UNIVERSITI PUTRA MALAYSIA

INVESTIGATION OF INTEGRATED MICROBUBBLE SYSTEM FOR TREATMENT OF PALM OIL MILL EFFLUENT (POME) WASTEWATER

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ABSTRACT

The Palm Oil Mill Effluent (POME) is a large concentration of nutrients and organic materials that, if not adequately handled, will cause environmental pollution. POME is also a heavily contaminating wastewater mainly attributed to its strong demand for chemical oxygen (COD) and need for biochemical oxygen (BOD) as well as total solids. Conventional methods of treatment require longer period (10-15 days) and higher operating costs. The microbubbles system used as the primary treatment in this project to remove the unwanted residue (sludge) from POME to obtain treated wastewater and to extract oil from the POME sludge trap. This technique is thought to be useful in improving POME's residual oil recovery and allowing an improvement in the mill's oil extraction rate. After the pre-treatment process, the content of total solid (TF), chemical oxygen demand (COD) and biochemical oxygen demand (BOD) show a reduction in value of 80.95%, 69%, and 26.76% respectively. The oil contain in the scum has been extracted show that 44.84% of oil was separated from the POME wastewater. The significant amount of reduction made show the success of the microbubble system as the pre-treatment for POME wastewater. The result may be increase by upgrading or improving the equipment the integrated microbubble system.

ABSTRAK

Efluen Kilang Kelapa Sawit (POME) adalah kepekatan nutrien dan bahan organik yang besar, jika tidak ditangani dengan baik, akan menyebabkan pencemaran alam sekitar. POME juga merupakan air sisa yang sangat mencemari terutamanya disebabkan oleh permintaan yang kuat untuk oksigen kimia (COD) dan keperluan oksigen biokimia (BOD) serta jumlah pepejal. Kaedah rawatan konvensional memerlukan jangka masa yang lebih lama (10-15 hari) dan kos operasi yang lebih tinggi. Sistem gelembung mikro digunakan sebagai rawatan utama dalam projek ini untuk membuang residu (enapcemar) yang tidak diingini dari POME untuk mendapatkan air sisa yang dirawat dan untuk mengeluarkan minyak dari perangkap enapcemar POME. Teknik ini dianggap berguna dalam meningkatkan pemulihan minyak sisa POME dan memungkinkan peningkatan dalam kadar pengekstrakan minyak kilang. Selepas proses pra-rawatan, kandungan jumlah pepejal (TF), permintaan oksigen kimia (COD) dan permintaan oksigen biokimia (BOD) menunjukkan penurunan nilai masing-masing 80.95%, 69%, dan 26.76%. Minyak yang terdapat di dalam sampah itu diekstrak menunjukkan bahawa 44.84% minyak dipisahkan dari air sisa POME. Jumlah pengurangan yang ketara menunjukkan kejayaan sistem gelembung mikro sebagai pra-rawatan air sisa POME. Hasilnya dapat ditingkatkan dengan meningkatkan atau memperbaiki peralatan sistem gelembung mikro yang terintegrasi.

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POME – Palm Oil Mill Effluent

TS – Total Solid

COD – Chemical Oxygen Demand

BOD – Biochemical Oxygen Demand

MPOB – Malaysian Palm Oil Board

FELDA – Federal Land Development Authority

FELCRA – Federal Land Consolidation and Rehabilitation Authority

CPO – Crude Palm Oil

CPKO – Crude Palm Kernel Oil

FFB – Fresh Fruit Bunch

FFA – Free Fatty Acid

TSS – Total Suspended Solid

VSS – Volatiles Suspended Solid

DAF – Dissolve Air Flotation

IAF – Induced Air Flotation

SAF – Suspended Air Flotation

CHAPTER 1

INTRODUCTION

1.1 Overview

Elaeis guineensis or oil palm has been introduced as a commercial plant in Malaysia in 1917 at the Tennamaram Estate in Selangor that has been an initial site for the development of palm oil industry in Malaysia by Frenchman Henri Fauconnier. In 1905, he came to Malaysia to establish the coffee plantation at Rantau Panjang, Selangor but the drop in price cause Fauconnier to start planted palm oil which then has become the first commercial development of palm oil industry in Malaysia. Surprisingly, in 1960, the plantation area for palm oil in Malaysia has increase from 55000 ha to 5.74 million hectares in 2016 (MPOB, 2017). A major expansion area take place during 1960s to 1970s which most of it was converted from rubber plantation to palm oil plantation due to the decreasing of rubber price. As the requirement for palm oil plantation was quiet similar to rubber plantation, the transition process save a lot of costing and can be seen to bring more profit rather than rubber plantation. Significantly grew with the plantation area, the outcome from the palm oil development also has increase from 100000 tonnes in 1960 to 17.32 million tonnes (Azman et al, 2018). In 1956, Federal Land Development Authority (FELDA) was

established by the government to increase the economic status and living standard of the rural area followed by Federal Land Consolidation and Rehabilitation Authority (FELCRA) in 1966 to eliminate inter-racial economic gap by creating more job opportunities, increase economic standard as well as the productivity (NST, 2017).

In the early stage, Europe has become the only main export which then the export market has expanded through the worldwide including India, Asia, and Africa. Start from the bottom, palm oil industries in Malaysia has successfully develop to be one of the Gross Domestic Product (GDP) in Malaysia at once increase the employment opportunities. For the last five year, this industry has contribute around RM 64.24 billion annually, which is around 5 % to 7 % of the country GDP where the income came from the production of crude palm oil (CPO) as well as process product from palm oil. This huge success are mostly affected by the introduction of Elaeidobius Kamerunicus weevil, a pollinating insect imported from Cameroon and has been applied at Mamor Estate at Kluang, Johor in 1981 which has reduce the pollination cost and become the turning point of increasing of free fruit bunch (FFB) extremely high.

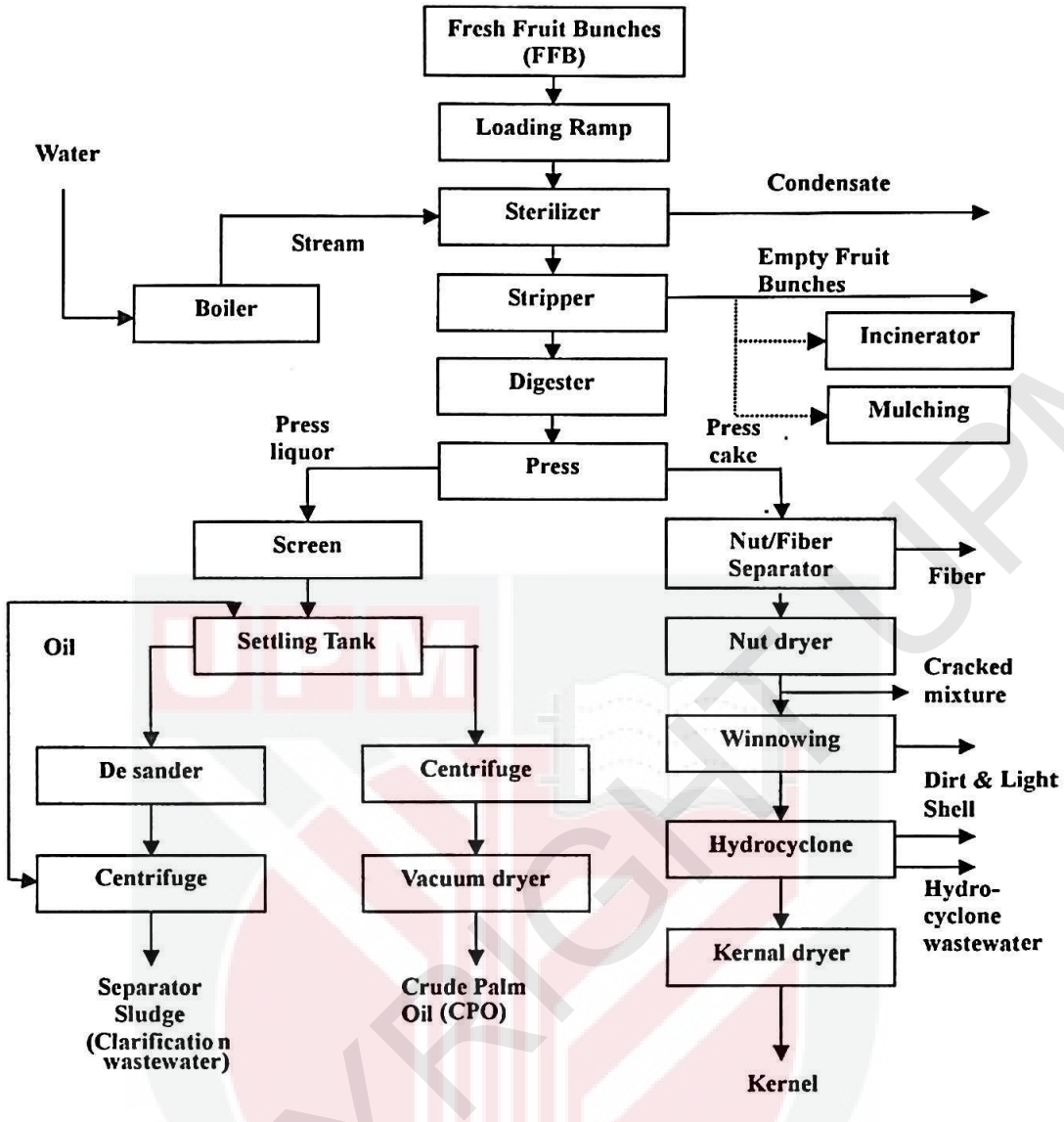


Figure 1.1.1 Process Flow of Oil Extraction

The extraction of oil from the fresh fruit bunch (FFB) after transported to the plantation area consist of a few unit procedure in which the main process can be found in Figure 1.1 above. There are two type of oil product that produced from the process which is crude palm oil (CPO) which was produce from mesocarp fibre and palm kernel oil produced from the palm kernel (endosperm). Roughly, the outer part of the palm fruit is the exocarp layer followed by mesocarp layer where the CPO is extracted. The third layer called endocarp (nut) consist of shell and kernel where the palm kernel oil was extracted (Figure 1.1.2).

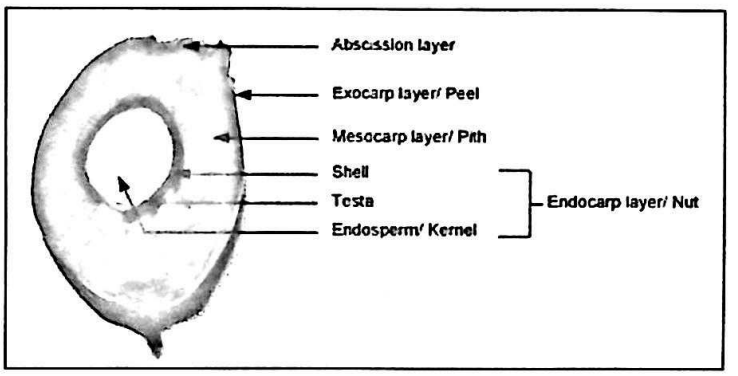


Figure 1.1.2 Cross-section of Palm Oil Fruitlet

The very first step in extracting oil from FFB is the sterilization process where the FFB collected was heated using a steam with temperature at 140°C for around 75-90 minutes inside the autoclave. There are a few purpose in this process which to loosen the fruitlet from the bunch, to deactivate the hydrolytic enzyme that used to breakdown the oil into free fatty acid. The breaking of oil cell will lead to coagulation. One of the main wastewater sources in this process is the steam condensate that comes out of the sterilizer. Next, the loosen fruitlet of the FFB will undergoes stripping process where the fruitlet is separated from the bunch using a rotary drum stripper. During the process, the FFB was lifted and dropped at a certain height to separate the fruit from bunch. The stripped fruit will come out from the space between the drum and the empty bunch will remain inside the drum and removed from the rotary drum stripper at the end of the process.

The next process is digestion. Digestion process take place in a digester, the cylindrical vessel embedded with rotating shaft in a vertical position. Inside the vessel, the fruitlets was mixed with hot water and the rotating shaft will rotate to mash the fruitlets. The hot water help in breaking the oil bearing cell of the mesocarp fiber of the fruitlets and foe easy flow of the oil extracted. The procedure includes fruits to be crushed and the pulp warmed up before oil is extracted. After the digestion process, it

will pass through a screw press break up unopened oil cells and remove additional oil. The best pressure is where a small amount of the nut split and the oil is around 8% dry from the pressing thread. Then, to remove the remaining solid and water, a vibrating screen is used to transport it into a hydrocyclone, and a decanters. Then, the CPO obtained was transferred to the clarification tank to skim out the oil at the top of the tank. Finally, by using the centrifuge and vacuum drier, the oil is purified and send to and keep in the storage at 60°C.

