BIOMETHANATION OF SEWAGE WASTEWATER USING ULTRASONIC MEMBRANE ANAEROBIC SYSTEM (UMAS)

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Thesis submitted in fulfilment of the requirements for the award of the Bachelor Degree in Civil Engineering

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ABSTRAK

Air kumbahan kumbahan adalah air kumbahan keras yang mengandungi permintaan oksigen kimia (COD), permintaan oksigen biologi (BOD), pepejal terampai (TSS) dan pepejal terampai (VSS) yang tidak menentu. Parameter penting ini perlu dirawat terlebih dahulu sebelum ia dilepaskan ke mana-mana laluan air. Terdapat banyak kaedah rawatan yang tersirat sehingga dekad ini dengan pendekatan tradisional. Sistem kolam adalah kaedah paling tersirat kerana kosnya yang rendah. Air buangan kumbahan merawat untuk menghasilkan gas metana dengan menggunakan kaedah pencernaan anaerobik membran. Tetapi akan ada masalah membran membran yang disebabkan oleh sejumlah besar jumlah pepejal yang dibawa oleh air kumbahan. Oleh itu kos operasi loji rawatan itu akan sangat tinggi. Oleh itu, Sistem Membran Anaerobik Ultrasonik (UMAS) digunakan sebagai kaedah alternatif untuk mengatasi masalah ini. Sisa air kumbahan dikumpul dari Kuantan Water Indah dan menyesuaikan diri selama 5 hari sebelum menjalankan reaktor. Barisan air sisa baris direkodkan, COD adalah 164.67 mg / L, dan BOD adalah 17.4 mg / L, dan TSS adalah 31 mg / L, dan VSS adalah 6mg / L, pH, tekanan dan suhu sentiasa berterusan semasa eksperimen dalam nilai 6.5 hingga 7.5, 2 hingga 3 bar dan 35 hingga 46 °C masing-masing. Prestasi Sistem Membran Anaerobik Ultrasonik UMAS dinilai berdasarkan keupayaan (UMAS) untuk merawat parameter ini. UMAS mesti dikendalikan setiap hari selama 5 jam operasi sehari. Percubaan dilakukan apabila UMAS mencapai keadaan mantap. Keadaan mantap dicapai pada hari 5. Prestasi UMAS menunjukkan kecekapan penyingkiran COD yang tinggi dengan 97.4%. dan kecekapan penyingkiran BOD dengan 92%, kecekapan penyingkiran TSS 97% dan kecekapan penyingkiran VSS 99% dan komposisi gas metana yang diperolehi adalah kira-kira 79%. Berdasarkan hasil yang diperoleh selepas sepuluh hari percubaan menunjukkan UMAS dapat merawat kekuatan dan air sisa yang rendah, tanpa masalah pengotoran membran dan menghasilkan gas metana dari air sisa kumbahan. Selain itu, kerja selanjutnya diperlukan untuk memberikan pemahaman yang lebih mendalam tentang mekanisme yang terlibat untuk memudahkan pembangunan sistem optimum yang digunakan untuk bidang loji rawatan air sisa.

ABSTRACT

Sewage wastewater is hard wastewater that contains a high amount of chemical oxygen demand (COD), biological oxygen demand (BOD), total suspended solids (TSS) and volatile suspended solids (VSS). These vital parameters should be treated first before it is discharged into any waterways. There are many treatment methods implied until this decade with the traditional approach. Pond system is the most implied method due to its low cost. The sewage wastewater was treating to produce methane gas by using membrane anaerobic digestion method. But there will be membrane fouling problem caused by high amounts of total solids carried by the wastewater. Therefore the operating cost of the treatment plant it will be very high. Thus, Ultrasonic Membrane Anaerobic System (UMAS) is used as an alternative method to overcome this problem. The sewage wastewater was collected from Kuantan Water Indah and acclimatizing for 5 days before running the reactor, The row wastewater parameter was recorded, COD was 164.67 mg/L, and BOD was 17.4 mg/L, and TSS was 31 mg/L, and VSS was 6mg/L, the pH, pressure and temperature was kept being constant during the experiment within the values of 6.5 to 7.5, 2 to 3 bar and 35 to 46 °C respectively. The performance of Ultrasonic Membrane Anaerobic System UMAS is evaluated on the ability of (UMAS) to treat these parameters. The UMAS must be operated daily for 5 hours operation per day. The experiment is done when the UMAS is achieving a steady state. The steady state is achieved on day 5. The performance of UMAS showed high COD removal efficiency with 97.4%. and BOD removal efficiency with 92%, TSS removal efficiency 97% and VSS removal efficiency 99% and the methane gas composition obtained was about 79%. Based on the result obtained after ten days of the experiment shows that UMAS can treat strength and low wastewater, with no membrane fouling problem and produce methane gas from sewage wastewater. Moreover, further works are required to provide more in-depth understanding of the mechanisms involved to facilitate the development of an optimum system applicable to the field of the wastewater treatment plant.

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LIST OF ABBREVIATIONS

AD	Anaerobic digestion
BOD	Biochemical oxygen demand
CH_4	Methane gas
COD	Chemical oxygen demand
HRT	Hydraulic retention time
MAS	Membrane anaerobic system
TSS	Total suspended solids
UMAS	Ultrasonic membrane anaerobic system
VSS	Volatile suspended solids

CHAPTER 1

INTRODUCTION

1.1 Introduction

Water is essential for life as it is one of the most critical natural resources on the planet. Wastewater, which is primarily used water, is also a valuable resource, especially with recurring droughts and water shortages in many areas in the world. However, wastewater contains many harmful substances and cannot be released back into the environment until it is treated. Thus, the importance of wastewater treatment is to restore the water supply and prevent the environment from pollution and temperature rising and to protect the planet from toxins. There are several types of wastewater produced every day in Malaysia like sewage wastewater and palm oil mill effluent (POME) wastewater, sugarcane wastewater, slaughter wastewater, brewery wastewater. Before treatment processes begin, the values of chemical oxygen demand (COD) and biological oxygen demand (BOD) of any wastewater should be examined and compared it to stander A and stander B according to the Department of Environmental (DOE). If the value of COD and BOD are greater than stander A and stander B, then treatment of wastewater should be taken compulsorily. Wwastewaters treatment plants require a large area and sophisticated treatment process, and their operational cost will be extremely very high (Tiruneh et al., 2014).

Malaysia has a population of 28.3 million based on the Report of Census 2010 by the Department of Statistics. The estimated volume of wastewater generated by municipal and industrial sectors is 2.97 billion cubic meters per year (Mat et al., 2013). The generation of municipal sewage wastewater is increasing every day in Malaysia. The municipal sector is consuming a large volume of water from natural water resources, and consequently generates a considerable amount of wastewater discharge. Municipal sewage, which contains both domestic and industrial wastewater, may differ from place to place depending upon the type of industries and industrial establishment (Iit and Kharagpur, 2000). Sewage wastewater is 99% water, and the rest is containing ions. Sewer is a mixture of water, and water carried wastes originating from homes, industrial facilities and commercial. Sewage wastewater is a major carrier of disease (from human wastes) and toxins (from industrial wastes). Untreated wastewater mainly contains high levels of organic material, numerous pathogenic microorganisms and nutrients and toxic compounds. The pollutant wastewater can cause many problems of pollution thus the safe treatment of sewage it is essential to the health of any community.

