CERTICATION OF APPROVAL

## OIL RECOVERY AND HEAVY METALS REMOVAL FROM PALM OIL MILL EFFLUENT BY USING CEIBA PENTANDRA (L.) AS A NATURAL SORBENT

by

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June 2010

# **CERTIFICATION OF ORIGINALITY**

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgement, and that the original work contained herein have not been undertaken or done by unspecified sources.

TENGKU FARIDAH TENGKU OMAR

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## **CHAPTER 1**

## **INTRODUCTION**

#### 1.1 Background of Project

Nowadays, the Malaysian palm oil industry is growing rapidly and becomes a very important agriculture-based industry, where the country is the world's leading producer and exporter of palm oil. However wet process of palm oil milling basically consumes a large amount process of water. It is estimated that for 1 tonne of crude palm oil produced, 5-7.5 tones of water will be used, and more than 50% of the water will end up as palm oil mill effluent (POME). The raw POME is a colloidal about 95% of water, 0.7% of oil and 5% of total solids [3]. If the effluent is discharged untreated, it can certainly cause considerable environmental problems due to its high biochemical oxygen demand, chemical oxygen demand, oil and grease, and total solids as well as suspended solids [3].

#### 1.2 Problem Statement

Palm oil industries in Malaysia has grown to become an important agriculture based industry and it is accounted for about 52% of the world palm oil output, which generated about RM 13 billions export earning for the country. By increasing the number of production of palm oil in the region, the disposal of the processes waste will raise and become a main problem in the future if it is not being treated properly starts from now. Palm oil mill waste or also known as POME is very polluting because it has high organic content. Malaysian Department of Environment (DOE) only allows the content of oil and grease about 50mg/l in POME but currently, most of POME has about 4000mg/l which is relatively high compared to DOE's requirement [1]. So, the main objective of this project is to recover the oil/metal from palm oil mill effluent (POME) waste by using natural sorbent. The development of natural absorbent as the main sorbent for the oil recovery process will be focused in this project. This is because natural source is cheap and available, as there would be no problem to increase its consumption.

## 1.3 Project Objectives

In this project, some objectives have been highlighted as shown below:

- a) To remove oil/heavy metals from palm oil mill effluent (POME) by using *Ceiba* pentandra (L.) as a natural sorbent.
- b) Study the capability of *Ceiba Pentadra (L.)* compared to other types of adsorbents to remove residue oil;
- c) To maximize the efficiency of natural absorbent for the oil recovery process from POME.

In order to achieve the objectives of this project, the steps that will be taken must be based on the time frame as per shown in Gann Chart (Appendix I). Besides that, this project will be divided into several stages within the period of time. The scope of study will include such identifying various types of engineering parameters and factors that contribute to oil recovery process.

#### 1.4 Relevancy of the Project

This study aims at removing oil/metals from palm oil mill effluent by using *Ceiba pentandra* (L.). *Ceiba pentandra*(L.) has been chosen as the basic materials for the oil sorbents. In addition, the increasing risk of chemical plants generate waste that contaminated with oil as waste will entrain various form of oils and it will pollute nearby bodies of water.

#### 1.5 Theory

Absorption process is defined as separation of components in fluid mixtures by transfer one or more components (the absorbents) into the internal porous solids (the absorbents) where there are held by molecular forces. In this case, the oil is known as absorbate and the kapok is defined as absorbents. This oil is mixed with the water and it is going to be separated by this process. The component of oil has been separated from the mixture and will be transferred into porous solid which is kapok. Basically, the absorption process can be divided into two main methods which are physical and chemical absorption (chemisorptions).

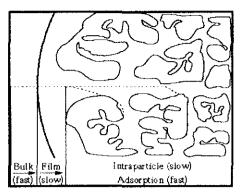


Figure 1 : Absorption Process

The absorption theory can be explained by four main steps that requires the movement of organic/inorganic molecule into surface sites which are:

- a) Bulk fluid transport
- b) Film transport
- c) Intra particle Physical attachment

The absorption of oil by human kapok is categorized as physical absorption because the process occurs rapidly since the chemisorptions process is a slow process. The absorption occurs at fast rate from the bulk region (mixture of oil and water) into surface of kapok but in film surface, the movement of oil entering the kapok interface becomes slow. The surface of kapok is not smooth but it is covered by pores where the oil may adhere and penetrate. This kind of hair surface will prepare a good surface to be wetted by non-polar liquid such as oil.

## 1.6 Oil absorption

Currently, the worldwide expansion of oil consumption is increasing since the sea is congested with the oil tankers carrying crude oil [1]. As a result, the percentage of oil contamination on water surface will also increase. Besides that, the industrial effluent from chemical plant especially and other chemical plant will produce various form of oil pollutant nearby the body of water. There are many methods had been developed to remove the oil floating and suspended in waters of all sorts. Mostly the oil-absorbing materials are divided into two groups which are synthetic high molecular substances and natural fibers. There are many examples of synthetic high molecular absorbing materials such as polypropylene, and polyurethane form. For natural sorbents, mostly consists of cotton fiber, coconut shell fibers, and peat fibers. Those made from high synthetic molecular substances; mostly have the fundamental defect because of lacking the resistance to oils. However, another disadvantages when the materials disposed of incineration in conjunction with absorbed oils, it gives rise to molten polymers which will do damage the incinerators and issues toxic gases [21]. Besides that, the oil adsorbing materials made of synthetic high molecular substances have a fatal drawback that they fail to exhibit any adsorbing activity on so called chocolate mousse and pitch on waves, form balls in consequence of the emulsification caused thereon by a small portion of entrapped sea area. The oil adsorbing materials made of synthetic high molecular substances also may cause the serious ecological problems if not thorough recovered after use on seas or oceans [21]. It is different with natural adsorbing materials because of their high hydrophilicity; it will exhibit a high water adsorbing capacity. However the materials must undergo preliminary treatment design to impart water repellency or waterproofness [21].

After various studies has been made on oil adsorbing materials, *Ceiba pentandra (L.)* has been discovered are free from disadvantages attendant upon the conventional oil adsorbent and exhibit an extremely high coefficient of oil adsorption and prove highly advantageous for the purpose of oil adsorption[21]. Ceiba pentadra (L.) excel than fibers of synthetic high molecular compound, particularly polyurethane, polypropylene, to some extent in hydrophilicity and therefore function advantageously upon and provide effective adsorption of chocolate mousse which defies adsorption by synthetic high molecular oil adsorbent [21].

When kapok fibers are used as the oil adsorbent in accordance with the present invention, the oil adsorbent has hydrophilicity such that it can be thrown in its unaltered form into water and it begins to adsorb oils floating on or suspended in water. Since kapok fibers have insufficient spin ability, it may be wrapped in net formed suitable fibers due to consideration focused to the suitability of their finished form to the condition in which oil are floating on or suspended in water [21].

Kapok fibers are classified as natural fibers which are composed by cellulose, lignin and pentosan, the oil adsorbent of the present invention which has served the purpose of oil adsorption and has been recovered from the water can be disposed by incineration without entailing any difficulty such as is involved in the case of synthetic fibers. Even if the oil adsorbent used on a sea or ocean should escape thorough recovery after use, there is no possibility that the residual oil adsorbent will give rise to any problem concerning the ecosystem because the adsorbent is made up of natural high polymers capable of spontaneous decomposition [21].

## **CHAPTER 2**

## LITERATURE REVIEW

#### 2.1 Palm Oil Industry

Currently Malaysia is the largest producer and exporter of palm oil products. The Malaysian palm oil milling and refining industries developed rapidly in the 1980's such that palm oil has emerged as one of the major oil commodities in the world oil and fats market. Approximately 11.9 million tonnes of crude palm oil (CPO) were produced that amounted to RM14.79 billion in the year 2002 (MPOPC 2003) [5]. Aside from being one of Malaysia's highest money earning industry, palm oil production is also one of the major potential, if untreated, organic polluters of the environment producing very high strength waste effluents.