In processing the palm oil into its product, it produce a lot of waste and one of it is liquid waste in which preferred as palm oil mill effluent (POME). It was generated by the oil extraction process as well as washing and cleaning of the plantation which contain cellulosic material, fat, oil and grease (Agamuthu, 1995). POME waste can be found at as few main process including sterilization (36%), clarification (60%), and hydrocyclone (4%). A tonnes amount of water is needed to extract the CPO from the FFB. The processing of 1 tonne crude palm oil was expected to require 5-7.5 tonnes of water, and over 50% of the water is extracted as POME. (Ahmad et al, 2003). Generally, POME wastewater contain an organic acid which makes low value of pH around 4.7 (Ma and A.N, 2000). It also contain high total solid, COD and BOD value that make it harmful towards the environment which is 40500mg/L, 50000mg/L, and 25000mg/L respectively (Table 1.1.1). Therefore, the palm oil mill plantation need to treat the POME wastewater before discharging into the river or reuse it back for the extraction process by following to the Environmental Quality Act 1974 which can be found in Table 1.1.2 (Pierzynski et al, 2005).

Table 1.1. 1 Characteristic of Raw POME

parameter	Value
Temperature, °C	80-90
pH	4.7
BOD_3, mg/L	25000
COD, mg/L	50000
Total Solid, mg/L	40500
Total Suspended Solid, mg/L	18000
Total Volatile Solid, mg/L	34000
Oil and Grease, mg/L	4000
Ammonia-Nitrate, mg/L	35
Total Kjeldahl nitrogen, mg/L	750

Table 1.1. 2 Effluent discharge standards for crude palm oil mills

parameter	Value
Temperature, °C	45
pH	5-9
BOD_3, mg/L	100
COD, mg/L	-
Total Solid, mg/L	-
Total Suspended Solid, mg/L	400
Total Volatile Solid, mg/L	-
Oil and Grease, mg/L	50
Ammonia-Nitrate, mg/L	150
Total Kjeldahl nitrogen, mg/L	200

Microbubble system is the concept of air flotation where bubble at the micro size at the range of $10\mu m$ to $50\mu m$ which was generated by the fabricated microbubble generator. The microbubble system is chosen because of it special characteristic that are able to treat the wastewater sample effectively. It has larger interfacial area with larger densities which is capable of attaching themselves to the particle of the wastewater sample in the contact zone. Other than that, it also has low rising velocity which allow the microbubble to have longer contact with the wastewater sample. Thus, the characteristic show by the microbubble make it the effective way to treat the wastewater.

In this paper, the microbubble was generated by the fabricated microbubble generator which came out from assemble of a few part. The first part of it is the centrifugal pump where it is supported by the HONDA Company as the collaboration with the department of Process and Food Engineering to study on the efficiency of the microbubble system to treat the palm oil mill effluent (POME). Next part is the tank where it is fabricated according to the desired size and the volume of sample that will be used. The tank was made up from steel wall and also a mirror on one side to inspect and view the treatment process. Then, the fabricated microbubble generator also has the inlet and outlet piping. Inside the piping system, the wastewater sample which is POME will enter and pumped up into the contacting chamber and at the same time, the air will be injected into the contacting chamber to form the microbubble. The microbubble generated is then flow back into the fabricated tank for the treatment process. It also has base to support the fabricated microbubble generator as well as the control panel to control the pump.

In this project, the sample used for the treatment process is the palm oil mill effluent (POME) which is highly contaminated of organic matter in the form of total suspended solids (TSS), volatile suspended solids (VSS), total solids (TS), oil and grease even though it is non-toxic liquid. The POME sample was collected from the discharge station at the palm oil mill at Pahang. The raw POME was heated before manually taken into container. Finally, after transported back to laboratory, the raw POME was keep on the freezer under 4°C temperature.

In the experimental design, the fabricated microbubble machine will be run first by using clean water to avoid from damaging the pump which will affect the efficiency of the microbubble system as well as cleaning the flow inlet and outlet and

after that, the raw POME sample will be filled into the fabricated tank. The treated sample in the tank will be collected in every 30 minutes for 6 hours. The collected treated sample is then keep in the freezer before the analysis process.

The organic matter of the POME sample was in the form of total suspended solids (TSS), volatile suspended solids (VSS), total solids (TS), oil and grease as well as the chemical oxygen demand (COD) and biochemical oxygen demand (BOD). Therefore, to ensure the efficiency of the microbubble system, the value of the organic matter inside the POME sample can be measure to get the percentage of the reduction of the organic matter in the treated sample. The simplest analysis will be on the total solid (TS) where the solid of the sample was measured after evaporating the water content of the treated sample. Three reading should be at least taken for an approximate reading.

There are a few weakness of the fabricated microbubble generator used for the experimental design at it is not equipped with the flow meter which are able to indicate the flow rate of the inlet flow of the sample into the system and the outlet flow of the microbubble produced which will discharge back into the fabricated tank.

1.2 Problem Statement

In the increasing of the worldwide population, the quantity of the clean water supply to the domestic and industrial area as well as the residential area has been increasing, and so does the wastewater produced every day. However the capacity of the river, lakes, and oceans to assimilate the wastewater is limited. Because of these reason, the water pollution occurs as the contamination inside the water keep

increasing day by day causing the contamination of the water by the discharge wastewater into the water sources become excessive. In fact, this has become a serious problem in the global context, especially in the developing area, that still in the process of economic development.

In the recent year, the environmental researcher, scientist, universities, engineering design unit has done a lot of work and a lot of facilities has been built in many sectors of industries across the countries, which is to control and increase the quality of the clean water. But because of the large variety of industries, products and production processes are complex and changeable, and the types of waste water pollutants are varied. Thus there are still many problems in the wastewater treatment needs to be resolved, especially small and medium enterprises are more complex and there are many problems for this type of wastewater treatment process research and design should be given more attention. The easier ways on handling this problem is that the management of every sector contribute to this problem by having their own effective way on handling their wastewater and should have at least having a good way to treat their wastewater as having a good treatment on wastewater will benefit them in the sector of costing.

Generally, in the industries sectors, most of the wastewater come from the company which having a bigger production estimated per year where the process involve required larger quantity of clean water used for the process, so does the wastewater produced. For example, the chemical industries such as paint industries which involve the liquid-based product, it is clearly involving the usage of water in its processes which will produce a lot of wastewater. Therefore, it must have a good and efficient ways on handling the wastewater to avoid from contamination in the

discharging process of the wastewater into the river or other sewage plant. The same things happen in the food industries as more quantity of the clean water needed for the process, normally the food based company has a preliminary treatment of clean water to ensure the quality of the water supply received and increase the quality of the product. The treatment of wastewater will not only reduce the contamination, but also help in reducing the costing for the clean water needed for every process in their plant. In this paper, the main focus is on the bio-process industries which is palm oil industries where a lot of wastewater was produces and it is highly contaminated when it is released from the discharge station.

Palm oil industries has been one of the important sector in Malaysia as one of the contribution to the production of palm oil fruit which are called fresh fruit bunch (FFB). There are three type of FFB which is overripe, ripe and unripe bunch. Each type has same process but required different value of the parameter as well as the clean water needed to completely process the FFB into the biomaterial which is crude palm oil (CPO) and crude palm kernel oil (CPKO). Along the process, many wastewater has been produces that come from the process such as sterilization on the FFB, digestion and pressing process. In palm oil processing, the wastewater produce are called Palm Oil Mill Effluent (POME). It is considered as highly contaminated because of the high quantity of organic matter in the form of total suspended solids (TSS), volatile suspended solids (VSS), total solids (TS), oil and grease which is the major contribution to the water resources pollution especially rivers or lakes if left untreated (Lam, Man Kee, and Teong Lee, 2011). In Malaysia, POME can be considered as one of the highest pollution load into river throughout the country (Wu, T. Y., Abdul Wahab Mohammad, J. Md Jahim, and Nurina, 2007). The high value of the organic matter content and the unpleasant smell of POME can cause a serious pollution and

environmental problem to the river and too many form of live underwater even though it is non-toxic liquid waste (Maygaonkar, Prajakta A., Pradnya M. Wagh, and Usha Permeswaran, 2012). Therefore, it need a proper treatment before it can be discharge into the river and for this purpose, various method and wastewater treatment has been design to reduce the high quantity of the organic matter as well as its unpleasant smell. For example, the conventional aerobic POME treatment has shifted to close digester tank which could reduce only 70% of BOD in POME.

As a conclusion, the microbubble system has been chosen to solve the problem base on its characteristic which has large liquid-gas interfacial, longer duration time in liquid phase, and fast dissolution rate (Rehman, Fahad, JD Medley, Hemaka Bandulasena, and BJ Zimmerman, 2015). Microbubble system also a new element as it produce small size of bubble as small as 50μm with long stagnation time, lower bubble rising speed and high interior pressure (Rehman, Fahad, JD Medley, Hemaka Bandulasena, and BJ Zimmerman, 2015). Therefore, microbubble system is very suitable as it is low in cost and environmental friendly because no chemical used.

1.3 Objective

3.1 To investigate the efficiency of the integrated microbubble system as the preliminary step to treat the Palm Oil Mill Effluent (POME) through the reduction value of total solid, chemical oxygen demand, and biochemical oxygen demand.

3.2 To study the important of the size of microbubble and the dilution of the sample as the controlling parameter for the integrated microbubble system to treat the Palm Oil Mill Effluent (POME).

1.4 Scope of the Study

The scope of this research project is within the separation of unwanted residue in the POME sample used such as solid particle (soil and dirt), fluid material (oil) and other microbial that can be found in the POME sample from the wastewater as the pre-treatment to obtain a clean water so that it can be discharge into the river or it can be reuse back for processing the palm oil.

In this study, it investigate the effectiveness of the microbubble system that act as the pre-treatment to reduce the unwanted residue from the POME sample.

The study cover the treatment of palm oil mill effluent (POME) that is collected from Labu Palm Oil Mill factory located at Labu, Negeri Sembilan.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction to the Microbubble System

Nowadays, reserving a safe and clean environment has become a social problem, and from this viewpoint, water is particularly important (Hiroshi 2006). In order to counter the problem, several efforts have been made to improve the quality of water in the midst of global environmental problems (Akiko at al. 2005). In the past study, the method of sedimentation is often used to separate solid impurities from water. However, there are weakness in the method as if the material density limits the process if the impurities have a density close to that of water, the treatment effect will not be achieved by the sedimentation separation method. In the treatment of POME, this method are completely useless as the organic matter of the wastewater contain various type of impurities that is denser than water. Therefore, a micro-bubble flotation experimental system that can be used indoors is built to treat oily wastewater with energy saving, low cost and simple operation. Development of the technology for oily wastewater treatment is the key of the environmental protection and water re injection in palm oil field.

In recent year, microbubbles have become relevant among water purification technologies (Burns et al.1997, Nakano et al. 2005, Matsuo et al. 2006, Usui 2006, Yamasaki et al. 2009 and 2010, Agarwal et al. 2010, Wen et al. 2011). Air, oxygen and ozone microbubbles are used widely in various applications for water treatment. In addition to water treatment, microbubbles were used in many other fields such as medical therapy, microbubbles were used to scan body organs, as well as a drug or gene carrier (Lindner 2004, Tsutsui et al. 2004, Matsumoto et al. 2005, Hernot and Klibanov 2008, Maliwal and Patidar 2008, Kurup and Naik 2010, (Dicker et al., 2011). It were used for aerobic and anaerobic antibacterial activities (Himuro et al. 2009). Microbubbles can be used to adsorb protein from aqueous solution, stabilized by different surfactants (Jauregi and Varley 1998). To stabilize microbubbles, both petroleum-based and biological surfactants were also used (Kukizaki and Baba 2008, Xu et al. 2011). Surfactants, nanoparticles, pharmaceuticals and bioactive molecules have functionalized the microbubbles system. Because of its small size, microbubbles are highly efficient in industrial separation processes such as removal of volatile pollutants and particulate matter in the aqueous phase (Ahmed and Jameson n.d).

Flotation processes using microbubbles are useful in eliminating water-borne particulate matter of low density (Terasaka and Shinpo 2007). Microbubbles serve as small particle carriers that are raised from the column's bottom. Based on the microbubble generation technique, the flotation process may be dissolved-air flotation, dispersed-air flotation or electroflotation (Ketkar et al. 1991, Liu et al. 2010). Microbubbles have also found use in cleaning (Himuro 2007, Akuzawa et al. 2010), soil washing (Roy et al. 1992), removal of oil from soil (Gotoh et al. 2006) and water (Xiaohui et al. 2011), fermentation (Kaster et al. 1990, Ago et al. 2005, Xu et al. 2011), marine fish farming (Tsutsumi, 2010), horticulture (Park and Kurata 2009), food

technology (Shen et al. 2008, Xu et al. 2008, Soli et al. 2010), absorption of acid gas (e.g., CO 2) by alkali (Akimov et al. 2011). Microbubbles can improve marine creatures ' growth rate, such as oysters and scallops, by providing a large interfacial gas-liquid area that facilitates air transfer interphase.

Due to its superior efficiency compared to conventional methods, water treatment with microbubbles has recently become a well-known technology for many industrial applications (Ohnari 1997, Jyoti and Pandit 2001). For oxidation, disinfection (Sumikura et al. 2007), decolouration and deodorization (Shin et al. 1999), ozone microbubbles were used. Ozone has a high oxidizing potential and is used to use it in water purification or waste water treatment for sterilization, colour and odour removal and organic product degradation (Camel and Bermond 1998). Thus, different properties of microbubbles, available technologies for microbubbles generation and their size calculation are discussed in this study. However, from the viewpoint of future applications and research directions, the main applications of microbubbles in the treatment of water and wastewater are addressed.

