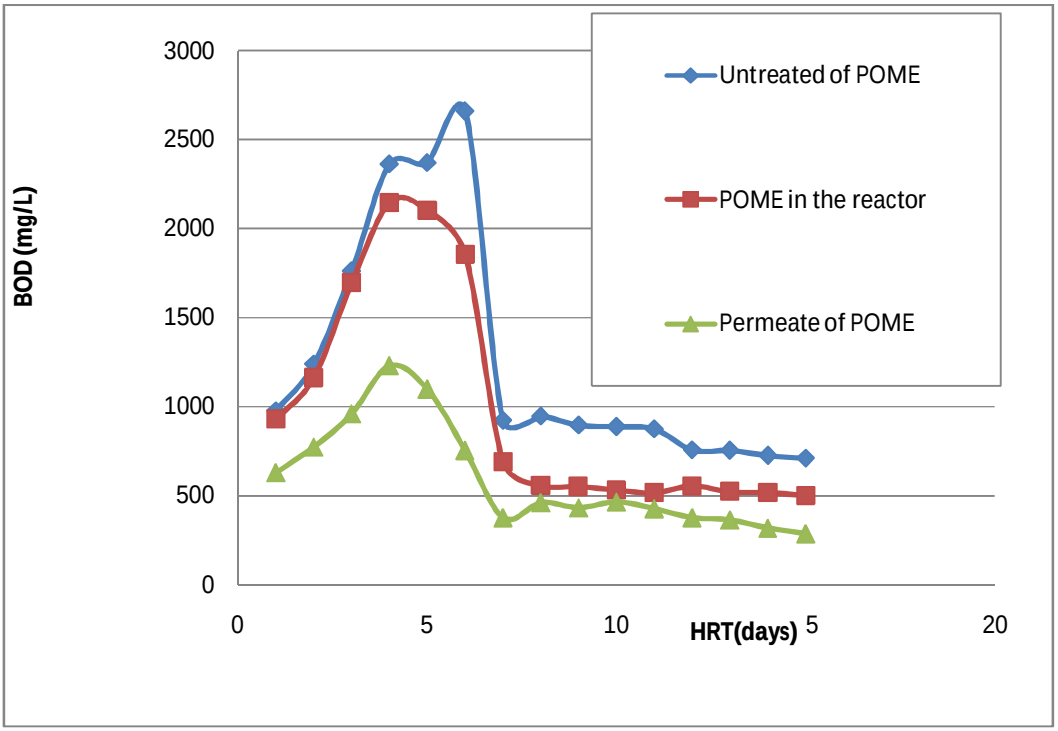

Figure 4.3: BOD VS HRT

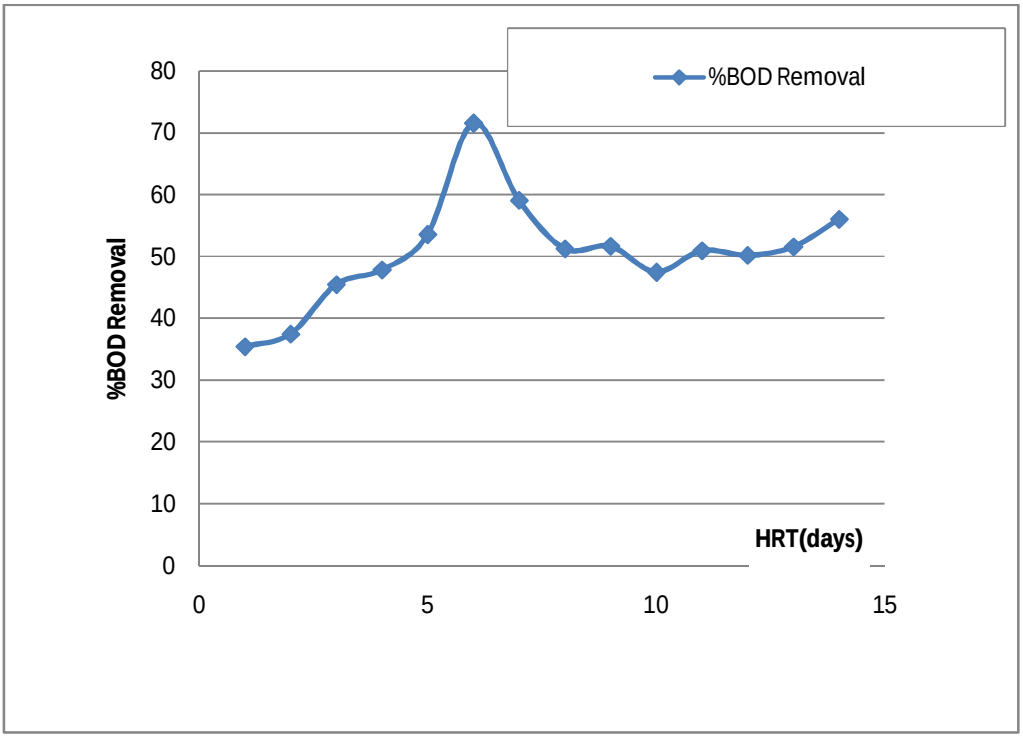

Figure 4.4: BOD Removal Efficiency

The results has shown the COD removal rate of the reactor increased from 90.82% to 95% which was highest on the 6th day as shown in **Figure 4.2**. On the other hand, BOD parameter recorded the same trend by increasing from 35.43% removal rate on the first day to the peak of 71.58% removal rate. This was due to the active utilization of the substrate by the microbial population for their growth as well as for methane generation. It can be observe that the VSS which serve as microbial population indictor develop to maximum on the 3rd day, the relationship between SS concentration and COD removal rate could be found whereby increase in SS in the system could improve COD removal. It is consistent with finding of Poh et al (2010) which the COD removal efficiency increases when the MLSS increase.