Palm oil processing is carried out using large quantities of water in mills where oil is extracted from the palm fruits. During the extraction of crude palm oil from fresh fruits, about half or 50% of water results in palm oil mill effluent (POME). In both conventional and modern milling setting, these solid waste products are all put to economically useful purposes such as fuel material and mulch in agriculture industries. But for the POME, it is usually discharged into the surrounding environment, either as raw materials or treated. In Malaysia itself, the palm oil industries contribute 83% of the single largest polluter and this situation is probably similar in other palm oil producing countries. This current situation needs us to look at the effect of raw POME on the environment.

The constituents of raw POME have been reported to be a colloidal suspension of 95-96% water, 0.6-0.7% oil and 4-5% total solids including 2-4% suspended [3]. The raw POME has an extremely high content of degradable organic matter, which is due in part to the presence of unrecovered palm oil. So, POME should be treated before discharged to avoid serious environmental pollution. In 1992, Malaysia produced about 6.7 million tonnes of crude palm oil that contributing to 15.9 million cubic meters of POME [5]. Raw POME has a high biochemical (BOD) which is about 100 times more that the sewage. In 1998, there were 330 palm oil mills

generated 32 million tonnes of POME per year [5]. Besides that, palm oil mills also generated large amounts of solid and liquid by products, empty fruit bunches (EFB), fiber, shell and palm oil mill effluent (POME). And it can be concluded that the total BOD load produced was about 1560 tonnes per day, equivalent to the domestic sewage generated by a population of 31.2 million people [5].

Based on Malaysian Sewage and Industrial Effluent Regulations 1979, the content of oil and grease inside the water that allowed by them is 10ppm. *Table 8* (Appendix II) has shown comparison of the current process that had been used for POME recovery and treatment. No single method is effective for the remediation of the oily water mixtures, but the use of oil sorbent has become commercially and operationally effective. The characteristics of hydrophobic-oleophilic for the oil sorbents may transform the liquid oil into solid or semi-solid phrase.

As mentioned on the objective of the project, the development of natural sorbents for oil recovery from POME is required. It is because natural sorbents are more environmentally-friendly, economically and the most important they are locally available. As a result, organic and inorganic natural product such as peat moss, cotton, rice husk, kenaf and *Ceiba pentandra* or kapok have become candidates of choices to replace synthetic sorbents.

Based on the studied, Kapok is naturally good oil sorbent among the others. Kapok or *Ceiba pentandra* (L) Gaertn is from *Bombaceae* and it is commonly named after certain countries, highland and lowland. In Malaysia, the local name for kapok is *kekabu*. Kapok is one of the natural sorbent that has a good oil absorption and exhibits excellent hydrophobic-oleophilic characteristics. So, the physical and chemical properties of kapok fibre as oil sorbent were characterized. Beside that, the oil types on the oil sorption characteristics of kapok as natural sorbent were studied in a batch system. The study on oil sorption capacity, oil entrapment stability, percentage of dynamic oil retention, saturation time, packing height reduction and reusability of kapok has been done.

#### 2.2 The Origin of Palm Oil Mill Effluent

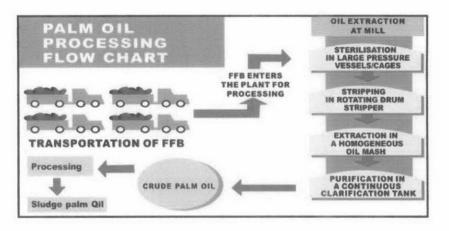


Figure 2 : The stages involved in palm oil processing plant.

Palm oil mill effluent is defined as a waste produced from the palm oil processing plants. This oily waste mostly produced in large volumes and contributes major problem to the palm oil processing mill's waste stream. The **Figure 2** above shows the stages involved in palm oil processing in the palm oil factory.

During the crude oil extraction process, large quantities of water are used. About 1.5m<sup>3</sup> of water is characteristically used to process one tone of FFB. From this quantity, about 50% of the water results in the POME, and the rest being lost as steam, mainly through sterilizer exhaust, piping leakages, as well as wash waters (Oil Palm & The Environment A Malaysian Perspective, 1999). POME comprises a combination of the wastewaters which are principally generated and discharged from the following major processing operations as seen in the **Figure 2** above.

- Sterilization of Fresh Fruit Bunch
   Sterilizer condensate is about 36% of total POME.
- Clarification of the extracted Crude Palm Oil
   It is about 60% of total POME for clarification wastewater.
- Hydro cyclone separation of cracked mixture of kernel and shell-hydro cyclone waste water is about is 4% of total POME.

The volume of the combined POME discharged mostly depends to a large extent on the milling operations. Besides that, there are other minor sources of relatively clean waste water that may be included in the combined mill effluent POME which is sent to the wastewater stream. All these include turbine cooling water and stream condensates, overflow from the vacuum dryers and some floor washing. Palm oil mills are traditionally located near rivers from which water is abstracted for their milling operations. A number of palm oil mill conveniently discharged their treated effluents into the river in an untreated or partially treated condition. This is because it is the cheapest method of POME disposal since POME was a non-toxic oily waste.

## 2.3 Chemical Bond

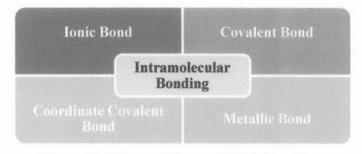


Figure 3 : Types of intermolecular bonding

Atoms are the basic building blocks of all types of matter. Atoms link to other atoms through chemicals bonds resulting from the strong attractive forces that exist between the atoms. The electrons that participate in chemical bonds are the valence electrons, which are the electrons found in an atom's outermost shell. When two atoms approach each other these outer electrons interact. Electrons repel each other, yet they are attracted to the protons within atoms. The interplay of forces results in some atoms forming bonds with each other and sticking together.

#### 2.4 Properties of Ceiba pentandra (L.)

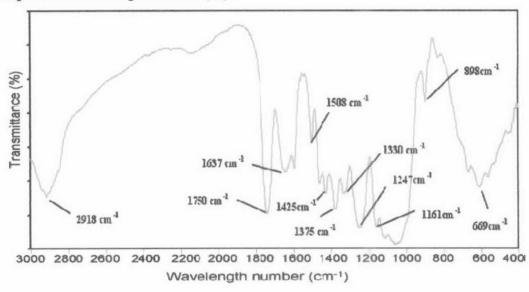


Figure 4 : FTIR Spectrum of raw kapok.

#### (Anisa Ur Rahman, 2009)

The Figure 4 above shows the FTIR spectra of raw kapok. This indicates all the components of Malaysian kapok as a lignocellulosic material, with hydrophobic waxy coating, similar to that reported previously [16, 17, and 18]. This can be associated with the presence of plant wax, which generally consists of n-alkanes, smaller portion of alcohols, fatty acids, aldehydes, ketones and n-alkyl esters [2, 18]. The bands around 1736 and 1242cm<sup>-1</sup> are due to the presence of carbonyl group (C O) in the ester bonds [18]. The C-O stretching vibrations are believed to be associated with the aliphatic aldehvdes, esters and ketones of kapok wax. The band around 1510cm<sup>-1</sup> corresponds to C-O stretching in lignin, while the band at 1050cm<sup>-1</sup> is within the region of carbohydrate or polysaccharides. An infrared spectrum pattern of cotton shows similar spectrum to our study with the band at 2891cm<sup>-1</sup> being suggested due to the CH stretching of CH2 and CH3, the bands at 1745 and 1234cm<sup>-1</sup>, respectively, being attributed to the presence of carbonyl C O stretching of ester and C-O stretching of acetyl group associated with the presence of plant wax, and the band at 1017cm<sup>-1</sup> for C-Ostretching being associated with the presence of cellulose, hemicellulose and lignin [21]. The presence of wax surface can be shown at transmittance through 1425 cm<sup>-1</sup> and 1375 cm<sup>-1</sup>. Besides that, the carbonyl group also observed from the spectrum 1750 cm<sup>-1</sup> and 1250 cm<sup>-1</sup>.