Microbubble is characterized as a very small bubble, typically a few hundred micrometres in diameter, which can be suspended uniformly in liquid (The American Heritage Dictionary of the English Language: Forth Edition, 2000). Microbubble also can be refer as a bubble with diameter of $10\mu m$ as microbubble (Ohnari, 2002). Overall, the correct microbubble sizes vary in different application areas (Tsuge and Li, 2006). Microbubbles are tiny spherical bubbles of approximately 50 μm in diameter. They differ from common omnipresent bubbles, not only in terms of size, but also in terms of physicochemical properties. These special physicochemical properties have made microbubbles especially useful in various treatment applications

for water and wastewater (Ohnari et al. 2002, Takahashi 2004, 2010, Tsuge 2010). Such characteristics are: low rising speed through water, high curvature surface, wide interfacial area between gas and liquid, and electrically charged interface between gas and liquid. Such properties are correlated with many of the useful features of microbubbles for the treatment of wastewater.

There is a circular shape of microbubbles. The region is minimal when the object has a spherical shape for a three-dimensional body with a given volume, which is represented mathematically by ' isoperimetric inequality ' (Osserman 1978). Therefore, when the bubble is spherical, gas-liquid interfacial energy is minimal. The diameter of microbubbles widely used in fermenters, gas-liquid reactors and ore flotation equipment is 2 – 5 mm. The ' microbubbles ' are about 50 μm in diameter (Takahashi et al. 2007a). Bubbles with a diameter of less than 200 nm are referred to as ' nanobubbles ' (Agarwal et al. 2010). The bubbles, between 200 nm and 10μm in diameter, are called ' micro-nanobubbles ' (MNBs) (Tsuge, 2010). Figure 1 A (Sumikura et al. 2007) displays a microbubble photomicrograph. After producing microbubbles, the photograph was taken 20 s. In water, as shown in Figure 1 B, microbubbles create a milky dispersion. Nevertheless, the dispersion of nanobubbles is transparent (Tsuge 2010). A surfactant-stabilized microbubble consists of two compressed gas spheres, in which a layer of water is sandwiched, according to Sebba (1988). In Figure 1C, this structure is shown. The inner gas core is about 50 – 60 μm in diameter and the inner water film is about 1μm in thickness (Bredwell and Worden 1998). The surfactant molecules give different repulsive interfacial forces (e.g., electrostatic double layer, steric and hydration forces) a stabilizing influence to the bubble against coalescence. The surfactant-stabilized microbubbles stability makes it

possible to pump them without collapse. Thus, in a small vessel, such microbubbles can be produced and then pumped into a much larger reactor.

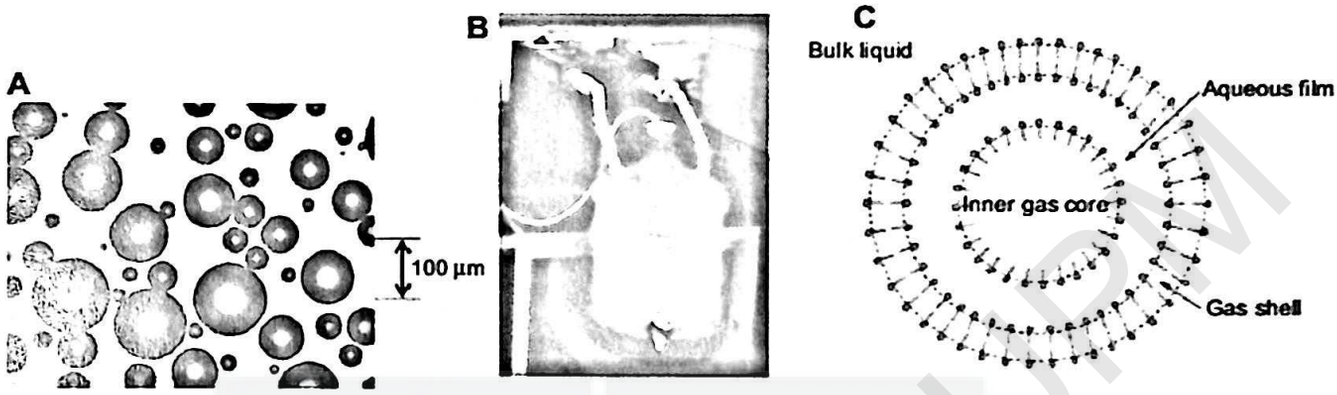


Figure 2.1. 1 The size and shape of microbubbles

Ultrasonically or by special generators, microbubbles have a wide distribution of size. Production of monodispersed microbubbles (Zhang and Li 2010) that are important for medical applications is a challenging task (Feshitan et al. 2009). Distribution of scale may be either unimodal or bimodal. A standard size distribution is shown in Figure 2 (Tsuge et al., 2009). The technique of microbubble generation has a substantial effect on microbubble size (Li 2006). Tsuge et al. (2009) studied single pore nozzle and microbubble generators of rotating flow and compared the size of the bubbles they formed. The bubbles are sheared and crushed with the spinning flow microbubble generator, resulting in smaller microbubbles. In addition, the pump bubbles of smaller size are generated with both generators at broad outlet pressure. This is due to the change in bubble formation pressure, which is greater and leads to smaller bubbles. The presence of active surface impurities and electrolytes can have a significant impact on the size of the microbubble. Walker et al. (2001) reported a decrease in the size of microbubbles as the concentration of NaCl increased to 1 mol / dm 3. By coalescence, pure liquids with small surface elasticity give rise to large

bubbles. Bubble size is decreased in aqueous solutions where surface elasticity is greater by inhibiting coalescence.

There are some methods in water to produce bubbles such as supplying gas through small pores or spinning blades to shear gas; however, effectively generating microbubbles smaller than 50μm in diameter is difficult. Figure 2 shows an effective generator of micro-bubbles in which liquid injected into the system by a pump is spiralled along the wall where the centrifugal force generated by diffusion induces a gas from the gas inlet and a vortex of gas is produced along the centre axis. The gas body at the outlet is divided into fine bubbles to form microbubbles.

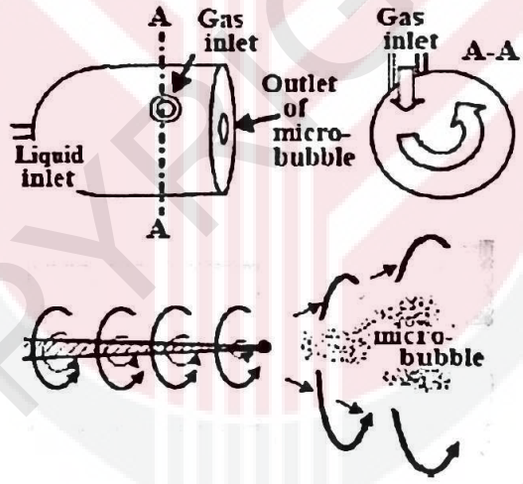


Figure 2.1. 2 Example of Microbubble Generator

Another example of an efficient generator is shown in Figure 3, where gas is introduced into the circulating liquid inside the vortex pump from the gas-inlet and dissolved into the super-saturated level by a high pressure in the spew-nozzle. Micro-bubbles are formed from the super-saturated gas-liquid by reducing the pressure outside the nozzle, which is intensified by the turbulent that is also generated by the

spew-nozzle. A lot of methods and devices are produced other than these two instances. An engineer will typically choose a suitable one for its cost-effectiveness.

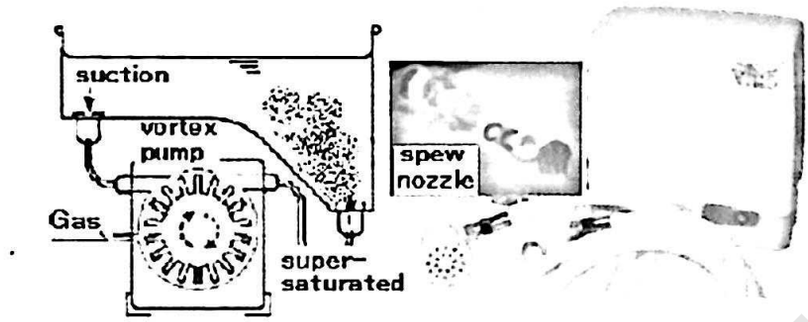


Figure 2.1. 3 Another Effective Microbubble Generator

Mixed-flow microbubble generator is a new device based on jet and air dissolution is show in Figure 4 to generate microbubble. An air compressor pumps air into the turbine when partial gas dissolves into pressurized wastewater. As the mixture of air and wastewater passes through the nozzle, the bubbles growing larger because of the decrease in pressure caused by the increase in velocity. At the same time, jet-generated velocity gradient, pulsation and surface wave, formed by high mixing speed, could produce churn flow. Due to the highly turbulent shear flow, the air and large bubbles are well separated into a large number of microbubbles.

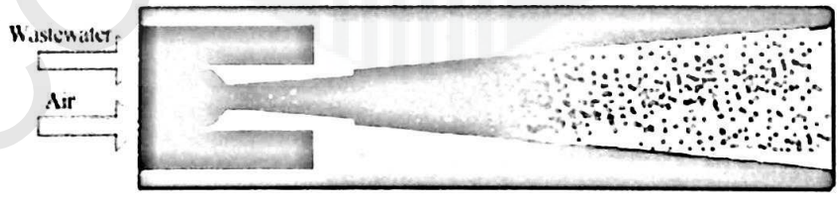


Figure 2.1. 4 Schematic Sketch of the Mix-Flow Generator

2.2 Mechanism of Microbubble System

In this experimental design, the generation of microbubble system started by diluting the POME wastewater sample which to increase the efficiency of the pump generator to create bubble as well to avoid the pump from malfunction due to the physical properties of the POME wastewater which very slurry and the contain of solid particle. The mechanism of the microbubble system shown in Figure 2.2.1 below.

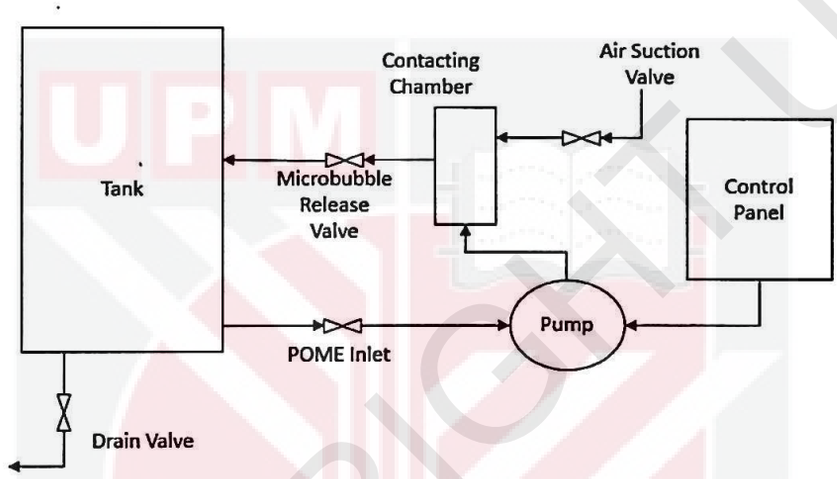


Figure 2.2. 1 Schematic Diagram of Microbubble System

After the dilution process, the POME inlet valve and microbubble release valve is fully open to allow the POME wastewater to flow into the system. Then the pump is turned on at the control panel and the bubble is generated. The air suction valve and microbubble release valve is then controlled to obtain the desired microbubble size. In the tank, the generated microbubble will attract the dirt and it will shrink from the water pressure and the collapses as the microbubble move upwards to the surface and separate the dirt from the POME wastewater. Figure 2.2.2 show the movement of microbubble generated in the tank.

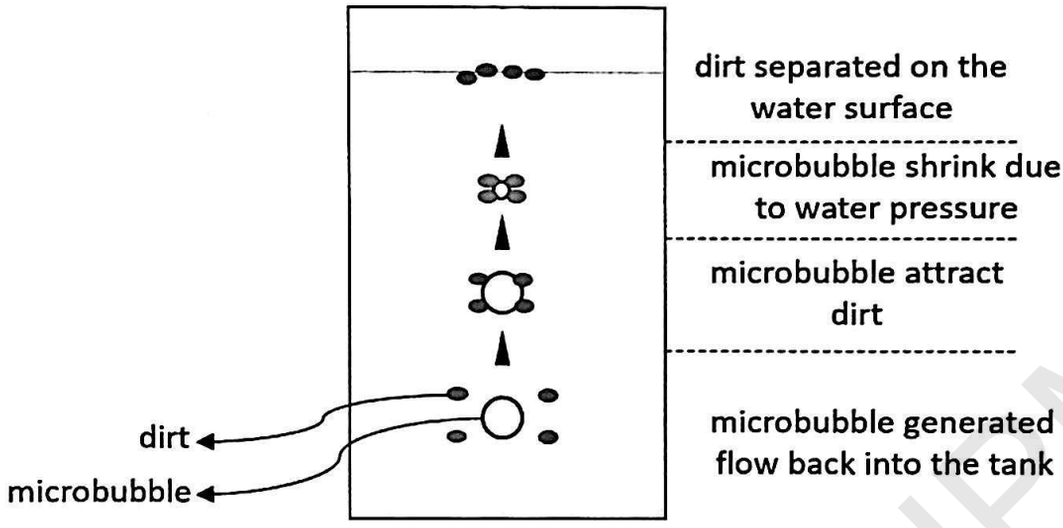


Figure 2.2. 2 Movement of Microbubble in the Tank

2.2.1 Electrical Properties of the Microbubble

There is a charge about the surface of microbubbles in water. If charged, as an electrical field is applied, microbubbles travel towards the oppositely charged electrode. This surface charge can be calculated in terms of the 'potential,' which is the 'plane of shear' potential (Hiemenz and Rajagopalan 1997). In the oxidation of harmful inorganic and organic compounds found in waste water, the charge at the gas-liquid interface plays a very important role in the stabilisation of microbubbles.