Besides, methane composition of 93.5% of biogas achieved on the the day. Hence, there was also a strong linear correlation between COD and methane gas production observed. The same observation reported by Basri (2010) where biogas and methane production increase by COD removal.

On the 9th day, the low digestion rate may associate with the reduction of microbial population as the growth restricted by the exhaustion of nutrient or substrate in the reactor. At this stage the total removal of COD achieved approximately 90% compared to the Influent feed at the beginning as there was no additional substrate added in as organic loading rate was not a parameter for this study, therefore substrate may served as the limiting factors. The similar experience was found in the research of (Poh et al, 2010), suggested that additional substrate must be added into system when substrate reduction up to 80% to prevent substrate to becoming limiting factor.

Ultra filtration membrane has play it part in increase the COD efficiency whereby the organic of molecular weight higher than 200kDa are susceptible to being absorbed into membrane hole (A.P.V Rajaletchumy,2010), leading to the high COD removal rate up to 95% and 73.2% of BOD removal based on **Figure 4.2** and **Figure 4.4** respectively.

As compared to the highest COD removal rate of 70% in the previous research done by (A.P.V Rajaletchumy, 2010) UMAS able to reach removal rate of 92.4% in the

same period of time. The performance difference may be contributed by the Ultrasonic equipped with the membrane which manage to emit ultrasonic irradiation in return creating turbulence flow which would triggered the removal of fouling particles from membrane surface in return retained the biomass in the system resulting in higher SRT for microbial degradation.

4.2.2 Total Suspended Solid (TSS) and Volatile Suspended Solid (VSS) removal Efficiency

Figures 4.5 and **4.6** had shown the Total suspended solid and volatile suspended solid profile of the bioreactor throughout the experiment respectively.