The chemical composition of kapok harvested from the Phillipines and Southern Vietnam, analyzed on the basis of dry fiber, suggest not only high amount of acetyl group content (13%), but also the presence of cellulose (35%), xylan (22%) and lignin (21.5%) [17]. Analysis on the basis of dry fiber for kapok from Phillipines and Southern Vietnam contain high amount of acetyl group (13%), cellulose (35%), xylan (22%) and lignin (21.5%) [18].

#### 2.5 Lignin and Cellulose

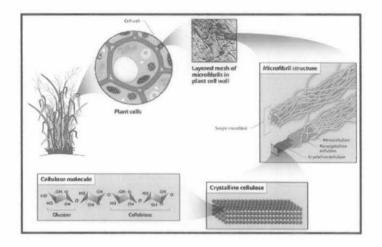


Figure 5 : Cellulose structure.

Microfibril is known as linear structures of high tensile strength and it is formed in the plant cell wall. The cell wall is made up from layers upon layers of micro fibrils. Basically each microfibril is about 10 to 20 nm in diameter and may consist of up to 40 cellulose chains. A micro fibril's crystalline and paracrystalline (amorphous) cellulose core is surrounded by hemicelluloses, a branched polymer composed of a mix of primarily pentose sugars (xylose, arabinose), and some hexoses (mannose, galactose, glucose). In addition to cross-linking individual micro fibrils, hemicellulose also forms a covalent association with lignin, a rigid aromatic polymer. Pretreatment of biomass with enzymes or acids is necessary to remove the surrounding matrix of hemicelluloses and lignin from the cellulose core prior to hydrolysis. The highly ordered, water-insoluble nature of crystalline cellulose makes access and hydrolysis of the cellulose chains difficult for the aqueous solutions of enzymes.

#### 2.6 Selection of Sorbents Materials

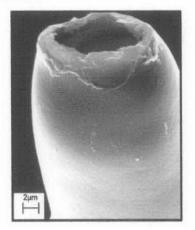


Figure 6 : The SEM image of single kapok fiber. (Anisa Ur Rahman, 2009)

**Figure 6** above shows the SEM image of single kapok fiber. In chemical nature of sorbents, there are presence of surface wax and molecular arrangement in order to determine the oil sorption characteristics of sorbent. The presence physical configuration such as hollow lumen, surface roughness, porosity and fineness is very important since all these characteristics are correlated to the oleophilic-hydrophobic characteristics of sorbents. Besides that, surface tension, density, contact angle and viscosity are parameters that characterized the sorbent-oil interaction. Hence, for better oil sorption performance, it is necessary to do sorbent pretreatment POME treatment as well.

The pretreatment for kapok conducted will include cleaning from visible impurities such as dust and lump. Application of kapok for oil-water mixture separation is successful with only invisible oil slick remains on the water surface. Without any treatment, kapok fibers prove excellent buoyancy and water repellency. Besides that, it shows higher sorption and retention capacity than other materials such as polypropylene, and others inorganic minerals sorbents.

## 2.7 Lignin Structure

The word lignin derived from Latin word lignum meaning wood. It is a main component of vascular plants. Indeed, lignin is second only to polysaccharides in natural abundance, contributing 24-33% and 19-28%, respectively, to dry wood weights of normal softwoods and temperate-zone hardwoods. In addition, lignin may be defined as an amorphous, polyphenolic material arising from enzymatic dehydrogenative polymerization of three phenylpropanoid monomers, namely (1) coniferyl alcohol, (2) sinapyl alcohol, and (3) p-coumaryl alcohol.

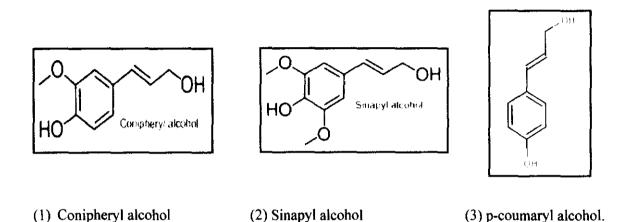


Figure 7 : Types of lignin.

Lignin is a complex structure that is important for mechanical support, water transport and defense in vascular. Lignin polymer will give the impact to compressive strength and hydrophobicity of xylem cell walls. This is important for the growth and development of the whole plant. The insolubility of lignin's structure makes it resists to microorganism. It can be concluded here that, lignin's structure play roles as plant defense. This essential role can be affected by the variation of lignin content, composition and location of lignin for normal xylem function.

Normal softwood lignins are usually referred to as guaiacyl lignins because the structural elements are derived principally from coniferyl alcohol (<90%), with the remainder consisting mainly of p-coumaryl alcohol type units. For normal hardwood lignins, the methoxyl content per phenylpropanoid unit is typically in the range of 1.2-1.5 [13]. Grass lignins are also classified as

guaiacyl-syringyl lignins. However, the grass lignins additionally contain small but significant amounts of structural elements derived from p-coumaryl alcohol. Moreover, grass lignins also contain p-coumaric, hydroxycinnamic, and ferulic acid residues attached to the lignin through ester and ether linkages [14].

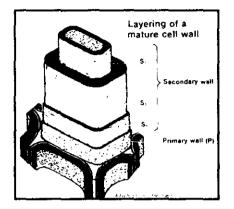


Figure 8 : Layer of a mature cell wall.

The distribution of lignin in individual cells of lignified wood has been well examined. The lignin concentration is rather uniform across the secondary wall, but there is a significant increase in lignin concentration at the boundary of the middle lamella and primary wall region [15]. This pattern of lignin distribution, with the highest concentration in the inter fiber region and lower, uniform concentration in the bulk of the cell walls, is typical for most wood cells. Thus lignin serves the dual purpose of binding and stiffening wood fibers through its distribution between and in the cell walls.

The distribution of lignin in individual cells of lignified wood has been well examined. The lignin concentration is rather uniform across the secondary wall, but there is a significant increase in lignin concentration at the boundary of the middle lamella and primary wall region [15]. This pattern of lignin distribution, with the highest concentration in the inter fiber region and lower, uniform concentration in the bulk of the cell walls, is typical for most wood cells. Thus lignin serves the dual purpose of binding and stiffening wood fibers through its distribution between and in the cell walls.

There are multiple function have been performed by lignin that are essential to the life of the plant. Lignin plays an important role in the internal transport of water, nutrients and metabolites. This will provide resistance to biological degradation and resistance to compression, impact and bending.

#### 2.8 Detection of Lignin

The characteristic color-forming response of lignified tissue and some lignin preparations on treatment with certain organic and inorganic reagents was recognized. There are more than 150 color reactions have now been proposed for the detection of lignin [7]. Reagents used in these reactions may be classified into aliphatic, phenolic and heterocyclic compounds, aromatic amines, and inorganic chemicals. Among the important reactions are the Wiesner and Maule color reactions.

## 2.9 Determination of Lignin Content

Lignin content in plants (wood) is determined by direct or indirect methods [8]. The direct method includes measurement of acid insoluble (ie, Klason) lignin after digesting wood with 72% sulfuric acid to solubilize carbohydrates [8]. The klason lignin contents of representative's lignified materials are shown in *Table 1*. In contrast to the direct determination of lignin content, indirect methods do not involve the isolation of a lignin residue. These include spectrophotometric methods and procedures that are based on oxidant consumption. An ultraviolet microspectrophotometric method has been used to determine the distribution of lignin in the various cell regions of softwoods [10]. Supplementing the uv-microscopic technique is a method in which lignin is brominated and the bromine uptake, which is proportional to the lignin content, is determined by a combination of scanning or transmission electron microscopy (sem or tem) and energy dispersive x-ray analysis (edxa) [12]. Among the solvents used to dissolve lignocellulosic material are sulfuric acid, phosphoric acid, nitric acid, cadoxene, and acetyl bromide in acetic acid. The acetyl bromide method appears to have gained the most widespread acceptance [12].