The ζ potential does not differ substantially with the diameter of the bubble, although the transient value of the ζ potential shows a substantial difference with time and, thus, with the diameter of the bubble as microbubbles shrink in water (Takahashi 2010). The ions in the air water interface are condensed into a smaller area as the bubble shrinks. Therefore, they begin from the interface to disperse into the bulk water. The rate of diffusion of the ions, however, is slower than the rate of bubble shrinkage. The ions can thus not disperse easily enough and are trapped near the interface, which

is manifested by the rise in ζ potential. Bubble boundary concentrated ions inhibit the escape of internal gas and stabilise nanobubbles (Takahashi 2009, Tsuge 2010). An ionic field of very high ion concentration is formed as the microbubble collapses, helping to form free radicals, which are the most reactive species in the oxidative treatment of waste water. The pressure within the bubble being inversely proportional to the diameter of the bubble contribute to a rapid reduction in pressure (pressure drop). If the rate of such an increase in pressure is high enough, due to adiabatic compression, the temperature inside the microbubble often rises dramatically. As a result, an environment with a pressure of several thousand atmospheres and a temperature of a few thousand degrees is formed at the moment of the microbubble collapse. This extreme reaction site is a very small region, but it has the capacity to cause the microbubble to decompose gas molecules, which produce free radicals (Takahashi 2010).

Due to the adsorption of OH- and H+ ions the charge is produced on the gas-water interface. The counterions have an electrostatic double layer and are drawn to the interface. In High pH, the OH − ions adsorb the air-water interface, where the solution is alkaline, which makes the microbubble negligible. The H+ ions are above the OH-ion at the interface in acidic solution. Based on their concentration, the presence of inorganic revenues decreases the potential of a sequence.

2.3 Air Flotation System

Flotation is a method whereby fine gas bubbles are introduced to separate solids or liquids from water. The bubbles bind to the particulate matter and the buoyant force of the combined particle and gas bubbles is sufficiently large to cause the particle to rise to the surface. Particles or liquids that are less dense than water like oil can rise

naturally, but particles that are denser than water will rise. Once the particles are floated to the surface, they can be collected by a skimming process. At the beginning, flotation separation was extended to mineral processing where it allows a selective classification of ore materials (Kitchener, 1984), and recently, water and wastewater treatment thought was given to water clarification (Kuiru and Schofield, 2001).

Air flotation was used as the alternative ways over sedimentation to remove particle, treating low turbidity, high coloured wastewater at low temperature, or removing algae from nutrient-rich stored water. The benefit of flotation over sedimentation in wastewater treatment includes when the water is unpredictable and some of the pollutants tend to rise or oil is present, a mechanism of sedimentation could be impaired as some of the waste floats naturally to the surface. Flotation is going to cause everything to go to the surface in the same direction. Other than that, It is possible to remove very tiny or light particles that would otherwise settle gradually more fully and in a shorter time by using air flotation system. Four key ways to incorporate the bubbles including Air injection while the liquid is under pressure, followed by pressure release. This is called dissolved air flotation (DAF) because at increased pressure the air actually dissolves into the water. Next, atmospheric pressure aeration, which is simply referred to as air flotation. Air is simply injected directly into the liquid through diffusers in the gas phase. This approach is not particularly effective when the particles are floating. The third ways is induced air flotation (IAF) means saturating wastewater with air either directly in an aeration tank or allowing air to reach a pump's or venture suction hand. The partial vacuum applied allows the dissolved air as minute bubbles to emerge from the solution. Finally, suspended air flotation (SAF) is a newer method in which a bubble generator creates bubbles using a surfactant. The bubble generator works at a relatively low pressure of 15–25 psig

and the minute bubbles produced are stated to be smaller than those produced by DAF's, are more numerous and rise faster as a mass.

Many believe that DAF is more effective in creating cleaner effluents than IAF. The air is actually dissolved under pressure into a stream of water, so it occurs as very minute bubbles when released, which can be directly attached to the particles. Generally a DAF effluent recycling flow is returned to a pressure tank where the air is applied and the tank is pressurized to 50 – 75 psig. The recycled water is combined with the raw power that makes intimate contact with the bubbles emitted and the important particles. The quantity of recycling required is 15 – 100% and depends on the total solids suspended. Laboratory and field studies on the size of the air bubble needed for successful liquid-solid separation have been carried out by many different companies and organizations. Generally speaking, the data show that the smaller the air bubble, the more successful the separation. IAF systems ' purpose was to try to reproduce a DAF's microbubble formation and to reach the same level of effectiveness as a DAF. In addition, the bubbles created by different techniques have a variety of sizes and can be quite varied.

2.3.1 Dissolve Air Flotation

Raw water is applied to the water treatment plant. In the pre-treatment process, which includes two steps: particle destabilization and particle flocculation, the fine particles in raw water are flocculated. Particle destabilization occurs after the injection of chemical coagulant in the flash mixer, where a high-speed stirrer disperses the coagulant evenly and rapidly. On the other hand, during the slow mixing stage in the flocculator, particle flocculation and aggregate growth occur. In the main component of the DAF process, part of purified water is recycled and saturated with air in the

range of 0.4~0.5MPa at high pressure up to 70~80 percent. The released gas bubbles adhere readily to the surface of floc particles forming agglomerates of bubble particles and rise to the liquid surface where a layer of sludge is formed that can be drained over a beach (Zlokarnik, 1998; Jameson, 1999; Shawwa and Smith, 2000).

The average diameter of microbubbles in DAF is 40-80μm (Han et al., 2002b; Haarhoff and Edzwald, 2004). The use of such small bubbles increases the performance of bubble-particle collisions, which is one of the most important factors in particle removal. Nevertheless, the DAF cycle has a range of inherent drawbacks, i.e. high demand for electrical power, complex system including a compressor, a saturator and a separate flotation tank and higher cost of operation (Schofield, 2001). In addition, since the theory of bubble generation in DAF is based on the Henry's law, the amount of air dissolved in water at a given pressure and temperature is limited and relatively low.

2.3.2 Induced Air Flotation

Induce Air Flotation (IAF) is occasionally found in wastewater treatment, where mechanical agitation or sparger air injection create relatively large bubbles with diameters of several hundred micrometers (Evans et al. 1992; Heindel and Bloom 2002). Some researchers proposed that IAF's large bubbles have correspondingly high terminal speeds leading to much more compact flotation systems. For water treatment, they wanted to use the IAF as an alternative to the DAF. Zlokarnik (1982 and 1998) created IAF with a specially designed, funnel-shaped nozzle that was self-aspirating and radically discharging. Jameson (1999), Yan and Jameson (2004) used a confined jet flotation cell, also known as a Jameson cell, to extract oils and fats from milk and slaughterhouse waste and blue-green algae from natural waters and ripening ponds.

Furthermore, the size of the bubble also influences the strength of the collision between bubble and particles. Big bubbles result in poor effectiveness in collisions (Shawwa et al., 2000). In addition, high shear in modern mechanical flotation cells typically leads to fragile particle breakage (Jameson, 1999; Zabel, 1992). Typically, these drawbacks are overcome by dosing other surfactants to create smaller bubbles and hydrophobicity to hydrophilic flocks (Jameson, 1999). Obviously, adding surfactant is not a good idea, as this will cause additional treatment for the sludge and complex operating system.

2.4 Pome Treatment Analysis

2.4.1 Chemical Oxygen Demand

Chemical oxygen demand (COD) is a way of calculating how much oxygen will be removed as a result of bacterial activity from a source of obtaining water. Whereas the BOD method is performed with a community of bacteria and other micro-organisms to try to replicate what will happen in a normal stream over a five-day cycle, the COD test utilizes a heavy chemical oxidizing agent to chemically oxidize the organic material in the freshwater sample under heat and high acid conditions. The COD test has the benefit of not being prone to hazardous substance contamination, and taking just two to three hours for completion of the study, as compared to five days for the BOD test. This has the drawback of being entirely artificial, which is still assumed to produce a conclusion which can be used as the basis for estimating a fairly precise and reproducible calculation of a wastewater's oxygen-demanding property (2007, Bennett).

The COD check oxidizes substance that cannot be metabolized by microorganisms in five days or which is poisonous. If the COD in raw wastewater is greater than the BOD, therefore the product is not readily biodegradable and can be

harmful to the organism. If COD is equivalent to BOD, otherwise the waste may be biodegraded readily (Scholz, 2006). The procedure used to achieve this measurement, which requires reflux of a sample into a strong acid with an abundance of potassium dichromate, does not directly quantify the organic content of the sample but instead the volume of oxygen needed for oxidation. Hence, this method offers a straightforward assessment of the possible effect of oxygen intake on the water body's oxygen quality (Walker, Baumgartner, Gerba & Fitzsimmon, 2019).

2.4.2 Biochemical Oxygen Demand

The Biochemical Oxygen Demand (BOD) is one of the most commonly employed parameters for determining water quality. It offers details on the ready-to-use biodegradable fraction of the organic water charge. However, this analytical procedure generally takes 5 days (BOD_5), and the tests that differ based on the laboratory (20 %), largely due to variations in the inoculum's microbial diversity. In 1936, the American Public Health Association Standard Methods Committee introduced this criterion as a guide measure for measuring the bio-degradation of contaminants and dangerous substances (Jouanneau et al, 2014). This parameter is known as the sum of oxygen, divided by the volume of the cell, taken up for a fixed time (5 days, BOD_5) by the respiratory activity of the microorganisms growing on the organic compounds present in the sample when incubated at a given temperature (20°C). It is a measure of that biologically degradable organic pollution of water.

The BOD_5 has three major implementations. First, it is a measure of the compliance of pollution disposal with the latest legislation and the sewage management protocol. Second, the ratio of BOD5 and COD in wastewater treatment facilities shows the biologically degradable proportion of an effluent. Thirdly, the

COD / BOD5 ratio is a measure of the size of a wastewater treatment plant needed for a particular site.

2.4.3 Total Solid

The measurement of total solid in POME sample determine all the dissolve, suspended, and colloidal solid that can be found in the wastewater sample. The value of total solid including silt and plankton as well as the dissolve salt such as sodium chloride. Based from the experiment in obtaining the total solid, it can be define as the remaining solid that is collected in the wastewater sample after completely loss the water content due to evaporating in the heating element (oven) at 105°C in a period of 8 to 24 hours. The procedures for the analysis including washing and drying of the crucible to remove any dirt and taking the weight of the POME sample with crucible before heating (with water molecule) and after heating (without water molecule). The value of total solid can be calculated by using these formula;

$$Total\ Solid = \frac{(Weight\ of\ the\ crucible + sample) - (Weight\ of\ empty\ crucible), g}{Volume\ of\ the\ sample, L}$$

2.4.4 Oil Extraction

Oil extraction was run towards the scum collected after the pre-treatment of POME wastewater. During the microbubble process all the unwanted particles including solid, oil and grease has been trapped inside the small microbubble and move upwards to the surface due to its lower density than the wastewater. The process of oil extraction from the scum involve the separation of oil from the dried scum by contacting it with the universal solvent which are more soluble and immiscible in water

and extraction condition (A.L Ahmad et al, 2003). The oil extraction process consist of few component which is the contact of between dried scum with the solvent, separation of oil from dried scum, and treatment of the solvent to remove the extracted oil. The oil extraction process can be run using the soxhlet extractor. It was designed by Franz Von Soxhlet in 1879. It consist of a round bottom flask, filter paper, distillation path, condenser, extractor, cooling water inlet, cooling water outlet, heat source and siphon tube (Gopalasatheeskumar, 2018). The dried scum is wrapped by filter paper and placed in the extractor. The extraction process takes six hours before continued with post extraction process. The extraction process cause the oil and the solvent chemically combined. Therefore, rotary evaporator and the distillation methods to separate the oil with the solvent, and also normal air drying process used to concentrate the oil are used by many researchers (Kaufmann B and Christen P., 2002). The solvent used for the oil extraction is n-hexane as it able to extract more oil from the dried scum sample in the condition of optimum solvent to POME ratio, optimum mixing time, optimum mixing speed, and optimum pH.

