KINETIC EVALUATION OF SEWAGE SLUDGE TREATMENT BY MEMBRANE ANAEROBIC SYSTEM (MAS)

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SUPERVISOR'S DECLARATION

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

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ABSTRACT

The application of Membrane Anaerobic System (MAS) process treating the sewage sludge and the overall MAS treatment efficiency was investigated. The MAS consists of a cross flow ultrafiltration membrane (Model Micro 240) for solid-liquid separation. Six parameters were investigated. The study showed a good trend for the Membrane Anaerobic System (MAS) treating raw sewage sludge. The removal efficiency of Chemical Oxygen Demand (COD) was 80 to 95 %, the removal efficiency of Total Suspended Solid (TSS) was 99 % and Volatile Suspended Solid (VSS) removal efficiency was 82 to 93 %. Throughout the study, the pH values were maintained from 7.5 to 7.8. The gas production ranged from 7200 ml/d to 16320 ml/d. The

percentage of the methane gas production was found to be 75.4 %. The treated sewage

sludge was achieved the environmental standard quality.

ABSTRAK

Aplikasi Sistem Membrane Anaerobik (MAS) dalam rawatan kumbahan mentah dan keseluruhan kecekapan rawatan MAS telah dikaji. MAS terdiri daripada membrane ultrafiltration aliran rentas (Model Micro 240) untuk pemisahan pepejal-cecair. Enam parameter telah dikaji. Kajian menunjukkan trend yang baik dengan menggunakan Sistem Membrane Anaerobik (MAS) dalam rawatan kumbahan mentah. Kecekapan penyingkiran Permintaan Oksigen Kimia (COD) adalah 80 hingga 95 %, kecekapam penyingkiran Jumlah Tahanan Pepejal (TSS) adalah 99 % dan kecekapan penyingkiran Meruap Tahanan Pepejal (VSS) adalah 82 hingga 93 %. Sepanjang kajian, nilai pH kekal dari 7.5 hingga 7.8. Penghasilan gas adalah dari 7200 ml/hari hingga 16320 ml/hari. Peratus penghasilan gas metana adalah 75.4 %. Kumbahan mentah yang dirawat telah mencapai piawai kualiti alam sekitar.

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LIST OF SYMBOLS/ ABBREVIATIONS

- BOD Biochemical Oxygen Demand
- CDM Clean Development Mechanism
- COD Chemical Oxygen Demand
- CRT Cell Retention Times
- CUF Crow-flow Ultrafiltration
- CWA Clean Water Act
- FSS Fixed Suspended Solids
- GHG Green House Gases
- IWK Indah Water Korsortium
- MAS Membrane Anaerobic System
- MF Microfiltration
- MWCO Molecular Weight Cut-Off
- OD Optical Density
- POME Palm Oil Mill Effluent
- TSS Total Suspended Solid
- TVSS Total Volatile Suspended Solid
- UF Ultrafiltration
- VSS Volatile Suspended Solid
- WSP Waste Stabilization Ponds

CHAPTER 1

INTRODUCTION

1.1 Background of Research

Sewage sludge is one of the organic wastes and commonly is the residual or semi-solid material effluent from industrial wastewater or sewage treatment processes. Sewage sludge is characterized based on the physical, chemical and biological properties. The physical properties of sewage sludge are colour, odor, solids and temperature. The chemical properties are divided into organic, inorganic and gases. The organic constituents of sewage sludge are carbohydrates, fats, oil, grease, proteins and surfactants. The inorganic components are pH, chlorides, nitrogen, phosphorus and sulphur. The gases elements of sewage sludge are hydrogen sulphide, methane and oxygen. Sewage sludge contents a high concentration of macronutrients, micronutrients and heavy metals (M. Nur Hanani *et al.*, 2010). Nutrients may cause eutrophication whereas heavy metals are toxic and may interfere with effluent reuse. Biological properties affect the microbial population, organic matter's decomposition, human health and environment.

Approximate 5 million m³ of sewage sludge is produced per year in Malaysia and is expected increase to 6.6 million m³ by the year of 2020 according Indah Water Konsortium Sdn. Bhd. (M. Nur Hanani *et al.*, 2010). The dramatic increase in the volume of sewage sludge is due to the increasing urbanisation and industrialisation, thus threatening the fresh water sources supply. Therefore, a solution of fresh water recovery is needed to solve the fresh water supply crisis. Conventionally, there are three sewage sludge treatment methods which are physical unit operations, chemical unit processes and biological unit processes (M. Nur Hanani *et al.*, 2010). Membrane Anaerobic System (MAS) on the other hand which is a treatment of waste using Ultrafiltration (UF) without oxygen has more advantages such as energy saving, biogas recovery, lower sludge production, clear final effluent, less usage of chemicals, small foot print and low maintenance cost. MAS method has been found to be more effective as compared to the conventional treatment methods (Liew, 2005). Membrane in the system can remove the suspended solids and the anaerobic system is the treatment of biodegradable organic and elimination of pathogenic organisms.