Material	Klason lignin (%)	
Softwoods	26-28.8	
Hardwoods	22	
Non fibers		
<ul> <li>Bagasse</li> <li>Bamboo</li> <li>Wheat straw</li> <li>Kenaf</li> <li>Sorghum</li> </ul>	19.6 22.2 17.0 10.9 7.9	
<ul> <li>Pulp</li> <li>Pine knaft</li> <li>Birch Kraft</li> <li>Spruce Kraft</li> <li>Birch acid sulfite</li> <li>Birch bisulfite</li> </ul>	4.8 5.0 2.8 3.2 4.0	

**Table 1 :** Klason lignin content of lignified materials

(Anisa Ur Rahman, 2009).

## 2.10 Lignin Composition and Distribution

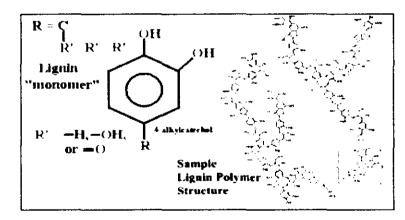


Figure 9 : Sample of lignin polymer structure.

Lignin heterogeneity could be directly related to enzymes diversity and specificity. However, lignin plant could be resulted from spatial and temporal distribution of isoforms for given steps in the biosynthetic pathway. Normally, the most distinct variation of lignin composition can be described between gymnosperms (softwoods) and angiosperms (hardwoods). Hypotheses to

account for differences in hardwood and softwood lignin are based on differences in the enzymes of the mono-lignol biosynthetic pathway (*Shimada et al., 1973*). Two major biosynthetic factors that account for difference in lignin compound are compositions between gymnosperms and angiosperms. In additional, mutants' also affecting lignin content and composition that have been characterized in maize, sorghums and *Arabidopsis thaliana*.

## 2.11 Lignin Heterogeneity

At some steps in lignin biosynthetic pathway, multiple genes and enzymes have been identified and that could carry out similar reaction during development or defense response (reviewed by Sederoff et al., 1994). Lignin heterogeneity could be accounted for at least in part, by different isoforms of monolignol biosynthetic enzymes that prefer different substrates. Heterogeneity in lignin can be generated based on timing and location of expression of given family.

In conifers, the composition of lignin changes during the formation of the xylem cell wall. It was showed that Terashima and Fukushima (1988, 1989) had examined the types of monomers that are deposited during the formation of xylem cell walls. The first lignin deposit during differentiation of xylem is contained in p-hydroxyphenyl lignin in the middle lamella and the cell corners. Moreover, there are guaiacyl lignin is deposited in the middle lamella and the secondary wall.

Compression wood is formed in gymnosperms in response to mechanical stress and it is characterized by higher wood density, increased lignin content and an increased proportion of p-hydroxyphenyl lignin (Kutsuki and Higuchi, 1981). Lignin content normally increases from 26% to 34% while methoxyl content is reduced from 15% to 12.6%. These are showing that the proportion of p-hydroxyphenyl lignin is increased (Kutsuki and Higuchi, 1981).

# **CHAPTER 3**

## **METHODOLOGY**

## 3.1 Work Flow

Refer Appendix I for Gann chart.

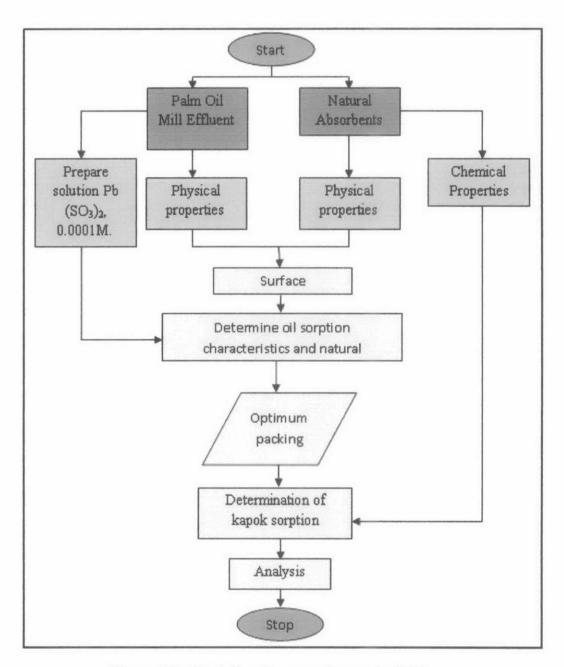


Figure 10 : Work flow for second semester FYP.

#### 3.2 Engineering Parameters

The possible interaction between sorbent and sorbate reversible Van der waals interactions or irreversible stronger interaction, like direct electron transfer. The difficulties of sorbent regeneration can be explained by those interactions. There are several characteristics that need to be considered in order to select the best sorbent for removing the contaminant;

- Highly porous materials with high surface area and compatible interaction with sorbent capacity. This because sorbent capacity will explain how much contaminant is sorbed per unit quantity of sorbent.
- 2) Sorbent selectivity which follows several mechanism:
  - i. Selective binding to the surface.
  - ii. Excluding certain component based on its geometry.
  - Differences in diffusion rates from the sorbate to the sorbent surface which facilitate by the intra particle diffusion.
- 3) Method chosen for sorbent regeneration in order to recover the sorbate inside the sorbent structure. It is because the regeneration and desorption will reduce the sorbent replacement cost, extent life duration, and will minimize the amount of waste disposal.
- Reduction of lifetime and sorption capacity will occur sorbent deactivation happens. This
  phenomenon occurs when the sorbents establish strong interaction with other component
  beside sorbate.
- Besides that, the main consideration in choosing the sorbent is operational cost. By using the less expensive, the cost parameter can be reduced.

### 3.3 Experiment Setup

Experiment Setup for Evaluation of Kapok Column Process Design Parameters for Oily Water Filtration.

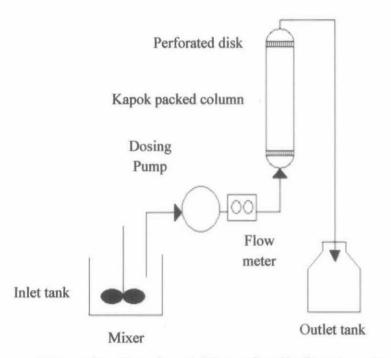


Figure 11 : Experimental Setup for oily filtration of kapok.

(Anisa Ur Rahman, 2009)

- a) The overall experiment setup is shown in Figure 11 above. Mixture of palm oil-water is blended about 15 minutes at high speed by using blender (Philips, UK) to produce 5% (w/w) concentration and total weight of 500 g is used in each set of experiment. This step is to produce a homogenous mixture.
- b) Before column loading, the palm oil-water is agitated with stirrer at 500 rpm (Heidolph, Germany). The step is to make sure a well-mixed palm oil-water mixture is produced.
- c) The liquid feed of palm oil-water mixtures were drawn from inlet tank out into the column by using dosing pump (Grundfos, Denmark).
- d) When the first drop of liquid feed enters the column, the experiment is considered starts.

e) Filtrate samples are collected every two minutes, starting from the water breakthrough. Two parameters such as packing density and flow rate will be take note in order to study the performance of kapok for oily water filtration.

### 3.4 Atomic- Absorption Spectroscopy

The equipment named Atomic-absorption (AA) spectroscopy is used to measure the concentration of gas-phase atoms. Only liquids and solids samples will be used. Since the liquids or solids sample will be used, the analyte atoms or ions must be vaporized in a flame or graphite furnace. The atoms absorb ultraviolet or visible light and make transitions to higher electronic energy levels. The analyte concentration is determined from the amount of absorption. It is difficult to apply Beer-Lambert law in atomic absorption spectroscopy because of the non uniformity of the concentration, path length of analyte atoms and variations in the atomization efficiency. Concentration measurements are usually determined from a working curve after calibrating the instrument with standards of known concentration. The sample palm mill effluent will be tested by using AAS in order to determine the existence of any metal ions in that waste.

