PRODUCTION OF METHANE GAS BY ULTRASONIC MEMBRANE ANAEROBIC SYSTEM (UMAS) USING SEWAGE SLUDGE AS SUBSTRATE

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A thesis submitted to the Faculty of Chemical and Natural Resources Engineering in
partial fulfillment of the requirements for the award of the Degree of
Bachelor in Chemical Engineering (Gas Technology)

Faculty of Chemical & Natural Resources Engineering
Universiti Malaysia Pahang

FEBRUARY 2013

SUPERVISOR'S DECLARATION

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

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STUDENT'S DECLARATION

"I declare that this thesis entitled "Production of Methane Gas by Ultrasonic Membrane Anaerobic System (UMAS) Using Sewage Sludge as A Substrate" is the result of my own research except as cited in references. The thesis has not concurrently submitted in candidature of any other degree."

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Special dedication to my parent and family that always inspire, love and stand beside me,

and to my beloved friends.

ACKNOWLEDGEMENT

In the name of Allah S.W.T the Most Beneficent and the Most Merciful. The deepest sense of gratitude to the Almighty for the strength and ability to complete this project. Infinite thanks we brace upon Him.

At this moment of accomplishment, first of all I pay homage to my supervisor, Dr Abdurahman Hamid Nour. This work would not have been possible without his guidance, support and encouragement. Under his supervision, I successfully overcame many difficulties and learned a lot. I hope to continue work with his noble thoughts in future.

My special thankful goes to my beloved parents, Mr Mohamad Asri bin Mohd Yusof and Mrs Wan Mariam Binti Wan Mahmood for their encouraged and supported me spiritually and financially during my year of study leading to completion of the thesis project. I know they always pray for my successful.

I would like to extend my heartfelt gratitude to the project coordinator, Miss Noraziah Bt Yazid and to all FKKSA lectures especially panels for presentation who offer tips, advice and endless cooperation. It is a special thanks to all teaching engineer and also lab assistant towards the accomplishment of my experiment. And sincere appreciation also extends to all my colleagues, housemates, and friends whom had provided assistance at various occasions.

PRODUCTION OF METHANE GAS BY ULTRASONIC MEMBRANE ANAEROBIC SYSTEM (UMAS) USING SEWAGE SLUDGE AS SUBSTRATE

ABSTRACT

A study was conducted to verify the status of the application of Ultrasonic Membrane Anaerobic System (UMAS) in wastewater treatment, efficiency of UMAS in methane production and kinetic parameters in sewage sludge on methane production from sewage sludge by using UMAS. UMAS consist of Ultrafiltration membrane for solid-liquid separation with 1.5 – 2.0 bar of operational pressure. The wastewater has been extensively analyzed for six parameters which are pH value, biochemical oxygen demand (BOD), chemical oxygen demand (COD), volatile fatty acids (VFA), total suspended solid (TSS) and volatile suspended solid (VSS). The reactor is operated at ambient temperature and wrapped with aluminum foil to avoid direct sunlight, which can affect the bacteria. The raw sewage sludge was obtained from Indah Water Air Putih, Kuantan. Sewage sludge is tested using jar test for the chemical requirements to remove small particles in the waste water. The digester operates 5 hours every 4 days for 12 days. The product that been produce contained carbon dioxide and methane gas. Both gases are collected into a syringe that connected with two rubber tube and a glass tube. Compare to aerobic digestion which is expensive method since it used oxygen and anaerobic digestion that requires larger area and slower process, UMAS was being innovated and offer great advantages. Carbon dioxide is removed by manipulated the syringe using 10 ml of sodium hydroxide. Methane gas is measured once every four days. This treatment showed the best solution to treat waste water such as reduction of COD contents and increased percentage of methane gas. Thus, make UMAS is a good alternative for treating wastewater.

PENGHASILAN GAS METANA OLEH SISTEM MEMBRAN ANAEROBIK BERULTRASONIK MENGGUNAKAN SISA KUMBAHAN SEBAGAI SUBSTRAT

ABSTRAK

Satu kajian telah dijalankan untuk membuktikan keberkesanan Sistem Membrane Ultrasonik anaerobik (UMAS) dalam rawatan air sisa, kecekapan UMAS dalam pengeluaran gas metana dan parameter kinetik dalam sisa kumbahan kepada pengeluaran metana daripada sisa kumbahan dengan menggunakan (UMAS). UMAS terdiri daripada membran ultrasonik untuk pemisahan pepejal-cecair dengan 1.5 - 2.0 bar tekanan operasi. Air kumbahan telah dianalisis selama enam parameter iaitu nilai pH, Permintaan Oksigen Biokimia (BOD), Permintaan Oksigen Kimia (COD), Asid Lemak Meruap (VFA), Jumlah Pepejal Terampai (TSS) dan Tidak Menentu Pepejal Terampai (VSS). Reaktor dikendalikan pada suhu ambien dan dibalut dengan kertas aluminium untuk mengelakkan cahaya matahari, yang boleh menjejaskan bakteria. Sisa kumbahan telah diperolehi dari Indah Water Air Putih, Kuantan. Air kumbahan ini diuji menggunakan Ujian Balang untuk keperluan kimia untuk mengeluarkan zarah kecil dalam air sisa. Reaktor ini beroperasi 5 jam setiap 4 hari selama 12 hari. Produk yang telah menghasilkan yang mengandungi karbon dioksida dan gas metana. Kedua-dua gas dikumpulkan ke dalam picagari yang disambung dengan dua tiub getah dan tiub kaca. Ber bandingkan dengan pencernaan aerobik yang kaedahnya yang mahal kerana ia menggunakan oksigen dan pencernaan anaerobik yang memerlukan kawasan yang lebih besar dan proses yang perlahan, UMAS telah diubahsuai dan menawarkan lebih banyak kelebihan. Karbon dioksida dikeluarkan oleh dimanipulasi picagari menggunakan 10 ml natrium hidroksida. Gas metana diukur setiap empat hari sekali. Rawatan ini menunjukkan penyelesaian terbaik untuk merawat air sisa seperti pengurangan kandungan COD dan meningkatkan peratusan gas metana. Oleh itu, membuat UMAS adalah alternatif yang baik untuk mengolah air sisa.

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LIST OF SYMBOLS

%	Percentage
°C	Degree Celsius
°F	Degree Fahrenheit
Mg/L	Milligram/Liter
Mg/dm^3	Milligram/cubicdecimetre
L	Liter
M^2	Meter Square
G	Gram
μm	Micrometer
kHz	Kilohertz
ppm	Parts per Million
mL	Milliliter

LIST OF ABBREVIATIONS

BOD	Biochemical Oxygen Demand
COD	Chemical Oxygen Demand
UMAS	Ultrasonic Membrane Anaerobic System
VFA	Volatile Fatty Acids
TSS	Total Suspended Solid
VSS	Volatile Suspended Solid
H_2S	Hydrogen sulphide
NH3	Ammonia
N2	Nitrogen
CO_2	Carbon Dioxide
CH_4	Methane
HRT	Hydraulic Retention Time
OLR	Organic Loading Rate
UF	Ultrafiltration
UASB	Upflow Anaerobic Sludge Blanket
EFB	Empty Fruit Bunch
FFB	Fresh Fruit Bunches
POME	Palm Oil Mill Effluent
TVFA	Total Volatile Fatty Acid
MSW	Municipal Solid Waste
AD	Anaerobic Digestion
OF	Organic Fraction
VS	Volatile Solids
TS	Total Solids
MS	Medium Solid
HS	High Solid
RVS	Refractory Volatile Solids
BVS	Biodegradable Volatile Solids
DO	Dissolved Oxygen
RO	Reverse Osmosis
EDR	Electro Dialysis Reversal
MF	Microfiltration

CUF	Cross Flow Ultra-Filtration Membrane
NTU	Nephelometric Turbidity Units

CHAPTER 1

INTRODUCTION

1.1 Introduction

This chapter will give the ideas about the significant of the research formulation. This first chapter will cover up the subtopic of background of research or information, problem statement, research objectives, research questions, scope of proposed research, and significance of the proposed research.

1.2 Research Background

Production of sewage sludge that increased lately have become an environment problem for engineers this field all over the world to dispose it into local rivers or lakes. The major organic loading originates from human excreta and is a complex mixture of fats, proteins, carbohydrates, lignin amino acids, sugars, celluloses, humic material and fatty acids. Primary sludge, material that settles out during primary treatment, often has a strong odor and requires treatment prior to disposal. Secondary sludge is the extra microorganisms from the biological treatment processes. The pH values were generally neutral which in range from 6.00 to 7.85. The Biochemical Oxygen Demand (BOD) and Chemical Oxygen Demand (COD) values were many higher before treatment process than after. The COD values of untreated sewage sludge amounted from 202 to 618 mg/dm^3, whereas BOD values are from 10000 to 20000 mg/L, 50 times more polluting than raw domestic sewage. On the other hand, high contents of organic matter and nutrients make sewage sludge a perfect material for fertilization and re-cultivation of degraded soils. The goals of sludge treatment are to stabilize the sludge and reduce odors, remove some of the water and reduce volume, decompose some of the organic matter and reduce volume, kill disease causing organisms and disinfect the sludge.

Anaerobic treatment which recently used is an economical option for municipal solid waste. Anaerobic is one of composting process; used to neutralize sewage sludge. The physical and chemical properties of sewage sludge are needed in designing and performance of anaerobic digestion which will affect the production of biogas. The benefits of anaerobic digestion are odor control and producing the biogas as a by-product. In the other hand, anaerobic processes require large area and have long retention time.

Membrane are used for separating two phases prevents the transportation of various chemicals in a selective manner. In membrane separation technology, feed stream into effluent streams called permeate and concentrate. The stream that is rejected by the membrane is called concentrate while the liquid that passes through the semi-permeable membrane is called as permeate. Ultrasonic produces wave that could prevent fouling in the process. Fouling is the accumulation of unwanted material on the solid surfaces that affect the function. Using Ultrasonic Membrane Anaerobic System (UMAS), the kinetic parameters that been observed are pH value, Biochemical Oxygen Demand (BOD), Chemical Oxygen Demand (COD), Volatile Fatty Acids (VFA), Total Suspended Solid (TSS) and Volatile Suspended Solid (VSS). As the production of methane, a lean water which satisfies standards that been enforced in Environment Quality Act 1974 either standard A or B. Clean water that satisfied the standard can be disposed into local river or lakes.