Chemical Oxygen Demand (COD), Biochemical Oxygen Demand (BOD), Total Gas Productin, Total Suspended Solid (TSS), Volatile Suspended Solid (VSS) and pH are the parameters in the study. Specifically, COD removal or reduction is the main concern of the study which indicates the level of decontamination of the wastewater. The parameter tests are to determine the quality of the sewage sludge. The environmental standards or regulations that should follow are Environmental Quality Act 1974 and Environmental Quality (sewage) Regulations 2009. The sewage sludge should achieve the standard as stated in the regulations before safely discharge into the environment. Besides, the parameter tests are used to determine the effectiveness of the system to treat the sewage sludge.

Anaerobic system is anaerobic digestion of the sewage sludge and converted into more stable organic compounds and gases with the existence of bacteria. It is an enclosed system that operated without aeration and promotes the growth of methane bacteria. Membrane is used to filter the sewage sludge to ensure the effluent or permeate is satisfy the standard of the regulations before discharge. The membrane system is suitable to treat different type of wastewater with a Chemical Oxygen Demand (COD) range from about 100 to more than 40000 mg/L and Hydraulic Retention Time (HRT) from 4 hours to numerous days (Thamer *et al.*, 2007). The high reduction of parameters such as Chemical Oxygen Demand (COD), Biochemical Oxygen Demand (BOD) and others can achieve by the combination of biological digestion with ultrafiltration membrane filtration. Hence, MAS is a process that ensures the safe disposal of sewage sludge and at same time to produce useful product which is methane gas.

1.2 Problem Statement

Water scarcity is occurred due to the growth of civilization, population rising and flourishing industrialization. Sewage sludge is one of the factors that contribute to the issue if discharge into environmental without pre-treatment. Besides the dramatic increases in sewage sludge production, the increases cost of land and public awareness of environmental and health issues encourage sewage sludge treatment. The treated sewage sludge is less contaminated with trace elements which include heavy metals and organic compound.

Sewage sludge is renamed compost due to its toxic nature of sewage sludge. Federal Clean Water Act (CWA) classifies sewage sludge as hazardous pollutant (Abby, 2002). Sewage sludge also contains pathogenic bacteria, viruses, protozoa and larger parasites for instance human roundworms, tapeworms, and liver flukes which are potentially hazardous to the health of humans, animals and plants. If the sewage sludge disposal to land without pre-treatment, such microorganisms can cause disease in human by inhaling sludge dust, digest food or drink contaminated by sludge. The facilities to treat and dispose the sewage sludge are limited. However, the producing of sewage sludge is increasing in every year due to the population is increasing. The sewage sludge treatment is complicated, taking longer time and occupied big area of land. Sewage sludge is hazard to environmental and public health if the excess sewage sludge is untreated due to limited treatment plant. Therefore, alternative sewage sludge treatment is needed to manage the large capacity of sewage sludge production.

In recent years, there has been an increasing interest worldwide in the production of alternative energy from the waste. Sewage sludge is one of the wastes that can pollute the environment if discharged without a treatment. Sewage sludge without any treatment can contaminate surface or underground water sources if it is discarded in water bodies or on land. To circumvent the problem, MAS is used to treat the sewage sludge before discharge to the earth. Sewage sludge treatment ensures the hygienic safety and sensory acceptability of the sludge. It is also a contribution to save the environment by reducing the waste in the world. The more sewage sludge is used as fuel in generating renewable energy, the more reduction in greenhouse gas emissions. By this way, sewage sludge can be utilized as a valuable resource rather than to be considered merely as a waste to be got rid of. Thus using the MAS to treat the waste and produce methane gas which is benefit to human by producing the methane gas.

1.3 Research Objectives

The following are the research objectives of the study

- 1.3.1 To determine the operating conditions or parameters of a design of a 20 Liter volume.
- 1.3.2 To examine the performance of the Membrane Anaerobic System (MAS) in reduction of Chemical Oxygen Demand (COD).

