"Treatment of POME via chitosan based flocculation" A studies of agitation speed and oil concentration in POME

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"I declare that this thesis is the result of my own research except as cited references. The thesis has not been accepted for any degree and is concurrently submitted in candidature of any degree".

Signature	:
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DEDICATION

In God I Trust.

Dedicated to all my beloved family especially to my father, mother, and my girlfriend Noreiera.

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ABSTRACT

Water pollution result waste from dismissal from work remains as processing plant palm oil is either pollution problem by is being faced our country. This polluted material removal already gets the attention since lately with use conventional method as reaction separation membrane, catalysis photograph, adsorption, flocculation, absorption and filtration. The cost to treat POME is very high lately. In order to find the alternative to solve the problem, this research is come out to study the effect of agitation speed and oil content concentration by using treatment POME via chitosan based on flocculation process. In this study, scope more focused to study agitation impact and oil content in POME. However there are the other parameter needs to be considered which is COD (chemical oxygen demand) and SS (suspended solid). There were three main processes viz preparation flocculent, study rate effect agitation and impact oil thickness and concludes with simulation experiment. In phase analysis, there were four analyses need to be alert, viz total suspended solid analysis, oil and grease analysis, turbidity analysis and COD analysis. Final result show that the best agitation speed is at 150 and the maximum percent oil removal is 88.2%. In conclusion chitosan has potential to be commercialized in wastewater treatment because it is safe and have many advantages.

ABSTRAK

Pencemaran air sisa akibat daripada pembuangan daripada sisa kilang seperti kilang pemprosesan minyak kelapa sawit merupakan salah satu masalah pencemaran vang sedang dihadapi oleh negara kita. Penyingkiran bahan tercemar ini telah mendapat perhatian sejak kebelakangan ini dengan mengunakan kaedah konvensional seperti tindak balas pemisahan membrane, foto pemangkinan, penjerapan, pengelompokan, penyerapan dan penapisan. Sejak akhir-akhir ini kos untuk perawatan sisa minyak kelapa sawit semakin meningkat. Oleh itu, kajian ini dijalankan untuk menkaji kesan kadar agitasi dan mengkaji kesannya ke atas kandungan minyak dalam sisa minya kelapa sawit. Dalam kajian ini, skop lebih tertumpu kepada mengkaji kesan agitasi dan kesan kepekatan kandungan minyak dalam sample. Namun begitu terdapat kajian lain yang harus diambil kira iaitu jumlah pepejal terampai dan analisis COD. Terdapat tiga peringkat utama proses iaitu penyediaan flocculent, mengkaji kesan kadar agitasi dan kesan kepekatan minyak dan diakhiri dengan analysis simulasi. Dalam peringkat analysis,terdapat empat analysis diambil kira iaitu jumlah pepejal terampai, analisis minyak dan gris, analisis COD dan kadar kekeruhan. Keputusan akhir kajian mencapai keputusan terbaik pada kadar agitasi yang ke 150 dan boleh menurunkan minyak sehingga 88.2% daripada kadungan sebenar. Kesimpulan yang dapat dibuat ialah chitosan berpotensi untuk dikomersialkan dalam bidang perawatan air kerana ianya selamat dan mempunyai banyak kelebihan.