CHAPTER 3

METHODOLOGY

3.1 Material and Apparatus

3.1.1 Sample of Raw POME

The raw sample of Palm Oil Mill Effluent (POME) was collected from palm oil mill at Pahang. The raw sample of POME was collected from the final discharge station and manually placed in the sterile container. The transportation of raw POME sample using a truck is then stored in the portable freezer at 4°C

3.1.2 Fabricated Microbubble Generator

The microbubble generator was fabricated by adding a few part which is the fabricated tank (where the microbubble is release and the treatment of the POME occurs), centrifugal pump, (for generation of the microbubble) and the piping inlet and piping flow of the raw POME sample as well as the air injected for the generation of microbubble.

3.1.3 Fresh Water

The fresh water play an important role for the experiment as the early stage of using the fabricated microbubble generator, the fresh water was used to run the fabricated microbubble generator before adding the raw POME sample and start the treatment process inside the fabricated tank. Other than that, the fresh water also needed for the cleaning process of the fabricated tank after the end of the treatment. The fresh water source is from the water pipe which is pumped into the fabricated tank using the centrifugal pump.

3.1.4 Oven

The oven used for the analysis of the sample collected during the treatment process for the analysis of the total solid reduce from the treatment process. The oven was used to eliminate the water content of the sample and leave only the solid content of the sample. The weight of the solid was calculated by the different of the empty crucible with the final weight of the crucible after the water content was removed from the sample by using oven at 115°C.

3.1.5 Minor Equipment

The minor equipment used in this experiment including the dipper (to collect the raw POME sample from the final discharge station from the palm oil mill and to collect sample of the treated POME sample from the fabricated tank), test tube (collected sample from the fabricated tank), freezer (the collected treated sample was placed in cold area before the analysis of the treated sample), crucible, electronic weighing balance, and other equipment and material that can be used for the treatment process as well as the analysis of the treated sample.

3.2 Methodology

3.2.1 Collecting the Raw Sample

The raw sample used for the experiment which is Palm Oil Mill Effluent (POME) was collected from the palm oil mill at Pahang. The raw POME sample was collected at the final discharge station and placed in the container. At the discharge station, the raw sample was heated up about 30 minutes before the discharging process of the POME. The raw POME was manually collected by using dipper and plastic jar into the container before loading it into the truck. One trip of the raw POME sample collection can obtain around 10 to 15 container of the sample accordingly. The raw POME sample is then transported back to the laboratory and was keep in the freezer under the temperature of 4°C

3.2.2 Generation of the Microbubble

As the early step of the experimental design, it is important to set up the fabricated microbubble generator before the real treatment process of the raw POME sample as the designed fabricated microbubble generator was not equipped with the flow meter which is to measure the flowrate of the sample that goes into the pump and the flowrate of the microbubble generated that was release back into the fabricated tank. Therefore, fresh water was used as the preliminary testing sample to set up the fabricated microbubble generator in order to achieve the desired size of the microbubble generated from the system. Firstly, the fabricated tank was filled with fresh water until the water level reach the above level of the inlet of the microbubble that was release in to the fabricated tank. The fresh water was pumped into the fabricated tank by using the centrifugal pump to increase the flowrate of the fresh water going into the fabricated tank. The fresh water also to dilute the sample before the

treatment process as the raw POME is highly contaminated and the preliminary step of the wastewater treatment should not be too efficient in the treatment of the wastewater. Therefore, to ensure the good data is obtain, the raw POME sample was first diluted and after the final result is obtain, it will be multiply with how much dilution has been made. Next, the inlet of the sample into the pump and outlet of the sample released back (microbubble generated) into the fabricated tank was fully open as well as the air inlet that used for the generation of the microbubble. Then, at the control panel, the switch was turned on to start up the pump and the pump speed was set at 50 hertz. Last but not least, control the inlet of the sample into the pump as well as the air injected into the system to control the size of the microbubble. There are some weakness in this method as the microbubble was measured only by the bare eyes because no indicator is used for the system to measure the size of the microbubble cause by the lack of costing. Finally, the raw POME sample will be filled into the fabricated tank for the treatment process. Before the time is set, the raw POME sample was let for 5 minutes inside the fabricated tank so that the raw POME sample can homogenously spreading through the fabricated tank. The flow process of generating microbubble can be describe in Figure 3.2.1.

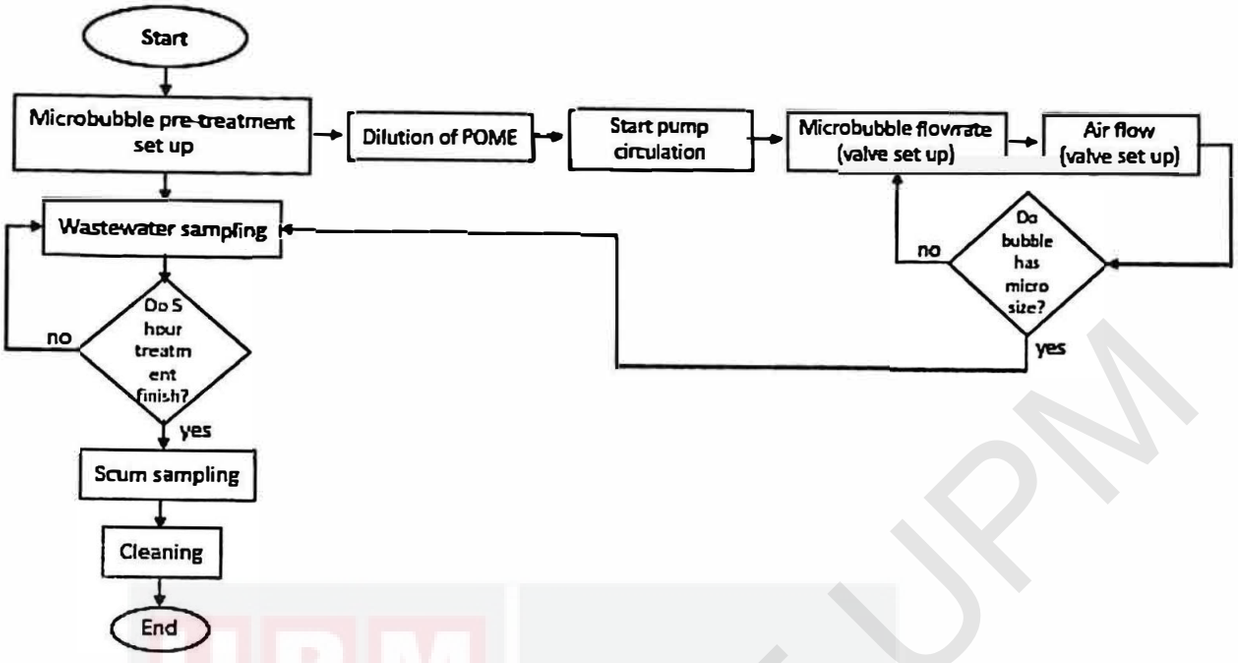


Figure 3.2. 1 Flow Chart of Microbubble Treatment

3.2.3 Sampling of the Treated Sample

The sampling process is the collection process of the sample after being treated in the fabricated tank by the microbubble system. The treated POME sample was manually collected from the fabricated tank by using the dipper. During the treated process of the POME sample, there are three layer form which is scum (first layer), solid impurities (second layer), and treated POME sample (third layer). The desired sample that need to be collected is the third layer that need to be analysed. The first and the second layer was skimmed aside and by using dipper, the sample from the third layer was taken out and poured into the test tube. Three sample was taken out to obtain more approximate result. The sample is then stored in the freezer for a short time before the analysis process. The process of collecting the sample from the third level was repeated for every 30 minutes in the duration of 6 hours.

3.2.4 Cleaning of the tank

Cleaning process is one of the important part of the process as to ensure that the fabricated microbubble generator are able to work for a long duration of time and to keep the efficiency of the pump used as if the cleaning process was not done correctly, the pump as well as the piping system may clogged by the sample and coagulation may formed. For the cleaning process, firstly the outlet valve was fully open to drain all the sample out of the fabricated tank. The tank is then washed using detergent to remove the solid particles as well as the impurities that left at the wall of the fabricated tank. Next, the tank was rinsed to remove the detergent through the outlet valve. Finally, the fresh water was filled into the fabricated tank until the water level reach the outlet of the microbubble connected to the fabricated tank. The fabricated microbubble generator was then run again to clean the piping of the system as well as the pump.

3.2.5 Total Solid Analysis

Total solid analysis is the analysis which is to measure the total solid inside the sample. The empty crucible was firstly weighted using the electronic weighing balance before pouring the 2 ml of the collected treated sample into the empty crucible. Each crucible will be labelled before placed it in to the oven at 115°C. The aim of the heating process is to remove the water content in the treated sample and leave only the solid inside the crucible. After 8 hours of the heating process, the sample was taking out of the oven as the water content was fully remove from the treated sample. The crucible with the heated sample was weighted using the electronic weighing balance and the data was recorded into the table. The value of the total solid content in the sample can be calculated by using the formula

$$Total\ Solid = \frac{(Weight\ of\ the\ crucible + sample) - (Weight\ of\ empty\ crucible), g}{Volume\ of\ the\ sample, L}$$

After the value of the total solid is calculated, the average value can be obtain based on the three sample collected in each time taken. The flow process of total solid analysis was shown in Figure 3.2.2.

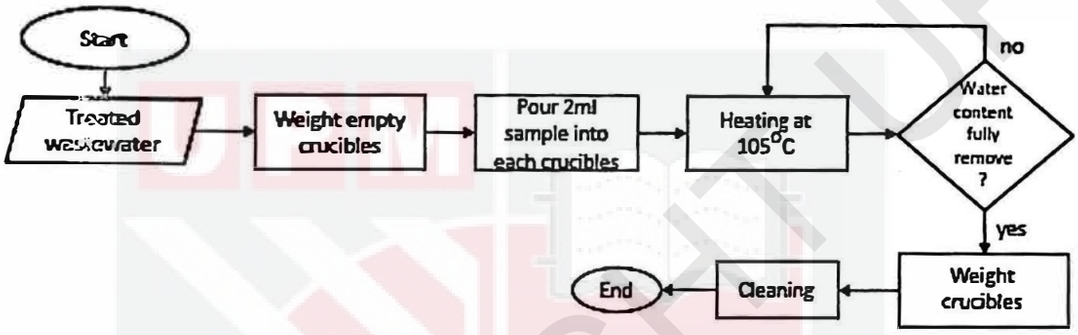


Figure 3.2. 2 Flow Chart of Total Solid Analysis

3.2.6 Chemical Oxygen Demand Analysis

A few equipment and preliminary step are required before taking the reading of the COD of the collected sample. Firstly, the heating block need to be preheated to 150°C. Next, the collected sample need to be diluted as the spectrophotometer cannot take the reading of the COD value which higher than 1500 mg/L. Therefore, to dilute the sample, 5 ml of sample is pipetted into 100 ml of volumetric flask, added with distilled water until reach 100 ml of the volumetric flask. The solution is then shacked well before taken out into 100 ml of beaker. Finally, 2 ml of the solution is pipetted into test tube containing the COD reagent vial and the dilution process is repeated for each sample of 0h, 1h, 2h, 3h, 4h, and 5h. All of the sample is then placed into the heating block for 2 hours including the blank sample which prepared by mixing 2 ml

of distilled water with the COD reagent vial. After 2 hours, take the reading of COD value of the heated solution by using spectrophotometer and record the data. Figure 3.2.3 below show the process flow of the analysis.