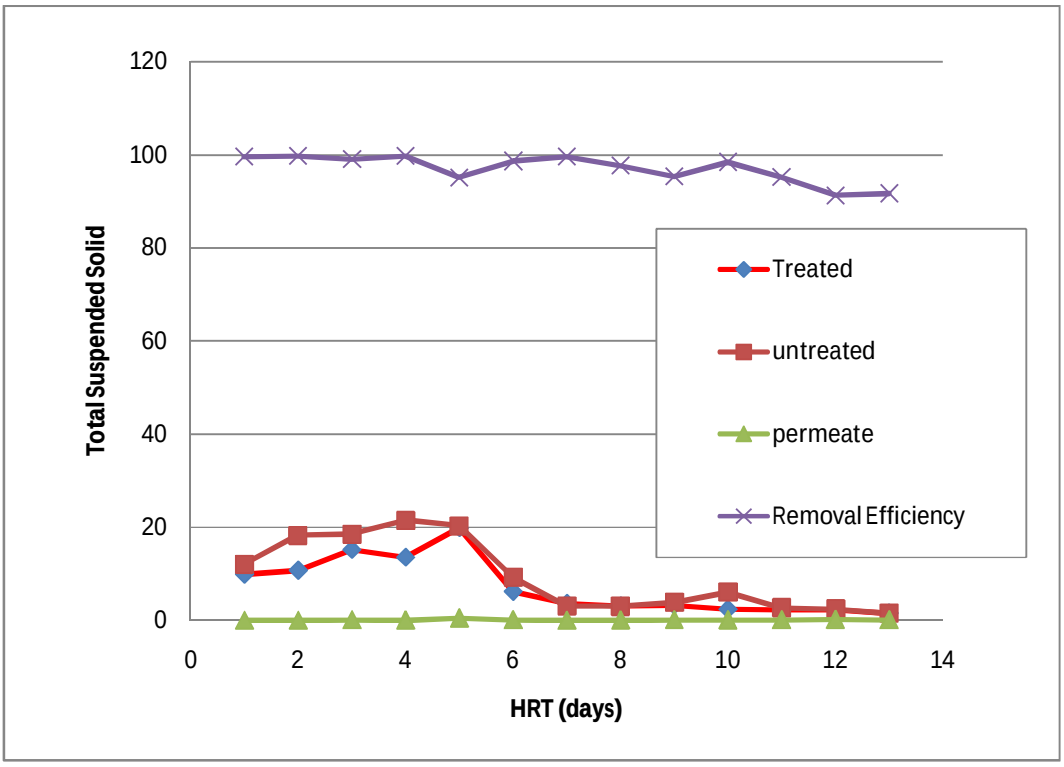

Figure 4.5 : Total Suspended Solid Profile and Removal Efficiency

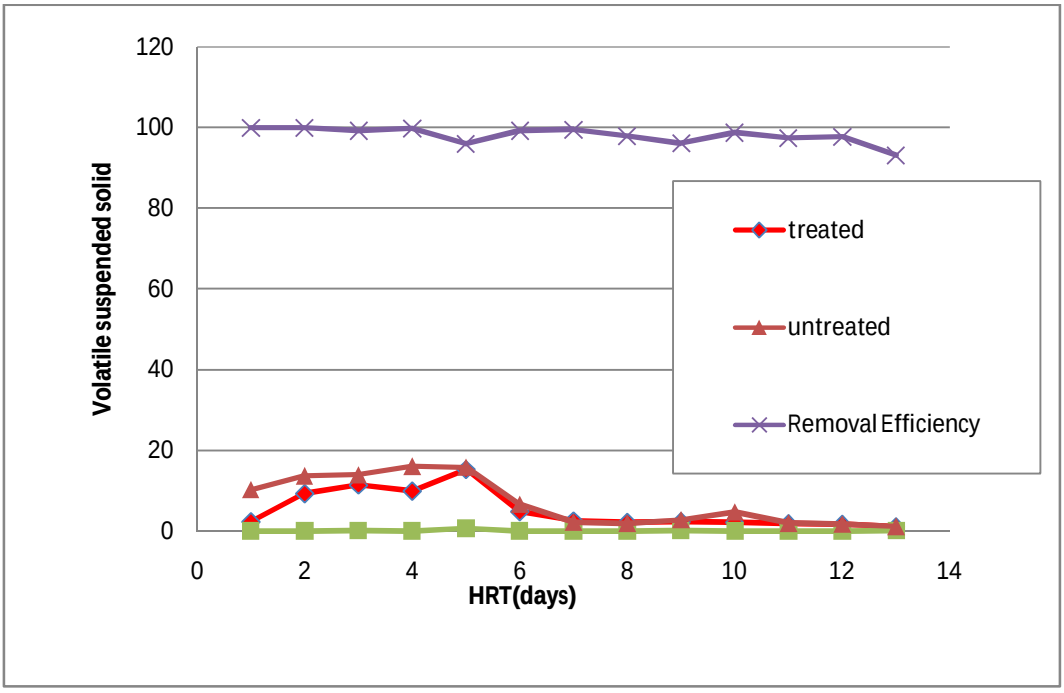

Figure 4.6: Volatile Suspended Solid and Removal Efficiency

The results showed that, the TSS content was increased from 9950mg/L in the first day to 19940mg/L on the 5th day. This was corresponded to organic matter that were not accumulated in reactor at early stage were hydrolyze and fermented into soluble form. (Rajaletchumy, 2010). Apart from that, it has also indicated that the mass of microbial cell that has developed in the system increased. However, not all the solid participates in the conversion of the organic substrate as there was inorganic fraction that does not play an active role in biological treatment. Therefore, this may be represented by VSS more accurately as not all the solid mass participates in the conversion of the organic fraction. The VSS results supported the TSS result whereby the VSS showing the same trend that the VSS increased from 2380 mg/L to 1529 mg/L on the 5^{th} day. Sanchez et al (2005) has also relied on VSS for the microorganism concentration estimation in their study. However VSS provided only estimation as the increase in volatile solids concentration may attribute by accumulation of compound such as fats, oil, and insoluble polysaccharide (Gerardi, 2006). VSS fraction obtained increasing drastically from day 4 to day 5 which indicate that the long Solid retention time of result from UMAS facilitates the decomposition of suspended solid. The same

experience reported by Abdurahman, (2010).It can be concluded that the microbial acclimatized well to the bioreactor environment.

TSS and VSS reduction rate always serve as an indicator on performance of anaerobic digestion. In this study, results showed that UMAS was able to remove TSS efficiently in permeate which recorded the removal rate of 98 to 99.8% of removal rate. This may attribute by the hollow fiber membrane which able to retained biomass back into the reactor effectively meanwhile giving permeates with minute amount of suspended solid. Besides, it has also prolonged the solid retention time which facilitated anaerobic digestion. The SS removal efficiency of present work was higher as compared to 81.43% reported by Raja et al (2005) who employed treatment by hollow fibre polyethersulphone membrane with 100000 MWCO and decanter system as pretreatment. It was largely contributed by the biological treatment that been integrated in the system.