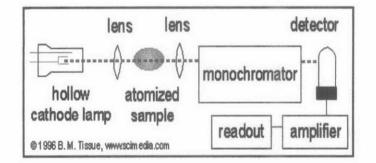


Figure 12 : Schematic of an atomic-absorption experiment.

The atomic absorption spectroscopy (AAS) involves two main steps which are atomization of the sample and the absorption of radiation from a light source by the free atoms. During the absorption of ultraviolet or visible light, the free atoms undergo electronic transitions from the ground state to excited electronic states. Atomic absorption spectroscopy is a technique for determining the concentration of a particular metal element in sample. The concentrations of metals in a sample running in a series of calibration standards can be found through the instrument. The instrument will record the absorption generated by a given concentration. Graph

absorption versus concentrations of standard can be plotted. The composition of metals in palm oil mill effluent has been investigated. Samples of effluent and standard solution were prepared for absorbance testing. Refer **Appendix V**.

### 3.5 Chemical Oxygen Demand (COD) Analysis

Chemical Oxygen Demand is defined as a test used to indirectly measure the amount of organic compounds in water. The main objective of COD test is to determine the amount of organic pollutants found in surface water. Besides that, COD is a useful method to determine of water quality. The unit of COD is mg/l which is indicates the mass of oxygen consumed per liter of solution. By using a strong chemical oxidant in an acid solution and heat, the organic carbon can be oxidized into  $CO_2$  and  $H_2O$ . Then, the amount of oil content inside the sample effluent was quantified by means of COD. In order to measure the value of COD, combination of some reagent as below is used:

a) *Potassium dichromate* in a 50% sulfuric acid solution as the oxidizing agent.

#### b) *Silver compound* as a catalyst.

This silver compound is to enhance the oxidizing of special class of organic compound such as straight chain-aliphatic.

## c) Mercury Compound as addition.

Mercuric compound is used to avoid the interference from oxidation of chloride ions. This is because the present of chloride ions will reduce the performance of the silver compound as a catalyst. If not, silver chloride will be formed and it is insoluble compound. So, mercuric sulfate will be added in order to avoid this phenomenon happen because chloride ions will be masked by this reagent.



Figure 13 : Spectrophotometer HACH DR 500 for COD Absorbance measurement.

## **Procedures:**

- a) Mixture of 1ml samples (POME) with 1ml of water are added into COD vials by using cylinder.
- b) After that, the mixture is heated in the Thermoreactor DRB 200 at 150°C.
- c) Then, the vials will be cooled at room temperature.
- d) Measure the COD value by using colorimetric method in a spectrophotometer at 420 nm (Spectrophotometer DR 5000, HACH, USA).

## 3.6 Oil Sorption Capacity

The procedure for determining oil sorption capacity generally followed the method F726-99 (ASTM, 1998c) [28]. Several packing densities were tested from 0.02, 0.04, 0.06, 0.08 g/ml [1]. After that, the test cell were soaked inside a 500 ml beaker filled with 400 ml of experimented oil, 30 minutes in diesel oil, 60 minutes in fresh engine oil, and 90 minutes in used engine oil. The speed of oil penetration will be different for each densities packing. Then the oil saturated test cell was then lifted and the oil was left dripping out from the test cell above the oil bath [1].

Oil sorption capacity : 
$$\frac{S_I - S_F}{S_A}$$

Where:

 $S_I$  = The weight (g) of oil before sorption inside the oil bath.

 $S_F$  = The weight (g) of oil inside the beaker inside the beaker at 1 min dripping time.

 $S_A$  = The dry weight (g) of kapok.

Then, the percentage of oil dynamic retention capacity was calculated [1].

Percentage of oil dynamic retention capacity = 
$$\frac{W_t}{W_{t=1}} \times 100\%$$

Where:

 $W_t$ The weight (g) of oil bath at 1 min dripping. $W_{t=1}$ The weight (g) of oil bath at t min of dripping, i= 2, 3, 4.....

For this experiment, the saturation point was detected from the appearance of the first oil droplet from the column tip. The saturation time is determined from the time the oil was poured into the column until saturation point. Then, the bed height difference was measured from the initial until the final height which no remaining oil were observed dripping out of the column [1]. The difference of bed height could show the effect of relation oil sorption within the kapok inside the column.

#### 3.7 Reusability of Kapok

About 100ml of diesel oil were poured into a column until saturation point. The saturation point was noticed from the first appearance of oil droplet from the column tip. A pressure different was applied by using vacuum pump (BUCHI Vacuum Controller V-800), free dripping of oil was allowed for 15 minutes. Besides that, the height of bed was observed for every cycle of sorption-desorption process [2].

#### 3.8 The Effect of Solvent Treatment

About 5 g of raw kapok was mixed with 200 ml of chloroform or 125 ml of NaOH at pH 13. Both chloroform and alkali treatments under reflux, the duration forsoxhlet extraction were taken about 4h and 8h. In order to remove waxy from the debris from the fiber, it was washed after the alkalization process. To separate extracted liquid and kapok, the filtration method was used by applying the vacuum suction about 10 minutes. After that, kapok was dried inside the drying oven at 60°C for 24h. Then the dried kapok was grounded by using blender to form the fiber fiber.

[1]. The final form of kapok was used to determine the oil retention capacity and sorption capacity.

## 3.9 Residual Oil

In any types of oil, fatty acids are the main constituents. A fatty acid is characterized as a longchain aliphatic hydrocarbon with a carboxylate group on one end. The naturally occurring fatty acids have common names such as oleic, linoleic, eleosteric, etc. There are two types of fatty acids, saturated and unsaturated. There are also branched-chain fatty acids with the most frequent branch being a methyl group, (-CH3) and also some which are attached to other functional groups (Genium Publishing Corporation, 2004).

Since fatty acid can be defined as a weak acid, it is actually very soluble in organic solvents and polar organic solvents like ether or alcohols. However, it is no soluble in non polar organic solvents such as hexane. The structural component of fatty acids mostly built in from triglycerides. Phospholipids is one of the family members where three fatty acids are esterifies to a phosphate, P (= O) (O-fatty acid) 3. Collectively the family is known as the lipids, which are a component of fat (Genium Publishing Corporation, 2004).

## **CHAPTER 4**

# **RESULT AND DISCUSSION**

## 4.1 Properties of Palm Oil Mill Effluent (POME)

In this project, the sample of palm oil mill effluent (POME) was taken from Felcra Nasaruddin, Perak and the typical characteristics of raw POME were shown in **Table 2** below. The final product for pretreatment process as shown below would be analyzed for parameters such as pH of POME, Chemical Oxygen Demand (COD), Total Suspended Solids (TTS), Total Dissolved Solids (TDS), and its turbidity.

**Parameters** Mean (mg/l) Range (mg/l) 3.4-5.2 pH 4.2 Oil and grease 6,000 150-18,000 BOD; 3 days, 30°C 25,000 10,000-44,000 COD 50,000 16,000-100,000 40,500 11,500-54,000 Total Suspended Solid Total Dissolved Solids 18,000 5,000-54,000

Table 2 : The characteristics of the raw POME obtained from local palm oil mill factory.

(A.L Ahmad\*, K.Sithamparam, M.M.D Zulkali and S. Ismail, 2003).