1.4 Scope of Study

The scope of the study is to convert the waste, sewage sludge to the alternative energy, methane by using the fermentation with the mix culture. 20 Litres is the amount of the sewage sludge used in the study. The amount of mix culture added into the sewage sludge is 10 % of the total amount of the capacity of sewage sludge using in the study. Hollow fiber ultrafiltration (UF) membrane is used to treat the sewage sludge so that can discharge the waste to environment safely. The six parameters were tested to determine the hazardous reduction of the sewage sludge. The six parameters were determined in these studies which are Chemical Oxygen Demand (COD), Biological Oxygen Demand (BOD), Total Suspended Solid (TSS), Volatile Suspended Solid (VSS), Total Gas Production and pH. The parameters used to be determined the achievement of the minimum COD in the treated sewage sludge and production of maximum methane gas in the shortest time.

1.5 Research Questions

The following are the research questions of the study

- 1.5.1 What are the operating conditions or parameters of a design of a 20 Litres volume?
- 1.5.2 What is the performance of the MAS in reduction of COD?

1.6 Significance of Study

The significant of the proposed study are environmental friendly, economic competitive, biodegradable and from waste to wealth. Fermentation is a good alternative to recover energy without oxygen supply and aeration. The environmental problem is

decreased by reducing the waste and from the waste to wealth. MAS is even a more effective, secure, worthy method to treat waste by decreasing its operating time. The addition of mix culture increase the digestion and methane gas production without purchase any bacteria strain. The plant using fermentation can be developed on biogas generation and the recovery of energy. This is a commercial project that can optimize methane to generate up to 500kW of electricity. Besides, methane can be utilized as a fuel to run boilers.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

The current concern of the whole world today is the environment issue-pollution. "Globally there is a great concern for reducing the emission of Green Houses Gases (GHG) through Clean Development Mechanism (CDM) projects for renewable energy" (Alawi *et al.*, 2009). "Climate change and rapid reduction of fossil fuel reserves have driven the world towards renewable energy source which are abundant, untapped and environmentally friendly" (Shahrakbah *et al.*, 2005). The current interest in energy supply from biomass has stimulated research in the application of anaerobic digestion of sewage sludge for gas production (Jasmine and Francesco, 1999). Thus, this study was carried out to optimize the production of methane by treating sewage sludge using MAS.

The renewable energy, methane gas, was used for electricity generation. "The recovery of waste material has priority over disposal" (Ravindra *et al.*, 2001). Thus, it is not a wise choice to dispose the sewage sludge. Instead, the sewage sludge needs to be treated to recover the waste material to generate methane. The keywords in this study are membrane anaerobic system, sewage sludge and kinetic evaluation. The study can be divided into six subtitles which are definition of parameters, Membrane

Anaerobic System (MAS), sewage sludge, anaerobic digestion of methane production, wastewater treatment and Palm Oil Mill Effluent (POME).

2.2 The Definitions of Parameters

Objectives of the study are to determine the optimal operating conditions of MAS and the performance of MAS for the treatment of the sewage sludge in a laboratory scale. The six parameters were determined in the study which was Chemical Oxygen Demand (COD), Biochemical Oxygen Demand (BOD), Total Suspended Solid (TSS), Volatile Suspended Solid (VSS), Total Gas Production and pH.

2.2.1 Chemical Oxygen Demand (COD)

COD is defined as the amount of a specified oxidant that reacts with the sample under controlled condition. The quantity of oxidant consumed is expressed in terms of its oxygen equivalence (Andrew *et al.*, 2005). COD is a measurement of pollutants in wastewater and natural waters. The unit of the COD parameters is in the unit of mg L^{-1} (Abdurahman *et al.*, 2010).

There are three type of COD measurement method such as open reflux method, closed reflux methods which are titrimetric method and colorimetric method (Andrew *et al.*, 2005). Open reflux method is suitable for a wide range of wastes where a large sample size is preferred. The closed reflux methods are more economical in the use of metallic salt reagents and generate smaller quantities of hazardous waste. However, closed reflux methods require homogenization of samples containing suspended solids to obtain reproducible results.

COD removal or reduction is the concern of the study which will indicate the decontamination of the wastewater. High COD removal will promote a progressive increase in methane production and the efficiency indicates good treatment performance for MAS (Abdurahman *et al.*, 2010). According to Environmental Quality (Sewage) Regulation 2009, the acceptable COD of sewage discharge is 120 mg/L for Standard A and 200 mg/L for Standard B. Standard A is applicable to discharges into any inland waters within catchment areas. Standard B is applicable to any other inland waters or Malaysian waters.