1.3 Problem Statement

Through anaerobic decomposition of organic matter, it produces combustible gas which is methane and carbon dioxide with a little bit amounts of hydrogen sulphide (H_2S), ammonia (NH_3) and nitrogen (N_2). The biogas produce is one of alternative energy sources.

Membrane separations that been used widely nowadays gas separations for gas purification. Removal carbon dioxide (CO_2) from methane gas (CH_4) is one of the applications of gas separation. A further process through membrane filtration can produce clean water.

Since wastewater can be treated to become some sort of energy such as methane, it also produces carbon dioxide which leads to greenhouse gases. As one of the scope that we want to achieve through this research, that is we want to obtain high purity of CH_4 gas, CO_2 must be remove by using the suitable membranes.

1.4 Research Objective(s)

1.4.1 To evaluate the application of Ultrasonic Membrane Anaerobic System (UMAS) in wastewater treatment

1.4.2 To determine the kinetic parameters in sewage sludge

1.4.3 To examine the efficiency of Ultrasonic Membrane Anaerobic System (UMAS) in methane production

1.5 Scope of Research

In order to prove that ultrasonic membrane anaerobic system is the best way on treating sewage sludge as substrate, the scope of research is more focusing on:

1.5.1 To design 200 L of Ultrasonic Membrane Anaerobic System (UMAS)

1.5.2 To study the effect of hydraulic retention time (HRT) on performance of UMAS

1.5.3 To study the effect of organic loading rate (OLR)

1.5.4 To study the kinetic parameters such as Biochemical Oxygen Demand (BOD), Chemical Oxygen Demand (COD), Total Suspended Solid (TSS), Volatile Suspended Solids (VSS), Volatile Fatty Acids (VFA) and pH

1.6 Research Question

What are significance when using open pond and closed pond (UMAS reactor) of anaerobic system?

1.7 Significance of Research

Sewage sludge is an end-product of biological wastewater treatment processes which contains pollutants such as heavy metals, organic pollutants and pathogens. This characteristic makes the sewage sludge being treated before disposal or recycling in order to reduce its content. From a waste to alternative energy sources, sewage sludge can be treated using anaerobic digestion to produce biogas which is methane and carbon dioxide. Since carbon dioxide leads to environmental problem: greenhouse effect, the removal of carbon dioxide done by using membrane.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

There are two main ways to treat raw sewage sludge. First with aerobic process and second is anaerobic process. Aerobic waste water treatment is a process where bacteria utilize oxygen to degrade organic matter (generally quantified as biochemical oxygen demand or BOD) to carbon dioxide and other pollutants involved in various production systems. The degradation of BOD is achieved through aerobic bacteria in a system. In contrast, this process quite costly which requires large number of oxygen been used to treat waste water. Thus, environmental engineers start to search and identify treatment that may benefits towards environment and human beings.

Anaerobic digestion is the natural breakdown of organic materials into methane and carbon dioxide gas. High rate anaerobic treatment of industrial wastewaters is a proven technology that offers many advantages such as high organic matter removal efficiency, recovery of energy, and excess sludge reduction (Recep, 2012). This takes place in Ultrasonic Membrane Anaerobic System (UMAS) reactor in which bacteria act without oxygen. Biogas is the name given to the mixture of gases formed during the anaerobic digestion of organic wastes (Monnet, 2003). Biogas consists of methane (70%) and carbon dioxide (30%). In this research, the main objective is to evaluate the application of ultrasonic membrane anaerobic system in wastewater (UMAS) treatment, determine the kinetic parameters in sewage sludge and to examine the efficiency of ultrasonic membrane anaerobic system (UMAS) in methane production. The methods of this study which includes the effect of hydraulic retention time (HRT) on performance of UMAS, effect of organic loading rate (OLR) and kinetic parameters such as Biochemical Oxygen Demand (BOD), Chemical Oxygen Demand (COD), Total Suspended Solid (TSS), Volatile Suspended Solids (VSS), Volatile Fatty Acids (VFA) and pH.

Anaerobic treatment solve problems in costing for oxygen supply but still give some obstacles in collecting the biogas that need to be separated from the effluent. This could results in low purity of biogas that been produced. Thus, membrane is used to separate the biogas from the effluent.

The membrane used is Ultrafiltration (UF) membrane. Ultrafiltration (UF) is a separation process using membranes with pore sizes in the range of 0.1 to 0.001 micron. Typically, UF membranes will remove high molecular-weight substances, colloidal materials, and organic and inorganic polymeric molecules. Low molecular-weight organics such as sodium, calcium, magnesium chloride, and sulfate are not removed by UF membranes. Because only high-molecular weight species are removed, the osmotic pressure differential across the UF membrane surface is negligible. Low applied pressures are therefore sufficient to achieve high flux rates from an Ultrafiltration membrane.

Fouling is one of the most restricting problems that occur in membrane processes. Fouling is deposition of solids on the membrane which is irreversible affected by operation parameters during processing. One of newest of fouling reduction is application of ultrasonic waves at which the high-velocity fluid movement associated with the ultrasonically generated microstreamers, microstreaming, microjets and acoustic streaming.

2.2 Definition of Sewage Sludge

Sludge is a residual in semi-solid material. Sewage is a mixture of water from the treatment process (wastewater) from domestic and industrial life. Sewage sludge is also known as bio solids. Sewage sludge is a mixture of solid materials in a slurry form, product from the treatment process of settling of solids and partial biological decomposition. Sewage sludge is formed from wastewater; a combination of municipal liquid-carried wastes discharged from residential, institutional, commercial and industrial activities. It may consist of undesirable components, including organic, inorganic, heavy metals and toxic substances, as well as pathogenic (Oleszczuk, 2007). The organic solids contain a portion of proteins, carbohydrates, fats and oils in wastewater.

2.3 Importance of Processing Sewage Sludge

The average production of sewage sludge being estimated around 40 – 60 g dry matter per inhabitant per day for urban sewage plants which leads to two pressing problem; the environmental crisis and the energy shortage. As it is expected to rise, an adequate management of sewage sludge is fundamental need (Ghazy, 2009). There are four main reasons wastewater cannot be disposed in untreated form. Firstly, biological decomposition of the organic materials in wastewater consumes oxygen and reduces the quantity available in the receiving waters for the aquatic. In addition, the decomposition release large amount of malodorous gases. Secondly, untreated wastewater contains pathogenic or disease causing micro-organisms that hazardous to human being (Kacprzak, 2005). Thirdly, toxic compounds such as heavy metals can be very dangerous to both plants and animals (Laturnus, 2007). Lastly, the presence of phosphates and nitrogen may cause irregular growth of aquatic animals. As an economical way, land application of sewage sludge become the most effective dispersive method that been widely used around the world. The organic compound such as nitrogen makes the wastewater suitable to be used as fertilizers. This would overcome the environmental crisis (Erbardt, 2001). Although there are these several ways, it is better to make use of these waste materials by turning them into a resource. Methane is become one of the energy sources that could be produced by wastewater by several processes.

2.4 Anaerobic Processes in Waste Treatment

Anaerobic digestion is one of biological processes of stabilization or disinfection of treatment. The stabilization aims at reducing the fermentation of the matter contained in waste and the emission of odors whereas disinfection for removing pathogens. The main objectives of this process are reducing the odor generation and reduce the pathogen content of the waste (Okuda, n.d.). In the other hand, anaerobic digestion as purposed reducing, stabilizing and partially disinfecting the treated volume of waste. Anaerobic digestion is a natural process which biodegradable waste will eventually go through in the absence of air at most active in two temperature ranges, 95 to 105 °F and 130 to 135 °F. It involves microorganisms breaking down the waste and producing combustible gas; methane gas and carbon dioxide as by product. It also called biogas and an alternative energy sources. Biogas needs to be scrubbed if it is for generating electricity. Anaerobic digestion is suitable for treatment of agriculture wastes, household waste, garden and park waste, sewage sludge and solid waste products from food that will produce beneficial end-products. In UK, anaerobic digestion has been limited to small on-farm digesters recently. In the other hand, Europe country have widely used while in Denmark, has a number of farm co-operative anaerobic plants which produce electricity and district heating for local villagers, biogas plant have been built in Sweden to produce vehicle fuel for fleets of town buses. In Germany and Austria, several thousand on-farm digesters have been built to treat mixtures of manures, energy corps and food waste as production of biogas to generate electricity. An anaerobic digester or sealed airless container is a device which accelerates the digestion process and so the production of biogas. The properties of the digester itself create the ideal conditions for the bacteria or microorganisms to ferment the organic material ("Anaerobic Digestion", 2007). It is fed with biodegradable waste, heated to the appropriate temperature and left to do its work. This process usually takes a couple weeks to a couple of months after which the residuals slurry can be removed, either continuous or batch-wise. Several options are available, ranging from simple digestion techniques to technologically complex designs on a household or municipal scale. Municipal scale is a large scale from a number of sources. A domestic anaerobic digestion technique `fixed dome type' consists of a simple biogas tank with a flat bottom and a round chamber covered with a dome shaped concrete gasholder. The gas is captured in the upper part of the digester. Gas pressure increases with the volume of gas stored, pushing the slurry into a separate outlet tank.

2.5 Anaerobic Treatment

Anaerobic digestion is affected by biological activity which relatively slowly reproducing methanogenic bacteria. These bacteria must be given sufficient time to reproduce, so that they can replace cells lost with the effluent and adjust their population size to follow fluctuations in organic loading. Maintaining a sufficient retention time for solids ensuring the bacterial cells remain in optimal concentration within the digester. The bacteria population in the digester will be eliminated of the system if the rate of bacteria lost from the digester with the effluent slurry exceeds the growth rate of the bacteria. Anaerobic digestion affected by the specific characteristics of feed substance, operational parameters such as hydraulic retention time (HRT), solid retention time (SRT) and environmental factors such as pH, temperature, design of reactor and available organic materials. Hydraulic retention time can be easily manipulated because it is possible to feed sufficient substrate by reducing it and increasing organic waste load. Designing a properly sized digester to obtain the maximum biogas production per unit of digester volume is important in maintaining low capital construction costs. The digester should be sized to achieve desired performance goals in both winter and summer, and must be large enough to avoid the population of bacteria being eliminated. Design goals for the digester maximizing gas production with minimal capital investment and a minimum of operational attention. Moreover, digester must achieving pollution control and reduction of pathogens. The most important target of designing digester is the production of sellable digested and composted biomass for use as a soil conditioner or fertilizer (Malakahmad, n.d.).