Toward the end of the experiment which starting from the 7th day onwards, the microbial degradation of total suspended solid has came to the bottleneck where the total suspended solid reduction started to slow down where 3690mg/L of total suspended solid remained in the system used up 5 days to reduce to 1580mg/L. The relative slow degradation possibly due to the fraction of the solid are relative complex molecules that are not directly used by a bacteria or poorly biodegradable, therefore required longer time to convert it into soluble matter. There was also a trend notable that VSS fraction reduced, from 77% to 70% which corresponds to inorganic matter accumulation. The same observation was found by Zhang et al (2007)

The SS removal efficiency of present work was higher as compared to 81.43% reported by SS Raja et al (2005) who employed treatment by hollow fibre polyethersulphone membrane with 100000 MWCO and decanter system as pretreatment. It was largely contributed by the biological treatment that been integrated in the system. In comparison to the MAS systems without ultrasonic conducted by A.P.V Rajaletchumy (2010), the maximum TSS removal rate was found to be 53.8% which was lower than the 99.8% on the same day. The effect may be indirectly or directly, where the membrane efficiency increased directly by the ultrasonic, while the

growth of microbial community contribute to TSS destruction has promoted by the higher solid retention time indirectly.

4.3 METHANE GAS COMPOSITION

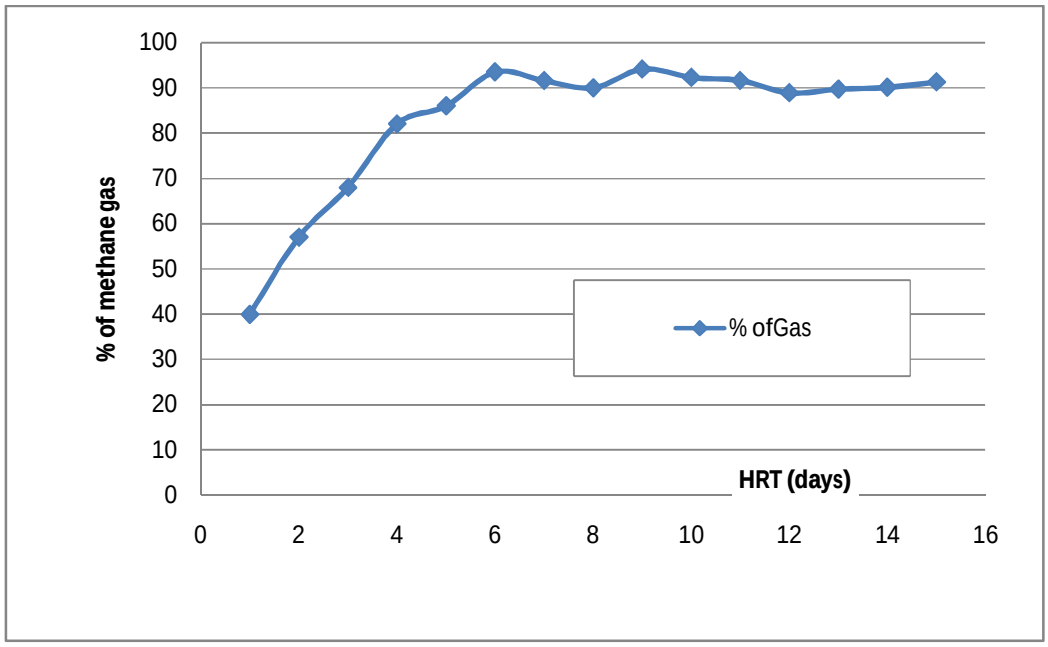

Figure 4.7: Methane Composition

Figure 4.7 showed the methane composition of biogas generated throughout the experiment. The biogas composition is an important parameter to evaluate the system balance whereby it reveals the ratio between acid former and methanogens. In this study, it can be found that the methane concentration was in a low level which was only 28% on the first day. The biogas composition was related closely to the microbial population mass, VSS results displaying the same trend where the microbial development started from 2.3mg/L to 15.29mg/L. From this point of view, the microbe in the system was acclimatizing to the reactor environment and started to develop in the system. However, the low concentration of the methane can be related to the oxygen contamination during the charging of inoculums into the system on that day which in return dilutes the gas and inhibiting the growth of methanogens. The similar problem encountered by (Basri et al, 2010) during loading of material into the bioreactor. The low percentage may also contributed by the high substrate concentration in the

beginning making the reaction favorable to acidogenesis in turn produced higher percentage of CO_2 compared to methane which can also be observe from the pH drop of 0.33 from 7.21 to 6.88.it was once again assuring that there was active assimilation of particulate organic matter. Similarly, the result of COD and BOD are also reflected the same things.

The system regain stable gradually achieving more than 82% on the 4^{th} day and maintained composition of 93.5-94.14% started from 6^{th} to 9^{th} day and decreased slightly to 76-77.7%. This was due to the increase of the SRT which was favorable for methanogenic bacteria and to obtain better adapting biofilm. (Sanchez et al, 2005) The same conclusion made by Rubia et al (2006) that more COD being used to generated methane when SRT increase due to the microbial population becoming adapted to new operational condition. The same observation found in pH in the system whereby the pH were increased and maintained in the ranged of 6.9 to 7.6 until the 11th day implying the system regains more alkalinity on the subsequent day and stabilized. These indicating that the volatile acid was converted into methane in the system. According to Poh et al (2010), rising of pH in the system indicate the methanogens have adapted to the environment.