An understanding of the nature of wastewater is fundamental for the design of appropriate wastewater treatment plants and the selection of effective treatment technologies. Anaerobic digestion is the most suitable method to treat sewage wastewater. High-rate anaerobic bioreactors have shown better treatment efficiency, and they are producing better-treated effluent with shorter retention times, these anaerobic bioreactors also require less space, as well as greater methane production. Anaerobic digestion happens in the absence of air (and thus molecular/free oxygen) by those microorganisms (also called anaerobes) which do not require air (molecular/free oxygen) to assimilate organic impurities. The final products of biological assimilation in anaerobic treatment are methane and carbon dioxide gas and biomass (Mittal, 2011). Utilizing anaerobic digestion technologies reduce the emission of landfill gas into atmosphere and emission greenhouse gasses and it can replace energy derived from fossil fuels and widely used as a source of renewable energy. Anaerobic digestion is an efficient wastewater treatment technology that harnesses natural anaerobic decomposition to reduce waste volume and generate biogas at the same time. It has been widely applied to the treatment of wastewater from agricultural and industrial operations. Anaerobic digestion has been touted as the best process for the treating of sewage wastewater than other treatment types.

1.2 Problem Statement

With growing concerns over climate change associated with fossil-fuel utilisation, anaerobic treatment of the sewage wastewaters receiving increased attention (Tiruneh *et al.*, 2014). Now a day, the anaerobic treatment system for treating the sewage wastewater before discharging into rivers and other water sources is meager

cost. But the biogases emitted during biodegradation of swage wastewater will be released into the atmosphere. These biogases especially methane gas has high potential to cause global warming and greenhouse effect.

Moreover, anaerobic treatment system requires a large area and has long hydraulic retention time (HRT) (Poh P.E, 2009). Membrane anaerobic system (MAS) can be used to treat the sewage wastewater but there will be membrane fouling problem caused by high amounts of total solids carried by the wastewater. The total suspended solids can reduce the membrane permeability and slow down the flow and decrease membranes performance and the quality of the water will be affected by severe flux declined will take place when the membrane fouling being occurs. To overcome this problem, the membrane can be taken out and cleaned using chemical cleaning method, but that solution will increase the operating cost of the treatment plant. The membrane can be suffered from fouling and degradation during it is continuous usage. Therefore another solution and more economical to overcome this problem, is adding ultrasonic device into the (MAS) system in treating POME wastewater and producing methane (Abdulrahman, 2014). However there is some development to be upgraded to improve the Ultrasonic Membrane Anaerobic System (UMAS) to produce the methane from sewage wastewater.

1.3 Research Objectives

The following objectives were considered in this research

- i. To evaluate the performance of Ultrasonic Membrane Anaerobic System (UMAS) in treating sewage wastewater.
- ii. To evaluate the retention time to the respective parameter (BOD, COD, TSS, VSS, PH).
- iii. To produce methane gas from sewage wastewater.

1.4 Scope of Study

In this study, four scopes were considered in in order to fulfil the research objectives.

i. To design a laboratory scale ultrasonic membrane anaerobic system (UMAS) with an effective 100 litre volume to treat sewage wastewater.

- To monitor parameter such as Biochemical Oxygen Demand (BOD), Chemical Oxygen Demand (COD), Total Suspended Solid (TSS), Volatile Suspended Solid (VSS), and Ph
- iii. To study the effect of the Organic Loading Rate (OLR) in the performance of UMAS
- iv. To evaluate the amount if methane gas produced by the volume of permeates.

1.5 Significant of Study

Inwardly, this research can produce another environment friendly method which is basically Ultrasonic Membrane Anaerobic System (UMAS) to treat sewage wastewater before discharging it into the environment. And can be consider one of the most cost affordable and alternative method compare to the conventional method for treating sewage wastewater. Moreover, the (UMAS) system generates the methane gas (CH4) as final product, which is can be consider as green technology and reduce the emission of the landfill.

CHAPTER 2

LITERATURE REVIEW

2.1 Wastewater Treatment Using Aerobic Bioreactors

The biological treatment process is an essential and integral part of any wastewater treatment plant that treats wastewater from either municipal or industry having soluble organic impurities or a mix of the two types of wastewater sources. The apparent economic advantage, both regarding capital investment and operating costs, of biological treatment over other treatment processes like thermal oxidation; chemical oxidation etc. has cemented its place in any integrated wastewater treatment plant. Biological treatment using aerobic activated sludge process has been in practice for well over a century. Increasing pressure to meet more stringent discharge standards or not being allowed to discharge treated effluent has led to the implementation of a variety of advanced biological treatment processes in recent years. Aerobic means in the presence of air (oxygen). Therefore, aerobic treatment processes take place in the presence of air and utilise those microorganisms (also called aerobes), which use molecular/free oxygen to assimilate organic impurities, i.e. convert them into carbon dioxide, water and biomass (Zia *et al.*, 2018).

2.1.1 Aerobic Activated Treatment Process

Aerobic activated sludge process has been in practice for well over a century. But both of these processes seems to depend on capital cost and skills labour required, which consume more energy and produce more sludge and do not allow recovery of valuable energy and nutrients (Nayono and Perencanaan, 2005). Activated sludge process is the most common method used in the treatment of municipal and industrial wastewaters, which produces a large amount of waste activated sludge (WAS) every year as the final product. The sludge contains a lot of organic matter and nutrients, and issues both related to its treatment and disposal have become very important. To resolve the problems on WAS, many technologies have been developed including the anaerobic digestion (AD) in sewage treatment plants (Li *et al.*, 2017). The merits and demerits of the aerobic treatment process are enumerated in Table 2.1.

6 6	
Advantages	Disadvantages
Volatile solids reduction approximately the same as anaerobic digestion.	Higher power cost associated with supplying oxygen.
Supernatant liquor with lower BOD concentrations.	Produces a digested sludge with poor mechanical.
Production of an odourless, humus-like, biologically stable end-product.	Dewatering characteristics.
Recovery of most of the basic fertiliser values in the sludge.	The process is significantly affected by temperature, location, and type of tank.
Lower capital cost	High operating cost.

 Table 2.1
 Advantages and disadvantages of aerobic wastewater treatment

2.1.2 Anaerobic Treatment Process

The anaerobic treatment process is increasingly recognized as the core method of advanced technology for environmental protection and resource preservation, and it represents, combined with other proper techniques, a sustainable and appropriate wastewater treatment system for developing countries. Anaerobic treatment of sewage is increasingly attracting the attention of sanitary engineers and decision makers (Gitis and Hankins, 2018). Anaerobic digestion of sewage sludge has been applied at wastewater treatment plants (WWTP) for decades. It is a well-known, efficient and environmentally sustainable technology which enables energy production as heat, electricity and vehicle fuel (Momayez et al., 2019). The process of anaerobic digestion starts with the hydrolysis of the sludge followed by fermentation, hydrogen-producing acetogenesis and homo-acetogenesis, until the final product biogas is obtained (Li et al., 2017). Typical sewage sludge comprises of primary sludge separated from wastewater during pre-setting and biological excess sludge from the activated sludge system. Characteristics of sewage sludge differ somewhat in different countries and areas, e.g. due to water consumption and local industry. Total solids (TS) content is usually low and sludge volume high unless some of the water is removed before sludge treatment. Biological stabilization of sludge aims at degradation of volatile solids (VS), the organic content of the sludge, and subsequent decrease in sludge volume.

Moreover, nitrogen and phosphorous contents are important, especially when the stabilized sludge is being reused as fertilizer or soil improver. Sewage sludge contains easily biodegradable materials and its typical methane production potential are approximately 300–400 m3/tVS Lipid-rich materials are known to have high methane production potentials, but their degradation products, long-chain fatty acids (LCFA), may be severely inhibitive to methanogenesis (Rodríguez-Méndez *et al.*, 2017). The inhibition was long assumed irreversible, but lately, it has been proved reversible with increasing consumption of acetate and butyrate indicating the recovery. The advantages and disadvantages of the anaerobic treatment process are enumerated in Table 2.2.