There are four types of anaerobic reactor that have been developed as follows:

- Up-flow Anaerobic Sludge Blanket (UASB) type units in which no special media have to be used since the high density sludge granules themselves act as the 'media' and stay in suspension. This will form a sludge blanket in the reactor. The waste is passed upward through the blanket. Because of its density, a high concentration of biomass can be developed in the blanket. These are commonly preferred.

- Fluidized bed units filled with sand or plastic granules are used with recirculation under required pressure to keep the entire mass fluidized and the sludge distributed over the entire reactor volume. Since the sand particles are small, a very large biomass can be developed in a small volume of the reactor. In order to fluidize the bed, high recycle is required. Their power consumption is higher.

- Anaerobic filter is generated on media. The bed is fully submerged and can be operated either up flow or down flow. High recycle is needed for high strength waste.

- Anaerobic Contact or an anaerobic activated sludge where the sludge is recycled from a clarifier or separator to the reactor. A vacuum degasifier is required in order to separate the gas from gas-liquid-solid mixture and avoid floating sludge in the clarifier.

2.6 Types of Anaerobic Process

Methanogenic bacteria are more sensitive to changes in temperature than other organisms present in digesters. The temperature effect also depends significantly on the solids concentration of the fermentation. Research has shown when high concentrations of organic loading were used (over 10 %), the tolerance for changes of 5 – 10 °C is much higher, and bacterial activity returns quickly when the temperature is raised again. Gas production efficiency generally increases with temperature, roughly doubling for every 10˚C rise between 15 ˚C and 35 ˚C. There are two types of digestion process which is mesophilic and thermophilic digestion.

2.6.1 Mesophilic Digestion

This digestion optimally operated at 25 to 37 °C (Sosa, 2012) or at ambient temperatures between 20 to 45 °C, where mesophiles are the primary microorganism present in the digester. Mean retention period of at least 15 to 23 days digestion. Mesophiles have a wider diversity of bacteria grow at mesophilic temperatures and they are also more adaptable to changes in environmental conditions than thermophiles. Mesophilic systems are, therefore, considered to be more stable than thermophilic digestion systems. It is essential for efficient operation to control temperature since the reaction rates drop off considerably as temperature falls and sharp drop off in activity at temperature above 45 °C. Thus, mesophilic bacteria become inhibited by the heat.

2.6.2 Thermophilic Digestion

Thermophilic digestion takes place optimally around 50 to 65 °C, or at elevated temperatures up to 70 C, where thermophiles are the primary microorganisms present. Mean retention period between 12 to 14 days (n.d.). Thermophilic offers the advantages of faster reaction rates compared to mesophilic digestion which leads to shorter retention time. Thermophilic digestion systems are considered to be less stable and the energy input is higher, more energy is removed from the organic matter. The increased temperatures facilitate faster reaction rates and, hence, faster gas yields. Operation at higher temperatures facilitates greater sterilization of the end digestate. In countries where legislation, such as the Animal By-Products Regulations in the European Union, requires end products to meet certain levels of reduction in the amount of bacteria in the output material, this may be a benefit. Thermophilic digestion also provides better pathogen kill due to the higher temperatures, although this is less important if the waste stream is treated. A drawback of operating at thermophilic temperatures is that more heat energy input is required to achieve the correct operational temperatures, which may not be balanced by the increase in the outputs of biogas from the systems. Therefore, it is important to consider an energy balance for these systems. Thermophilic systems are usually more expensive to operate as they require additional energy to maintain the higher operating temperatures. Another drawback of thermophilic systems is the greater sensitivity to operational and environmental conditions. For feedstocks rich in nitrogen where ammonium or ammonia can result in inhibition of the digestion process, thermophilic operation is less recommended.

2.7 Application of Anaerobic Digestion

2.7.1 Palm Oil Mill Effluent (POME)

Malaysia is the largest palm oil producer and exporter in the world which corresponding a large amount of wastes such as empty fruit bunch (EFB) (23 %), mesocarpfibre (12 %), shell (5 %), and palm oil mill effluent (POME) (60 %) for every tonne of fresh fruit bunches (FFB) being processed in the mills (Najafpour, 2006). In 2005, it was estimated that about 75.5 million tonnes of FFB has been processed in the country (Baharuddin et al., 2010). Palm oil mill effluent is a highly polluting wastewater that pollutes the environment if discharged directly due to its high oxygen demand (COD) and biochemical oxygen demand (BOD) concentration (Abdurahman, 2011). It is also acidic and thick brownish liquid. POME is composing of high organic content mainly oil and fatty acids which enable to support bacterial growth. Anaerobic process is a suitable treatment method due to the organic characteristic of POME. In Malaysia, the most popular treatment method for POME is open pond system or anaerobic digestion which utilized by more than 85 % of the mills since 1982. This is due to its low capital and operating costs (Rupani, 2010). Anaerobic treatment is one of the successful and powerful biological methods for POME treatment that contains high organic substances which non-toxic and biodegradable. It gives excellent pollutant destruction efficiency of above 95 %. In anaerobic digestion, a group of microorganisms catalyzing a complex series of biochemical reactions that mineralize organic matter which producing methane and carbon dioxide. The suspended and colloidal components are easily decomposed biologically or by other conventional due to buoyancy characteristic; floating on the surface of the wastewater having an impact on the microbial and reactor performance (Meesap, 2011). This is the major obstacle faces in treatment process. Particularly, the key factors in biological anaerobic digestion, microorganism plays an important role and core factor of the system to control reactor performance or design of the reactor and stability. The parameters that should be experimented are hydraulic retention time (HRT), organic loading rates (OLR), pH, temperature, inhibitor concentration, concentration of total volatile fatty acid (TVFA) and substrate composition. These parameters should be maintained at or near optimum conditions.

Table 2.1: Parameter Limits for Watercourse Discharge for Palm Oil Mill Effluent (Lang, 2007)

Parameter	Concentration*
pH	4.7
Temperature	80 – 90
BOD 3-day, 30°C	25,000
COD	50,000
Total Solids	40,500
Suspended Solids	18,000
Total Volatile Solids	34,000
Ammoniacal-Nitrogen	35
Total Nitrogen	750
Phosphorus	18
Potassium	2,270
Magnesium	615
Calcium	439
Boron	7.6
Iron	46.5
Manganese	2.0
Copper	0.89
Zinc	2.3

Nowadays, the anaerobic hybrid sludge bed and fixed film reactor (AHR) is applied to POME treatment for solving cell washout and material cost. In a palm oil mill processing system, the wastewater is discharged at relatively high temperatures about 80 to 90 °C. Thus, it is feasible to treat using mesophilic or thermophilic temperature. Various POME treatments were studied in order to meet the stringent water course discharge regulation. POME treatment using membrane technology with physical chemical pretreatment showed a reduction in turbidity, COD and BOD up to 100 %, 98.8 % and 99.4 % respectively. Two-stage up-flow anaerobic sludge blanket system (UASB) could work efficiently up to 30 g COD/L/day while methane yield and COD reduction greater than 90 %. COD removal efficiencies greater than 94 % obtained in single anaerobic tank digester and single stage anaerobic ponding system after 10 days of retention time. This showed COD removals higher than 90 % in both anaerobic filter and anaerobic fluidized bed reactor at loading of 10 g/COD/L/day. COD removal up to 88 % was obtained with 55h HRT using attached growth on the rotating biological contactor (Najafpour, 2006). A 95% COD reduction is achieved using treatment of tropical marine yeast with 2 days retention time (Lang, 2007). The end product of anaerobic digestion using POME as substrate gives a mixture of biogas (65% CH_4, 35% CO_2 and traces of H_2S) from research studies and approximately 28m^3 of biogas can be obtained from 1 tonne of POME. Anaerobic pond has particular disadvantages such as long hydraulic retention time of 45 to 60 days, solids accumulation that deactivate the activated sludge and also large land requirement.

2.7.2 Municipal Solid Waste Treatment (MSW)

In Malaysia, 16,000 tons of domestic waste has been generated daily by population of over 25 million. A new law has been introduced by Malaysia government on solid waste management and also drafting in Strategic Plan for Solid Waste Management in Peninsular Malaysia. When treating municipal waste, AD can be used to process specific source separated waste streams such as soil improver. To minimize the impact on the climate, compostable and recyclable material should be separated at source for treatment or processing, using AD where suitable. Municipal solid waste contains an easily biodegradable organic fraction (OF) of up to 40% (Malakahmad, 2004). Conventional MSW management has been primarily disposal by land filling (Zakarya, 2008).

2.8 Microbiology and Biochemistry of Anaerobic Digestion

In anaerobic digestion, organic matters are degraded to methane and carbon dioxide in discrete steps by the combined action of several different metabolite groups of microorganism. The major reactions of the anaerobic digestions are shown in **Figure 2.1**.

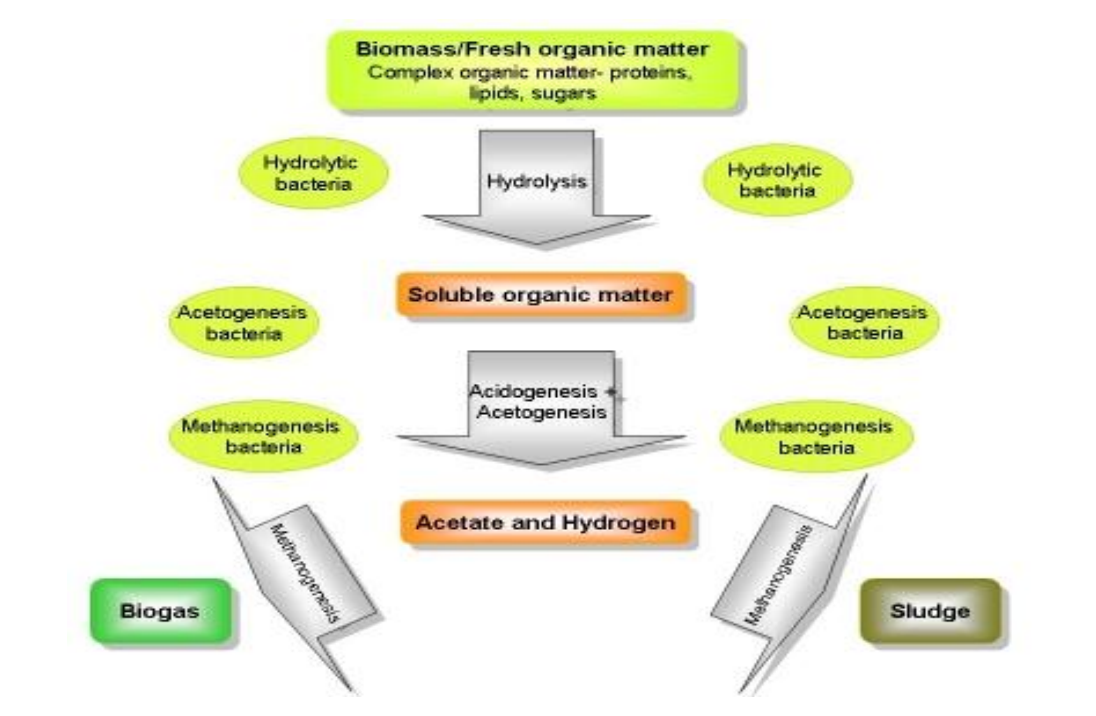

Figure 2.1: Process flow of the Degradation of Organic Material through Anaerobic Digestion ("Anaerobic Digestion", 2009)

