### **CERTIFICATION OF APPROVAL**

## PROPERTIES AND UTILIZATION OF JATROPHA CURCAS OIL FOR FXORMATION DAMAGE CONTROL

by

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A project dissertation submitted to the Chemical Engineering Department Universtiti Teknologi PETRONAS in partially fulfillment of the requirement for the Bachelor of Engineering (Hons) Chemical Engineering

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## **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 as references and acknowledgements, and that the original work contained herein have not been undertaken nor done unspecified sources or persons.

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### ABSTRACT

This report basically discusses the preliminary research done and basic understanding of the chosen topic, which is Properties and Utilization of Jatropha Curcas Oil for formation Damage Control. Jatropha seed is the seed from the jatropha trees. The seeds from this tree will be used as a research product. Jatropha trees can be easily plant at the khalatulistiwa area. The trees are very easy to plant and its have very strong physical properties. The oil from jatropha seeds can be produced by two ways: mechanical pressing and chemical extraction. In general there two types of products from jatropha oil: as cosmetic and biodiesel. For biodiesel, there are a lot of researches on producing the biodiesel. Then, this research basically is about to expand the used of the jatropha oil as a part of drilling fluid in the oil and gas industry. The oil will be used as an oil base mud for drilling. Nowadays the type of oil that used as an oil base mud in drilling is diesel and palm oil and of course these two types of oil are very high demand in the market. Formation damage is an undesirable operational that frequently happened during drilling activities. The damage of the formation will reduce the permeability and the porosity of the core to transfer the crude oil from wellbore to the well. The damage may cause by several factors including chemical, physical, biological and thermal interactions of formation and fluids, and deformation of formation under stress and fluid shear. For the physical mechanism of formation damage clay swelling, fines migration, solid invasion and geochemical transformation will be the major factor of formation damage. For the chemical part the type of mud will be a major cause of this phenomena. Then, it is important for the mud engineer to determine the right composition of the mud that used as a drilling fluid. The objective of the project is to determine the properties of the jatropha curcas fruit in order to apply it to substitute the surfactant that used nowadays in the reservoir by determine the rheology of the mud using the drilling lab equipment such as mud balance to determine the density of the mud and the viscometer to determine the other important parameter like Yield Point and Plastic Viscosity. The standard for all this parameter will refer to the API standard. The research will also investigate the reduction of formation damage by this mud using FDS-800-10000 equipment at UTP drilling lab.

The parameter that involve in this activities will be the change of the permeability and porosity of the core. The condition of temperature and the pressure will be set equally as in the reservoir before the experiment being applied. The hypothesis for this research is the oil can be used as part of the oil base mud in during drilling activities.

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# CHAPTER 1 INTRODUCTION

## **1.1 BACKGROUND OF STUDY**

Formation damage is an undesirable operational and economic problem that can occur during the various phases of oil and gas recovery from subsurface reservoirs including production, drilling, hydraulic fracturing and workover operations (Civan 2005). As expressed by Amaefule et al.(1988), "formation damage is an expensive headache to the oil and gas industry". Formation damage assessment, control and remediation are among the most important issues to be resolved for efficient exploitation of hydrocarbon reservoirs. Such damage is caused by various adverse process including chemical, physical, biological and thermal interactions of formation and fluids, and deformation of formation under stress and fluid shear. Formation damage indicators include permeability impairment, skin damage and decrease of well performance.

Near wellbore mud filtrate and fines invasion during drilling operations and the resulting formation damage and filter cake formation are amongst the most important problems involving the petroleum exploitation. Drilling of wells into subsurface reservoirs is usually accompanied with the mud circulation in order to remove the frictional heat generated as the drill bit penetrates the rock, to provide lubrication for reduction of the frictional effects, and to transport the cutting of the rock produced during drilling. However, mud fines and filtrates can damage the near welbore formation. Typical drilling muds maybe water based, oil based or water oil emulsion types. Usually, certain types of fine solid particles are added as weighting agents. Drilling muds are usually non-Newtonian fluids.

### **1.2 PROBLEM STATEMENT**

There are a lot of types of liquid that used in the drilling mud in order to make sure that drilling is not overheated and fluent. A part of this liquid is from diesel, palm oil and sarapar oil. Unfortunately these 3 categories of oils are very expensive to use when drilling especially the palm oil: because of the demand of the palm oil also as a food for human. Between both types of oil, palm oil and sarapar oil is effective than diesel when drilling.

Therefore, this project intends to study another type of oil that can be used as a drilling mud which is it will be cheaper and more reliable. The type of oil that will be used for this experiment is biodiesel which is extraction from jatropha fruits. Comparing this oil from another 2 type of oil (diesel and palm oil), this oil is not as highly demand as those.

## **1.3 OBJECTIVE AND SCOPE OF STUDY**

The main objectives of this research are:

- To measured the characteristic of jatropha curcas oil
- To perform field trials and customize formulation in Formation Damage Control.