Advantages	Disadvantages
High efficiency: Good removal efficiency can be	Low pathogen and nutrient removal:
achieved in the system, even at high loading rates and low temperatures.	Pathogens are only partially removed, except helminthic eggs, which are adequately captured in the sludge bed. Nutrients removal is not complete, and therefore a post-treatment is required.
Simplicity: The construction and operation of these reactors are relatively simple.	Long start-up: Due to the low growth rate of methanogen organisms, the start-up takes longer as compared to aerobic processes, when no good inoculum is available
Flexibility: Anaerobic treatment can easily be applied on either a very large or a very small scale.	Possible bad odours: Hydrogen sulphide is produced during the anaerobic process, especially when there are high concentrations of sulphate in the influent. Proper handling of the biogas is required to avoid bad smell.
Low space requirements: When high loading rates are accommodated, the area needed for the reactor is very small.	The necessity of post-treatment: Post-treatment of the anaerobic effluent is generally required to reach the discharge standards for organic matter, nutrients and pathogens.
Low energy consumption: As far as no heating of the influent is needed to reach the working temperature and all plant operations can be done by gravity, the energy consumption of the reactor is almost negligible. Moreover, energy is produced during the process in the form of methane.	
Low sludge production: The sludge production is low when compared to aerobic methods, due to the slow growth rates of anaerobic bacteria. The sludge is well stabilized for final disposal and has good dewatering characteristics. It can be preserved for long periods without a significant reduction of	
activity, allowing its use as inoculum for the start-up of new reactors. Low nutrients and chemicals requirement:	
Especially in the case of sewage, an adequate and stable pH can be maintained without the addition of chemicals. Macronutrients (nitrogen and	
phosphorus) and micronutrients are also available	

 Table 2.2
 Advantages and disadvantages of anaerobic wastewater treatment

2.2 Chemical Oxygen Demand (COD)

Chemical oxygen demand (COD) and it is a quantity of oxygen required to oxidize all organic material into carbon dioxide and water and ammonia completely. In another word Chemical oxygen demand is defined as the amount of a specified oxidant that reacts with the sample under controlled conditions. The quantity of oxidant consumed is expressed regarding its oxygen equivalence. Because of its unique chemical properties, the dichromate ion (Cr_2O_7) is the specified oxidant and is reduced to the chromic ion (Cr^{+3}) . Both organic and inorganic components of a sample are subject to oxidation, but in most cases, the organic component predominates and is of the greater interest. Thus the COD is a measure of oxygen equivalent of the organic matter as well as microorganisms in wastewater. If the value of COD is greater than the value of BOD value, the sample contains a large amount of organic compounds that are very difficult to degrade (Seghezzo *et al.*, 1998)

2.3 Controlling Factors in Anaerobic Digestion

Anaerobic digestion is affected by several components are temperature, pH, retention time, the chemical composition of wastewater, the competition of methanogens with sulphate-reducing bacteria, and the presence of toxicants (Rajesh et al., 2000).

2.3.1 Temperature

Anaerobic digestion is strongly influenced by temperature and can be grouped under one of the following categories: psychrophilic (0-20°C), mesophilic (20-42°C) and thermophilic (42-75°C). The details of the bacterial processes in all the three temperature ranges are well established though a large section of the reported work deals with the mesophilic operation. Changes in temperature are well resisted by anaerobic bacteria, as long as they do not exceed the upper limit as defined by the temperature at which the decay rate begins to exceed the growth rate. In the mesophilic range, the bacterial activity and growth decrease by one half for each 10C drop below 35°C. Thus, for a given degree of digestion to be attained, the lower the temperature, the longer is the digestion time. The effect of temperature on the first stage of the digestion process (hydrolysis and acidogenesis) is not very significant, as among the mixed population there are always some bacteria which have their optimum within the range concerned. The second and third stages of decomposition can only be performed by certainly specialized microorganisms (acetogenic and methanogenic bacteria), and thus, these are much more sensitive towards temperature change. However, an important characteristic of anaerobic bacteria is that their decay rate is very low at temperatures below 15°C. Thus, it is possible to preserve the anaerobic sludge for long periods without losing much of its activity. This is especially useful in the anaerobic treatment of wastewater from seasonal industries such as sugar mills (Rajeshwari *et al.*, 2000). The municipal wastewater treatment plant, anaerobic digestion is carried out in the mesospheric range at a temperature (25°C to up to 40°C), with an optimum at approximately 350 C. Thermophilic digestion operates at temperatures range of (50 -65°C). It allows higher loading rates and is also conducive to greater destruction of pathogens. One drawback it is higher sensitivity to toxicant (Koster and Leopold, 1988).

2.3.2 pH

Anaerobic reactions are highly pH dependent. The optimal pH range for methane-producing bacteria is (6.8-7.2), and the process may fail if the pH near 6.0, while for acid-forming bacteria, a more acid pH is desirable. The pH of an anaerobic system is typically maintained between methanogenic limits to prevent the predominance of the acid-forming bacteria, which may cause VFA accumulation. It is essential that the reactor contents provide enough buffer capacity to neutralize any eventual VFA accumulation, and thus prevent the build-up of localised acid zones in the digester. In general, sodium bicarbonate is used for supplementing the alkalinity since it is the only chemical, which gently shifts the equilibrium to the desired value without disturbing the physical and chemical balance of the fragile microbial population (Rajeshwari *et al.*, 2000).

2.3.3 Retention Time (RT)

Tian et al., (1994) reported that It is well known that the hydraulic retention time (RT) of a digester is one of the most critical factors for the control of anaerobic digestion systems. The Hydraulic Retention Time (HRT) which depends on the wastewater characteristics and environmental conditions, must long enough to allow metabolism by anaerobic microorganisms indigestion. The Retention Time of

thermophilic and mesophilic digesters are ranged between 25 to 35 days but can be less time. Hydraulic Retention Time is effectively how long the liquid/biomass remains in the reactors. The HRT doesn't have to equal SRT, but when HRT equals SRT, the HRT must be \geq Biomass generation time, typically greater than 15 days. Lower HRT higher throughput means better treatment times; longer SRT will result in higher Solids concentration (more biomass), a higher percentage of COD conversion to methane, lower daily methane production rate and greater stability. The HRT in days is equal to the volume of sludge in the digester (m³⁾ divided by the volume of digested sludge withdrawn daily (m³/day).

2.3.4 Toxicants

As an efficient waste treatment technology that harnesses natural anaerobic decomposition to treat waste, reduce waste volume and generate biogas as well, anaerobic digestion has been widely used as a source of renewable energy. However, anaerobic digestion can be inhibited to varying degrees by toxic materials present in the system; these substances may be components of the influent waste stream or byproducts of the metabolic activities of the digester bacteria. Inhibitory toxic compounds include organics, ammonia, sulfide, heavy metals, and the emerging nanomaterial's, and are often present in the processing of wastes from agricultural and industrial operations such as molasses fermentation, petroleum refining and the tanning industries. These toxic compounds principally obstruct the activities of the sensitive obligate hydrogen producing acetogens and methanogenic portions of the digester population, as well as cause retarded methane formation, a decrease in the methane content of biogas, or can even cause complete failure of methanogenesis (Rodríguez-Méndez et al., 2017). However, because of the difference in anaerobic microorganisms and waste composition, results from previous studies on the inhibition of anaerobic processes vary substantially. Also, better understanding the mechanism(s) of inhibition or toxicity of different toxicants in an anaerobic digester provides insights into overcoming these toxic effects and possible solutions or strategies to cope with it properly, successfully apply anaerobic digestion and significantly improve waste treatment efficiency. On the other hand, measuring the toxicant concentration and monitoring them is an essential precautionary strategy (Chen et al., 2014).