2.8.1 Hydrolysis

The first step for most digestion process is hydrolysis during which, organic matters are converted to insoluble compounds that can be hydrolyzed further to simple monomers to be subsequently utilized by fermentative bacteria (Lorestani, 2006). The organic materials are hydrolyzed to monomers such as glucose, fatty acid and amino acids. Fats are hydrolyzed into fatty acid or glycerol; proteins are hydrolyzed into amino acids or peptides while carbohydrates are hydrolyzed into monosaccharides and dissacharides. The group of nonmethanogenic microorganisms responsible for the fermentation process consists of facultative and obligate anaerobic digestion bacteria. Extra cellular excreted by the fermentative bacteria catalyze the hydrolysis reactions. As no mineralization of organics is involved, this conversion results in no reduction in COD. Although most biopolymers are readily degradable, has been shown to be resistant to hydrolysis. The rate of hydrolysis is a function of factors such as pH, temperature composition and particle size of the substrate (Lorestani, 2006). The hydrolytic activity may become rate limiting due to its significant role in wastes with high organic content. In order to reduce the digestion time, certain chemicals can be added, thus producing higher methane yield.

2.8.2 Acidogenesis and Acetogenesis

In the acidogenesis step, the hydrolysis products are absorbed by the cells of fermentative bacteria to be fermented or anaerobically converted into compounds such as alcohols, volatile fatty acids, aldehydes, formic acid, water, carbon dioxide, hydrogen, ammonia and sulfide (Lorestani, 2006). Volatile fatty acids with more than four-carbon chain could not be used directly by methanogens (Rapport, 2008). Thus, it will be further oxidized to form organic acid, acetic acid and hydrogen by acetogenic (acid formers) bacteria through a process called acetogenesis. Production of acetate from hydrogen and carbon dioxide also includes in acetogenesis. Acidogenesis and acetogenesis could be combined together as one stage. The organic substrates serve as both the electron donors and acceptors. The final products of the metabolic activities of these bacteria depend upon the initial substrate as well as the environmental conditions.

2.8.3 Methanogenesis

In final stage, methane can be formed by bacteria called methane formers or methanogens through conversion of acetate to carbon dioxide and methane by acetotrophic organisms or reduction of carbon dioxide with hydrogen by hydrogenotrophic organisms. Production of methane is higher from reduction of carbon dioxide but limited hydrogen concentration in digesters. Methanogenic bacteria (as obligate anaerobes) have a limited substrate spectrum. Methanogenesis reactions expressed as follows:

$$CH_3COOH \rightarrow CH_4 + CO_2$$

$$CO_2 + 4H_2 \rightarrow CH_4 + 2H_2O$$

2.9 Anaerobic Digestion Operational and Environmental Conditions

2.9.1 Temperature

Anaerobic digestion can occur under two main temperature ranges:

- Mesophilic conditions, between 20 to 45°C, usually at 35°C
- Thermophilic conditions, between 50 to 65°C, usually at 55°C

The optimum temperature of digestion may vary depending on samples composition and type of digester, but in most AD processes it should be maintained relatively constant to sustain the gas production rate. Thermophilic digesters are more efficient in terms of retention time, loading rate and gas production, but they need higher heat input and have greater sensitivity to operating and environmental variables, which makes more obstacles than mesophilic digestion (Monnet, 2003). The disadvantages of thermophilic anaerobic fermentation are the reduced process stability and reduced dewatering properties of the fermented sludge and the large requirement of energy for heating (Vindis, 2009).

2.9.2 PH and Alkalinity

The optimal pH values for acidogenesis, acetogenesis, methanogenesis stages are different. During acidogenesis, lactic and propionic are formed and, thus the pH falls. Low pH can inhibit acidogenesis and pH below 6.4 can be toxic for methane forming bacteria. The retention time of digestate affects the pH value and in a batch reactor, acetogenesis occurs at a rapid pace. Acetogenesis may lead to accumulation of large amounts of organic acids resulting in pH below 5. Methanogens are sensitive to the acid concentration and their growth can be inhibited by acidic conditions. The optimal range for methanogenesis process is between 6.6 and 7.0. Reduction in pH can be reduced by adding lime or recycled filtrate obtained during residue treatment. During methanogenesis stage, pH value could be increase due to ammonia concentration that increases to above 8. An optimal pH range for all is between 6.4 and 7.2.

2.9.3 Retention Time

Retention time is the time required to achieve the complete degradation of the organic matter. Hydraulic retention time (HRT) gives major impact to distribution of products effluent. The retention time varies with process parameters, such as process temperature, technologies and waste composition. The biodegradability of carbohydrates increased with HRT, continued by proteins and lipids. The retention time for waste treated in a mesophilic digester ranges from 15 to 30 days and 12 to 14 days for thermophilic digester. Lower retention time means an inefficient extraction of methane, so full revenue is not realized which operated at thermophilic range. Too long retention time means too much was spent on surplus capacity or not enough substrate is being added to maximize revenue.

2.9.4 Organic Loading Rate (OLR)/Volatile Solids (VS)/Total Solid Content (TS)

There are three different ranges of solid content; low solid (LS) anaerobic systems contain less than 10 % Total Solis (TS), medium solid (MS) from 15 to 20 % and high solid systems (HS) range from 22 to 40 %. When increasing the total solid content, the volume of the digester decreases, due to lower water requirements (Monnet, 2003).

OLR is a measure of the biological conversion capacity of the AD system. Substrate fed of system above its optimum OLR, results in low biogas yield and organic matter removal due to accumulation of inhibiting matters such as volatile fatty acids in the digester slurry (Lang, 2007). Under such conditions, the feeding rate of the system must be reduced. Methanogenic cannot consume hydrogen at high OLR. Thus, results in increasing of hydrogen partial pressure along with decreasing of methane yield. Degradation of carbohydrates at all loading rates but for protein and lipids decreased as increasing in loading. OLR is a particularly important control parameter in continuous systems. Many plants have reported system failure due to overloading. OLR is expressed in kg Chemical Oxygen Demand (COD) or Volatile Solids (VS) per cubic meter of reactor. It is related with retention time for any particular feedstock and anaerobic reactor volume. Volatile Solids (VS) represents the organic matter in a sample which is measured as a total solid content minus ash content, as obtained by complete combustion of the feed wastes. VS consist of biodegradable VS (BVS) and refractory VS (RVS). High VS content with low RVS is best suitable for AD. The composition of wastes affects yield and biogas quality as well as the compost quality.

2.9.5 Mixing

The purpose of mixing in a digester is to blend the fresh material with digestate containing microbes (Verma, 2002). Furthermore, mixing discourage scum formation, prevent accumulation of inhibiting materials, clearing of grit, dense solid, encourage to release gas and avoids development of temperature gradients within the digester. In addition, it also promotes close contact between the micro-organisms (raw) and substrate (digested sludge) and bacterial population ability to obtain nutrients (Monnet, 2003). Non-mixing reactor has closer microbial contact than others. However, excessive mixing can disturb the microbes. Thus, slow mixing is preferred. The type of mixing equipment and amount of mixing varies with the type of reactor and the solids content in the digester. Unmixed digester has shown a faster start-up than mixed digester and demonstrated higher methane yield than continuous mixed digester.

2.10 Advantages of Anaerobic Digestion

A traditional ways of composting waste will always have a role but does not allowed to gives extra benefits of energy extraction. Thus, AD encourages a number of advantages and potential ("Anaerobic Digestion of Farm and Food Processing Residues", 2000)

- Less energy demands
- Minimum sludge formation

- No unpleasant odor
- Great potential for rapid disintegration of organic matter to generate biogas
- Low coasts
- Relatively small space requirement
- High tolerance to unfed conditions
- High organic removal rates

2.11 Comparison with Aerobic Treatment

Organic and inorganic compounds of industrial wastewater are hazardous to the environment. In order to remove these compounds, many methods have been introduced depending on the characteristics of the wastewater. There are three main treatment systems; physical, chemical and biological unit processes.

Secondary treatment methods are commonly used the chemical and biological treatments. In chemical treatments, it relatively highly cost and may require additional treatment for neutralizing the effect of added chemicals. As in biological treatments, it is an interesting process which more sophisticated and development if efficiency bioreactor in the area of wastewater treatment with activated sludge.

Aerobic treatment of organic wastes also named composting which is conventional and centuries old agriculture technique. Simply put, aerobic wastewater treatment refers to the removal of organic pollutants in wastewater by bacteria that require oxygen to work. Water and carbon dioxide are the end products of the aerobic wastewater treatment process. Processes include trickling filtration, activated sludge, and rotating biological contactors. Bacteria that thrive in oxygen-rich environments work to break down and digest the wastewater inside the aerobic treatment plant or system (Buchanan, 2004). This process is called aerobic digestion. By maintaining appropriate dissolved oxygen (DO) reduction, the amount of oxygenation must be equal to or excess than the consumption of oxygen by the microbes. This conditions which efficient removal of pollutants by biodegradation in reducing pollution. One of the most simple and potential technique of high mixed liquor suspended solids (MLSS) concentration was considered in order to achieve highest possible chemical oxygen demand (COD) reduction. There are some disadvantages of aerobic process which are relatively high energy consumed and high excess sludge production, which requires handling, treatment and disposal (Karkare, 2012). In other hand, anaerobic treatment have relatively low construction and operational cost, no oxygen requirements, low nutrient requirements, low production of excess sludge, production of energy in form of biogas and many more. Other than that, anaerobic systems are suitable for the treatment of high strength wastewater (biodegradable COD concentrations over 4000 mg/L) (Chan et al., 2012). Therefore, highly polluted industrial wastewaters are preferably treated in an anaerobic reactor due to the high level of COD, potential for energy generation and low surplus sludge production.