2.2.2 Biochemical Oxygen Demand (BOD)

BOD is one of the important parameters and most commonly used in the environmental treatment process such as waste water which is similar to sewage sludge treatment with a unit of mg L⁻¹ (Abdurahman *et al.*, 2010). The BOD values indicate the amount of biochemically degradable organic material and the oxygen used to oxidise inorganic material such as sulphites (SO₃³⁻), sulphides (S²⁻) and ferrous ion (Fe²⁺). Besides, oxygen used to oxidise reduced forms of nitrogen can also be measured by using BOD (Jing *et al.*, 2005).

The BOD is measured by determining the oxygen consumed from a sample placed in an air-tight container and kept in a controlled environmental for a period of time. There are three measurement of oxygen consumed which are 5 days test period at 20°C (5-d BOD or BOD₅, 5210B), oxygen consumed after 60 to 90 days of incubation (ultimate BOD or UBOD, 5210C), and continuous oxygen uptake (respirometric method, 5210D). According to the Environmental Quality (Sewage) Regulation 2009, the acceptable BOD₅ of sewage discharge of Standard A is 20 mg/L whereas Standard B is 50 mg/L.

The dilution of the samples is prepared according to the range of BOD. Table 2.1 is shown the ranges of BOD covered by various dilutions.

By using percent mixtures		By direct pipetting into 300 mL bottle	
% mixture	Range of BOD	mL	Range of BOD
0.01	20000-70000	0.02	30000-105000
0.02	10000-35000	0.05	12000-42000
0.05	4000-14000	0.10	6000-21000
0.1	2000-7000	0.20	3000-10500
0.2	1000-3500	0.50	1200-4200
0.5	400-1400	1.0	600-2100
1.0	200-700	2.0	300-1050
2.0	100-350	5.0	120-420
5.0	40-140	10.0	60-210
10.0	20-70	20.0	30-105
20.0	10-35	50.0	12-42
50.0	4-14	100.0	6-21
100.0	0-7	300.0	0-7

Table 2.1: Ranges of BOD Values Covered by Various Dilutions (Sawyer and Mccarty,1978).

2.2.3 Total Suspended Solid (TSS)

Total solids are all the solids in the water, suspended and dissolved, organic and inorganic. The sources of the suspended solids are from domestic use, industrial wastes and erosion by infiltration or inflow. It is used to measure the quality of the wastewater. A gravimetric test is the test for measuring solids involving the mass of residues. The sample is evaporating to dryness and weighing the residue to measure the parameter. The residue is expressed as milligram per liter (mg L^{-1}) on a dry-mass-of-solids basis.

The liquid and the water adsorbed can be removed by drying at a temperature above boiling which is 105°C. The occluded water can evaporate at a temperature of 180°C.

TSS is the nonbiodegradable material, nonvolatile residue or fixed suspended solids (FSS). Suspended solids may consist of organic, inorganic or immiscible particles. Organic substances for instance plant fibers and biological solids are the common constituents. Clay, silt and soil are the common components of inorganic solids. Sewage sludge is domestic wastewater which generally contains large amount of organic suspended solids. Suspended solids may cause sludge deposits and anaerobic conditions in aquatic environment.

Filtration is used to remove the suspended solids. The most common parameter evaluated in TSS is in the unit of milligrams per liter (mg L⁻¹) which is expressed as dry mass per volume. Dissolved solids are filterable residues that pass through the filter. Suspended solids are nonfilterable residues that retained on the filter. TSS measurement uses a number of different methods which to measure suspended solids in wastewater (James *et al.*, 2006). "Total Volatile Suspended Solid (TVSS) is used to estimate the total mass of organic matter in a water sample by a method of dry ashing TSS samples" (James *et al.*, 2006). In general, the correlations between TSS and Total Volatile Suspended Solids (TVSS) are as TSS concentrations increase, the fraction of TVSS will decrease.

2.2.4 Volatile Suspended Solid (VSS)

VSS is the solid lost in ignition which is useful to the treatment plant operator due to a rough approximation in the amount of organic matter present in the solid fraction of wastewater, activated sludge and industrial wastes (John *et al.*, 2005). The unit of VSS is mg L⁻¹ (Abdurahman *et al.*, 2010).

2.2.5 Total Gas Production

Total gas production is the time of measurement of the average length of time that gas produce in a constructed bioreactor. The equation of total gas production is shown as below

Total gas production =
$$\frac{\text{Total volume of permeate,V}}{\text{Time,hr}} = \frac{ml}{hr}$$

The units of the total gas production can be ml/day by multiple 24 hours/day. The volume of permeate flow out is equal to the production of gas in the reactor. The total gas production is increasing as the volume of permeate increasing. As the gas produce in the reactor, the pressure in the reactor is increasing and thus the permeate flow out due to the increasing pressure in the reactor.