Table 3 : The result for	Chemical	Oxygen De	mand Experiment 1

Samples	Content	Result (mg/l) 0	
1	Potassium dichromate + 2ml distilled water		
2	Potassium dichromate + pure POME	Absorbance > 3.5	
3	Potassium dichromate + filtered pure POME Over measuring r		
4	Potassium dichromate + pure POME + kapok	Absorbance > 3.5	

Samples	COD (mg/l)	Absorbance	% Trans
0	0	0	100
1	3182	1.403	4.0
2	1218	0.537	29.1
3	2556	1.127	7.5
4	994	0.438	36.5
5	386	0.170	67.6

Table 4 : The result for Chemical Oxygen Demand Experiment 2

When POME fresh, it is a thick brownish in color colloidal slurry of water, oil and cellulosic fruit residues. Normally POME is charged at a temperature of between 80°C and 90°C after the related process. This charged is quite acidic because the pH is between 3 and 4. *Table 2* above shows the POME has a very Biochemical Oxygen Demand (BOD) and Chemical Oxygen Demand (COD), which is 100 times more than the domestic sewage. The waste for each sample had been diluted within the range 10-10000 times factor (**Appendix V**). This is because without the dilution process, the reading for Spectrophotometer HACH DR 5000 mostly over the measuring range. Based on the *Table 4*, the lowest COD (mg/l) is 386 mg/l where the samples had been diluted about 10000 times from the pure POME. It has shown that, even the Sample 1 had been diluted about 10 times, the COD measurement still give the high reading which is 3182 mg/l. For information, POME is a non-toxic waste because no chemical is added during the oil extraction process.

#### 4.2 Chemical and Physical Properties of Ceiba pentandra (L.) as natural sorbent.

Based on SEM and OM analysis, significant amount of oil penetrates the hollow lumen inside kapok fiber while high surface tension of kapok fiber surface against air at 7.2x 10<sup>-4</sup> N/cm at 21°C inhibit water from penetrating the hollow tubes [1]. Besides that, the presence of large and non-collapsing hollow lumens also determined the oil sorption capacity of kapok. In comparison with kapok, milkweed and cotton, higher amount of wax content are reported in kapok and

milkweed as compared to cotton [1]. This higher amount of wax content will promote the interactions between oil and sorbent through hydrophobic and Van der Waals forces.

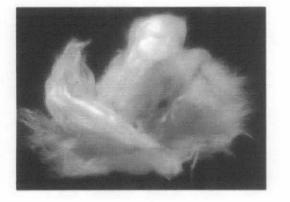


Figure 14 : Image of raw Ceiba pentandra (L.)

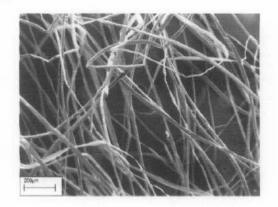


Figure 15 : SEM Image for untreated Ceiba pentandra (L.)

(Abdullah et al, (2010), (Anisa Ur Rahmah, Z. Man, 2009)

#### 4.3 Effect of Absorbent Loading

For oil sorption process, the effect of absorbent loading is a very important criterion. It was found that when the amount of absorbent increase, the amount of oil absorbed by the absorbent also gradually increase. Adsorption itself is known as surface related phenomenon and the surface selectivity can be classified as hydrophobic and hydrophilic characteristic (Keller et al., 1987). Kapok's surface has a hydrophobic surface that can strongly adsorb oil and grease. In addition, the internal porosity of the kapok also induces adsorption process (Frederick, 1990) as well as an adsorption-surface area. This is very important because these forces will cause large and small molecules of dissolved organic molecules to be deposited and adsorbed from the solution onto the molecules-size pored. In equilibrium, mostly the oil and grease molecules in the solution were adsorbed on the surface and in the intermolecular pores of the kapok.

#### 4.4 Effect of Mixing Time

By evaluating the effect of mixing time, the relation between contact times on residual oil with the natural sorbent must be focused. When contact time is increased, it means that the residual oil molecules have a longer residence time for adsorption purpose on the kapok's surface. However, at the equilibrium the surface of kapok was covered with oil molecules and therefore no longer mixing time does not increase the sorption oil form palm oil mill effluent [1].

#### 4.5 The Effect of Solvent Treatment.

As the **Figure 15** above shows the SEM image for untreated Ceiba pentandra (L.), there is no such different with the SEM image of chloroform treated on **Figure 16**. The buoyancy and fluffiness was retained even the silky appearance of chloroform treated was reduced and some parts of the fibers were flattened. This condition indicates the increment of wax removal during the equipment. Other study has similarly reported this reduction of silky cluster of kapok after only 1 h chloroform treatment [16].

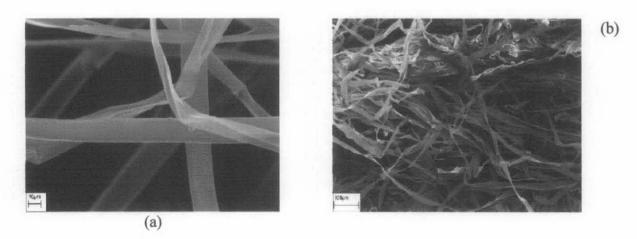


Figure 16 : Image of (a) *Ceiba pentandra (L.)* after chloroform treated; (b) *Ceiba pentandra (L.)* after alkali treated.

(Abdullah et al, 2010) and (Anisa Ur Rahmah, Z. Man, 2009)

From the Figure 16, it was observed there was a total disappearance of air entrapment inside kapok fiber and the structure became completely flattened, similar to a flat ribbon-like structure with increased density [1]. The silky and smooth structure had also changed into hardened structure and the color changed to dark from originally fluffy white. This could be due to excessive removal of hemicelluloses and lignin [6].

The contamination of plant wax will determine the plant wet ability, optical appearance, water repellency and the solubility among organics and polar solvents [1]. Since wax content of kapok (about 3%) which is also higher than cotton (0.4–0.8%), and high acetyl content (13%) may be responsible for its high hydrophobicity [24]. With reduction of meager amount of surface wax, the slightly increased retention capacity in chloroform treatment confirm our earlier suggestion that wax is not the only factor governing the draining out of extra-lumen liquids. However, as the presence of surface wax caused the tubular structure to provide sufficient capillary pressure that can hold up the absorbed oil, significant removal as in alkali-treated kapok, could have reduced capillary pressure inside the tubular structure and hence the retention capacity. Chemical treatment to modify the surface properties of natural fibers could improve the desired properties such as to produce fiber with better mechanical properties for the polymer reinforcement material.

#### 4.6 Oil sorption and retention capacity.

There is no difference of oil retention in oil sorption capacity between chloroform treated kapok and untreated kapok [1]. The same condition was also applied for kapok with alkali treatment. Comparison between both conditions, there is no such huge difference except for 8h alkali treatment; the oil retention capacity was reduced from 17.7 g oil/g fiber (4h) to 15.4 g oil/g fiber (8h) [1]. The are many factors that effect the oil sorption and retention capacity of sorbent such as molecular arrangement, the surface roughness, porosity, fineness and the amount of wax. However, the waxy content is not the main factor that contributes into oil sorption and retention capacity. Another method also has been tested to prove the oil sorption and oil retention capacity.

Weight of kapok + beaker (g)	Weight of beaker (g)	Weight of kapok used (g)	Amount of water used(ml)	Amount of oil used(ml)
512	412	100	2000	250
562	412	150	2000	375
612	412	200	2000	500

Table 5: Amount of oil can be absorbed by Ceiba pentandra (L.)

Table 6 : Percentage of water trapped after oil sorption.

Weight of kapok (g)	Weight of kapok after sorption process (g)	Oily Water trapped in kapok (g)	Percentage water trapped (%)
100	698.9	698.9-412 = 286.9	14.35
150	795.1	795.1-412 = 383.1	19.66
200	886.7	868.7 - 412 = 456.7	22.84

From the experiment, it is shown that 100 grams of kapok totally absorb 286.9 grams of oily water, 150 grams of kapok absorb 383.1 grams of oil and 200 grams of kapok absorbed 456.7 grams of oil. In this experiment, since the crude oil is not available to get from laboratory, used motor oil is considered as crude oil for this process. From the data obtained by using the motor oil, 1 gram of kapok absorbs about 2.5 grams of oil. Crude oil and used motor oil may have different chemical composition that kapok hinders from absorbing. As the amount of kapok increase, the amount of oil adsorbed will also increase proportionally. The amount of water collected during absorption is considerably small compare to the total amount water that has been used. Gradually, this will increase the rate of oil absorption efficiency.