It is notable that biogas composition increased with the total COD and BOD removal. For instance, the BOD and COD removal rate on the 6th day recorded the highest removal giving us the methane composition of 93.5% on the same day. Despite of the reduction in COD and BOD removal rate on the subsequent day, the biogas composition was only affected on the 11^{th} day. As discussed earlier, there was only low energy yield obtained from volatile acids by methane forming bacteria so the amount of substrate utilization per unit of methane forming organism is high hence the COD utilization is rapid. On the 12^{th} and 13^{th} day the decomposition rate of complex organic compounds not as rapid as the methane conversion rate hence hydrolysis stage became rate limiting factor. Basri et al (2010) had also found that a considerable portion of COD was not being degraded in the digester due to it complex nature of plant cell walls which are difficult to hydrolyze microbiologically. The explanation has found to be consistent with literature review of (Gerardi, 2003).

In this study, the highest methane composition was found to be 94.14% .The high percentage is contributed by the membrane system that able to separate the hydraulic retention time and solid retention time by recirculation of biomass. The prolonged solid retention time of the UMAS has allowed for the decomposition of the suspended solid and subsequent conversion to methane. (Abdurahman, 2010) Besides, sludge recirculation create modest mixing which enhanced the digestion process by distributing bacteria, substrate, and nutrients throughout the digester as well as equalizing temperature. The metabolic activities of acetate forming bacteria and methane forming bacteria require that they be in close spatial contact. (Gerardi, 2003)

The highest methane composition of 94.14% was found to be higher than the conventional method which recorded 54.4% and 36% for the anaerobic pond and open digesting tank. (Poh et al, 2009) The result was comparable to that achieved by Najafpour et al (2006) which reported the range of 62-82% for both of the system and present system were superior in term of biomass retention.

4.4 ULTRASONIC SYSTEM

In this study, the effectiveness of the Ultrasonic can be seen from the permeability yield, permeate quality and the pressure drop of the system. The permeability yield improvement can be seen from the permeate volume collected in the experiment which was direct proportional to the membrane flux. The flux of the membrane throughout the experiment have calculated by measuring the quantity of permeate collected in 5 hour period and divided by the effective membrane area for filtration which was $0.048m^2$ for four membrane (Wu et al.,2007). It was found that the flux reduction accounted for 37.7% in 8 days operation compare with the flux in the beginning of the experiment. The similar study has been done by Sui et al (2008) using different approach by measuring the filtration resistance. It was found that the total filtration resistance was only 30% of that without ultrasonic after 28 days of operation which shown better performance. However, the performance difference can be explained as properties of wastewater used was vary, synthetic wastewater was used by Sui (2008) which was lower strength wastewater compared to POME with high

suspended solid and organic content. In the mean time, the pressure drop of the system was not significant throughout the experiment.

There were some improvements of results in TSS and COD removal parameter as compared with the experimental result done on the system without ultrasonic by (Rajaletchumy, 2010). It was found that the highest COD removal and TSS removal achieve by Rajaletchumy (2008) were 70% and 53.8% respectively. In comparison with the current results the COD removal reached up to 92.4% and TSS removal of 99.8% on the same HRT 4 day. The performance may attribute by Ultrasonic equipped with the membrane which manage to emit ultrasonic irradiation in return creating turbulence flow which would trigger the removal of fouling particles from membrane surface in return retained the biomass in the system resulting in higher SRT for microbial degradation. The same explanation can be applied when comes to comparison with the methane content obtained by Abdurahman et al (2010) which was 76.3% as compared with 82% found. Hence, it could be found that the permeate improvement relied more on the microbial digestion rather than physical separation result from reduction of fouling problems. It is reported by Wang et al (2008) that the membrane fouling did not affect the water quality as foulant does not change and destroy the properties of membrane. Some researcher even found that membrane fouling layer increased the resistance for organic matter to pass through in return causing lower concentration of COD and BOD in the permeate.(Ahmad et al,2005) (Wu et al,2007). Although the increases in COD and BOD removal efficiency in fouling phenomena, fouling still an unfavorable condition as it reduced membrane permeability incurs higher cost for high membrane surface area and capital cost in replacing membrane. There were also no damage found on the membrane and also no negative effects that on the bacterial activity which was not consistent with the finding of Sui et al (2008). It was found that ultrasonic irradiation has slight negative effect on bacterial activity in the study of Sui et al (2008). Besides, the operating frequency of 25 kHz and an adjustable power output of 250W were found to be effectively reduced the fouling layer on membrane.

4.5 pH

The performance of an anaerobic digester was highly dependent on the pH as the enzymatic activity of the microbial community was very sensitive to pH changes. Besides, it would also affect the toxicity of a number of compounds such as sulfide indirectly. Generally, the methane producing microorganism has optimum growth in pH range within 6.6-7.4 although the stability may be achieved in a wider range within 6-8. Hence, the pH of the anaerobic digester was maintained within the range. Before starting the experiment the raw POME was poised to pH 7.2 to prevent the pH drop out from the optimum range result from production of volatile acid in the system causing. As expected at the beginning stage of the experiment, pH was dropped from 7.16 from the first day to 6.82 on the 4th day. The system regains more alkalinity on the subsequent day and stabilized this indicating that the volatile acid was converted into methane in the system. The pH were maintained in the ranged of 6.9 to 7.4 until the 11th day. According to finding of Poh P.E et al (2010), pH rise in the system indicate the methanogens have adapted to the environment. The pH increased again reached up to 7.8 which correspond to the fast utilization of $CO_{2\ in}$ the system and also the methane formation rate exceeding the hydrolysis rates which delay the further formation of volatile acid.