The scope of work for this project is to investigate the porosity and permeability of the reservoir using the oil base mud. Once the mud had injected to the model of FDS (Formation Damage System) the change of permeability and porosity will be taking as a result of the experiment. These tests shall simulate the performance the rocks formation in a well where it will be subjected to porosity and permeability.

The properties of the oil and the rheology of the mud need to be identified first and determine the correct amount of oil that will be mixed with the mud to reduce the formation damage of the core.

At the end of the research, the project will justifies these two (2) outcomes:

- 1. To evaluate rheological properties (Mud Weight, Plastic Viscosity, Yield Point, Gel Strength),and filtration loss (static) properties of drilling fluid
- 2. To evaluate the effects of drilling fluids to the porosity and permeability impairment with core flow experiment.

#### **CHAPTER 2**

#### LITERATURE REVIEW

### **2.1 JATROPHA CURCAS TREE**

Jatropha curcas seeds that have been used as raw material have been purchased from CV. Tanah Karo Simalem, Lausolu, Indonesia. Meanwhile, The following chemicals which have been used to synthesize the biodiesel from jatropha curcas seed have been supplied by ZatiKimia Sdn Bhd (Perlis, Malaysia) such as : pro analysis methanol Merck (CH<sub>3</sub>OH, purity > 99.9%), pro analysis hexane Merck (CH<sub>3</sub>(CH<sub>2</sub>)<sub>4</sub>CH<sub>3</sub>, purity > 99.9), pro analysis sodium hydroxide Merck (NaOH, purity > 99%), pro analysis isopropanol Merck (CH<sub>3</sub>CH(OH)CH<sub>3</sub>, purity > 99.8%), sodium methoxide 30 wt.% solution in methanol Acros Organics (CH<sub>3</sub>NaO), analytical grade potassium hydroxide GENE Chemical (KOH, purity > 85%), ethanol absolute GENE Chemical (C<sub>2</sub>H<sub>5</sub>OH, purity > 99.7%) and sodium sulphate anhydrous GENE Chemical (Na<sub>2</sub>SO<sub>4</sub>, purity > 98%).

Moreover, the reference standards for fatty acid methyl ester with gas chromatography grade were purchased from Supelco, USA. These standards consist of methyl laurate, methyl myristate, methyl palmitate, methyl stearate, methyl oleat, methyl linoleat, methyl linolenat, monoolein, diolein, triolein, 1,2,4 butanetriol, and tricaprin. In addition, N-Methyl-N-trimethylsilyltriflouroacetamide (MSTFA) Sigma Aldrich, pyridine Fluka (99%), glycerine, and n-heptane were supplied by RH Oilfield (Kuantan, Malaysia). Meanwhile, the standards for fatty acid ethyl ester with chromatographically purity were supplied by ETD Makmur (M) Sdn Bhd (Selangor, Malaysia). These ethyl ester standards include ethyl laurate (99%), ethyl myristate (98%), ethyl palmitate, ethyl stearate (95%) and ethyl oleat (98%). All the above ethyl esters standards were brand of Merck Schuchardt OHG Hohenbrunn, Germany. In addition, ethyl linoleat standard (99%) was purchased from Sigma Aldrich.

## **2.1.1 Classification**

The jatropha seeds is part of the Euphorbiaceae, one of the plant family with the carrot. The classification of the jatropha tree as below :

Division : Spermatophyta Subdivision : Angiospermae Class : Dicotyledonae Ordo : Euphorbiales Family : Euphorbiaceae Genus : Jatropha Species : Jatropha Curcas Linn

## 2.1.2 Morphology

The jatropha tree can be grow untill 1-7 m, and the branch is not arranged orderly. The stalk is cylinder. The parts of the jatropha tree as below:

### Leaves

The leaves is part of all the segment of the tree and it stick to the branch. The colour of

the leaves is green with the dark green on the top while pale green at the bottom of the leaf. The shape of the leaf is like the heart with the end of the leaf is sharp-pointed. The wide of the leaf is 5-15cm wide. Its have 5-7 bones leaves and the leaves is connect to the tree with the stem and the stem is a about 4-15cm



#### Flower

The flowers in the form of a number of clusters and the colours yellow+green. The



flowers arrangement is arrange like a cup at the end of the branch and each of the clusters of the flower have 5 purple sheath with the length is about 4mm. There are more than 15 flowers for each of the bunch. Jatropha is part of the monoecious plant and the flower is unisexual.

### Fruits

The shape of the fruits is round with 2-4 cm diameter. The length of the fruits is 2 cm with the thick is about 1 cm. The colur of the fruits is green at the young and become grey + chocolate tint when the fruit matured. The jatropha fruits have 3 part of hollow space and each of them is fill by the seed.



The seed shape like straight and slender and the colours is

chocolate + black. This is where the oil come from with 30-50% oil, with the level of the acid in this seed is very high and sure it cannot be eaten.