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LIST OF SYMBOLS

NOMENCLATURE

SS Suspended solids

COD Chemical Oxygen Demand

CHAPTER 1

INTRODUCTION

1.1 Background

To this century various efforts have been made to reduce pollution of nature rate caused by remnants industry. Among the efforts was done is to find alternative a more effective to stem this problem. Most of the research carried out more converging towards find method a more convenient, cheap, effective, easy and safe. There are various type of pollutant such as solid waste, hazardous waste, toxic waste and liquid waste. One of the pollution components is oil waste. The concentration of oil in effluents from different industrial sources is found to be as high as 40,000mg/l (Guo, J. and Lua, A.C. 2000). It is important to remove oil from the waste water in order to: (1) Prevent interfaces in water treatment units; (2) reduce fouling in process equipment; (3) avoid problems in biological treatment stages; (4) comply with water discharge requirements. There are many methods for wastewater treatment such as coagulation/flocculation, filtration, membrane process; sedimentation and biological process. Every of this method have their advantages and disadvantages. Flocculation process is one the effective method widely used in waste water treatment. Normally flocculation of waste oil using chemical flocculent such as alum, aluminum chlorohydrate, aluminum sulfate, calcium oxide, iron(III) chloride, iron(II) sulfate, sodium aluminate, silicate. But the problem is some of chemical flocculent produce activated sludge which is extremely hazardous for example Aluminum's sulphate (alum) and polyaluminum chloride (PAC). Some of natural flocculants that are

commercially used are Moringa oleifera, Isinglass, Papain, and chitosan (Fatima Hammy, Guy Mercier and Jean-François Blais[,])

Therefore chitosan is believed to be the best natural flocculent to remove oil from wastewater. It is a natural, hydrophilicity, biocompatibility, adsorption property, flocculating ability, non-toxic, polyelectolysity, anti bacterial, and biodegradable. Chitosan is available as solution, flake, fine powder, bead and fiber. Study continued to be carried out so that application of chitosan in wastewater treatment can be increased. In this study, focus more focusing to determine the best agitation speed and maximum percent oil can be removed in the sample.

1.2 **Problem Statement**

Palm oil industrial development highly evolved to this century. Yet lie behind stated achievement, exist second question that arise. Problem driving at is waste production palm oil to environment. This problem had caused disruption to environmental ecosystem, it especially habitat aquatic life. Therefore study continued to be carried out to stem this problem. Studies carried out include how to treat oil waste and then how to produce it become more useful to mankind. For example, palm oil waste can be purified to produce drinking water (Berita Harian, 16 Mac 2007).

Nevertheless usage of chitosan in the field waste treatment oil-palm still phased study. Much research as evidence those carried out by researchers cover modification aspect, improvement and cost reduction treatment. Problem that happen includes instrumentation need high cost. For example, using activated carbon as adsorbent, using chitosan is cheaper than activated carbon because chitosan is more readily available. Furthermore chitosan easy to be obtained because it originated from remnants marine life such as prawn and crab. Therefore this study carried out to study the potential of chitosan as flocculent.

To determine the potential chitosan as agent collector, over study focused towards modification and analyze outcome of the study. The studies are more concentrates to get the best performance of chitosan especially on agitation speed and oil content analysis. The other analysis to be alert is suspended solid, turbidity, and chemical oxygen demand.

1.3 Objectives of the study

• To study the effect of agitation speed and oil content concentration by using treatment of POME via chitosan based on flocculation process.

1.4 Scope of the research

Several scopes have been identified in order to achieve the objective:

- To investigate the potential and effectiveness of chitosan as a flocculent to flocculate the suspended solid and residue oil from POME.
- To analyze total suspended solid before and after treatment process.
- To analyze water quality after treatment process.

CHAPTER 2

LITERATURE REVIEW

2.1 Palm Oil Mill Effluent (POME)

2.1.1 Palm oil issue

Palm oil is a form of edible vegetable oil obtained from the fruit of the oil palm tree. Palm oil is a natural food that has been consumed for more than 5,000 years. Palm oil is produced from the fruit of the oil palm, or Elaeis Guinnesis tree, which originated in West Guinea. While the tree was introduced into other parts of Africa, South East Asia and Latin America during the 15th century, it was first introduced 1870 as an ornamental plant. Large commercial planting and cultivation of the plant in Malaysia did not begin until the mid- 1990's. The world's largest producer and exporter of palm oil today is Malaysia, where it maintained dominance of the palm oil trade, with 49.3% of the market share (Global Oil & Fats Business Magazines).



Source: MPOB * North East – Japan, South Korea, North Korea and Taiwan

Note: The summation of export data does not tally with the total exports due to exclusion of exports to other countries.