4.6 PROBLEM FOUND DURING EXPERIMENT

Problem obtained on the 5^{th} day where leakage of treated POME found to happen in the fitting joint of membrane and housing. The leakage was probably resulted by the degradation of membrane after a period of time of frequent chemical cleaning where the membranes are found soften and hard to fitted in the housing when the pressure exerted on the membrane the membrane fallout from the housing causing a gap where leaking happen. The contaminated permeate are found to have higher suspended solid. The colors of permeate is darker than usual. Hence, the experiment is stopped and replace with new membrane.

CHAPTER 5

CONCLUSIONS & RECOMMENDATIONS

5.1 CONCLUSIONS

UMAS was found to be an effective system in treating POME and producing methane gas effectively in a short period. From this study, the COD removal rate found to be in the range of 86.80% to 95.55%. The performance of the system has also implied in the BOD removal rate which fall within 45.48% to 71.59%. Another added value of the system was the high methane composition that was produced where recorded highest methane composition of 93.5%. This showed that the system has overcome the problems of slow anaerobic grow rate which appear to be a disadvantage in the conventional POME treating method. The system has successfully separated the hydraulic retention time and solid retention time by equipping membrane in the system whereby the filtrate was discharged and the sludge was recycled back into the system. In this way, the solid free effluent can be reducing in the mean time COD removal rate can be greatly improved. Besides, due to the high solid retention time and the recycling of the slow growing bacteria the microbial mass in the system can be maintained in considerable amount.

In additional, the Ultrasonic attached to the membrane has solved one of the most critical problems during the membrane anaerobic system which was membrane fouling. Membrane fouling has reduced the membrane flux so reduce the membrane efficiency and shortened the life of membrane therefore increase the capital cost. The turbulence that created by the ultrasonic during operation has removed particles that blocking the pore efficiently. As a result, the quality of the effluent was elevated with lower COD as well as the higher biomass retention efficiency.

From the parameter that were evaluated, the UMAS was good alternative in treating high strength wastewater .The objective of this study was attained where the efficiency of the reactor has been increased by using ultrasonic as compared with the previous results by higher COD removal rate improvement by 32% as well as 71% higher TSS removal rate.

5.2 RECOMMENDATIONS

(i) Equipped the system with mixing induced more even distribution of substrate and microbe as well as equalizing temperature.

(ii) Conduct the experiment under thermophilic condition as higher temperature may increase the microbial activity hence increasing the methane production rate.

(iii) Install gas analyzer for more accurate reading and purging system to prevent oxygen contamination.

(iv) Volatile fatty acid as a parameter indicating process stability as parameter such as pH, volatile solid destruction and gas composition often too slow for optimal detection of sudden changes.

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APPENDIX 1

EXPERIMENTAL DATA

1) ANALYTICAL RESULT

A. Chemical Oxygen Demand (COD) Profile

DAY	Untreated of POME	POME in the reactor	Permeate of POME	% COD Removal
1	25650	9650	2355	90.8187135
2	32400	20550	2655	91.8055556
3	30700	17505	2436	92.0651466
4	28950	15350	1925	93.3506045
5	28600	13800	3105	89.1433566
6	26200	10350	1165	95.5534351
7	17550	8750	1545	91.1965812
8	11150	2060	1243	88.8520179
9	10620	1920	1041	90.1977401
10	10780	3080	1329	87.6716141
11	9780	1600	1079	88.9672802
12	9369	1061	914	90.2444231
13	8460	1130	952	88.7470449
14	8072	1052	940	88.3548067
15	7157	1012	945	86.7961436

B. Biochemical Oxygen Demand (BOD) Profile

DAY	Untreated of POME	POME in the reactor	Permeate of POME	%BOD Removal
1	978	930.5	631.5	35.4294479
2	1240.5	1164.5	775.5	37.4848851
3	1764.5	1698	962	45.480306
4	2364	2147	1233	47.8426396
5	2372	2104	1101	53.5834739
6	2661	1856	756	71.589628
7	924	692	378	59.0909091

8	948	560	462	51.2658228
9	898	554	434	51.6703786
10	889.5	533	467	47.4985947
11	876	518	429.5	50.9703196
12	759	557	378	50.1976285
13	756	524	366	51.5873016
14	728	519	320	56.043956
15	712	502	288	59.5505618