2.12 Membrane Separation Process

The most recent development in high rate anaerobic treatment is using membranes to separate biomass from the effluent. Membrane processing is a technique that allows concentration and separation without use of heat. Particles are separated on the basis of their molecular size and shape with the use of pressure and specially designed semi-permeable membranes. When a solution and water are separated by a semi-permeable membrane, the water will move into the solution equilibrate the system. This is known as osmotic pressure. If a mechanical force is applied to exceed the osmotic pressure up to 700 psi, the water is forced to move down the concentration gradient. Permeate describes the liquid passing through the membrane and concentrate describes the fraction that not passing through the membrane. Membrane material should be configured in such way so as to allow water to pass through (Singhania, 2012). For the last few decades, membranes that can respond to pH, temperature, electric fields or ionic strength have been importantly focused. Some of membrane applications including controlled drug release, wastewater treatment, chemical sensors and separation of biological macromolecules such as proteins (Feng et al., 2012). The application of using membrane application in separating processes has been seriously considered because of lower energy consumption than other separation processes.

2.13 Membrane Filtration Applications

2.13.1 Reverse Osmosis (RO)

RO is also known as hyper-filtration as the finest filtration known. This process will allow the removal of particles as small as ions from a solution. Reverse osmosis is commonly used to purify water and remove salts and other impurities in order to improve the color, taste or properties of the fluid while rejecting the contaminants that remain.. It can be used to purify fluids such as ethanol and glycol, which will pass through the reverse osmosis membrane, while rejecting other ions and contaminants from passing. It is used to produce water that meets the most demanding specifications that are currently in place. Pure water flows from a dilute solution through reverse osmosis uses a membrane that is semi-permeable to pass through it, while rejecting the contaminants that remain. Semi permeable acting as a barrier to dissolved solids from a less concentrated to a more concentrated solution. Reverse osmosis is capable of rejecting bacteria, salts, sugars, proteins, particles, dyes, and other constituents that have a molecular weight of greater than 150-250 daltons.

2.13.2 Nanofiltration (NF)

A pressure-driven membrane processes that lies between UF and RO. This process operated at pressure range between 10 to 50 bar. While processing water, it is rejecting ions with more than one negative charge such as SO_4^{2-} and CO_3^{2-}. Monovalent ions like sodium, potassium, chlorine, urea and lactic acid is removed during the process is called partial demineralization. Some benefit of using NF is reduce salty taste and removed acid. This filter is widely used for food processing applications for simultaneously concentration and monovalent ion.

2.13.3 Ultrafiltration (UF)

Ultrafiltration (UF) is process of separating extremely small particles and dissolved molecules from fluids based on their molecular size. The permeability of a filter medium can be affected by the chemical, molecular or electrostatic properties of the sample. Materials ranging in size from 1K to 1000K molecular weight (MW) are retained by certain ultrafiltration membranes, while salts and water will pass through (Hashsham, 2006). UF starts to be recognized as the best pre-treatment before Reverse Osmosis (RO) because it removes, in only one physical filtration stage, all the suspended solids and biological materials. Some advantages of UF compare to conventional pre-treatment are the small footprint of the installation (Lorain, 2007). For UF, pore sizes commonly range from 0.01 to 0.05 µm or less.

2.13.4 Microfiltration (MF)

Microfiltration contains two filter modules that connected in series which are retentate circulation pump and permeate circulation pump. In this process, the removal of particles or biological entities from fluids by passage through a microporous medium such a membrane filters. Generally, MF membranes have pore size range of 0.1 to 0.2 µm considerably. Since there are some exceptions, the pore size can be up to 10 µm.

2.14 Factors Affecting Membrane Performance

2.14.1 Concentration Polarization

A concentration layer builds up at the membrane surface is called concentration polarization. This results in depletion of the permeating solute on the feed side of the membrane and its concentration is lower than in the bulk fluid. A concentration gradient is formed in the fluid adjacent to the membrane surface. In order to minimize concentration polarization, a suitable membrane should be selected to the process. Other than that, the flow should be maintained across the membrane to reduce the film thickness.

2.14.2 Fouling

Fouling is one of the most restricting problems that occur in membrane processes. Fouling is deposition of solids on the membrane which is irreversible affected by operation parameters during processing. It can decrease the permeate flow at given driving force, lower the permeate quality (purity) and increase energy consumed to maintain a given permeate flow. Some causes of fouling to be occurred are membrane characteristics, sludge characteristics, feed properties and operation parameters (Mirzaie[a] & Mohammadi[b], 2012). There are two types of fouling as following:

- **Surface (temporary) fouling**

Surface fouling occurs when foulant appears an evenly deposited layer on the membrane surface. It can be easily removed by cleaning solution. Permeation rate of membrane can be generated by cleaning. The most common type of fouling is in UF plant.

- **Pore (permanent) fouling**

Particulate matter that diffuses in to the membrane is called pore fouling. It also could be caused by the poor quality of the cleaning water. Flux cannot be regenerated by cleaning.

2.15 Advantages of Membrane Processes

There are many significant advantages on using membranes for industrial processes. Some advantages showed by membrane usage are:

- Do not involve phase changes or chemical additives
- Simple in concept and operation
- Modular and easy to scale up
- Low energy efficiency for raw materials use and potential for recycling of by-products
- Equipment size may be decreased

2.16 Ultrasonic

2.16.1 Theory of Acoustic

Acoustics is the interdisciplinary science that deals with the study of all mechanical waves in gases, liquids, and solids including vibration, sound, ultrasound and infrasound. A scientist who works in the field of acoustics is an acoustician while someone working in the field of acoustics technology may be called an acoustical engineer. The application of acoustics can be seen in almost all aspects of modern society with the most obvious being the audio and noise control industries. Vibrations of frequencies greater than 20 kHz which beyond range of the audible for human is called ultrasonic. Some animals like dogs can hear part ways into this range. One of the application using ultrasonic is in medicine field; ultrasounds for pregnant women. The term sonic is applied to ultrasound waves of very high amplitudes. Hyper sound is sound waves frequencies higher than 10^{13} hertz. The molecules of material in solid or liquid in which the longitudinal waves could not propagate at all of frequency about 1.25×10^{13} hertz. This is because the waves cannot pass the vibration along rapidly enough at such high frequencies to propagate efficiently.

2.17 Ultrasonic Treatment

One of newest of fouling reduction is application of ultrasonic waves at which the high-velocity fluid movement associated with the ultrasonically generated microstreamers, microstreaming, microjets and acoustic streaming. Ultrasonic treatment is a non-conventional and environmentally friendly. Ultrasonic waves have frequencies more than 16 kHz and widely used as a surface cleaning technique and is a promising strategy for the prevention of membrane fouling. In this treatment, no chemical cleaning reagent is required and a high permeate flux can be maintained throughout a medium; the medium is subjected to a series of compression and rarefaction cycles. Thus, the formation of cavitation bubbles is occurred where the bubbles collapse and generate a variety of physical and chemical phenomena.

2.18 Transducer

An ultrasonic transducer is a device used to convert some other type of energy into an ultrasonic vibration. There are several basic types, classified by the energy source and by the medium into which the waves are being generated. Ultrasonic transducers convert electrical or mechanical energy into high frequency sound. Mechanical and electrical transducers are limited to low ultrasonic frequencies.

2.19 Types of Transducer

2.19.1 Electromechanical Transducers

a) Magnetostrictive Transducer

This device use an effect found on some materials which reduce in size when placed in a magnetic field and return to normal dimensions when the field is removed.

b) Piezoelectric Transducers:

This device contains special crystals that will produce a voltage if pressure is applied to them in one direction.

2.20 Applications of Ultrasonic

- Ranging and navigating
- The Doppler Effect
- Materials testing
- Chemical and electrical uses
- Medical applications
- Infrasonic

CHAPTER 3

MATERIALS & METHODS

3.1 Chapter Overview

In order to study the production of methane by Ultrasonic Membrane Anaerobic System (UMAS) using sewage sludge as substrate, there are several methods that must be done. This chapter will discuss about the list of materials, chemical reagents, equipment and instrumentations used to determine the parameters in presence of UMAS. Next section will explained on experimental procedures and method to analyze raw sample.

3.2 List of Material Chemical Reagent, Equipment and Instrumentation

3.2.1 Materials

The main samples that been used in this process is sewage sludge which is collected from anaerobic pond at Air Putih Indah Water, Kuantan at about 50 L. The sample is filtered through a strainer before been placed into the reactor and also to avoid membrane fouling. Next, the sample is analyzed for COD, BOD, TSS, VSS, pH and turbidity. Jar test is also conducted to compare its result with the product from UMAS.

3.2.2 Chemical Reagents

In order to complete this research, there are certain chemical reagents that been used such as Digestion Solution for COD (20-1500ppm) of high range, Sodium Hydroxide (NaOH), Ammonia Salicylate Reagent (powder), and Ammonia Cyanurate Reagent (powder).

3.2.3 Equipment and Instrumentations

The main reactor is used for the entire process is Ultrasonic Membrane Anaerobic System (UMAS). A syringe, glass tube and two pair of rubber tube are used to collect the methane gas directly from one of the valve of the reactor. Other equipment used for analyzing data is COD Digestion Reactor and Jar Test Apparatus.

3.3 Experimental Procedures

3.3.1 Reactor Set-Up

The schematic diagram of the UMAS reactor is shown in **Figure 1**. Raw sewage sludge was treated in a Laboratory digester with an effective 200 L volume. This reactor consists of a Cross Flow Ultra-Filtration Membrane (CUF) apparatus, a centrifugal pump, and an anaerobic reactor.