Figure 2.1: Malaysian Palm Oil Export to Major Destination (tones)



Source: Tabulated from MPOB data

Note: The summation of regional data does not fally with MPOB figure on total exports due to exclusion of data from Malaysia.

Figure 2.2: World's Largest Importer of Palm Oil in Year 2005-2006

Oil palm is an important crop in Malaysia. Approximately in 2006, 15.60 million tones of crude palm oil (CPO) were produced from 4.172 million hectares planted areas and 3,740 million hectares matured areas over the Malaysia (Edoardo, et al., 2002). The process to extract the oil actually requires large quantities of water for steam sterilizing the palm fruit bunches and clarifying the extracted oil. It is estimated that for one tone of crude palm oil produced, 5-7.5 tones of water are required, and more than 50% of the water will end up as palm oil mill effluent (POME). The complete palm oil and palm kernel oil extraction process flow is illustrated in Figure 2.5. Thus, while enjoying a most profitable commodity, the adverse environmental impact from the palm oil industry cannot be ignored. Based on the current annual rate of oil palm harvesting, some 4.5 million metric tones of effluents are being produced and discharged into the rivers.



Figure 2.3: The complete palm oil and palm kernel oil extraction process flow

2.1.2 Palm Oil Mill Effluent (POME)

POME is a colloidal suspension of 95-96% water, 0.6-0.7% oil and 4-5% total solids including 24% suspended solids originating from the mixture of a sterilizer condensate, separator sludge and hydro cyclone wastewater . The raw or partially treated POME has an extremely high content of degradable organic matter, which is due in part to the presence of unrecovered palm oil. POME characteristic and standard discharge limit is illustrated in Table 1.0. In order to regulate the discharge of effluent from the crude palm oil industry as well as to exercise other environmental controls, the Environmental Quality (Prescribed Premises) (Crude Palm Oil) Order, 1977, and the Environment Quality (Prescribed Premises) (Crude Palm Oil) Regulations, 1977, were promulgated under the Environmental Quality Act, 1974. Due to these factors, the palm oil industry faces the challenge of balancing the environmental protection, its economic viability and sustainable development. There is an urgent need to find a way to preserve the environment while keeping the economy growing.

This highly polluting wastewater can make severe pollution of waterways due to oxygen depletion and other related effects. The presence of oil-waste makes the bio-degrading process of the effluents difficult as oil inherently difficult to disintegrate; so that will lead to disturb the lake or river ecosystem (Sharon, P, 2005). But actually the recent studies have been trickling that single tropical oil out of saturated fat milieu which confers resistance to rancidity, also makes it less prone to oxidative polymerization, excellent stability at high temperatures. Palm oil contains components with nutritional and beneficial health properties where these nutrients have many advantages include water soluble antioxidants , for the example flavonoids and phenolic acids which generally known as a powerful free radical scavenger better than vitamins C,E and beta-carotene to prevent skin aging and inhibits UV radiation, penetrate the blood-brain barrier to help protection of the brain and nerve tissue from oxidation, enhance capillary strength and vascular function, alleviate PMS problems edema from injury or trauma, leg swelling and retinopathy, varicose veins (Yeoh, B.G ,2004). There are as many as 5 mg of antioxidant polyphenols in every 10 grams of palm oil.

	ADVANTAGES	DISADVANTAGES
1	Produce biogas- methane gas	- Difficult to disintegrate by bacteria.
2	Produce animal feed supplement.	-Ruin the river or lake ecosystems
3	Used for fertilizer	-unpleasant smell
4	Rich in phytonutriens (antioxidant)	

Table 2.1: Advantages and Disadvantages of POME

 Table 2.2: Characteristics of POME and its respective standard discharge limit by the

 Malaysian Department of the Environment

(Abdul Latif et al., 2003)

PARAMETERS	STANDART LIMIT	CONCENTRATION mg/L
	mg/L	
pH	5-9	4.7
Oil and grease	50	4000
BOD	100	25000
COD	-	50000
Total Solids	-	40500
Suspended solids	400	18000
Total nitrogen	150	750

All values, except pH and temperature, are expressed in mgL-1.