2.2.6 pH

pH is used in alkalinity, carbon dioxide measurements and other acid-base equilibria. The characteristic of a solution either acidic or basic is indicated by pH or hydrogen ion activity. pH is defined as $-\log [H^+]$ by Sorenson which mean "intensity" factor of acidity. Pure water is very slightly ionized and at equilibrium the ion product is

$$[H^+][OH^-] = K_w = 1.01 \times 10^{-14} \text{ at } 25 \text{ °C}$$

$$[\mathrm{H}^+] = [\mathrm{OH}^{--}] = 1.005 \text{ x } 10^{-7}$$

Where

[H⁺] is activity of hydrogen ions, moles/L,

[OH⁻⁻] is activity of hydroxyl ions, moles/L,

 $K_w = ion product of water$

pH meter is the device use to measure the pH of a solution. pH meter consist of potentiometer, a glass electrode, a reference electrode and a temperature-compensating device. Electrometric pH determines the activity of the hydrogen ions by potentiometric measurement using a standard hydrogen electrode and a reference electrode.

The pH of the sewage sludge in the anaerobic digester should in the range of 6.6 to 7.6 to ensure the efficiency of the system. The pH value should not less than 6.2 due to the methane formers cannot function below this pH.

2.2.7 Temperature

Chemical reaction rate, solubility levels of chemicals, solubilities of the gases and activity rate of biological species present in water is affected by the temperature. Generally, the diversity of biological species is wider if cooler water. The rate of biological activity such as growth, reproduction and utilization of food supplies is slower at the lower temperature. The biological activity will increase as the temperature increase with the ratio of double the biological activity for an increase of 10°C if the essential nutrients are present.

Temperature also affects physical properties of water such as viscosity of water increases with decreasing temperature. Temperature is one of the environmental factors that will affect the efficiency of the digestion system. Mesophilic sludge digestion is optimum at the temperature range of 30°C to 38°C while thermophilic sludge digestion is optimum at the temperature range of 49°C to 57°C (Gurjar., 2001). Sharp and frequent fluctuations in temperature affect the methanogens bacteria. Thus, digester temperature changes must less than 0.6°C. The advantages of thermophilic anaerobic digestion over mesophilic digestion are

- i) Faster reaction rates
- ii) Better destruction of phatogens

The disadvantages of thermophilic anaerobic digestion consist of

- i) Higher energy requirements for heating
- ii) Higher odour potential
- iii) Low-quality supernatant which containing large quantities of dissolved materials
- iv) Low process stability due to the thermophilic bacteria are more sensitive than mesophilic bacteria to temperature fluctuations
- v) Poor dewaterability

2.3 Membrane Anaerobic System (MAS)

MAS is a combination of anaerobic bioreactor and a membrane unit which offers additional advantage than conventional anaerobic digesters. MAS can substitute the activated sludge process and the final clarification stage in the municipal wastewater treatment. MAS is a novel reactor design which enable Cell Retention Times (CRT) to be separated from the Hydraulic Retention Times (HRT) thus making it possible to anaerobically treat sewage sludge at short retention time. MAS can treat sewage sludge efficiently at low HRT (Cheng *et al.*, 1999).

4.2.1 pH

The pH test was done on raw sewage sludge, treated sewage sludge and untreated sewage sludge. pH was used to indicate contamination and acidification. Besides, acidity or pH of the sewage sludge was crucial to achieve the proper digestion of organic materials and yield the maximum amount of methane gas. The pH range of the raw sewage sludge was from 6.93 to 8.06. This proved that methanogenic activity was possible in the process but higher level of pH may be toxic to the bioactivity. Ammonical nitrogen such as NH₄⁺NH₃⁻N was inhibitory or toxic to anaerobic digestion and the toxicity of ammonia was highly dependent on pH which was the predominant form at higher pH. Methane producing bacteria require a neutral to slightly basic condition which was pH from 6.8 to 8.5 in order to produce methane gas. Thus, the raw sewage sludge was in a good environment to produce methane gas. The pH range of the untreated sewage sludge was from 6.98 to 7.12. This shows the increasing of sewage sludge digestion into methane gas as the methanogenic activity was active. This was because of most anaerobic bacteria will perform well in the pH range of 6.8 to 7.2. The pH range of the treated sewage sludge was from 7.45 to 7.76. This illustrates that the treated sewage sludge was nearly neutral and was safe to discharge.