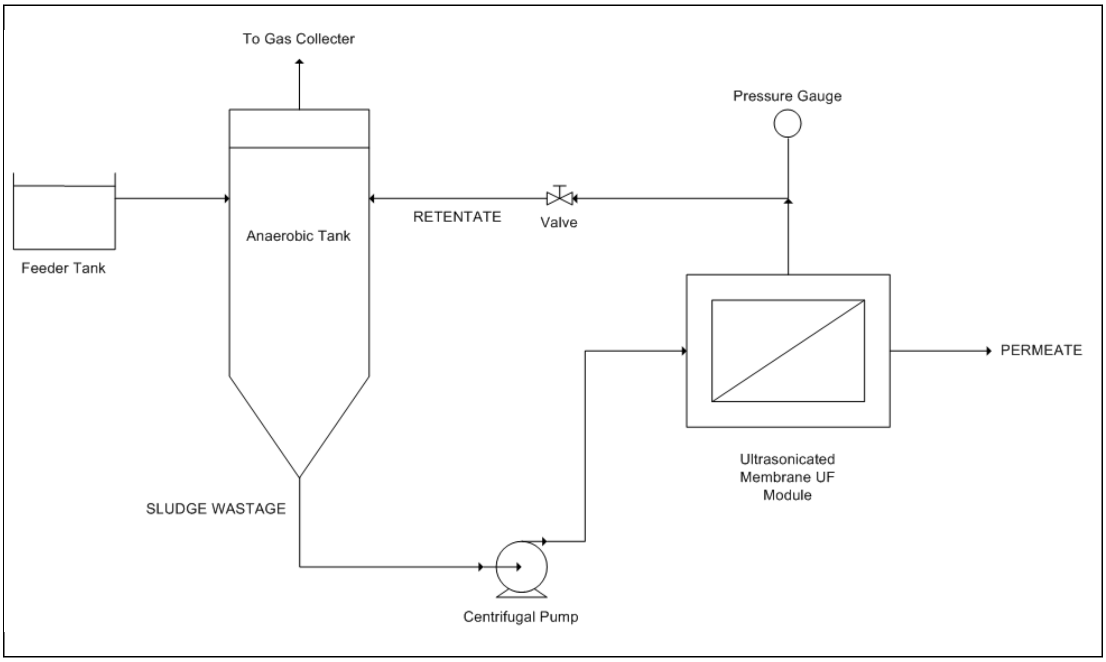

Figure 3.1: Experimental Set-Up (Abdurahman, 2011)

3.3.2 Sample Preservation

Raw sewage sludge samples are preserved in a cold room at about 4°C because bacteria have a very short holding time that may affected the result and the product of this experiment.

The sewage sludge was treated by UMAS in the digester with approximately 40 L volume. The dilution of the sample is done before enter the reactor to identify the value of COD. The effluent flow from the anaerobic digester is also undergoing same process to record the readings.

3.3.3 Reactor Operation and Monitoring

Under ambient temperature of 30 to 35 ˚C, the reactor is operated using sewage sludge. Sewage sludge is fed into the side flow into the anaerobic reactor with continuous up-flow feeding. By controlling the flow rate of the influent feed, the HRT was adjusted volumetrically. After 5 hours, the effluents samples were taken to be analyzed by kinetic parameters such as COD, pH, alkalinity, suspended solids and volatile suspended solids based on the American Public Health and Association (APHA) standard methods for water and wastewater analysis (APHA 2005). In acclimization phase, the feed flow rate of 40 L is corresponding to HRT of 4 days to allow all the microorganisms present in the mixed liquor perfectly acclimatized to the new environmental.

3.3.4 Acclimization Process

When the anaerobic bacteria is allowed to mixed liquor entirely this will acclimatize to the new environment in the reactor, the acclimization process of the UMAS reactor I done with the feed of 40 L. After 4 days of loading period in the anaerobic reactor, parameters of sewage sludge is measured and recorded. Parameters for the treated sewage sludge (permeate) are measured after 5 hours later.

3.3.5 Collect Products

First, the apparatus for collecting the products are a syringe, two pair of rubber tube and a glass tube. The set-up of the apparatus is as follows:

Figure 3.2: Apparatus Set-Up for Methane Gas Collection

The end of the rubber tube is placed at one of the valve as the valve is opened. Then, the gases are sucked out from the laboratory digester. The gases are consisting of carbon dioxide and methane gas. In order to remove the carbon dioxide gas, 10 mL of sodium hydroxide (NaOH) is used to absorb the gas. By manipulating the syringe, the gas can be absorb and remove. The methane gas is left in the syringe is measured by its length before (L_1) and after (L_2) carbon dioxide been removed. The formulae used are as follows:

$$\text{Total methane gas} = \frac{L_2}{L_1} \times 100\%$$

3.4 Determination of Parameters

3.4.1 Determination of BOD_5

The reagents are prepared in advance in the stock bottles. Reagents grade or better for all chemicals and distilled or equivalent water are used. The reagents are phosphate buffer, magnesium sulfate, calcium chloride, ferric chloride and acid and alkali solutions. There are 3 types of phosphate used; KH_2PO_4, $Na_2HPO_4 \cdot 7H_2O$, andNH_4CI. For preparing KH_2PO_4 solution, 8.5 g and 21.75 g is needed. While for $Na_2HPO_4 \cdot 7H_2O$, and NH_4CI reagent, 33.4 g and 1.7 g needed respectively. Each type of reagents are dissolved in about 500 ml distilled water and diluted to 1L. The pH should be 7.2 without further adjustment. Next, the following reagents that been dissolved in distilled water and diluted to 1L are $MgSO_4 \cdot 7H_2O$ (22.5 g), $CaCI_2$ (27.5 g), $FeCI_3 \cdot 6H_2O$ (0.25 g), concentrated sulfuric acid (28 mL) and sodium hydroxide (40.0 g).

In preparing of dilution water, 1mL each of phosphate buffers, magnesium sulfate, calcium chloride, ferric chloride solution is added into 1L volumetric flask with distilled water is added to 1L. Then, 10 ml of wastewater sample (refer Appendix) and 300 mL of dilution water is added into 500ml beaker. Value of pH is adjusted to 6.5 to 7.5 by adding acid or alkali. Dilution water of 300 mL as control is prepared in another 500 mL beaker. After that, each of prepared samples is placed and control into different incubation bottle. Dissolved oxygen (DO) concentration for each sample is measured and recorded using Dissolved Oxygen Meter. The water is added to the flared mouth of bottle and covered with aluminum foil. All the bottles are being put in BOD Incubator for five days. The temperature is set at 20°C. The final DO value is measure after five days. BOD_5 is calculated according to the formula below:

$$BOD_5\text{, mg/L} = (D_1 - D_2) / P$$

Where;

D_1 = DO value in initial sample

D_2 = DO value in final sample

P = Decimal volumetric fraction of sample used

OR;

BOD_5, mg/L = $(D_1 - D_2)$ x Dilution factor

Dilution factor = Bottle volume (300mL) / Sample volume

3.4.2 Determination of TSS

The filter disk in the oven was dried at 103°C to 105°C for an hour and then cooled in a desiccator before weighed. Filtering apparatus and filter were assembled before suction was begun. 50 mL of mixed-well water sample was pipetted onto center of filter disk in a Buchner flask using gentle suction. It was mixed to ensure the homogeneity. The filter was washed with three successive 10 mL volumes of distilled water for allowing complete drainage between washing and then was continued with suction for about three minutes after the filtration was complete.

After that, with careful, filter was removed from filtration apparatus and transferred to aluminium weighing dish as a support. Filter was dried at least for an hour at 103°C to 105°C in an oven and then cooled in a desiccator to balance the temperature before weighed. The cycle of drying, cooling, desiccating and weighing were repeated until a constant weight was obtained.

$$mg\frac{TSS}{L} = \frac{(A-B)\times 1000}{Sample\ volume, mL}$$

Where

A = weight of filter + dried residue, mg

B = weight of filter, mg

3.4.3 Determination of COD

A sample volume is homogenized for 30 seconds in a blender. For samples containing large amounts of solids, increase the homogenization time. The homogenized sample is poured into a 250 mL beaker and stirred gently with a magnetic stir plate in order to improve accuracy and reproducibility of other ranges. The above step is valid for samples that contain suspended solids. Next, COD reactor is turned on and preheated to 150°C. The safety shield is placed in front of the reactor. The caps from two COD Digestion Reagent Vials are removed. Using a clean volumetric pipette, 2.00 mL of sample is added to the vial. This is the prepared sample. Then, 2.00 mL de-ionized water is added to the vial using a clean volumetric pipette. This is the blank. Both vials are rinsed with de-ionized water and wiped with a clean paper towel. The vials are hold by the cap over a sink an inverted gently several times to mix. After that, both vials are placed in the preheated COD Reactor. Leave the vials to be heated for two hours. After two hours, the reactor is turned off and the vials are left about 20 minutes to cool to 120°C or less. While the vials still warm, both of it is inverted several times and placed into a rack until it cooled to room temperature. Touch Hach Programs. Program 430 COD LR (Low Range) or 435 COD HR (High Range/High Range Plus) is selected. Touch Start. The outside of the vials is cleaned with tissue to remove fingerprints or other marks. A 16 mm adapter is installed and the blank sample is placed into the adapter. Touch Zero. The display will show: 0 mg/L COD. Then, the sample vial is placed into the adapter after the time beeps. Touch Read. Results are appeared in mg/L COD.

3.4.4 Determination of VSS

The aluminum dish and filter is weighted. Then, the dish is placed in the furnace to ignite the residue on the filter. The dish is leaved about 20 to 30 minutes at 550 °C for the residue is ignited in the furnace (VSS_B). Let the dish cooled and the dish is weighted again (VSS_A). Lastly, volatile suspended solids are calculated using following equation:

$$\text{mg/L volatile suspended solids} = [(VSS_A - VSS_B)] \times 1000 / \text{sample mL}$$

Where

VSS_A: weight of residue + dish and filter before ignition, mg

VSS_B: weight of residue + dish and filter after ignition, mg

3.5 Jar Test

A stock solution of aluminum sulfate (alum) is prepared using 1 g of alum that been placed into 1 L volumetric flask. Distilled water is poured until it reaches 1 L mark. The alum is dissolved by mixing the solution with magnetic stirrer. The raw material of 1 L is placed in a beaker. The remaining five beakers are repeated as before. Each sample is measured for its pH and turbidity (Alum is most effective between pH ranges of 5.0 – 7.5).the beakers is placed in the stirring machine. With a measuring pipette, a correct dosage of alum solution is added to each beaker as rapidly as possible. With the stirring paddles lowered into the beakers, the stirring machine is started and operated it for 1 minute at a speed of 80 RPM. While the stirrer operated, the appearance of the water in each beaker is recorded. The presence or absence of floc, the cloudy or clear appearance of water, and the color of the water and floc is noted and recorded. The stirring speed is reduced to 20 RPM and continued stirring for 30 minutes. A description of the floc in each beaker 10, 20 and 30 minutes after addition of the chemicals is recorded. The stirring apparatus is stopped and allowed the samples in the beakers to settle for 30 minutes. A description of the floc in each beaker after 15 minutes of settling and again after 30 minutes of settling is recorded. The turbidity for each sample is measured using a turbidity meter. A pipette is used to draw a portion from the top of each beaker without disturbing the sample. A graph of turbidity (before and after alum is added) versus coagulant dose is plotted to determine the most effective dose of coagulant that gives the least turbid results.