Statutory incubation conditions

This additional limit is the arithmetic mean value determined on the basis of a minimum of four samples taken at least once a week for four weeks consecutively.

Value of filtered sample

Therefore, the main issue in this case was to find ways to make use of palm oil waste effluent in the wise condition which was described as a very important step forward in environmental conservation and technological advancement which could benefit both the environment and the public.

2.2 Wastewater treatment

2.2.1 Effect of wastewater to the environment.

Wastewater is any water that has been adversely affected in quality by any anthropogenic influence. It therefore includes liquid waste discharged from domestic houses, industrial, agricultural or commercial processes. It does not include rainwater uncontaminated by human activities.

There has been a widespread awareness for a cleaner environment. The major sources of pollution can be from water and air. Toxic chemicals, harmful bacteria, unwanted solid waste and most of these can be classified as organic & inorganic waste. When organic waste enters into the stream, it will stimulate bacterial growth that affects aquatic life forms by depleting the quantity of oxygen level in the water. The stream becomes septic due to bacteria undergoing anaerobic metabolism, producing odors and darkening the water appearance. The inorganic wastes such as copper, nickel, lead, when entered into the stream, are basically toxic substances that cannot be further broken-down by the bacteria and can be poisonous to human health & aquatic life forms. There is some common type of pollution effected by waste water:

Total Solids is the sum of suspended solids and dissolved solids. Suspended solids are those that can be removed by filtration. Biodegradable organic. Those that can be broken down by micro-organism to form stable compounds such as CO2 and water. Effluent is the liquid product discharged or emerging from a process. COD (Chemical Oxygen Demand) It is the amount of oxygen (usually measured in mg/1 (milligram per litre) required to oxidize both organic and oxidizable inorganic compounds. BOD (Biochemical Oxygen Demand) It is the amount of oxygen (mg/1) required by micro-organism to consume biodegradable organic in waste-water.

2.2.2 The principle of commercialize waste water treatment

Wastewater treatment is the process that removes the majority of the contaminants from waste-water or sewage and produces both a liquid effluent suitable for disposal to the natural environment and sludge. To be effective, sewage must be conveyed to a treatment plant by appropriate pipes and infrastructure and the process itself must be subject to regulation and controls. Other wastewaters require often different and sometimes specialized treatment methods.



Figure 2.4: Typical 'Waste Water Treatment Plant Flow

Firstly for commercialize waste water treatment raw wastewater will flows into the equalization tank then into the primary process (Physical Chemical Treatment). This process will basically involve in two chemicals. A coagulant, usually an inorganic substance, is used to destabilize the electrostatic repulsive forces which tend to keep colloidal and soluble suspension particles apart and prevent them from gathering together. After that, the flocculants, usually organic in nature, must be added to combine the particles into bigger flocs, which can then be more easily floated or settled. In a well-designed system there should have proper flowrate that allows the coagulant to have sufficient time to destabilize the repulsive forces before the flocculant is added. In the aeration (Biological Treatment) basin, organic matter will acts as food supply for bacteria to metabolize the waste solids resulting in absorbing oxygen (BOD) and releasing CO2. The process removes organic matter from solution by synthesis into microbial cells. Then it will transfer to a clarifier for separation of the sludge and effluent. The sludge is pollutants then converted into concentrated form. This sludge has to be further treated in order to prevent further pollution of waterways. The basic aim of sludge treatment is to reduce the volume and destroy or stabilize sludge solids before final disposal. The chlorination of water and wastewater is to disinfect & destroy Pathogens and control microorganisms. Chlorine is also used for oxidation where the oxidant used is iron and manganese. In general when the water appeared to be cloudy as a result of many suspended particles floating & the electrical repulsive charge is actually holding the particles in suspension. After that, the next process occur called coagulation and flocculation process.