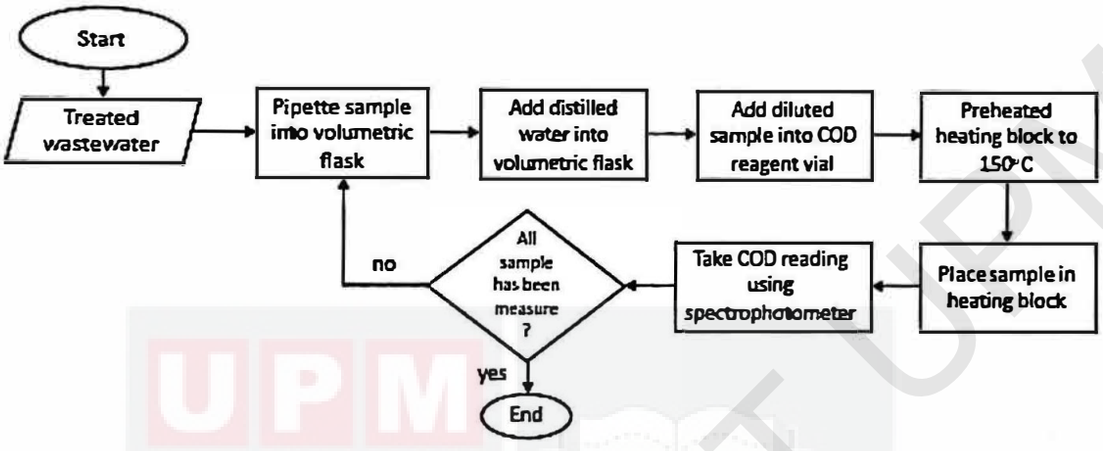


Figure 3.2. 3 Flow Chart of Chemical Oxygen Demand Analysis

3.2.7 Biological Oxygen Demand

The analysis of BOD towards the treated POME wastewater is to measure the amount of dissolved oxygen demanded by the organic biological organism in the collected sample to break down the organic material. Figure 3.2.4 show the flow process of BOD analysis. The analysis is carried out by mixing 5 ml of wastewater sample with 250 ml distilled water into 300 ml of BOD bottle. Both sample and distilled water is mixed well before taking the initial reading D_i by using the Dissolve Oxygen Meter (DO Meter). The DO meter is immersed into the mixed sample about 5 minutes to obtain a stable and precious reading. Then, the sample need to be incubate in the incubator for 5 days with 20°C temperature in a dark condition. It is to avoid the light for photosynthesis or growing of algae in the sample. After 5 days, the sample is taken out from the incubator and the final reading, D_f is taken using the DO meter. The BOD value of the sample can be calculated by using formula;

$$BOD = \frac{D_i - D_f}{P}$$

Where D_f= dissolve oxygen after 5 days

D_i= initial dissolve oxygen

P = fraction of wastewater sample

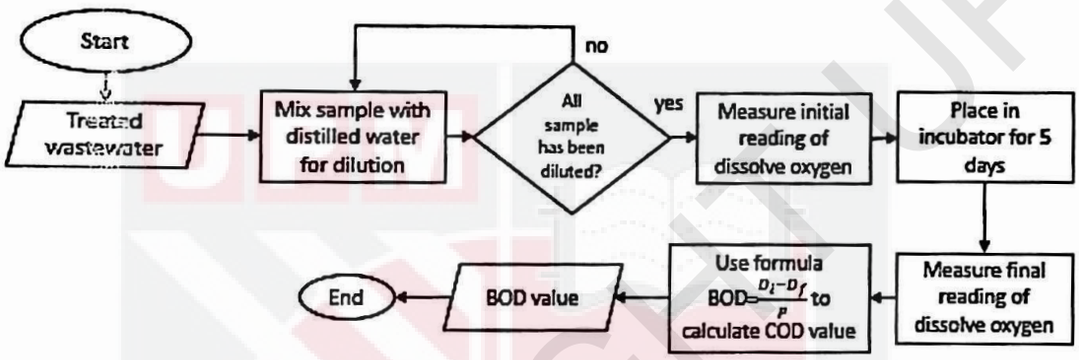


Figure 3.2. 4 Flow Chart of Biochemical Oxygen Demand Analysis

3.2.8 Oil Extraction from Collected Scum

In collecting the oil from the scum, a soxhlet experiment can be done where the sample is wrapped in a filter paper and soaked it into hexane. Firstly, before the soxhlet experiment can be run, the wet scum need to be dried by using oven at 105°C to 115°C. An amount of wet scum is placed into tray wrapped by aluminium foil to simplify the work for collecting the dried scum. The wet scum in the tray is then placed in the oven for 8 to 24 hours to make sure that the water content in the wet scum is fully removed. After a certain time, the scum is taken out of the oven and the dried scum is collected and kept in a container.

To start the soxhlet experiment, firstly, weight 10 gram of dried sample using electronic balance and wrapped it by using filter paper and placed the wrapped dried scum into the extractor. The process of wrapping the dried scum is repeated to obtain 6 sample of wrapped dried scum. After the sample is placed in the extractor, 160 ml of hexane is poured into distillation flask and connect it with the bottom of the extractor. Then, the top of the extractor is connected to the condenser (connected to the water supply). Next step is the water supply is opened to supply the condenser with tap water and the heat supply also is turned on. The voltage for the heat supply is controlled to ensure that the hexane is heated wisely and the oil extraction process can be done. The extraction process occurs slowly. Therefore, the process is run for 6 hours before the heat supply is turned off. After the experiment is done, dismiss the distillation flask, extractor as well as the condenser all together. Pour the excessive mixture in the extractor into the distillation flask and take out the wrapped dried scum from the extractor and keep it in a separated container. Clean up the extractor and wrap the closure of the distillation flask using aluminium foil.

The aim of the oil extraction is to collect the oil from the dried scum but the sample collected in the distillation flask is the mixture of oil and hexane. Therefore, rotary evaporator is used to eliminate the hexane from the oil collected. To use the rotary evaporator, firstly the Microprocessor Control (MPC) need to be turn on for 30 minutes before the experiment start and the temperature on MPC is set at 14°C. Then switch on the rotary evaporator and set up the temperature of water bath at 40°C. The next step is collect the mixture of oil and hexane sample into the evaporator flask and clip it at the end tunnel on top of water bath and clip the collector flask at the end of the condenser. Then, set the rotary evaporator downward to ensure the sample in the evaporator flask is completely immerse into the water bath. Then, the rotary speed is

set between 75 rpm to 100 rpm. Before start the evaporation process, turn the vacuum pump and close the pressure relive valve. During the process, hexane is evaporated from the sample and collected in the collector flask and left the oil sample in the evaporator flask. Regularly check the process to ensure that the hexane is not completely remove from the evaporator flask to easily remove the sample out. After the process is done, switch off the vacuum pump and open the pressure relive valve. Bring the evaporator flask out from the water bath and take out the sample from the evaporator flask into the centrifuge tube and keep it in the oven at 68°C (hexane boiling point). Finally, pour out the collected hexane from the collector flask into the waste disposal container and clean out the evaporator and collector flask. Figure 3.2.5 show the flow process to extract the oil from scum.

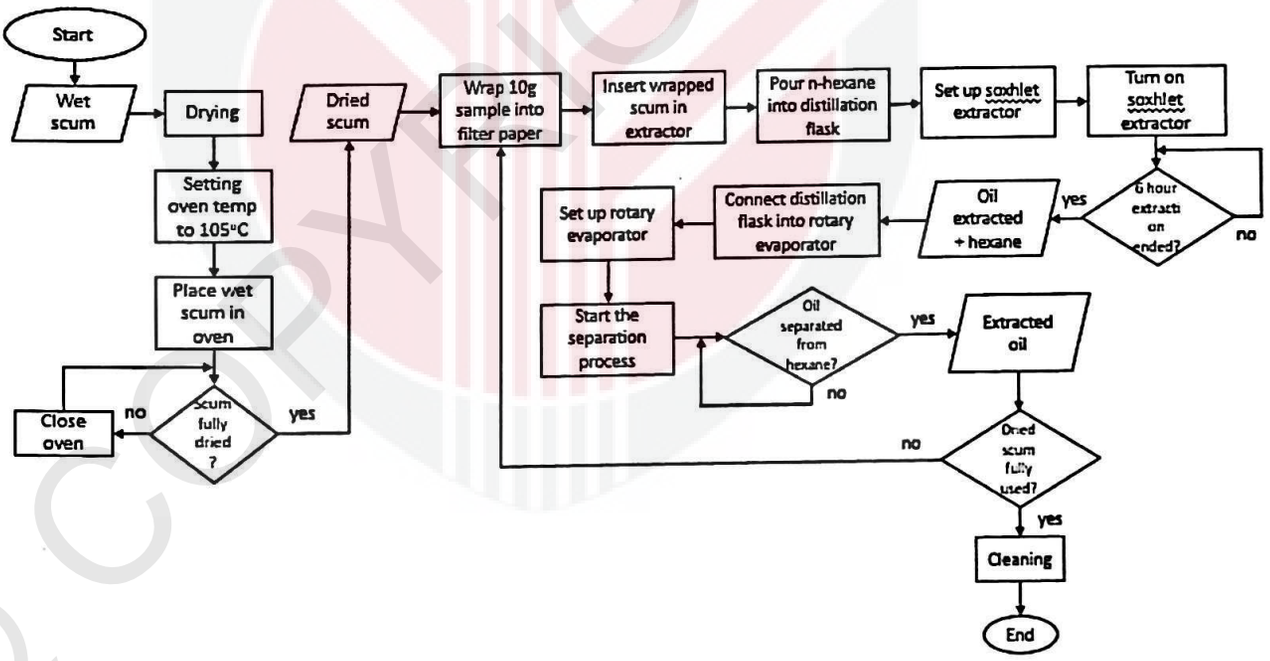


Figure 3.2. 5 Flow Chart of Oil Extraction Analysis

CHAPTER 4

RESULT AND DISCUSSION

The result obtained below represent the content of the treated POME wastewater on a different parameter which is total solid (TS), chemical oxygen demand (COD), and biochemical oxygen demand (BOD) to measure the reduction in above parameter compare to its initial value. The value for TS and COD was compared between three different dilutions made during the microbubble treatment with the previous experiment. The comparison was made between 5 times dilution (current experiment) with 10 times and 30 times dilution. The result also measure on the oil extracted that contain in the scum collected from the surface of the POME wastewater after the microbubble treatment. Finally, the data will be compared to other pre-treatment that commercially used by palm oil plantation.

4.1 Total Solid Analysis

Table 4.1.1 and Figure 4.1.1 show the reduction value of total solid for 5 times dilution of raw POME wastewater. The data sample was taken from the initial time (0 hour) before the treatment process begin until five hours of treatment. The sampling process taken with one hour interval. From the sample, the value of total solid was

calculated with 21000 mg/L of initial value and the TS value drop to 4000 mg/L after five hour show the reduction of 80.95 % from the initial value.

Table 4.1.1 Total Solid Content for 5x Dilution

Sample	Total solid (g/L)	Total solid (mg/L)	Actual Total Solid (mg/L)
0 H	4.2	4200	21000
1 H	3.8	3800	19000
2 H	2.2	2200	11000
3 H	2.0	2000	10000
4 H	1.6	1600	8000
5 H	0.8	800	4000

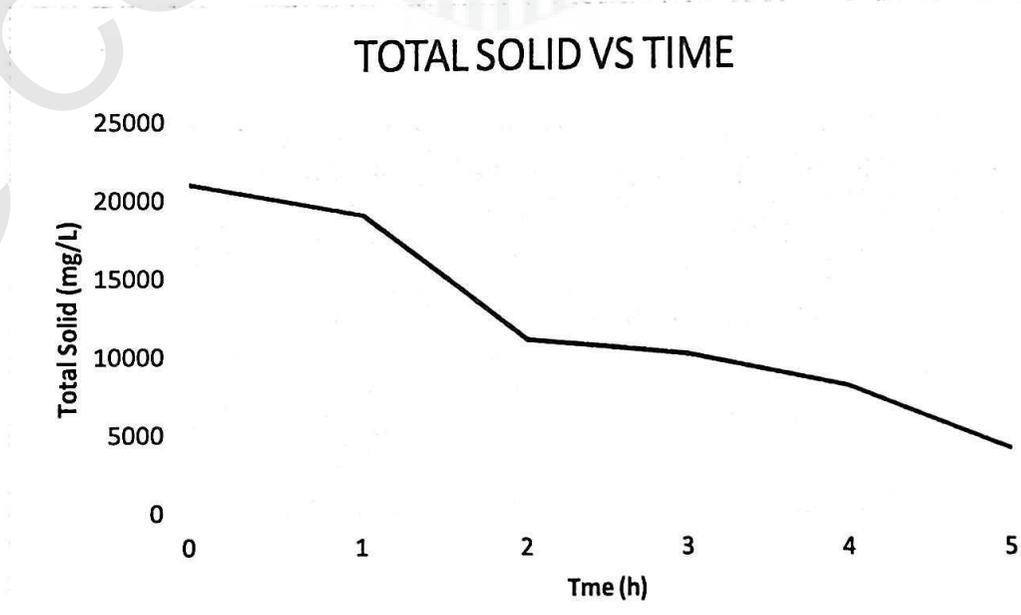


Figure 4.1. 1 Graph of Total Solid for 5x Dilution after 5 Hour

Table 4.1.2 Total Solid Content for 10x Dilution

Min	Sample	Total solids (g/L)	Total solid (mg/L)
0	A	2.0	2000
0	B	1.6	1600
30	A	1.6	1600
30	B	1.4	1400
60	A	1.2	1200
60	B	1.0	1000
90	A	0.8	800
90	B	0.8	800
120	A	0.8	800
120	B	0.8	800
150	A	0.6	600
150	B	0.4	400
180	A	0.2	200
180	B	0.2	200

Table 4.1.3 Average Total Solid for 10x Dilution

Min	0	30	60	90	120	150	180
Total solid (mg/L)	1800	1500	1100	800	800	500	200

Actual Total Solid (mg/L)	18000	15000	11000	8000	8000	5000	2000

Table 4.1.2 show the data of total solid taken from raw POME wastewater with 10 times dilution. Two sample (A and B) was taken for every 30 minutes for duration of three hours. Table 4.1.3 show the average data taken by formula $\frac{sample\ A+sample\ B}{2}$. From the data, the initial value at 0 hour is 18000 mg/L and reduce to 2000 mg/L show 88.89% reduction of total solid by 10 times dilution.