CHAPTER FOUR

RESULTS & DISCUSSIONS

4.1 Results and Discussion

After done with the experiments and analysis, the values getting from the analysis of jar test and kinetic parameters such as BOD, COD, TSS and pH is being recorded into the table. The jar test result is tabulated in table and a graft of Turbidity versus Coagulant dose is plotted.

In jar test result, there are 5 tables that been studied to determine the correct dosage for treating waste water in order to overcome some problems. Jar test is done separately from the raw sample that been placed in the laboratory digester. Then, kinetic parameters are analyzed by following equation:

For BOD calculation,

$$BOD_5\text{, mg/L} = (D_1 - D_2) / P$$

Where;

D_1 = DO value in initial sample

D_2 = DO value in final sample

P = Decimal volumetric fraction of sample used

OR;

BOD_5, mg/L = (D_1 – D_2) x Dilution factor

Dilution factor = Bottle volume (300mL) / Sample volume

For VSS calculation,

mg/L volatile suspended solids = [(VSS_A - VSS_B)] × 1000 / sample mL

Where

VSS_A: weight of residue + dish and filter before ignition, mg

VSS_B: weight of residue + dish and filter after ignition, mg

For TSS calculation,

$$mg\frac{TSS}{L} = \frac{(A - B) \times 1000}{Sample\ volume, mL}$$

Where;

A = weight of filter + dried residue, mg

B = weight of filter, mg

4.2 Study the Jar Test

Experiment has been done to determine optimum coagulant concentration using jar test. In this experiment, the raw samples have same volume (1 L) is held constant while the coagulant dosage is being manipulated. This experiment is done in 90 minutes. Six beakers with same volume of raw samples with differ coagulant dose are being prepared. Below are the results and the graph plotted in determining optimum coagulant concentration using jar test. For **Table 4.1**, different dosages of alum are manipulated in each beaker. The pH value is recorded using pH meter after the alum is added. Next, **Table 4.2** showed the appearance of floc in jars during fast stir (80 RPM) for 1 minute. Based on **Table 4.3**, the appearance of floc in jars is recorded during slow stir (20RPM) for 30 minute. The appearance is recorded every 10 minute for each beaker. Next, in **Table 4.4** the appearance of floc is recorded during the settling which is after the stirring process ends. The appearance is recorded every 15 minute in each beaker. Lastly, **Table 4.5** showed the value of turbidity before and after the settling for each beaker.

Table 4.1: Amount of Alum Dosages

	Amount of Alum Dosages					
	Jar 1 (control)	Jar 2	Jar 3	Jar 4	Jar 5	Jar 6
Volume of raw water, L	1	1	1	1	1	1
pH	6.74	6.71	6.66	6.64	6.64	6.62
Alum dose, mg/L	-	10	15	20	25	30

Table 4.2: Appearance of Floc in Jars during Fast Stir

	Appearance of Floc in Jars During Fast Stir					
Times after alum addition	Jar 1 (control)	Jar 2	Jar 3	Jar 4	Jar 5	Jar 6
First appearance	-Less floc -Very cloudy	-Less floc -Cloudy	-Less floc -Cloudy	-More floc	-More floc	-More floc

Table 4.3: Appearance of Floc in Jars during Slow Stir

	Appearance of Floc in Jars During Slow Stir					
Times after alum addition, min	Jar 1 (control)	Jar 2	Jar 3	Jar 4	Jar 5	Jar 6
10	-Cloudy -No changes	-Cloudy -Less floc	-Less floc	-More floc	-More floc -Settling appeared	-More floc -Settling appeared
20	-Cloudy -No changes	-Floc start to form	-Less floc -Cloudy	-More floc -Colorless	-More floc -Colorless	-More floc -Colorless
30	-Cloudy -No changes	-More settling	-More settling -More colorless	-More settling -More colorless	-More settling -More colorless	-More settling -More colorless

Table 4.4: Appearance of Floc on Jars during Settling

	Appearance of Floc on Jars During Settling					
Times after stirring ends, min	Jar 1 (control)	Jar 2	Jar 3	Jar 4	Jar 5	Jar 6
15	-Cloudy -No changes	-Fully floc settled	-More floc settled -Floc static -More colorless	-More floc settled -Floc static -More colorless	-More floc settled -Floc static -More colorless	-More floc settled -Floc static -More colorless
30	-Cloudy -No changes	-Fully floc settled	-More floc settled -Floc static -More colorless	-More floc settled -Floc static -More colorless	-More floc settled -Floc static -More colorless	-More floc settled -Floc static -More colorless

Table 4.5: Turbidity

	Turbidity, NTU					
	Jar 1 (control)	Jar 2	Jar 3	Jar 4	Jar 5	Jar 6
Before	23.4	23.2	24.5	23.7	23.8	24.6
After settling	41.2	35.7	29.0	23.0	17.7	15.0

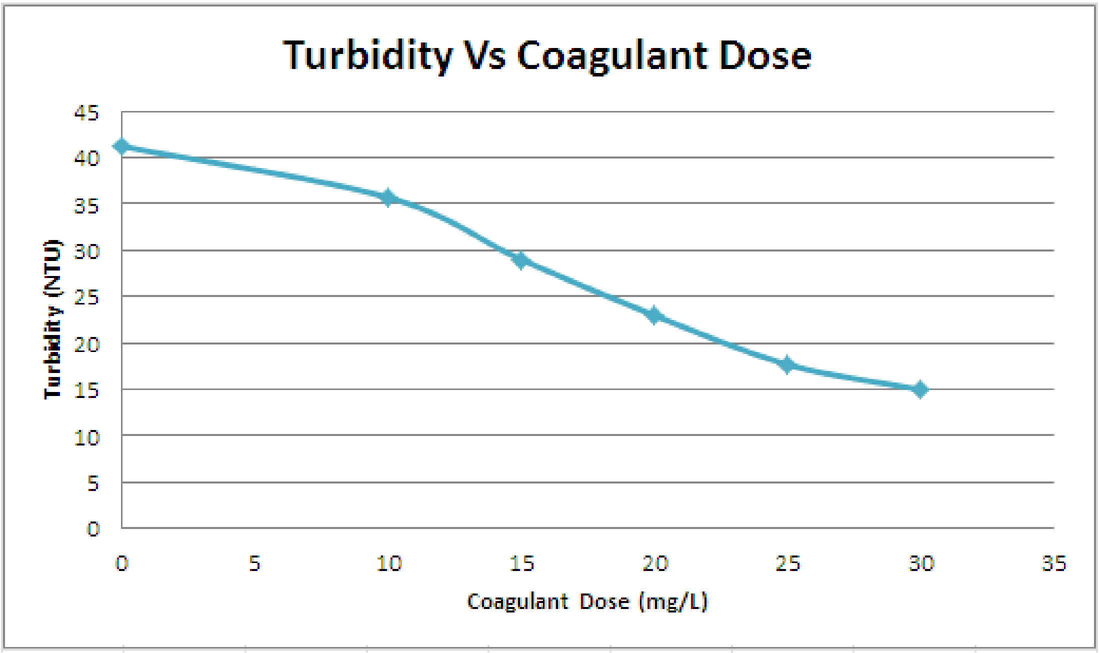

Figure 4.1: Graph Turbidity versus Coagulant Dose

For **Figure 4.1,** the line was created and this shows that when coagulant dosage increased, the turbidity of the raw sample is decreased. The highest coagulant dose value is at 30 mg/L and the turbidity is 15.0 NTU. The lowest coagulant value is 0 mg/L while the turbidity vale is 41.2 NTU.

The jar test is used to determine dosage requirements for chemicals added to remove small particulates from water or wastewater. Graph above shows different dosage of alum that mimics coagulation and flocculation. Jar testing should be done as seasons change, weather changes, raw water characteristics change and whenever the system is shut down or when part of the system is taken out of service for maintenance. A jar test should be run to save money. By optimizing coagulation and flocculation, a common problem; overdosing which may leads to hurt the quality of the water can be avoided.

4.3 Study the Kinetic Parameters

In this experiment, kinetic parameters such as BOD, COD and TSS is been analyzed. For BOD experiment, the raw sample is diluted referred to Appendix below.

Table 4.6: Estimated BOD Sample Dilutions

Estimated BOD_5 (mg/L)	Suggested sample volumes (mL)
<5	200, 250, 300
<10	100, 150, 200
10-30	25, 50, 100
30-60	15, 25, 50
60-90	10, 15, 25
90-150	5, 10, 15
150-300	3, 5, 10
300-700	1, 3, 5
700-1500	0.5, 1, 3
1500-2500	0.25, 0.5, 1

Notes**:** *Standard methods provide additional guidance as follows: use less than 3mL for strong industrial wastes, 3-15mL for raw and settled wastewater, and 15-75mL for biologically treated effluent.*

Then, the raw sample dilution been mixed with four different types of chemicals; phosphate buffer, magnesium sulfate, calcium chloride and ferric solution. Thus, Dissolved Oxygen (DO) value is being measured. After being stored for 5 days, the sample is been analyzed for Biochemical Oxygen Demand (BOD) reading. The sample is analyzed before entered the reactor and after 4 days are processed by the laboratory digester as effluent. The recorded reading is tabulated in **Table 4.7**.

Next, experiment to analyzed Chemical Oxygen Demand (COD) value by placed the raw sample in the COD Digestion Reagent Vial. The sample is measured the value of COD in the COD Digestion Reactor. The sample is analyzed before entered the reactor and after 4 days are processed by the laboratory digester as effluent. The recorded reading is tabulated in **Table 4.7**.

Then, experiment to determine Total Suspended Solids (TSS) in the raw sample. The sample is filtered by filter paper using vacuum pump. The particulate that left on the filter paper is dried and weighted to obtain the results. The sample is analyzed before entered the reactor and after 4 days are processed by the laboratory digester as effluent. The recorded reading is tabulated in **Table 4.7**.

For collecting the biogas, the apparatus used is a syringe connected to a rubber tube, a glass tube and another rubber tube. The product from the digester consists of carbon dioxide and methane gas. Once carbon dioxide been removed using sodium hydroxide, methane gas can be measured as in table below.