2.3 Flocculation

2.3.1 Introduction

One of the most important treatment processes in surface water treatment is flocculation. Flocculation is a condition in which clays, polymers or other small charged particles become attached and form a fragile structure, a floc. In dispersed clay slurries, flocculation occurs after mechanical agitation ceases and the dispersed clay platelets spontaneously form flocs because of attractions between negative face charges and positive edge charges. Flocculation process very important because all waters, especially surface waters, contain both dissolved and suspended particles Flocculation processes are used to separate the suspended solids portion from the water. Total suspended solid are solid materials, including organic and inorganic, that are suspended in the water. These would include silt, plankton and industrial wastes. The suspended particles vary considerably in source, composition charge, particle size, shape, and density. Correct application of flocculation processes depend upon understanding the interaction between these factors. The small particles are stabilized (kept in suspension) by the action of physical forces on the particles themselves. One of the forces playing a dominant role in stabilization results from the surface charge present on the particles. Most solids suspended in water possess a negative charge and, since they have the same type of surface charge, repel each other when they come close together. Therefore, they will remain in suspension rather than clump

together and settle out of the water. The function of flocculants is to facilitate the settling of suspended solids in solution.

2.3.2 Flocculation mechanism



Coagulation mechanism



Like charged particles repel





Removal of charge can enable particles to approach close enough to coagulate



Polymer chains can "bridge" particles to create larger masses that settle out

2.3.3 Flocculent

Flocculent is one of the important components in the flocculation mechanism. Flocculants, or flocculating agents, are chemicals that promote flocculation by causing colloids and other suspended particles in liquids to aggregate, forming a floc. Flocculants are used in water treatment processes to improve the sedimentation or filterability of small particles. There are two types of flocculent which is chemical flocculent and natural flocculent.

Examples of the chemical flocculent:

alum aluminium chlorohydrate aluminum sulfate calcium oxide iron(III) chloride iron(II) sulfate sodium aluminate Examples of the natural flocculent:

<u>Chitosan</u> <u>Moringa oleifera</u> seeds <u>Papain</u> A species of <u>Strychnos</u> (seeds) <u>Isinglass</u>

Some of the chemical flocculent such as Aluminum sulphate (alum) and polyaluminum chloride (PAC) produce activated sludge which is very hazardous. Hence, it is suggested that a biodegradable flocculent can be a better alternative .The best alternative for flocculent in this study is chitosan.

2.4 Chitosan

2.4.1 Definition and Composition of Chitosan

Chitosan is a fiber-like substance derived from chitin, a homopolymer of β -(1 \rightarrow 4)-linked *N*-acetyl-D-glucosamine. Chitin is the second most abundant organic compound in nature after cellulose (Ruiz-Herrera, 1978). Chitin is widely distributed in marine invertebrates, insects, fungi, and yeast (Austin, et al., 1981) However; chitin is not present in higher plants and higher animals. Generally, the shell of selected crustacean was consisting of 30-40% protein, 30-50% calcium carbonate and calcium phosphate, and 20-30% chitin (Knorr, D., 1984). Chitin is widely available from a variety of source among which, the principal source is shellfish waste such as shrimps, crabs, and crawfish. It also exists naturally in a few species of fungi.

For the structure, chitin is associated with proteins and, therefore, high in protein contents. Chitin fibrils are embedded in a matrix of calcium carbonate and phosphate that also contains protein. The matrix is proteinaceous, where the protein is hardened by a tanning process (Ruiz-Herrera, 1978). Studies of Asford and co-workers (1977) demonstrated that chitin represents 14-27% and 13-15% of the dry weight of shrimp and crab processing wastes, respectively.

By refer to their chemical structure; chitin and chitosan have similar chemical structure. Chitin is made up of a linear chain of acetylglucosamine groups while chitosan is obtained by removing enough acetyl groups (CH3-CO) for the molecule to be soluble in most diluted acids. This process is called deacetylation. The actual difference between chitin and chitosan is the acetyl content of the polymer. Chitosan having a free amino group is the most useful derivative of chitin.