2.4 Microbial Communities

Anaerobic digestion consists of a series of microbial processes that convert organics to methane and carbon dioxide and can take place under psychrophilic (20 °C), mesophilic (25-40°C) or thermophilic (50-65°C) conditions, although biodegradation under mesophilic conditions is most common. It also enables higher loading rates than aerobic treatment and greater destruction of pathogens. (Chen *et al.*, 2014) The microorganisms that function within the Anaerobic Digestion process can be mainly classified as hydrolytic, fermentative, acetogenic, and methanogenic. As shown in Fig 2.1 (Li et al., 2011).





2.4.1 Hydrolytic Bactria

In the hydrolysis phase, hydrolytic bacteria reduce complex particulate compounds to soluble monomeric or dimeric substrates. Generally, most of the soluble organic material in the reactor medium is converted to volatile organic acids through fermentation and eventually processed into biogas through methanogens. Hence, hydrolysis is a critical rate-limiting step that determines the conversion efficiency of the biomass feedstock. Cellulose, found in many agricultural and municipal wastes, is an example of an insoluble compound that undergoes enzymatic hydrolysis. Cellulolytic bacteria such as *Cellulomonas, Clostridium, Bacillus, Thermomonospora,*

Ruminococcus, Bacteroides, Erwinia, Acetovibrio, Microbispora, and Streptomyces can produce celluloses that hydrolyse cellulolytic biomass.

2.4.2 Fermentative Bacteria

Fermentative bacteria are responsible for consuming the soluble created from hydrolysis and producing various intermediates such as VFAs, carbon dioxide, hydrogen gas and alcohols. Some of the fermentation pathways that occur during Anaerobic Digestion, along with their corresponding microorganisms, are shown in Table 2.3. Among the products of fermentation, acetate and carbon dioxide contribute the most to methane production.

c c	-	-
Fermentation	Genera	Major products
pathway		
Acetate	Acetobacterium, Clostridium,	Acetate, CO_2
fermentation	Sporomusa	
Alcohol	Saccharomyces	Ethanol, CO_2
fermentation		
Butyrate	Butyribacterium, Clostridium	Butyrate, butanol, isopropanol,
fermentation	-	ethanol, CO ₂
Lactate	Lactobacillus, Streptococcus	Lactic acid, CO_2
fermentation	-	
Propionate	Clostridium	Propionate, acetate, CO_2
fermentation		-

Table 2.3Major genera of fermentative bacteria in anaerobic digestion

2.4.3 Acetogenic Bacteria

Acetogenic bacteria, or acetogens, are differentiated from acetate-forming fermentative bacteria mainly because of their capability to reduce carbon dioxide to acetate using a wood-Ljungdahl pathway. There are bacterial genera that are exclusively acetogenic, such as Acetobacterium and Sporomusa, and there are also genera that contain both acetogenic and nonacetogenic bacteria, such as Clostridium, Ruminococcus, and Eubacteria. A combination of the vital role of acetate as a methanogen substrate as well as the ubiquity and diversity of acetogens makes Anaerobic Digestion a naturally robust phenomenon. However, acetogens are obligate hydrogen producers that cannot survive in high partial hydrogen pressures. Thus a symbiotic relationship exists between acetogens that produce hydrogen and methanogens that consume it (Li et al., 2011).

2.4.4 Methanogens

The final stage of methanogens produces methane by two groups of methanogenic bacteria: the first group splits acetate into methane and carbon dioxide and the second group uses hydrogen as an electron donor and carbon dioxide as an acceptor to produce methane. Appels, et al., (2008) reported that the methanogens in most of the cases, the rate-limiting step of the overall process. Anaerobic digestion of organic matter in environmental releases approximately 500 million tons of methane per year into the atmosphere, representing about 0.5% of the organic matter derived from photosynthesis.

2.5 Scales of Anaerobic Process

There are majorly two types of anaerobic process, and these include the mesospheric and thermophilic digestion as discussed in the preceding sections.

2.5.1 Mesospheric Digestion

Mesophilic are microorganisms such as some species of Bacteria, Fungi, and even some Archaea that are best active at median temperatures. For instance, bacterial species involved in biodegradation (i.e., digestion and decomposition of organic matter), which are more active in temperatures ranging from approximately $70^{\circ} - 90^{\circ}$ F (approx. 15° – 40° C), are termed mesophilic bacteria. They take part in the web of micro-organic activity that forms the humus layer in forests and other fertile soils, by decomposing both vegetable and animal matter. In general, mesospheric anaerobic digestion of sewage sludge is more widely used compared to thermophilic digestion, mainly because of the lower energy requirements and higher stability of the process. Moreover, another disadvantage of mesophilic digestion is that it does not reduce the pathogen concentrations enough to produce Class A biosolids, a biosolid that contain no detectable levels of pathogens (Gavala et al., 2003).

2.5.2 Thermophilic Digestion

The anaerobic digester that operates at the higher thermophilic temperature range (50 - 65° C) is known as thermophilic digestion. Interest in the thermophilic digestion developed based on the facts that higher temperatures reduce pathogens and

thermophilic temperatures provide more rapid reaction rates than mesophilic temperature. Class A quality bio-solids can be produced from thermophilic digestion. Thermophilic anaerobic digestion, in general, are more efficient in biogas production but associated with higher maintenance cost. However, the thermophilic anaerobic digestion process is usually characterized by accelerated biochemical reactions, the higher growth rate of microorganisms and accelerated interspecies hydrogen transfer resulting in an increased methanogenic potential at lower hydraulic retention times. Also, the enhanced hygienization effect of the thermophilic process complies with the EU policy for the elimination of pathogens, originating mainly from humans and animals; it has been reported that thermophilic anaerobic digestion of sewage sludge can lead to EPAs class The bio-solids, which are suitable for subsequent land application, thermophilic anaerobic digestion of sewage sludge can lead to EPAs class A bio-solids, which are suitable for subsequent land application (Xue *et al.*, 2015).

2.6 Membrane Technology

Membrane bioreactors were initially developed in the 1960s when commercialscale UF and MF membranes became available. The original process was introduced by Dorr-Olivier Inc. (Milford, Connecticut) and combined a cross-flow membrane filtration loop with an activated sludge bioreactor. A membrane is defined as a thin selective barrier between two phases (gas or liquid), which is impermeable to the transfer of specific particles, molecules or substances, colloidal, and dissolved chemical species other than water or solvent. A material of reasonable mechanical strength that maintains a high throughput of a desired permeates with a high degree of selectivity is ideal for the production of membranes. Usually, a thin layer of material with a narrow range or domain of pore size and a high surface porosity affect the physical structure of the membrane. The physical structure of a thin layer membrane leads to the separation of dissolved solutes in liquid streams and the separation of gas mixtures for membrane filtration. Membrane processes or productions are categorised based on (1) the driving force which is used for separation of impurities such as pressure (P) temperature (P), concentration gradient (C), partial pressure (p), or electrical potential (E), (2) the mechanism of separation, (3) the particular application of membrane, (4) the size of the retained material, and (5) the type of membrane.

2.6.1 Membrane Bioreactor Configurations

There are two membrane configurations used in the membrane system. The first configuration is side-stream (external) membrane bioreactors (Figure 2.2) and the second one is submerged MBR (the membrane is immersed directly into bioreactor) (see Fig. 2.2). The second one is more applicable in wastewater treatment than the first one because it has many advantages such as lower energy.



Figure 2.2 Side-stream membrane bioreactor with the external pressure-driven membrane unit



Figure 2.3 Submerged Membrane Bioreactor with internal vacuum pressure-driven membrane filtration

The side-stream configuration is possibly used for wastewater treatment, with wastewater (feed) is pumped into the membrane and part of the permeate is collected while the other part is returned to the MBR. Side-stream configuration can control membrane fouling significantly; resulting in constant flux but the energy consumption and intricate design are the significant limitations. The arrangements of MBR are based on geometry (either cylindrical or planar). There are five major membrane configurations currently used in practice viz: hollow fibre (HF), spiral-wound, plate-and-frame (FS), pleated filter cartridge and the tubular (Ladewig et al., 2017).