Table 4.7: Kinetic Parameter Values and Methane Gas Collection

Parameter \ Day	Day 0	Day 1	Day 2	Day 3
BOD	35.72	32.68	28.64	25.80
COD	1524.00	1509.00	1485.00	1413.00
TSS	27.2	28.30	29.10	28.70
Methane Gas Collection (%)	-	32.92	44.35	63.49

For the table above, the data shows Day 0 which is the value of raw sample before entering laboratory digester while Day 1, Day 2 and Day 3 are the effluent from the digester.

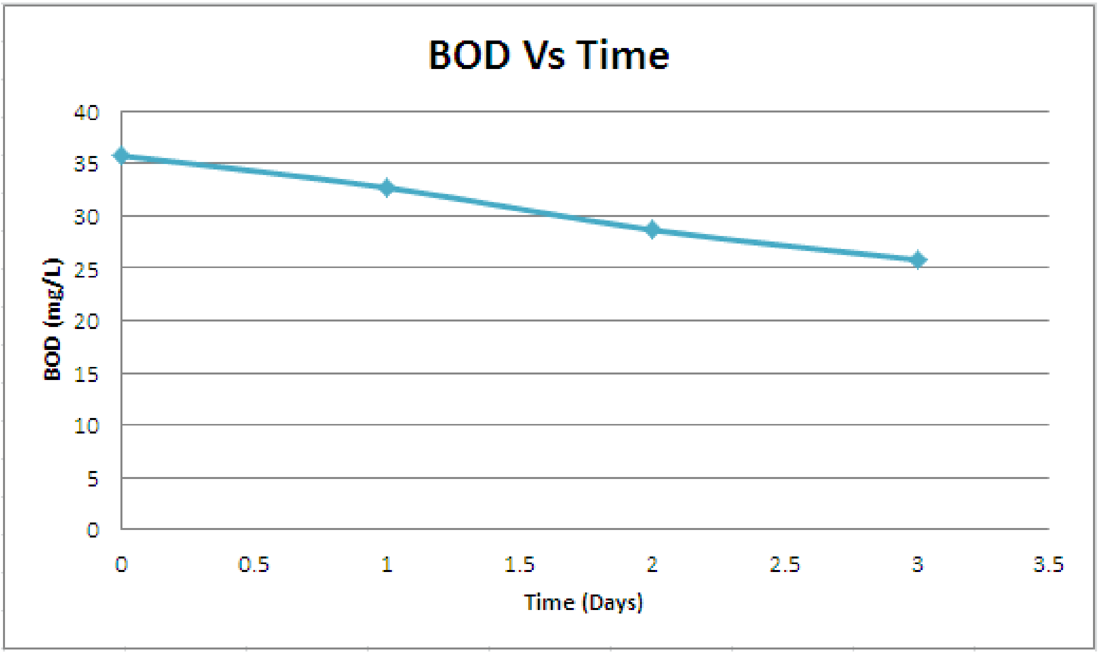

Figure 4.2: Graph Biochemical Oxygen Demands versus Time

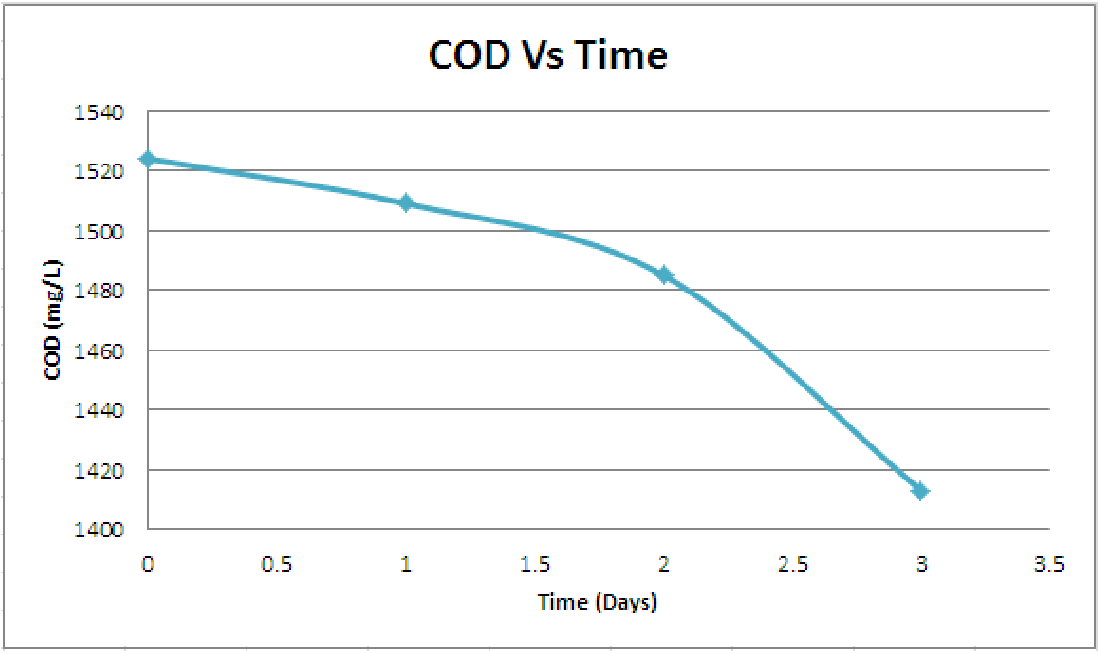

Figure 4.3: Graph Chemical Oxygen Demands versus Time

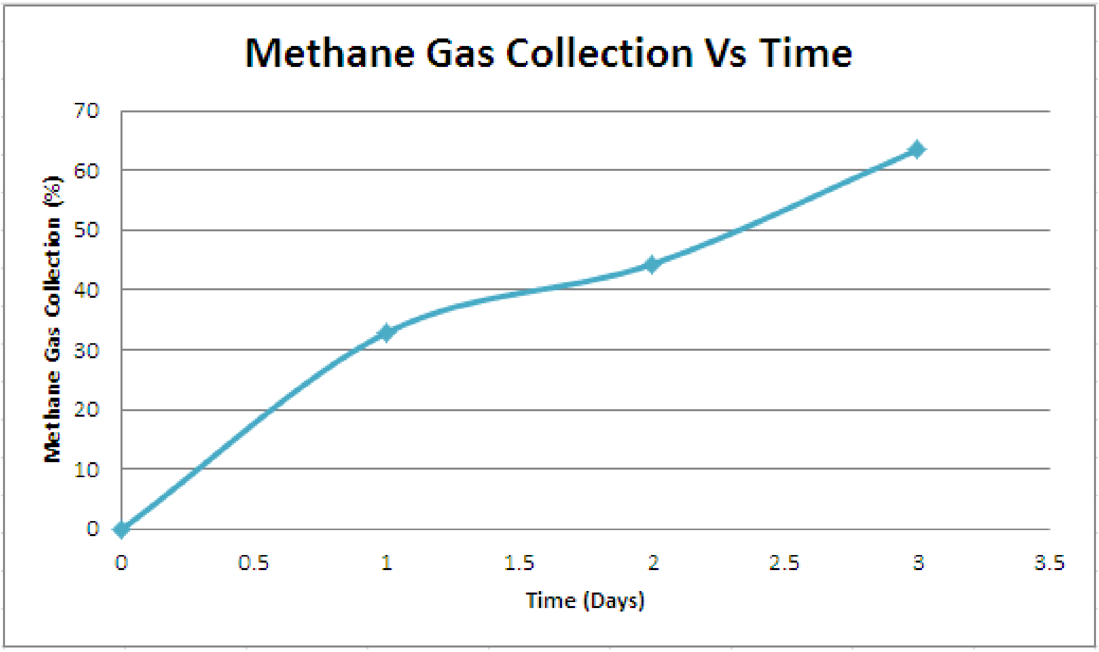

Figure 4.4: Graph Methane Gas Collections versus Time

Based on **Figure 4.2**, the line was created and this shows that when the time increased, the value of BOD will decreased. The lowest value of BOD is at Day 3 which is 25.8 mg/L while the highest value of BOD is 32.68 mg/L at Day 1 .As a summary, this figure showed decreasing value of BOD of 21.1 % from Day 1 to Day 3. Too much BOD can cause excessive growth of bacteria in the wastewater. Low oxygen in the wastewater also encourages the growth of anaerobic bacteria (bacteria which do not require oxygen for growth). Many anaerobic bacteria produce a mucilaginous coating which can quickly clog the leaching field. Thus, excess BOD in sewage can cause a leaching field to function poorly and even to fail prematurely.

In the **Figure 4.3**, the line was created and this shows that when the time increased, the value of COD will decreased. The lowest value of COD is at Day 3 which is 1413 mg/L while the highest value of COD is 1509 mg/L at Day 1 .As a summary, this figure showed decreasing value of COD of 6.4 % from Day 1 to Day 3.

In the **Figure 4.4**, the line was created and this shows that when the time increased, total for methane gas collection is increasing. The lowest value of methane gas collection is at Day 1 which is 32.92 % while the highest value of methane gas collection is 63.49 at Day 3. From the figure, we can concluded that the kinetic parameter; pH is been controlled as the methane gas been captured is more than carbon dioxide. The lower pH value (acidic), the higher percentage of VFA in the wastewater. From this experiment, shows that the percentage of VFA is decreasing with time. As a summary, this figure showed increasing value of methane gas collection of 48.1 % from Day 1 to Day 3.

CHAPTER FIVE

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

As a conclusion from the research, the findings and experiment that have been done and the result obtain will help researcher to improve Production of Methane Gas by Ultrasonic Membrane Anaerobic System (UMAS) using Sewage Sludge as Substrate. The results obtained from these experiments really help the researcher to find the best way to treat waste water. The batch process of this study is a lab scale where this research can be continued by using continuous process. Lastly, these experiments are considered successful. The raw sewage sludge can be treated by using Ultrasonic Membrane Anaerobic System (UMAS).

5.2 Recommendations

Some recommendations have been made to improve the result for future references.

1. Make sure the raw sample is stored and used for experiment not later than 2 weeks which may affect the results.

2. The laboratory digester must be fully covered with aluminum foil to avoid direct sunlight to the bacteria and the valve is fully shut to avoid volume loss.

3. Be careful when collecting biogas from the valve of the reactor that may contain the sample because of the pressure in the reactor. Choose the best valve to collect the biogas.

4. The biogas that been collected should be analyzed for ensuring the contents of the biogas using scientific analysis such as gas chromatography.

5. Another method should be invented to measure the amount of methane gas collected accurately.

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