CHITOSAN



Figure 2.5 structures of chitin and chitosan

2.4.2 Characteristics of Chitosan

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Chitosan is a non toxic, biodegradable polymer of high molecular weight, and is very much similar to cellulose, a plant fiber. This can be proof by refer to the figure 2.2.



Figure 2.6 Structures of Cellulose, Chitin, and Chitosan

As seen in Figure 2.2, the only difference between chitosan and cellulose is the amine (-NH2) group in the position C-2 of chitosan instead of the hydroxyl (-OH) group found in cellulose. However, unlike plant fiber, chitosan possesses positive ionic charges, which give it the ability to chemically bind with negatively charged fats, lipids, cholesterol, metal ions, proteins, and macromolecules (Tolaimate et al., 2000). In this respect, chitin and chitosan have attained increasing commercial interest as suitable resource materials due to their excellent properties including biocompatibility, biodegradability, adsorption, and ability to form films, and to chelate metal ions.

CHAPTER 3

METHODOLOGY

3.1 Introduction

Flocculation, a gentle mixing stage, increases the particle size from submicroscopic microfloc to visible suspended particles. Figure 3.1 shows the flocculator used during the study. There are six spindles and the speed is uniform for every spindle for certain analysis. The microflocs are brought into contact with each other through the process of slow mixing. Collisions of the microfloc particles cause them to bond to produce larger, visible flocs called pinflocs. The floc size continues to build through additional collisions and interaction with inorganic polymers formed by the coagulant or with organic polymers added. Macroflocs are formed. High molecular weight polymers, called coagulant aids, may be added during this step to help bridge, bind, and strengthen the floc, add weight, and increase settling rate. Once the floc has reached it optimum size and strength, the water is ready for the sedimentation process.



Figure 3.1 Design contact times for flocculation range from 15 or 20 minutes to an hour or more.

3.2 Material

3.2.1 Apparatus

Apparatus used for the study including the process to flocculate and to run analysis after treatment. For example flocculator used for flocculating process while homogenator used to homogenize water and oil. For oil and grease analysis the main apparatus needed is separator funnel. The others apparatus used for COD analysis, suspended solid analysis and turbidity analysis.

1Separatory funnel, 2-L, with TFE* stopcock. Distilling flask, 125-mL. Liquid funnel, glass. Filter paper, 11-cm diam.[†] Flocculator Test tubes, 50-mL, glass. Homogenator Water bath, capable of maintaining 85°C. Vacuum pump or other source of vacuum. Distilling adapter with drip tip. Ice bath. Waste receptacle, for used solvent. Desiccator. Oven Glass fiber filters Filtering flask Atomic spectrophotometer

3.2.2 Reagents

A reagent or reactant is a substance or compound consumed during a chemical reaction. More of the reagents used during analysis period. For example Acetic acid used to activate the sample while n-Hexane used for emulsion oil and grease. For COD analysis the reagent that used for analysis is potassium dichromate and mercury powder. More of the reagent used for sample treatment, analysis after treatment and simulation.

Hydrochloric or sulfuric acid, 1:1: Mix equal volumes of either acid or reagent water.

n-Hexane, boiling point 69°C. The solvent should leave no measurable residue on evaporation; distill if necessary. Do not use any plastic tubing to transfer solvent between containers.

Methyl-tert-butyl ether (MTBE), boiling point 55°C to 56°C. The solvent should leave no measurable residue on evaporation; distill if necessary. Do not use any plastic tubing to transfer solvent between containers.

Sodium sulfate, Na₂SO₄, anhydrous crystal.

Solvent mixture, 80% n-hexane/20% MTBE, v/v.