2.6.2 Advantages and Limitations of Membrane Bioreactors

Membrane bioreactors have attracted extensive attention as a result of their numerous advantages over CASP. The advantages of MBRs include excellent treated water quality, high biodegradable efficiency, small footprint and reactor requirements, absolute biomass retention and ease of stable operation. They can also display high effluent quality, flexible operation, absolute removal of bacteria, high volumetric loading up to 20 kg COD/m³ per day, excellent disinfection capability and turbidity less than 0.5 NTU (number transfer unit), low sludge production, compactness, enable high removal efficiency of biological oxygen demand (BOD) and chemical oxygen demand (COD). As a result, the MBR process has now become a viable alternative for the treatment and reuse of municipal and industrial wastewaters. MBRs are therefore considered a promising tool for future wastewater treatment (Meng et al., 2009). However, alongside these advantages, MBR technology is affected by crucial issues that severely hamper the performance and the widespread applications of MBRs. Membrane fouling, that is the undesirable deposition of retained particles, colloids, macromolecules and salts on the membrane surface or the membrane pores, is the most critical disadvantage. Specifically, membrane fouling results in a reduction of separation process output, diminishes process productivity, severe decline of the permeation flux or rapid trans-membrane pressure increase (TMP), leading to high energy consumption, frequent membrane cleaning or replacement, which consequently leads to the increase in operating and maintenance cost (Huang et al., 2018).

2.6.3 Membrane Fouling

While filtrating of source waters, the membrane is prone to losing permeability because of the accumulation of impurities (physical, chemical, and bio-substances) on or inside the membrane matrices. It is named membrane fouling which is the most limiting factor for wider application of the membrane. Membrane fouling and contaminant removal efficiency constitute. Membrane fouling which is just responsible for the permeability yields no effect on the water quality. The possible explanation is that the fouling layer does not change or destroy the properties of the membrane fouling and the quality of treated water. Identification of foulants is important to membrane fouling and the quality of membrane fouling. Based on the existing knowledge, fouling can be categorized according to the type of foulants as follows:

- i) Particles are fouling: This is considered to be formed by two classical blocking laws. Firstly, accumulation of larger particles on the membrane surface and smaller inside the pores of the membrane; secondly, cake formation with more and more particles precipitated on the initial layer to the level of creating high resistance of membrane flux.
- ii) Organic fouling: This is considered to be caused by natural organic matter (NOM) from the source waters, is not well elucidated. NOM is ubiquitous in natural waters, and its removal is still at issue. It can be classified by molecular weight or different hydrophobicity. Concerning molecular weight (MW), the fouling order of NOM is: media to low MW fraction of NOM; then high MW fraction blocked on the surface. Some researchers are devoted to the effect of the hydrophobicity of NOM on organic fouling; it was found that the mechanisms accounting for the fouling by a hydrophobic fraction, hydrophilic part component were concentration polarization, adsorptive fouling and cake layer deposition, respectively. The dissolved organic matter (DOM) gave rise to more severe fouling than the sum of fouling from each independent DOM. It was demonstrated that a possible adverse interaction existed, though the fouling modes of DOM were not totally independent.
- iii) Moreover, studies of organic fouling are also concentrated on the fractionation methods to investigate which fraction of NOM is responsible for the fouling.

Gel permeation chromatography series resins, different pore size membranes, dialysis bag, and size exclusion chromatography, are used to fractionate the NOM according to MW, hydrophobicity or other characteristic of the NOM. However, for the different property of the ultrafiltration membrane, NOM fraction presents different influence. The specific effect of the properties of NOM on fouling of ultrafiltration is not yet elucidated due to the complex or unknown speciation in NOM from the natural waters. Limited details about the behaviour of NOM.

iv) **Bio-fouling**: This stems from aquatic organisms, such as algae, which can form colonies and then cause bio-fouling. Due to the lack of data (possibly because of the periodic cleaning with chlorine, which may kill the organisms before the fouling happen) concerning bio fouling of membrane, it is unclear what the specific or possible bio-fouling mechanism is. Hiroshi et al. classified the membrane fouling as physically reversible fouling which can be totally eliminated by physical cleaning or certain pre-treatment and physically irreversible fouling which cannot be entirely counteracted by physical cleaning or certain pre-treatment. The irreversible fouling can explain the gradual increase of membrane filtration resistance after running a long period, although the physical cleaning and effective pre-treatment are routinely implemented. The study of fouling models is significant for the better understanding of the fouling mechanism and better predicting of fouling formation and would provide a useful tool for practical design and operation. For fouling models of low-pressure membranes, related issues have been extensively reviewed (Gao et al., 2011).

2.6.4 Membrane Process Classification

Table 2.4 shows the different types of the membrane process, specifications and their applications.

Type of	Size of material	Driving	Type of	Application
process	retained	force	membrane	
Ultrafiltration	1-100 nm	1-10 bar	Microporous	-Separation of
	macro-			protein and virus
	molecules			-The concentration
				of oil in water
				emulsions
Microfiltration	0.1-10 µm	0.5-2 bar	Porous	Separation of
	microparticles			bacteria and cells
				from solutions
Nanofiltration	0.5-5 nm	10-70 bar	Microporous	-Separation of Dye
	molecules			and sugar
_				-Water softening
Reverse	<1 nm	10-	Nanoporous	-Desalination of the
osmosis	molecules	100bar		sea and brackish
				water
				-The process of
D' 1 '	.1		ъ <i>с</i> :	water purification
Dialysis	<1 nm	ΔC	Microporous or	Purification of blood
F1	molecules		Nanoporous	C
Electrodialysis	<1 nm	ΔE	Microporous or	Separation of
Democration	molecules		Nanoporous	Debudration of
Pervaporation	-	ΔC	Nanoporous	Denydration of
				solvents
Gas separation	-	Partial	Nanoporous	Hydrogen recovery
		pressure		from process gas
		difference		streams, dehydration
		(1-100		and separation of air
		bar)		
Membrane	-	ΔT	Microporous	Water purification
distillation				and desalination

 Table 2.4
 Classification of membranes and their inherent applications

CHAPTER 3

METHODOLOGY

3.1 Introduction

In this chapter, specific focus was given on describing the experimental design, the operational methods used and the procedures of different experiments, which needed to be done for this research. It is also together with sample collection and preservation, the analytical methods and calculations for the sewage wastewater samples.

3.2 Experimental procedures

The Ultrasonicated membrane Anaerobic System (UMAS) consisted of a crossflow ultra-filtration membrane (CUF) with 2 membranes, a centrifugal 54 pump, an anaerobic digester of effective volume of 180 L and 6 ultrasonic transducers that were fasten to the membrane unit holder and linked to one unit of 250 watts 25 KHZ Crest's Genesis Generator as shown in Figure 3.1 and Figure 3.2. The UF has 2000 Daltons of molecular weight cut-off (MWCO), a mean pore size of 0.1 µm with each tube was 30 cm at length and a diameter of 1.25 cm. The overall area of the membrane was 0.048 m2. The ultimate operating pressure on the membrane was 55 bar at 70°C, and it works at all pH ranges. The reactor was consisting of a heavy-duty reactor with 15 cm inner diameter and 100 cm length. The operating pressure in this research was preserved in the range of 1.5 bar and 2 bar by tampering on the gate valve at the retentate line after the CUF unit.



Figure 3.1 A schematic diagram for the ultrasonicated membrane anaerobic system.