C. Total Suspended Solid (TSS) Profile

Day 1		Before	After	Difference	mg/L	TSS	%
U	1	0.1534	0.2692	0.1158	11580	12080	99.67
	2	0.1525	0.2783	0.1258	12580		
T	1	0.1524	0.198	0.0456	4560	9950	
	2	0.151	0.2049	0.0539	5390		
P	1	0.1521	0.1525	0.0004	40	40	
Day 2		Before	After	Difference	mg/L	TSS	%
U	1	0.1528	0.3214	0.1686	16860	18270	99.78
	2	0.1527	0.3495	0.1968	19680		
T	1	0.1521	0.246	0.0939	9390	10765	
	2	0.1536	0.275	0.1214	12140		
P	1	0.1522	0.1526	0.0004	40	40	
Day 3		Before	After	Difference	mg/L	TSS	%
U	1	0.1539	0.3701	0.2162	21620	18580	99.17
	2	0.1536	0.309	0.1554	15540		
T	1	0.1545	0.3168	0.1623	16230	15225	
	2	0.154	0.2962	0.1422	14220		
P	1	0.1521	0.1552	0.0031	155	155	
Day 4		Before	After	Difference	mg/L	TSS	%
U	1	0.1542	0.3791	0.2249	22490	21515	99.77
	2	0.1549	0.3603	0.2054	20540		
T	1	0.1528	0.2965	0.1437	14370	13600	
	2	0.1533	0.2816	0.1283	12830		
P	1	0.1533	0.1538	0.0005	50	50	
Day 5		Before	After	Difference	mg/L	TSS	%
U	1	0.1525	0.2754	0.1229	12290	20360	97.54
	2	0.1538	0.2381	0.0843	8430		

T	1	0.1533	0.3252	0.1719	17190	19940	
	2	0.1524	0.3793	0.2269	22690		
P	1	0.1518	0.1618	0.01	1000	1000	

Day 6		Before	After	Difference	mg/L	TSS	%
U	1	0.1545	0.2489	0.0944	9440	9330	98.71
	2	0.1534	0.2456	0.0922	9220		
T	1	0.1535	0.2472	0.0937	9370	6170	
	2	0.1516	0.1813	0.0297	2970		
P	1	0.1515	0.1527	0.0012	120	120	
Day 7		Before	After	Difference	mg/L		%
U	1	0.1525	0.1857	0.0332	3320	3085	99.67
	2	0.1531	0.1816	0.0285	2850		
T	1	0.1528	0.1902	0.0374	3740	3590	
	2	0.152	0.1864	0.0344	3440		
P	1	0.1531	0.1533	0.0002	20	20	
Day 8		Before	After	Difference	mg/L		%
U	1	0.1536	0.1838	0.0302	3020	3065	97.71
	2	0.1535	0.1846	0.0311	3110		
T	1	0.1545	0.1885	0.034	3400	3055	
	2	0.1544	0.1815	0.0271	2710		
P	1	0.1539	0.1546	0.0007	70	70	
Day 9		Before	After	Difference	mg/L		%
U	1	0.1526	0.1843	0.0317	3170	3895	95.38
	2	0.1512	0.1974	0.0462	4620		
T	1	0.1522	0.1838	0.0316	3160	3285	
	2	0.1532	0.1873	0.0341	3410		
P	1	0.1528	0.1546	0.0018	180	180	
Day 10		Before	After	Difference	mg/L		%
U	1	0.1525	0.2102	0.0577	5770	6095	98.52
	2	0.1529	0.2171	0.0642	6420		
T	1	0.1537	0.1704	0.0167	1670	2425	
	2	0.1534	0.1852	0.0318	3180		
P	1	0.1517	0.1526	0.0009	90	90	
Day 11		Before	After	Difference	mg/L		%
U	1	0.153	0.1793	0.0263	2630	2760	95.29
	2	0.1532	0.1821	0.0289	2890		
T	1	0.1521	0.1862	0.0341	3410	2300	

	2	0.1528	0.1647	0.0119	1190		
P	1	0.152	0.1533	0.0013	130	130	
Day 12		Before	After	Difference	mg/L		%
U	1	0.1529	0.1803	0.0274	2740	2420	91.32
	2	0.1509	0.1719	0.021	2100		
T	1	0.1523	0.1734	0.0211	2110	2350	
	2	0.152	0.1779	0.0259	2590		
P	1	0.1508	0.1529	0.0021	210	210	
Day 13		Before	After	Difference	mg/L		%
U	1	0.1511	0.1672	0.0161	1610	1580	91.77
	2	0.1521	0.1676	0.0155	1550		
T	1	0.1518	0.1676	0.0158	1580	1560	
	2	0.1518	0.1672	0.0154	1540		
P	1	0.1495	0.1508	0.0013	130	130	

D. Volatile Suspended Solid (VSS) Profile

Day 1		After	Difference	mg/L	VSS	VSS Fraction	%
U	1	0.1738	0.0954	9540	10300	78.97350993	100
	2	-	-	-			
T	1	0.1849	0.0131	1310	2375	23.86934673	
	2	0.1705	0.0344	3440			
P	1	0.1525	0	0	0	0	
Day2		After	Difference	mg/L		VSS	%
U	1	0.2011	0.1203	12030	13660	74.76737822	100
	2	0.1966	0.1529	15290			
T	1	0.1701	0.0759	7590	9300	86.39108221	
	2	0.1649	0.1101	11010			
P	1	0.1526	0	0	0	0	
Day 3		After	Difference	mg/L		VSS	%
U	1	0.2053	0.1648	16480	13910	74.86544672	99.28
	2	0.1956	0.1134	11340			
T	1	0.1944	0.1224	12240	11410	74.94252874	
	2	0.1904	0.1058	10580			
P	1	0.1532	0.002	100	100	6.451612903	