#### 4.6 Atomic Absorption Spectroscopy

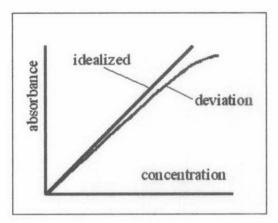


Figure 17: Example of calibration curve

From the Beer's law, the absorbance of an absorbing analyses is directly proportional to its concentration. However, there are many factors that lead into deviation from linearity (**Figure 17**) such as stray light, and unabsorbed radiation.

It is impossible to eliminate totally the curvature but it can be minimized. Besides that, an alternative wavelength that has a lower absorptive can be used in order to make the absorbance works into the optimum working range. Next, the absorbance also can works in optimum range by reducing the path length by rotating the burner hand. On the other hand, the sample can be diluted in order to get the better result interpretation.

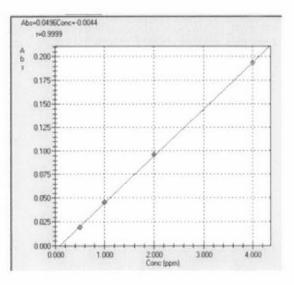


Figure 18: Graph for Plumbum Absorption within POME

After plotting the graph, deviations from linearity usually occur, especially as the concentration of metallic analytes increases due to various factors. These factors includes such as unabsorbed radiation, stray light, or disproportionate decomposition of molecules at high concentrations. **Figure 17** shows an idealized and deviation of response curve. The curvature could be minimized, although it is impossible to be avoided completely. Based on **Figure 18**, the value for r is 0.9999 which is ideal of r value. This problem could be handled by diluting the samples within 10 and 100 factor of dilution. Some of the metals were detected by the equipment, however, sample of diesel produce negative reading shows that there are no metals contaminated within the sample.

Sampling Point	Duplicate No	Conc.(ppm)	Conc. (mg/l)
Pure POME	1	0.0338	67.6
Diesel	1	0.0218	40.52
Diluted POME	1	0.2657	53.14

Table 7 : AAS Result for Iron

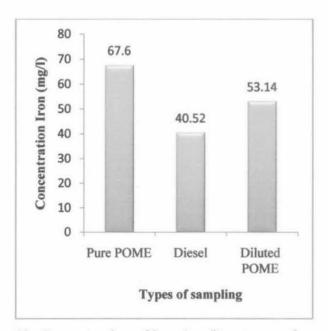


Figure 19: Concentration of iron (mg/l) vs types of sampling.

Sample Point	Conc.(ppm)	Conc. (mg/l)
Diluted		
POME	0.0338	6.76
Diesel	0.0218	4.36
Pure POME	0.2657	53.14

Table 8: AAS Result for Plumbum

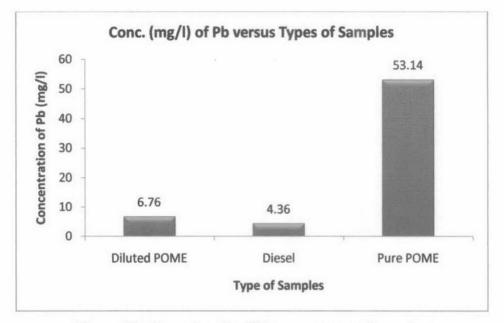


Figure 20: Conc. (mg/l) of Pb versus types of samples

The study has shown that, the palm oil mill effluent also contaminated with heavy metals. The higher content in waste must be reduced so that the impact to environmental directly will reduce However there are others method to bring the absorbance into optimum working range if the concentration of sample is too high to permit accurate analysis in linearity response range which are sample dilution, using an alternative wavelength that having lower absorptive, and reducing the path length by rotating the burner hand. It is desirable to work in the linearity response range. The rule of thumb is that a minimum of five standards and a blank should be prepared in order to have sufficient information to fit the standard curve appropriately. Manufacturers should be consulted if a manual curvature correction function is available for a specific instrument.

#### **CHAPTER 5**

#### **CONCLUSION AND RECOMMENDATION**

#### 5.1 Conclusion

In conclusion, kapok fiber significantly hydrophobic and does not wet with water. The hydrophobic-oleophilic characteristics of kapok fiber surface were believed to be responsible for the excellent selectivity in the experiment. The addition of kapok fiber as an absorbent to a sample POME reduces the residual oil content of the effluent. There are several benefits possible to agricultural industries, energy and environment by using kapok fiber as natural sorbents. The analysis of kapok fiber based on the chemical and physical treatment will provide the maximum condition for sorption process. This study shows that kapok was promising as commercial absorbent for oil recovery from POME. The overall sorption characteristics of kapok imply that Malaysian kapok is a potential lignocelluloses material for oil removal with high oil sorption and retention characteristics and stable for prolonged use [2]. The capability of kapok to absorb the oil had been approved by several researches and experimental work. However, further study must be continued in part of removing metals from waste by using kapok.

#### 5.2 Recommendation

There are so many aspects that should be look deeper inside and to be improved in order for the conducted research to be implemented and the objective of developing kapok as natural sorbent for oil recovery from POME. Here is some of the improvement that could be conducted in the future in order to achieve better result. These studies hope can be a basis and would help to elevate the potential of kapok as the main sorbent of recovery oil from POME.

- 1) Set the equipment based on the requirement so that the outcomes will not be affected by the equipment's error human's error.
- 2) Prepare the fresh palm oil mill effluent for experiment.
- 3) Consider the other types of effluent to make comparison of Ceiba pentandra (L.) capability as natural sorbent to recover oil from waste.
- 4) Do collaboration with Chemical Engineering Department because some of the equipment will need to be tested at Block 4 and Block 5.

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### APPENDICES

## Appendix I

### Table 9 : Gann chart for FYP 2

			Week													
No.	No. Activity		1	2	3	4	5	6	7	8	9	10	11	12	13	14
					•						•					
1	Brainstorming	Brain storming Session														
		Discussion with supervisor														
2	Planning	Experiments														
3	Progress Report 1	Task Completing									,		,			,
4	Implementation	Researching												:		
		Materials Collecting				,										
		Laboratory														
		Equipment Testing											-			
5	Progress Report 2	Final Result														
6	Final Evaluation	Progress Report Final Report	 													
		Oral Presentation														

# Appendix II

Process	Process Anacrobic Contact Anaerobic Lank		Exaporation	Pending
Description	& Acration	Land Application		
Acreage &	Large land area	Large land area	Modular but	Large land
Facilities			complicated	area
Controllability				Difficult
Organic	Small	High	Moderate	Small
Loading				
Sludge	Large	Large	None	Large
Generation				
Energy	High	High	High	Minimal
Requirement				
Requirement of	Yes	Yes	Yes	Yes
further				1
treatment				
Resource	No	No	Yes	No
Recovery				
<b>Operating</b> Cost	Cheap &	Cheap &	Cheap &	Cheap &
& Maintenance	Intensive	Intensive	Moderate	Intensive

## Table 10 : Comparison of current treatment and recovery of POME

Parameter / Element	<b>Concentration</b> *
pH	4.7
Oil and grease	4000
Biochemical Oxygen Demand (BOD)	25 000
Chemical Oxygen Demand (COD)	50 000
Total solids	40 500
Suspended solids	18 000
Total volatile solids	34 000
Ammonia cal nitrogen	35
Total nitrogen	750
Phosphorus	180
Potassium	2270
Magnesium	615
Calcium	439
Boron	7.6
Iron	46.5
Manganese	2.0
Copper	0.89
Zinc	2.3

Table 11 : The common characteristic of palm oil mill effluent

Source: A.L Ahmad\*, K.Sithamparam, M.M.D Zulkali and S. Ismail (2003).