3.3 Steady State Determination and Feed Preparation

Seven steady states are achieved using the ultrasonicated membrane anaerobic system (UMAS) with the operating conditions were the temperature in the mesophilic range 25°C-35°C, which is the room temperature and pH in the range 6.5-7.8, which are the optimum conditions for the anaerobic reactions (Bhargava, 2016). The laboratory digester is completely mixed-semi continuous following steady-state operation. The raw sewage wastewater is being fed continuously from feeder tank on the top of the reactor by gravity flow. The sewage wastewater was screened through screener before being added to the digester to avoid clogging and pump damage. Then, daily samples were analyzed to determine the TSS, VSS, COD, BOD, pH and for the raw feed permeate and the content from inside the reactor.

3.4 Analysis of Feed Wastewater

3.4.1 Biochemical Oxygen Demand (BOD)

The biochemical oxygen demand was specified by analysing the oxygen depletion after sample incubation at 20°C for five days. As described in (the standard method; 5210B). BOD is the amount of oxygen consumed by organisms for breaking down the organic materials, which are usually found in a wastewater plant. During the BOD test, the sewage wastewater sample was poured in a 300 mL BOD bottle. The bottle was then topped up with dilution water satiated in oxygen and rich with the

necessary nutrients needed for biological growth. The concentration of the BOD for the sample was determined as Eq. (3.1)

$$BOD = \frac{D1 - D2}{P}$$
(3.1)

Where BOD is Biochemical Oxygen Demand (mg/L), D_1 is D_0 of the diluted sample at the time of dilution (mg/L), D2 is DO of a diluted sample after five days' incubation at 20 °C (mg/L), P is a fraction of sample volume to the total volume.

3.4.2 Chemical Oxygen Demand (COD)

The Chemical Oxygen Demand (COD) for all samples were determined by the dichromate reflux (HACH Water Analysis Method). Chemical Oxygen Demand is a measurement for the quantity of oxygen needed for the oxidation of compounds in the water. The experiment of Chemical Oxygen Demand (COD) is applied widely for mensuration of the quantity of organic compounds in water (organic strength of wastewaters) indirectly. The experiment concept is that most of the organic components could be oxidized by a strong oxidizing agent in high acidic conditions. The chemical oxygen demand has been tested directly utilizing HACH equipment LR (3-150) mg/L COD and HR (20-1500) mg/L COD. For COD test, the original sample was diluted to close the vial results. The sample vials and blank vials were identified and shaken strongly, then were put in COD reactor at 150°C for two h. Following their removal from the reactor, the vials were specified by utilizing the Spectrophotometer program DR 3900 by setting the blank vial as zero. The removal efficiency Removal (%) was calculated from the Eq. (3.2).

Removal percentage (%) =
$$\frac{(C_0 - C_t)}{C_0} X100$$
 (3.2)

Where C_0 = is the initial COD of sample; C_t = is the COD of sample at time (t).

The dedicated temperature profiles HI 839800 was used to heat the vial reagent COD HR (Figure 3.2). Also the



Figure 3.2 Dedicated temperature profiles HI 839800.

The HACH DR3900 Spectrophotometer (Figure 3.3) was used to analyse the COD HR value of the vial reagent.



Figure 3.3 DR3900 portable spectrophotometer

3.4.3 Volatile Solids

The sample was fully shaken and then strained over a weighed standard glass fibre filter. The residual on the filter was dried up to a stable weight in an oven at (103-105) °C and then cooled down in a desiccator to balance temperature, and weighed again. After that, the dried solids burned for one hour at (550 ± 50) °C in a furnace. The difference between the weights of ash and dish is the weight of suspended solids. The VSS can be calculated using the Eq. (3.3).

$$VSS = \frac{(A-B)X1000}{sample \ volume.mL}$$

where VSS is volatile suspended solids (mg/L), A is the weight of dried remain + filter + dish before burning (mg), B is the weight of dried residue + filter + dish after burning (mg).

(3.3)

3.4.4 Total Suspended Solid (TSS)

Before the TSS experiment is done. 2 filter disks (for treated and permeate sample) are first discharged in the oven for about an hour at a temperature of 103 °C to 105 °C. The filter disks are then cooled down inside the desiccators for a few minutes weighed using the analytical balance the filtering was set up at the suction was started by switching on the vacuum pump 50 ml of each sample were permeate onto the filter disk through a Buchner flask, using gentle suction (under vacuum). The filter disk was washed with 10 ml of distilled water allowing a complete drainage between washing, and the sockets continue for about 3 minutes after the filtration is completed. The filter disks are then placed inside the aluminium weighing disks for support and were left in the oven for 1 hour with the same temperature range of $103 \,^{\circ}C - 105 \,^{\circ}C$. Then, both samples were left inside the desiccator to cool down before it is weighed again. The total suspended solid is calculated using the Eq. (3.4).

$$TSS = \frac{(A-B)}{\text{sample volume mL}} x1000$$
(3.4)

Where TSS is suspended solids (mg/L), A is weight of dish + paper + dried residue (mg), B is weight of dish + paper (mg).

3.4.5 Gas Measurement

The percentage of methane gas in the produced biogas was measured using J tube gas analyzer as shown in Figure 3.4. The produced biogases comprise mainly CO2 and CH4. Then, sodium hydroxide was absorbing the CO2. The residual volume is methane gas CH4. The device consisted of a glass-tube connected by a flexible hose to a syringe. The syringe was initially full of 0.5 M NaOH solution, the glass tube was inserted into the gas area of the reactor, and then, a column of biogas entered the glass-

tube until it reaches a certain mark. Then, the end of the tube was immediately submerged in water. By tampering the syringe many times, the NaOH solution absorbed the carbon dioxide CO2, leading to a decrease in the extent of the biogas column, and then the biogas column was measured again.

$$Methane (\%) = \frac{Final \ length \ of \ gas \ column}{Initial \ length \ of \ gas \ column} x100$$
(3.5)

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Results at Pre-acclimation and Acclimation

The initial test for BOD, COD and TSS, VSS, pH and temperature were conducted on the row water the result shows in Table 4.1. The anaerobic reactor was prepared and fully covers by the aluminium foil to avoid sunlight entering which could make an effect for the reaction as described in *Appendix A1*. The reactor was loaded with 99L of sewage wastewater and kept inside the reactor for five days where the acclimation process took place. And the parameter was tested after that for BOD, COD and TSS, VSS, pH and temperature were found to be as shown in Table 4.2

 Table 4.1
 Results from pre-acclimated feed wastewater

T (°C)	pН	BOD	COD	TSS	VSS
		(mg/L)	(mg/L)		
39	6.89	15	167	46	

Table 4.2Results from acclimated of the feed wastewater

T (°C)	pН	BOD (mg/L)	COD (mg/L)	TSS	VSS
35	7.05	17.4	164.7	31	35

The properties of the water have slightly change from the row water (Figure 4.1) has fully described the differences between the row water and the row water after the acclimation that happen inside the reactor. Before the experiment started, the pressure, pH and temperature were kept constant with 1.5-2.0 bar, 6.5-7.5 and 32 °C- 49 °C

respectively. For hydraulic retention time (HRT) of 5 days, the experiment was run for five days and removal efficiency was obtained for the BOD, COD and TSS, VSS from the reactor and permeates water and methane gas was measured for every run of the experiment. The total time for investigation was ten days which is divided into two periods, the first five days was using Membrane Anaerobic System (MAS) and other five days was using Ultrasonic Membrane Anaerobic system (UMAS) to be comparative with each other.



Figure 4.1 Feed wastewater before and after acclimation



Figure 4.2 Clean water after filtration

4.2 Parameters Obtained from MAS and UMAS Removal Activities

In this study, the reactor was loaded with 99L of sewage wastewater and entirely covered by aluminum foil to avoid sunlight from entering the reactor which may affect the reactions. For the hydraulic retention time (HRT) of 5 days, the experiment was run for 5 hours daily, during the experiment the pressure and pH and temperature were kept constant with 1.5 to 3 bar and 6.5 to 7.5 and 35 to 46 °C respectively. The first five runs was using Membrane Anaerobic System (MAS) and the second run was using Ultrasonic Membrane Anaerobic system (UMAS) as carefully discussed in the proceeding sections.