Table 4.1.4 Total solid content for 30x dilution

Sample	Min	Total solids (g/L)	Total solid (mg/L)
A	0	1.8	1800
B	0	1.4	1400
A	60	1.0	1000
B	60	0.8	800
A	120	0.6	600
B	120	0.6	600
A	180	0.4	400
B	180	0.2	200

Table 4.1.5 Average Total Solid for 30x Dilution

Hour	0	60	120	180
Total solid (mg/L)	1600	900	600	300
Actual Total Solid (mg/L)	48000	27000	18000	9000

Same condition with the 10 times dilution, the sampling for treated wastewater for 30 times dilution also taken twice (sample A and sample B) for every one hour for duration of three hour was shown in Table 4.1.4. In Table 4.1.5 show the average data calculated and multiplied by 30 times dilution where 48000 mg/L was calculated as initial value of sample (raw sample) and 9000 mg/L as the calculated data for treated sample after three hour pre-treatment. The total of 81.25% of reduction was calculated from the experiment which show small different with reduction for 10 times dilution of sample but can be considerately high reduction shown.

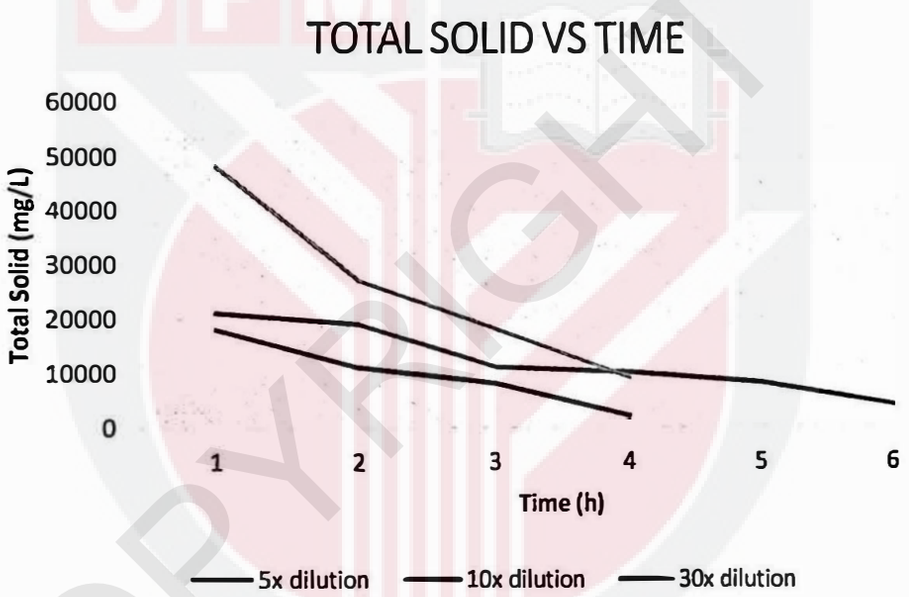


Figure 4.1. 2 Graph of Total Solid for Three Different Dilution

Figure 4.1.2 show the different of reduction for three type of dilution which is five times, 10 times, and 30 times dilution made for the raw POME wastewater before the pre-treatment process. Based from the calculated data, the highest reduction done by 10 times dilution followed by 30 times and 5 times dilution with value of 88.89%, 81.25%, and 80.95% respectively. The respective data show that the microbubble system used as the pre-treatment for POME wastewater give out more than 80% reduction of total solid content compare to the initial value.

4.2 Chemical Oxygen Demand (COD) Analysis

Table 4.2.1 and Figure 4.2.1 show the decreasing of COD value from the POME wastewater sample after dilute the sample five times. The average value of COD obtained was multiplied by 20 times for the dilution made for the sample as the spectrophotometer cannot take the reading exceed 1500 mg/L. The COD value also was multiplied by 5 times for the dilution made before the pre-treatment process. Based from the actual reading, the initial value of COD before the treatment process was run is 22600 mg/L reduce to 7000 mg/L after five hour of treatment which shows 69% reduction of COD content in the wastewater compare to the initial value.

Table 4.2.1: Reading of COD for 5x Dilution

Sample	Average COD Reading (mg/L)	Actual Reading (mg/L)
O H	226	22600
1 H	189	18900
2 H	131	13100
3 H	128	12800
4 H	99	9900
5 H	70	7000

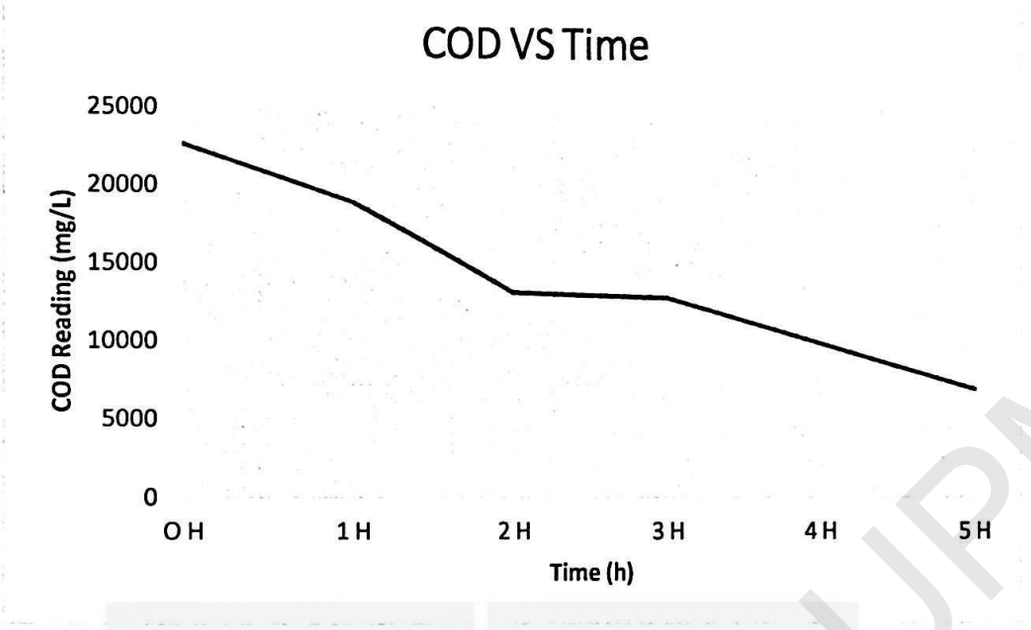


Figure 4.2. 1 Graph of COD for 5x Dilution after 5 Hour

Table 4.2.2 show the reading taken by the spectrophotometer for the POME wastewater treated with microbubble system after 10 times dilution. The initial reading for raw POME wastewater after dilution was 75585 mg/L and after three hour of microbubble treatment, the COD value reduce to 30250 mg/L. From the calculation, the experiment show that 50.3% reduction of COD value was achieve through three hour of microbubble treatment. If the reduction occurs constantly through every hour, it can be estimated that more than 80% reduction can be achieve.

Table 4.2.2 Reading of COD for 10x Dilution

Hour	COD Reading (mg/L)	
	Average	Actual Reading
0	151.17	75585
1	103.20	51600
2	75.17	37585
3	60.5	30250

Table 4.2.3 Reading of COD for 30x Dilution

Hour	COD Reading (mg/L)	
	Average	Actual Reading
0	229.34	344010
1	142.34	213510
2	71.00	106500
3	57.34	86101

Table 4.2.3 describe the data obtained during the pre-treatment the raw POME wastewater after being diluted 30 times. Based from the data, the three hour period of the pre-treatment process show 74.97% reduction of COD value from the initial value at 344010 mg/L to 86101 mg/L after three hour pre-treatment process. Figure 4.2.2 show the comparison of the reduction occur between three different dilutions made for the raw POME wastewater before the pre-treatment process. The gradient show the reduction percentage of the COD value during the pre-treatment process where higher gradient show higher percentage of reduction. Based from the graph, the highest reduction can be obtained after 30 times dilution followed by 10 and five times dilution.

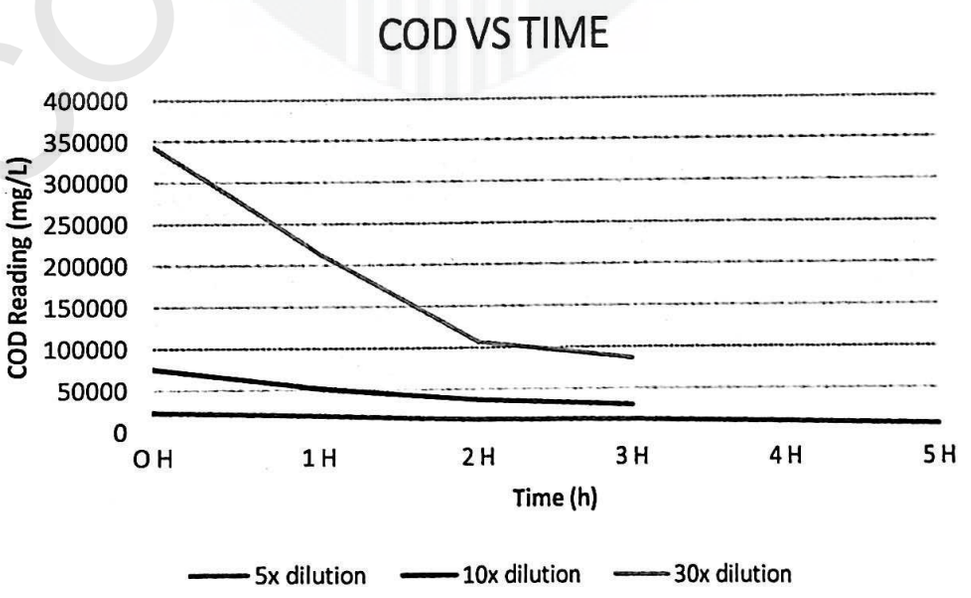


Figure 4.2. 2 Graph of COD against Time for Three Different Dilution

4.3 Biological Oxygen Demand (BOD) Analysis

Table 4.3.1 and Figure 4.3.1 show the data of BOD value obtained for five times dilution of the raw POME wastewater. The BOD value was calculated by using formula $BOD = \frac{D_i - D_f}{P}$ where the initial reading of the dissolve oxygen was subtracted by the value of dissolve oxygen after five days and divided by the fraction of treated wastewater sample. The data show that the value drop from the initial of 12670 mg/L to 9280 mg/L. After five hour of pre-treatment process, the BOD value show 26.76% reduction compare to the initial value.

Table 4.3.1 Reading of BOD for 5x Dilution

Sample	BOD Reading (mg/L)	Actual Reading (mg/L)
O H	126.70	12670
1 H	123.30	12330
2 H	102.00	10200
3 H	98.50	9850
4 H	98.50	9850
5 H	92.80	9280

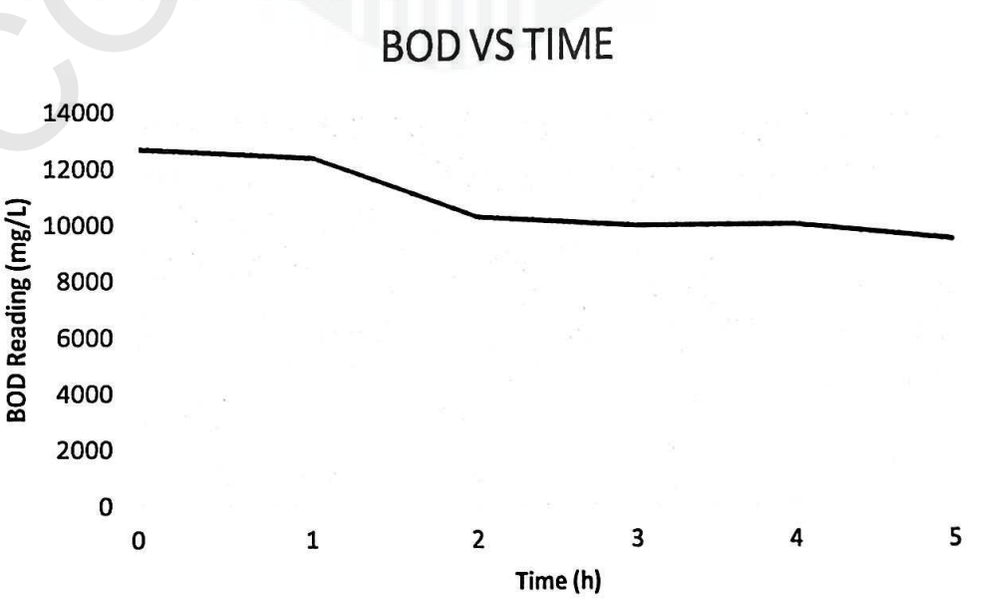


Figure 4.3. 1 Graph of BOD for 5x Dilution after 5 Hour

4.4 Oil Analysis

The scum collected by scrubbing it at the end of the microbubble system contain water, solid particles, grease and oil. The oil content in the scum was extracted by eliminating the water content to obtain the dried scum by using soxhlet extractor and n-hexane as the solvent, the oil was extracted and chemically combine with n-hexane. The separation of oil and n-hexane was done by the rotary evaporator and oil was keep in the oven at 69°C (n-hexane boiling point)

Table 4.4.1 Drying Wet Scum for Oil Extraction

Scum retrieve after 5 hours of microbubble pre-treatment for 5x dilution: 5.03kg		
Sample	Wet Scum (g)	Dry Scum (g)
1	764.60	36.30
2	1727.25	84.36
3	1636.21	78.08
4	902.78	43.76
Total	5030.84	242.5
Dried Scum Retrieve (%)	4.82	

The wet scum was dried in oven at temperature 105°C which slightly higher than the boiling point for water (100°C) at random weight. The random sample was labelled from 1 to 4 show high water content in the wet scum as only 4.75%, 4.88%, 4.77%, and 4.85% of dried scum was collected for each sample respectively. Therefore from the total of 5.03 kg of wet scum collected, only 242.5 g of dried scum was obtain which around 4.82 %.