Chitosan powder (500 g)

Cooking oil (500 ml)

Palm oil mill effluent (POME)

Distilled water

Acetic Acid

Sodium Hydroxide

potassium Dichromate

mercury powder

Sulfuric Acid

3.3 Experimental procedure

3.3.1 Preparation of flocculent

To prepare flocculent we need to mix 7% of acetic acid into distilled water and then mix together chitosan powder so that the solution will became 2% in that acid solution. The mixture was stirred in a blender for 2 min to extract the active ingredients. The resulting suspension was filtered through a muslin cloth.

3.3.2 Methodology

- 1. POME was allowed to sediment for 1 h with the supernatant being analyzed for its residue oil content before and after sedimentation. A conventional jar apparatus (Stuart Science Flocculator model (SW1)) was used to flocculate the samples of POME with chitosan. This apparatus could accommodate six beakers by add the same quantity of chitosan (flocculent).
- 2. The contents of each beaker can be simultaneously stirred at the different speed with six-spindle of steel paddles for the first analysis and the parameter of palm oil mill effluent concentration is constant for each beaker. Each beaker was filled with 1 l of POME.
- After adding the flocculent into the suspension, the beakers were rapidly mixed at various mixing time (5–60 min). The effect of sedimentation time was analyzed at best condition at pH (3 – 6) to remove residue oil and suspended solid from POME.

- 4. It is same thing with the temperature where every part of the experiment the temperature was set at 26–30 ∘C as it is the best condition and standard for this experiment. In this part the agitation speed was set at the different set point at 100,150,200 and 250 rpm of mixing rate, 1 h of mixing time and 1 h of sedimentation time at pH 4.5.
- 5. After that the residue oil content was measured using the oil and grease method recommended by APHA Standard Method of Examination of Water and Wastewater (Sharon and P, 2005), with *n*-hexane being used as the oil-extraction solvent. The oil and grease content in the suspension was determined for each sample of POME both before and after experiment.
- 6. Three replicates of each test were undertaken with the mean value obtained for residual oil content being calculated from the replicates. The procedure for second variable is same thing but the palm oil mill effluent concentration was varying for every six beaker and meanwhile the agitation speed was constant at standard speed at 100 rpm. All the another standard parameter such as pH, time and temperature are same thing like the procedure before. After that the result will analyzing.



Figure 3.2 The experiment procedure were summarize as figure below

3.3.3 Oil and grease method

Dissolved or emulsified oil and grease was extracted from water by intimate contact with an extracting solvent. Some extractable, especially unsaturated fats and fatty acids oxidize readily; hence special precautions regarding temperature and solvent vapor displacement are included to minimize this effect. Organic solvents shaken with some samples may form an emulsion that is very difficult to break. This method includes a means for handling such emulsions. Recovery of solvents is discussed. Solvent recovery can reduce both vapor emissions to the atmosphere and costs.

Procedure

- 1 Mark sample bottle at the water meniscus or weigh the bottle, for later determination of sample volume.
- 2 If sample has not been acidified previously, acidify with either 1:1 HCl or 1:1 H₂SO₄ to pH 2 or lower (generally, 5 mL is sufficient for 1 L sample).
- 3 Using liquid funnel, transfer sample to a separatory funnel. Carefully rinse sample bottle with 30 mL extracting solvent (either 100% *n*-hexane, or solvent mixture) and add solvent washings to separatory funnel.
- 4 Shake vigorously for 2 min. Let layers separate.
- 5 Drain aqueous layer and small amount of organic layer into original sample container.
- 6 Drain solvent layer through a funnel containing a filter paper and 10 g Na₂SO₄, both of which have been solvent-rinsed, into a clean, tared distilling flask. If a clear solvent layer cannot be obtained and an emulsion of more than about 5 mL exists, drain emulsion and solvent layers into a glass centrifuge tube and centrifuge for 5 min at approximately 2400 rpm.
- 7 Transfer centrifuged material to an appropriate separatory funnel and drain solvent layer through a funnel with a filter paper and 10 g Na₂SO₄, both of which have been prerinsed, into a clean, tared distilling flask. Recombine aqueous layers and any remaining emulsion or solids in separatory funnel.