4.2.1 Comparative Study of BOD, and COD Removal Efficiencies Using MAS and UMAS

The initial of COD for the sewage wastewater was measured, where 164.67 mg/L was recorded after five days of acclimation process. Figure 4.2 showed that the reactor was run for 5 hours continuously, and the COD removal efficiency was 11.9% in the first run of the experiment. The COD removal efficiency reached 85.5% in 5th run by using (MAS). But when the (UMAS) was used the COD removal efficiency was 46.9 % in the first run of the experiment. The COD removal efficiency reached 98 % in 5th run when the UMAS was used. There was a large increase in the COD removal efficiencies from day 1st to day 5th, which is attributed to the un-acclimatized of the effluent consisting mostly of aerobic bacteria. This requires some time to adapt to the anaerobic condition in the reactor before it accumulates a large enough anaerobic bacteria population to breakdown the influent COD as reported by Cronin (1991). The result obtained showed a greater in COD removal efficiency of UMAS when compared with that of MAS. This showed a greater performance when an ultrasonic was incorporated into the reactor.



Figure 4.3 COD Removal Efficiency for MAS and UMAS

In Figure 4.3 the BOD removal efficiencies increase in direct proportionality with the HRT for the treatment of the sewage wastewater. Using MAS, the initial BOD for the first run of the experiment was calculated to be 15.15 and 19.53 mg/L for the permeate and wastewater, respectively. This increases correspondingly until it reached a threshold value of 5.07 and 16.29 mg/L for the permeate and wastewater, respectively. However, by using UMAS, the initial BOD for the first run of the experiment was calculated to be 5.73 and 11.31 mg/L for the permeate and wastewater, respectively. This increases correspondingly until it reached a threshold value of 2.0 and 14.3 mg/L for the permeate and wastewater, respectively. This increases correspondingly until it reached a threshold value of 2.0 and 14.3 mg/L for the permeate and wastewater, respectively. In both cases, the higher BOD removal efficiency was obtained as 93 % at the 5th day when using UMAS when compared with MAS (69.6 %). The result obtained showed a greater in BOD removal efficiency of UMAS when compared with that of MAS. This indicated a greater performance when an ultrasonic was incorporated into the reactor.



Figure 4.4 BOD Removal Efficiency for MAS and UMAS

4.2.2 Comparative Study of TSS, and VSS Using MAS and UMAS

Figure 4.4 showed that TSS reached 93.8 % removal efficiency was attained when the MAS has was used while 98.0 % removal efficiency achieved when UMAS applied at the 5th run. The TSS, therefore, showed very high removal efficiency when UMAS was incorporated into the reacting system.



Figure 4.5 TSS Efficiency for MAS and UMAS

Moreover, in the case of VSS, a threshold removal efficiency of 97.6% and 99.5 % was achieved when MAS and UMAS were used, respectively after the 5th run as depicted in Figure 4.5 below.



Figure 4.6 VSS Efficiency for MAS and UMAS

4.2.3 Comparative Study of Methane Gas Obtained via MAS and UMAS

For the stability of the anaerobic reactor, it is essential to determine the composition of methane gas inside the reactor. Figure 4.6 shows the percentage of methane composition after the 5th run within MAS and UMAS system as 75% and 79%, respectively. This indicated that the application of ultrasonic membrane results in improved performance of the reactor about the methane gas produced.



Figure 4.7 Methane Gas for MAS and UMAS

CHAPTER 5

CONCLUSION

5.1 CONCLUSION

Based on the result obtained, it shows that membrane fouling does not occur while using the ultrasonic device as support for membrane anaerobic system (MAS). UMAS is only adequate for the biological treatment of high strength wastewater such as sewage wastewater and POME and suitable to treat low strength wastewater. The COD removal efficiency reached 85.5% in 5th run by using (MAS). But when the (UMAS) was used the COD removal efficiency was 46.9% in the first run of the experiment. The COD removal efficiency reached 98% in 5th run when the UMAS was used. There was a large increase in the COD removal efficiencies from day 1st to day 5th, which is attributed to the un-acclimatized of the effluent consisting mostly of aerobic bacteria.

Moreover, higher BOD removal efficiency was obtained as 93 % at 5th day when using UMAS when compared with MAS (69.6 %). The TSS on the other hand showed very high removal efficiency when UMAS was incorporated into the reacting system. In the case of VSS, a threshold removal efficiency of 97.6% and 99.5 % was achieved when MAS and UMAS were used, respectively after the 5th run. Additionally, the percentage of methane composition in MAS and UMAS system as 75% and 79%, respectively. The overall result indicated that the application of ultrasonic membrane results in an improved performance of the reactor in relation to the methane gas produced.

5.2 Recommendation

Based on the results in this study, it is suggested that the following recommendations be considered for any future work:

- The pH control should be taking into consideration during and after the acclimatization process to ensure an improved methanogen activities and reduction of fatty acid formation.
- Temperature control is another important factor to consider. This is so pertinent because a decrease in the temperature will aid the reduction in fatty acid accumulation within the system. This in turn has the capacity to reduce the COD removal efficiency in the reactor, thereby affecting the methane gas generated within the system.
- Loss in the methane gas should be avoided during collection as this has the possibility of affecting their composition when estimating their percentage efficiency.

REFERENCES

Appels, L. *et al.* (2008) 'Principles and potential of the anaerobic digestion of waste-activated sludge', *Progress in Energy and Combustion Science*, 34(6), pp. 755–781. doi: 10.1016/j.pecs.2008.06.002.

Bhargava, D. A. (2016) 'Physico-Chemical Waste Water Treatment Technologies: An Overview', *International Journal Of Scientific Research And Education*, (September). doi: 10.18535/ijsre/v4i05.05.

Chen, J. L. *et al.* (2014) 'Toxicants inhibiting anaerobic digestion: A review', *Biotechnology Advances*. Elsevier Inc., 32(8), pp. 1523–1534. doi: 10.1016/j.biotechadv.2014.10.005.

Gao, W. *et al.* (2011) 'Membrane fouling control in ultrafiltration technology for drinking water production: A review', *Desalination*. Elsevier B.V., 272(1–3), pp. 1–8. doi: 10.1016/j.desal.2011.01.051.

Gavala, H. N. *et al.* (2003) 'Mesophilic and thermophilic anaerobic digestion of primary and secondary sludge. Effect of pre-treatment at elevated temperature', *Water Research*, 37(19), pp. 4561–4572. doi: 10.1016/S0043-1354(03)00401-9.

Gitis, V. and Hankins, N. (2018) 'Water treatment chemicals: Trends and challenges', *Journal of Water Process Engineering*, 25(May), pp. 34–38. doi: 10.1016/j.jwpe.2018.06.003.

Huang, S., Ras, R. H. A. and Tian, X. (2018) 'Antifouling membranes for oily wastewater treatment: Interplay between wetting and membrane fouling', *Current Opinion in Colloid and Interface Science*. Elsevier Ltd, 36, pp. 90–109. doi: 10.1016/j.cocis.2018.02.002.

lit, N. and Kharagpur (2000) 'Characteristics Of Sewage And Overview of Treatment Methods Lecture 16: Characteristics Of Sewage And Overview of Treatment Methods', *NPTEL IIT Kharagpur Web Courses*, Module 13, pp. 1–13.

Koster, K. L. and Leopold, A. C. (1988) 'Sugars and Desiccation Tolerance in Seeds', *Plant Physiology*, 88(3), pp. 829–832. doi: 10.1104/pp.88.3.829.