Table 4.4.2 Oil Extraction

242.5 g dried scum = 24 soxhlet sample	
No	Oil Retrieve (mL)
1	14.50
2	33.00
3	26.25
4	16.00
5	16.00
6	14.00
Total	119.75

After the extraction process oil using the soxhlet extractor and rotary evaporator, the μoil sample was heated in the oven at temperature 69°C to eliminate the remaining n-hexane contain in the solution. Table 4.4.2 show the volume of oil extracted from the dried scum. The rotary evaporator give out six sample of oil and kept it the centrifuge tube before heating. From 242.5 gram of dried scum only produce 119.75 mL or 108.73 g of oil. Therefore, the yield of oil extraction calculated is 44.84% where it show how much of oil extracted.

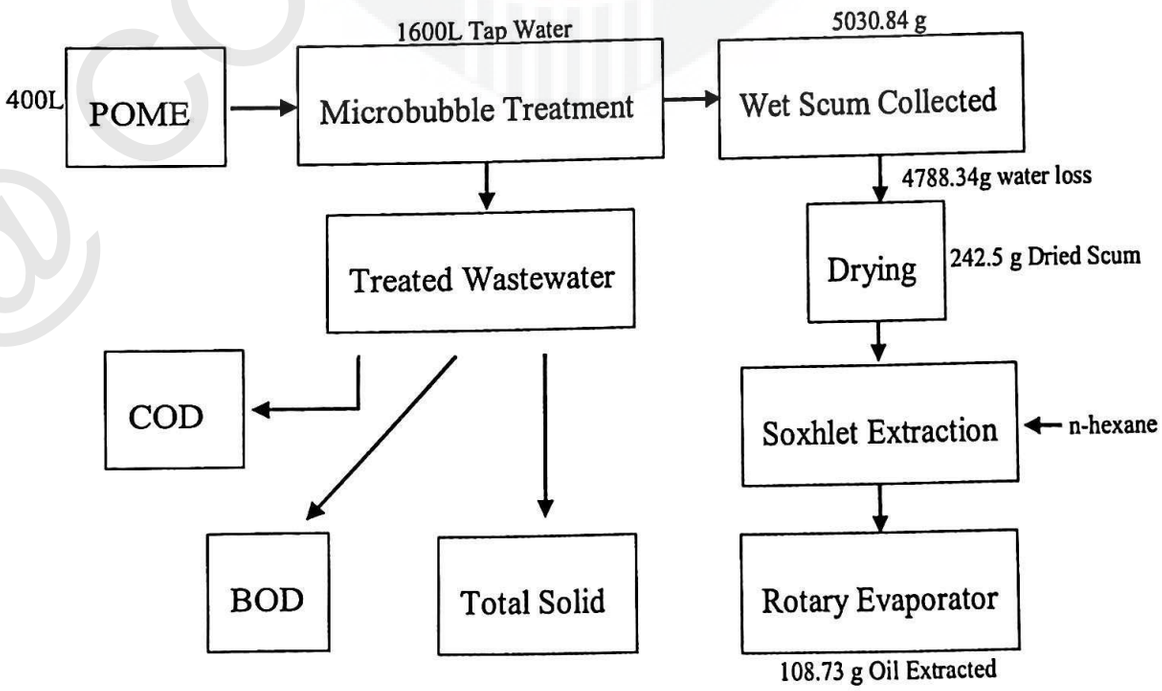
As the pre-treatment step of treating the POME wastewater, the microbubble system has shown a good sign as the analysis run by the fabricated machine featuring a tank with volume up to 2 m^3 combined with a centrifuge pump that able to produce bubble with micro size around 10μm to 50μm. After the process with duration 5 hours occurs, the value of the total solid (TS), chemical oxygen demand (COD), and biochemical oxygen demand (BOD) has shown a significant reduction for all of the analysis. The experiment show that more than 80% reduction for the total solid

content, 69% for the COD content, and 26.76% reduction for BOD content in the treated POME wastewater.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

Based from the result of total solid for 5x, 10x, and 30x dilution, the percentage of the total solid decrease from the pome is 80.95%, 88.9%, and 81.25% respectively. For the value of the chemical oxygen demand (COD), the percentage remaining after 3 hour of pre-treatment is 56.6% for 5x dilution, 40% for 10x dilution and 25% for 30x dilution of the sample. Next for the biochemical oxygen demand, the result show that 77.7% of BOD left in the treated sample after 3 hours. Finally, from the total of 242.5g of dried scum collected, 44.84% of oil manage to be extracted. The outcome of the experiment can be shown in a block flow diagram below;



Therefore, a conclusion can be made where the usage of microbubble system as the pre-treatment for the POME show a good sign as it manage to reduce the total solid, COD, and BOD value inside of the sample. Other than that, the dilution made before running the treatment help the treatment to be more efficient as the water added to the tank able to improve the pump efficiency to run the system.

Some recommendation can be suggested to solve the current problem existed at the fabricated microbubble generator that may give out effect towards the experimental design in producing an accurate size of bubble for further study. There are few problems that affect the production of the bubble with size including the lack of flow meter, no filling system, unsystematic skimming process for scum sampling, and ineffective sampling process for the wastewater treated.

Firstly, the flow meter can be installed at the piping system before the microbubble entering the tank. Then, the study on the effect of microbubble flowrate on the microbubble size is very appropriate to be learn as is the size of the bubble produced by the system is smaller than the expected size (10μm to 50μm), the pre-treatment can be considered as not achieving the target. Next, the filling system can be installed for easier process of transferring the POME wastewater sample into the fabricated tank. Other than that, the current skimming process using the water dipper also seems not very convenient and suggested to be replace by skimming net where it allow the wastewater with small particle pass through it but the bigger solid particle will remain on the skimming net. Finally, for collecting the wastewater sample, the tap or faucet can be installed at the bottom of the fabricated tank. This instalment is to

ensure that the wastewater sample are not mixed with the scum where the problem might occur if the sample is taken on top of the tank.

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APPENDICES

Sample Calculation

A. Total Solid Content for 5x Dilution

Sample	Weight of sample + crucible, g	Weight of crucible after dry, g
0 H	12.129	12.15
1 H	11.581	11.60
2 H	14.089	14.10
3 H	9.630	9.64
4 H	13.292	13.30
5 H	8.746	8.75

$$Total\ Solid = \frac{(Weight\ of\ the\ crucible + sample) - (Weight\ of\ empty\ crucible), g}{Volume\ of\ the\ sample, L}$$

$$= \frac{(12.15 - 12.129)g}{0.005\ L}$$

$$= 4.2\frac{g}{L} \times \frac{1000mg}{1g}$$

$$= 4200\frac{mg}{L}$$

$$Actual\ Total\ Solid = Total\ Solid \times Dilution\ in\ Tank$$

$$= 4200\frac{mg}{L} \times 5$$

$$= 21000\frac{mg}{L}$$

B. Total Solid Content for 10x Dilution

Min	Sample	Weight of crucible, g	Weight of sample + crucible, g	Total solids (g/L)	Total solid (mg/L)
0	A	9.408	9.418	2.0	2000
0	B	9.704	9.712	1.6	1600
30	A	13.158	13.166	1.6	1600
30	B	13.447	13.454	1.4	1400
60	A	13.257	13.263	1.2	1200
60	B	9.749	9.754	1.0	1000
90	A	13.981	13.985	0.8	800
90	B	13.198	13.202	0.8	800
120	A	13.109	13.113	0.8	800
120	B	8.903	8.907	0.8	800
150	A	12.462	12.465	0.6	600
150	B	9.243	9.245	0.4	400
180	A	13.024	13.025	0.2	200
180	B	13.519	13.520	0.2	200

$$Total\ Solid$$

$$= \frac{(Weight\ of\ the\ crucible + sample) - (Weight\ of\ empty\ crucible), g}{Volume\ of\ the\ sample, L}$$

$$= \frac{(9.418 - 9.408)g}{0.005L}$$

$$= 2.0\frac{g}{L} \times \frac{1000mg}{1g}$$

$$= 2000\frac{mg}{L}$$

$$Average\ Total\ Solid = \frac{Total\ Solid\ Sample\ A + Total\ Solid\ Sample\ B}{2}$$

$$= \frac{2000\frac{mg}{L} + 1600\frac{mg}{L}}{2}$$

$$= 1800 \frac{mg}{L}$$

$$Actual\ Total\ Solid = Average\ Total\ Solid \times Dilution\ in\ Tank$$

$$= 1800 \frac{mg}{L} \times 10$$

$$= 18000 \frac{mg}{L}$$

C. Total Solid Content for 30x Dilution

Sample	Min	Weight of crucible, g	Weight of sample + crucible, g	Total solids (g/L)	Total solid (mg/L)
A	0	57.842	57.851	1.8	1800
B	0	51.307	51.314	1.4	1400
A	60	47.913	47.918	1.0	1000
B	60	54.925	54.929	0.8	800
A	120	61.448	61.451	0.6	600
B	120	43.907	43.910	0.6	600
A	180	50.012	50.014	0.4	400
B	180	49.752	49.753	0.2	200

$$Total\ Solid$$

$$= \frac{(Weight\ of\ the\ crucible + sample) - (Weight\ of\ empty\ crucible), g}{Volume\ of\ the\ sample, L}$$

$$= \frac{(57.851 - 57.842)g}{0.005L}$$

$$= 1.8\frac{g}{L} \times \frac{1000mg}{1g}$$

$$= 1800\frac{mg}{L}$$

$$Average\ Total\ Solid = \frac{Total\ Solid\ Sample\ A + Total\ Solid\ Sample\ B}{2}$$

$$= \frac{1800\frac{mg}{L} + 1400\frac{mg}{L}}{2}$$

$$= 1600\frac{mg}{L}$$

$$Actual\ Total\ Solid = Average\ Total\ Solid \times Dilution\ in\ Tank$$

$$= 1600\frac{mg}{L} \times 30$$

$$= 48000\frac{mg}{L}$$

D. COD Reading for 5x Dilution

Sample	1st Reading (mg/L)	2nd Reading (mg/L)
O H	220	232
1 H	180	189
2 H	140	122
3 H	146	110
4 H	105	93
5 H	78	62

$$Average\ COD\ reading = \frac{1st\ reading + 2nd\ reading}{2}$$

$$= \frac{(220 + 232)\frac{mg}{L}}{2}$$

$$= 226\frac{mg}{L}$$

Actual COD Reading = Average COD Reading × Sampling Dilution × Dilution in Tank

$$= 226\frac{mg}{L} \times 5 \times 20$$

$$= 22600\frac{mg}{L}$$

E. COD Reading for 10x Dilution

Sample	1st Reading (mg/L)	2nd Reading (mg/L)
O H	157.67	144.67
1 H	111.67	95.33
2 H	79.67	70.67
3 H	66.33	54.67

$$Average\ COD\ reading = \frac{1st\ reading + 2nd\ reading}{2}$$

$$= \frac{(157.67 + 144.67)\frac{mg}{L}}{2}$$

$$= 151.17\frac{mg}{L}$$

Actual COD Reading = Average COD Reading × Sampling Dilution × Dilution in Tank

$$= 151.17 \frac{mg}{L} \times 10 \times 50$$

$$= 75585 \frac{mg}{L}$$

F. COD Reading for 30x Dilution

Sample	1st Reading (mg/L)	2nd Reading (mg/L)
O H	233.67	225.00
1 H	146.67	138.00
2 H	71.67	70.33
3 H	62.00	52.67

$$Average\ COD\ reading = \frac{1st\ reading + 2nd\ reading}{2}$$

$$= \frac{(233.67 + 225.00) \frac{mg}{L}}{2}$$

$$= 229.34 \frac{mg}{L}$$

Actual COD Reading = Average COD Reading × Sampling Dilution × Dilution in Tank

$$= 229.34 \frac{mg}{L} \times 30 \times 50$$

$$= 344010 \frac{mg}{L}$$

G. BOD Reading for 5x Dilution

Sample	D_i (mg/L)	D_f (mg/L)
O H	4.88	0.65
1 H	4.70	0.58
2 H	3.69	0.53
3 H	3.63	0.22
4 H	3.51	0.28
5 H	4.70	0.20

$$BOD\ Reading = \frac{D_i - D_f}{P}$$

$$= \frac{(4.88 - 0.65)}{\frac{5}{300}}$$

$$= 253.8 \frac{mg}{L}$$

$$Actual\ BOD\ Reading = BOD \times Sampling\ Dilution \times Dilution\ in\ Tank$$

$$= 253.8 \frac{mg}{L} \times 10 \times 5$$

$$= 12670 \frac{mg}{L}$$