# Appendix IV

Substances	Content (% of ODM)				
	Philippines	Vietnam			
Ash	0.8	0.5			
Lignins		I			
Klason lignin	17.4	15.4			
Acid-soluble lignin	4.1	3.8			
Total	21.5	19.2			
AcBr lignin	20.8	18.8			
Neutral Sugars	· · · · · · · · · · · · · · · · · · ·	L			
Rhamnose	0.4	0.5			
Arabinose	0.3	0.2			
Xylose	21.9	22.8			
Mannose	0.8	0.6			
Galactose	0.4	0.3			
Glucose	35.1	38.5			
Total	58.9	62.9			
Acetyl Group	13.0	12.5			

# Table 12: Chemical Properties of Ceiba pentandra (L.)

# Appendix V Atomic Absorption Spectroscopy

<b>Information</b>	Measurement	
Detection Limit		
• 0.1 ppm	40 ppm (0.1; 0.2; 0.4; 0.8)	
• 0.5 ppm	200 ppm (0.5;1.0;2.0;4.0)	
Volumetric Flask	25 ml	
Test Tube	5 ml	

Table 13: Addition	al Information fo	or samples dilution
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### Example of calculation

a) Plumbum Detection (0.5 ppm)

Concentration (ppm)	Calculation
0.5	$C_1V_1=C_2V_2$
	$(200) V_1 = (0.5) (5.0)$
	$V_1 = 12.5 \ \mu l$
1.0	$C_1V_1=C_2V_2$
	$(200) V_1 = (1.0) (5.0)$
	$V_1 = 25.0 \ \mu l$
2.0	$C_1V_1 = C_2V_2$
	$(200) V_1 = (2.0) (5.0)$
	$V_1 = 50.0 \ \mu l$
4.0	$C_1V_1 = C_2V_2$
	$(200) V_1 = (4.0) (5.0)$
	$V_1 = 100.0 \ \mu l$

 Table 14 : Standard Solution for Plumbum

# b) Magnesium Detection (0.1 ppm)

Concentration (ppm)	Calculation
0.1	$C_1V_1 = C_2V_2$
	$(40) V_1 = (0.1) (5.0)$
	$V_1 = 12.5 \ \mu l$
0.2	$C_1 V_1 = C_2 V_2$
	$(40) V_1 = (0.2) (5.0)$
	$V_1 = 25.0 \ \mu l$
0.4	$C_1V_1 = C_2V_2$
	$(40) V_1 = (0.4) (5.0)$
	$V_1 = 50.0 \ \mu l$
0.8	$C_1V_1 = C_2V_2$
	$(40) V_1 = (0.8) (5.0)$
	$V_1 = 100.0 \ \mu l$

Table 15 : Standard Solution for Magnesium

# c) Potassium Detection (0.5 ppm)

Concentration (ppm)	Calculation
0.5	$C_1 V_1 = C_2 V_2$
	$(200) V_1 = (0.5) (5.0)$
	$V_1 = 12.5 \ \mu l$
1.0	$C_1V_1 = C_2V_2$
	$(200) V_1 = (1.0) (5.0)$
	$V_1 = 25.0 \ \mu l$
2.0	$C_1V_1 = C_2V_2$
	$(200) V_1 = (2.0) (5.0)$
	$V_1 = 50.0 \ \mu l$
4.0	$C_1 V_1 = C_2 V_2$
	$(200) V_1 = (4.0) (5.0)$
	$V_1 = 100.0 \ \mu l$

Table 16 : Standard Solution for Zinc

## Appendix VI

Details for Chemical Oxygen Demand (COD) Testing for Palm Oil Mill Effluent

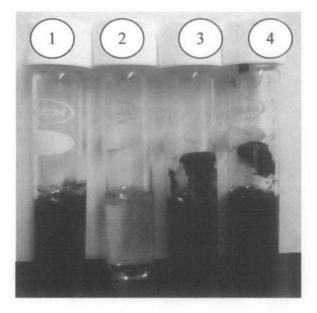


Figure 18 : Mixture of samples with COD Reagent

Table 17	Sample	variation	for	COD	Testing	1
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Samples	Content		
1	Potassium dichromate + pure palm oil mill effluent		
2	Potassium dichromate + 2ml distilled water		
3	Potassium dichromate + filtered pure palm oil mill effluent		
4	4 Potassium dichromate + pure palm oil mill effluent + kapok		

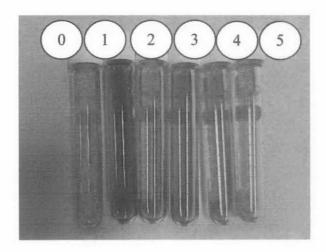


Figure 19 : Mixture of samples and COD Reagent

Samples	Content	Explanation
0	Potassium dichromate + 2ml distilled water	-
1	Potassium dichromate + filtered pure palm oil mill effluent + kapok	POME is diluted by factor 10
2	Potassium dichromate + filtered pure palm oil mill effluent + kapok	POME is diluted by factor 100
3	Potassium dichromate + pure palm oil mill effluent	POME is diluted by factor 10
4	Potassium dichromate + pure palm oil mill effluent	POME is diluted by factor 100
5	Potassium dichromate + filtered pure palm oil mill effluent + kapok	POME is diluted by factor 10000

 Table 18 : Sample variation for COD Testing 2

#### **Appendix VII**

#### a) Experimental Flow



Figure 20 : Location where Palm Oil Mill Effluent was collected at Felda Nasaruddin, Perak

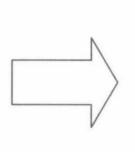
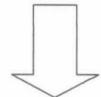




Figure 22 : Collected waste



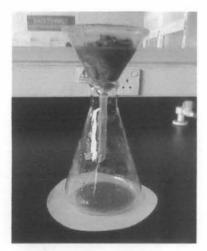


Figure 21 : Filtrations process by using filtered paper and kapok

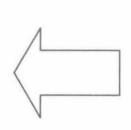




Figure 23 : Palm Oil Mill Effluent

### b) Experimental Flow For AAS

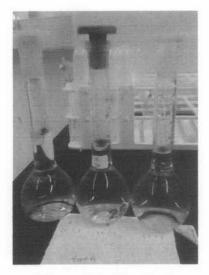


Figure 24 : Standard solution was prepared for AAS purpose

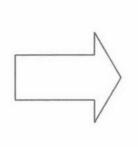
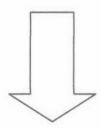




Figure 25 : One of samples POME for AAS



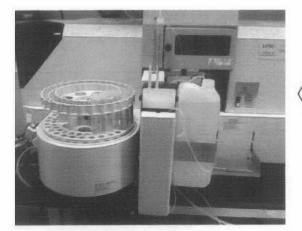


Figure 27 : AAS Equipment

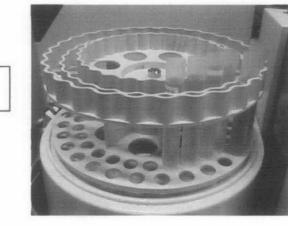


Figure 26 : Standard and samples were combined

c) Experimental Flow For COD Testing



Figure 29 : One of sampels POME prepared



Figure 28 : Heat the sampels with 150'C about 2 hr

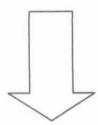
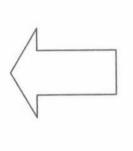




Figure 31 : COD Testing Equipment.



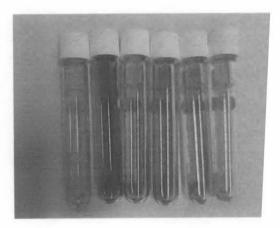


Figure 30 : Cooled the sampels about