Li, X. *et al.* (2017) 'Anaerobic digestion using ultrasound as pretreatment approach: Changes in waste activated sludge, anaerobic digestion performances and digestive microbial populations', *Biochemical Engineering Journal*. Elsevier B.V. doi: 10.1016/j.bej.2017.11.009.

Li, Y., Park, S. Y. and Zhu, J. (2011) 'Solid-state anaerobic digestion for methane production from organic waste', *Renewable and Sustainable Energy Reviews*, 15(1), pp. 821–826. doi: 10.1016/j.rser.2010.07.042.

Mat, E. A. T., Shaari, J. and How, V. K. (2013) 'Wastewater production, treatment and use in

Malaysia', Safe Use of Wastewater in Agriculture: 5th Regional Workshop for Southeast and Eastern Asia, p. 6. doi: 10.1016/j.watres.2014.11.002.

Meng, F. *et al.* (2009) 'Recent advances in membrane bioreactors (MBRs): Membrane fouling and membrane material', *Water Research*. Elsevier Ltd, 43(6), pp. 1489–1512. doi: 10.1016/j.watres.2008.12.044.

Mittal, B. A. (2011) 'Biological Wastewater Treatment', pp. 2–9.

Momayez, F., Karimi, K. and Taherzadeh, M. J. (2019) 'Energy recovery from industrial crop wastes by dry anaerobic digestion: A review', *Industrial Crops and Products*. Elsevier, 129(November 2018), pp. 673–687. doi: 10.1016/j.indcrop.2018.12.051.

Nayono, S. and Perencanaan, J. dan (2005) 'Anaerobic Treatment of Wastewater from Sugar Cane Industry', *Jurnal Inersia*.

Rajeshwari, K. V *et al.* (2000) 'State-of-the-art of anaerobic digestion technology for industrial wastewater treatment', *Renewable & Sustainable Energy Reviews*, 4, pp. 135–156. doi: 10.1016/s1364-0321(99)00014-3.

Rodríguez-Méndez, R. *et al.* (2017) 'Long chain fatty acids (LCFA) evolution for inhibition forecasting during anaerobic treatment of lipid-rich wastes: Case of milk-fed veal slaughterhouse waste', *Waste Management*, 67, pp. 51–58. doi: 10.1016/j.wasman.2017.05.028.

Seghezzo, L. *et al.* (1998) 'A review: The anaerobic treatment of sewage in UASB and EGSB reactors', *Bioresource Technology*, 65(3), pp. 175–190. doi: 10.1016/S0960-8524(98)00046-7.

Tian Cheng Zhang and Noike, T. (1994) 'Influence of retention time on reactor performance and bacterial trophic populations in anaerobic digestion processes', *Water Research*, 28(1), pp. 27–36. doi: 10.1016/0043-1354(94)90116-3.

Tiruneh, A. T., Fadiran, A. O. and Mtshali, J. S. (2014) 'Evaluation of the risk of heavy metals in sewage sludge intended for agricultural application in Swaziland', *International Journal of Environmental Sciences*, 5(1), pp. 197–216. doi: 10.6088/ijes.2014050100017.

Xue, Y. *et al.* (2015) 'Effects of thermal hydrolysis on organic matter solubilization and anaerobic digestion of high solid sludge', *Chemical Engineering Journal*. Elsevier B.V., 264, pp. 174–180. doi: 10.1016/j.cej.2014.11.005.

Zia, S., Graham, D. and Dolfing, J. (2018) 'Wastewater treatment: Biological', *Water Research*, 20(14), pp. 2645–2655. doi: 10.1081/E-EEM-120046063.

APPENDIX A EXPERIMENTAL PROCEDURE



Figure A. 1 Acclimation Operation



Figure A. 2 TSS Filter Paper



Figure A. 3 COD Experiment



Figure A. 4 Methane Gas Measurement

APPENDIX B MAS EFFICINCEY REMOVAL (%)

HRT	BOD	MAS _C	TSS	VSS	METH	
(DAYS)	%	OD (%)			ANE	
1	12.9	11.9	80	93	77.68	
2	54	17.3	93.4	96	77.78	
3	68.2	58.3	89.5	82.9	75.34	
4	68.8	79.4	97.6	97.5	77.33	
5	69.6	85.5	93.8	97.6	75	
HRT	BOD	COD	TSS	VSS	PH	ТС
(DAYS)	mg/L	mg/L	mg/L	mg/L		dgree
First		0	0			0
run						
CLEA	15.15	145	6	1.05	6.78	30.6
N water						
Wastew	19.53	147.33	46.0	11	7.43	47
ater						
HRT	BOD	COD	TSS	VSS	PH	ТС
(DAYS)	mg/L	mg/L	mg/L	mg/L		dgree
2 nd run			0			0
CLEA	8.64	113	4	1.0	7.08	32.7
N water						
Wastew	18.81	136.67	61	25	6.5	45
ater						
HRT	BOD	COD	TSS	VSS	PH	ТС
(DAYS)	mg/L	mg/L	mg/L	mg/L		dgree
3 rd run						
CLEA	5.64	20	2.0	1.1	7.25	27
N water						
Wastew	17.7	48	19	6.43	6.53	46
ater						
HRT	BOD	COD	TSS	VSS	PH	ТС
(DAYS)	mg/L	mg/L	mg/L	mg/L		dgree
4 th run	<u> </u>					
CLEA	5.07	23	1	0.1	7.2	27.2
N water						
Wastew	16.29	112	42	3.571	7.26	46
ater						
HRT	BOD	COD	TSS	VSS	PH	ТС
(DAYS)	mg/L	mg/L	mg/L	mg/L		dgree
5 th run	Ŭ	U				
CLEA	4.56	20	3	0.4	6.76	26
N water		_ •	-			_ ~
Wastew	7.77	138	49	16.666	7.59	46
ater				7		

APPENDIX C UMAS EFFICINCEY REMOVAL %

HRT	BOD	COD	TSS	VSS	METH	
(DAYS)	%				ANE	
1	49.3	46.9	95.2	95.5	78	
2	63.6	95.2	95.6	96	69	
3	65.9	97.4	95.2	96.8	76	
4	78.3	97.9	96	97.5	77	
5	93	98	98	99.5	79	
HRT	BOD	COD	TSS	VSS	PH	ТС
(DAYS)	mg/L	mg/L	mg/L	mg/L		dgree
First	<u> </u>					
run						
CLEA	5.73	6	2	0.35	7.3	36
N water						
Wastew	11.31	81	42	7.7777	7.45	44
ater						
HRT	BOD	COD	TSS	VSS	PH	ТС
(DAYS)	mg/L	mg/L	mg/L	mg/L		dgree
2 nd run	-					
CLEA	2.43	4	2	0.7	7.03	35
N water						
Wastew	6.69	85	46	17.5	7.57	45
ater						
HRT	BOD	COD	TSS	VSS	PH	T C
(DAYS)	mg/L	mg/L	mg/L	mg/L		dgree
3 rd run						
CLEA	3.03	3	2	0.4	6.81	32
N water						
Wastew	8.85	116	42	12.5	7.55	45
ater						
HRT	BOD	COD	TSS	VSS	PH	T C
(DAYS)	mg/L	mg/L	mg/L	mg/L		dgree
4 th run						
CLEA	2	2	1.5	0.3	6.89	34
N water						
Wastew	9.2	95.23	42	12	7.65	49
ater						
HRT	BOD	COD	TSS	VSS	PH	TC
(DAYS)	mg/L	mg/L	mg/L	mg/L		dgree
5 th run						
CLEA	2	1	1.0	0.1	6.75	34
N water						
Wastew	14.3	50	50	20	7.48	48
ater						