Day 4		After	Difference	mg/L		VSS	%
U	1	0.2116	0.1675	16750	16030	74.50615849	99.81
	2	0.2072	0.1531	15310			
T	1	0.1925	0.104	10400	9970	73.30882353	
	2	0.1862	0.0954	9540			
P	1	0.1535	0.0003	30	30	60	
Day 5		After	Difference	mg/L		VSS	%
U	1	0.1825	0.0929	9290	15800	77.60314342	95.88
	2	0.1762	0.0619	6190			
T	1	0.1909	0.1343	13430	15290	76.68004012	
	2	0.2078	0.1715	17150			
P	1	0.1553	0.0065	650	650	65	
Day 6		After	Difference	mg/L		VSS	%
U	1	0.1819	0.067	6700	6640	70.33898305	99.25
	2	0.1798	0.0658	6580			
T	1	0.1728	0.0744	7440	4790	77.63371151	
	2	0.1599	0.0214	2140			
P	1	0.1522	0.0005	50	50	41.66666667	
Day 7		After	Difference	mg/L		VSS	%
U	1	0.1619	0.0238	2380	2210	71.636953	99.55
	2	0.1612	0.0204	2040			
T	1	0.1631	0.0271	2710	2590	72.1448468	
	2	0.1617	0.0247	2470			
P	1	0.1532	1E-04	10	10	50	
Day 8		After	Difference	mg/L		VSS	%
U	1	0.1685	0.0153	1530	1965	64.11092985	97.96
	2	0.1606	0.024	2400			
T	1	0.1621	0.0264	2640	2195	71.84942717	
	2	0.164	0.0175	1750			
P	1	0.1542	0.0004	40	40	57.14285714	
Day 9		After	Difference	mg/L		VSS	%
U	1	0.1609	0.0234	2340	2855	73.29910141	96.15
	2	0.1637	0.0337	3370			
T	1	0.1619	0.0219	2190	2315	70.4718417	
	2	0.1629	0.0244	2440			
P	1	0.1535	0.0011	110	110	61.11111111	
Day 10		After	Difference	mg/L		VSS	%

U	1	0.1607	0.0495	4950	4750	82.32235702	98.74
	2	0.1716	0.0455	4550			
T	1	-	-	-	2180	89.89690722	
	2	0.1634	0.0218	2180			
P	1	0.152	0.0006	60	60	66.66666667	
Day 11		After	Difference	mg/L		VSS	%
U	1	0.1602	0.0191	1910	1980	75.2851711	97.47
	2	0.1616	0.0205	2050			
T	1	0.1572	0.029	2900	1905	82.82608696	
	2	0.1556	0.0091	910			
P	1	0.1528	0.0005	50	50	38.46153846	
Day 12		After	Difference	mg/L		VSS	%
U	1	0.1606	0.0197	1970	1730	71.48760331	97.68
	2	0.157	0.0149	1490			
T	1	0.1587	0.0147	1470	1665	70.85106383	
	2	0.1593	0.0186	1860			
P	1	0.1525	0.0004	40	40	19.04761905	
Day 13		After	Difference	mg/L		VSS	%
U	1	-	-	-	1160	73.41772152	93.10
	2	0.156	0.0116	1160			
T	1	0.1562	0.0114	1140	1130	72.43589744	
	2	0.156	0.0112	1120			
P	1	0.15	0.0008	80	80	61.53846154	

2) PERCENTAGE OF METHANE GAS COMPOSITION

Day	**% Gas**
1	40
2	57
3	68
4	82
5	86
6	93.5
7	91.6
8	90
9	94.14
10	92.3
11	91.6
12	88.9
13	89.7

3) pH

Day	U	T	P
1	7.2	7.11	7.81
2	7.01	6.98	7.77
3	6.96	6.86	7.72
4	7.22	7.40	7.84
5	7.23	7.35	8.14
6	7.32	7.38	8.06
7	7.41	7.48	7.98
8	7.35	7.43	8.05
9	7.29	7.37	8.00
10	7.44	7.52	8.17
11	7.48	7.81	8.27
12	7.34	7.44	8.12
13	7.43	7.52	8.26

4) MEMBRANE FLUX

Day	Permeate volume (L)	Flux (L/m3.h)	% reduction
1	3.05	9.33	0
2	2.84	8.46	0.09375
3	2.62	7.54	19.19642857

4	2.64	7.63	18.30357143
5	3.25	10.17	0
6	3.07	9.42	7.37704918
7	2.96	8.96	11.8852459
8	2.69	7.83	22.95081967
9	2.47	6.92	31.96721311
10	2.39	6.58	35.24590164
11	2.33	6.33	37.70491803
12	2.36	6.46	36.47540984
13	2.33	6.33	37.70491803