## 2.2. JATROPHA OIL EXTRACTION

The plants of jatropha are produces the seeds that consist of 60% beans and 40% of the shell in weight percent. The beans of jatropha have about 50% of the oil and can be extract from the beans with mechanical or chemical extraction. The composition of the oil is likely oil of the soil beans.

The oil is more viscous compare to the others oil as it is. The biggest component of this oil is Oleic and Linolenic acid.

## 2.2.1. Pressing Equipments

The equipment of the jatropha is used the screw type model to extract the oil from the seeds. The specification of the model as below:

Capacity : 50-100 kg / hr Screw rotation: 30 rpm Resistance: Flange screw type Engine: Diesel engine type with electric starter Force: 12hp Fuel burner: Solar/Diesel



To start the machine is simply start the engine with the electrical starter. The concept of this machine is simply used the screw mechanism principle with all the materials will be force top flow to screw type machine. The material just simply put in the boxes at the top of the machine and its automatically will be force to the machine. At the end of the day all the seeds will be pushed to extract the oil content in the seeds.

From the mechanical extraction that using this machine, there will be some of the silt residue that still left in the oil. Then, this oil need to filtrate to get out all the silt to make sure that the oil will not affected the other process.

The filtration can be done simply just put the wire cloth and the hydraulic pressing for small and medium scale or using the flat and frame for big scale.

### 2.2.2. Pressing process

There are 2 method that can be done to extract the oil from the seeds, mechanical (rendering) and chemical extraction (solvent extraction). The common method that currently used is using the mechanical extraction which is more cheap than chemical extraction.

There are 2 common method that used in mechanical extraction; hydraulic pressing and expeller pressing.

## Hydraulic Pressing

This method is using the force about 140.6 kg/cm that can be used to extract the oil. The bigger force will expel more the oil from the seeds. Before the pressing been done the seeds need to be heat in the oven or to steam the seeds using the steam to make sure that all moisture in the seeds will get out.

Commonly, for this technique (hydraulic pressing), about 80% of the oil will get from the seeds.

## **Expeller** Pressing

This technique is more efficient to expel more oil from the seeds. It have been using in most of the industry. With this technique, all the seeds will be force with the continuous screw press flow. This method is commonly used because the seeds did not need to put in the oven or to steam it out. The dry matured seeds are just put to the machine to extract it.

There are 2 type of the machine; single screw press and twin screw press.

About 21-24% of extract oil is produce from single screw press and 24-27% is produce from twin screw press.

The advantage that using this technique is that the process is continuous and the capacity is more bigger



Flow diagram Extraction oil from the seeds using twin screw press and solvent extraction

#### 2.2.3. Purification of Jatropha oil

The major reason on the purification of the jatropha oil is to make sure that the oil is to endure the oil ruined. Beside that, the purified jatropha oil is needed for making the biodiesel to make sure that all the residue is expel from the jatropha oil. The residue will make the quality of the biodiesel decreased plus it will destroyed the engine that used diesel as a fuel. For example, the free fatty acid in the biodiesel oil can make the diesel engine get corroded and will form a crust at the surface of the injector. The Gum in jatropha oil will increase the viscosity of biodiesel produced. The purification is consist of degumming, neutralization. Beside that bleaching (the process to change the colour of the oil) and deodorization (to reduce the smell of the oil) need to done.

### Degumming

The gum separation is the process that separate the gum or sticky that consist of phosphate, protein, carbohydrate, water and resin without reduce the fatty acid in the oil. Commonly this process is done by adding the phosphate acid in the oil, then heat it until it will form a phospholipids that easier to separate it. After that, the centrifuge of the oil will take place.

#### Neutralization

It is the process where the oil is separate the free fatty acid from the oil. The reaction between fatty acid and the catalyst until it form the foam. Beside that deasidification can also take place to distill the free fatty acid from the oil.

## Bleaching

Bleaching is the process to clearer the colour of the jatropha oil. It can be done by adding some of the adsorbent like fuller earth, activated clay and active coal. The adsorbent will absorb the all the substances until the water will be clear.

## Deodorization

It is the process to make sure all the bad smell is take out from the oil. The principle of this process is with the steam is been applied at atmospheric pressure or vacuum.

### 2.3. BIODIESEL PRODUCTION PROCESS

Metil Ester (biodiesel) from jatropha oil can be produce by transesterication process. The process is like hydrolysis, but a different is, transesterication process used alcohol instead of water. The catalyst for this process is NaOH or KOH.

Mehtanol is commonly used in this process because it is cheaper and easy to recovery. Transification is the equilibrium reaction, then more alcohol need to make sure that all the reaction is moving forward to the product site. The main factor that influence the reaction is the molar ratio between and alcohol and the type of catalyst used for the reaction. The operating temperature, reaction time and the content of water and the content of free fatty acid in the triglyceride. The another factor that effect the reaction are glycerol content, the type of alcohol used in the reaction, the number of catalyst used and foam content.

The byproduct of transesterication process is the glycerin that can used for making a foam (moisturizing). The flow diagram for making the biodiesel from jatrohpa oil as below:



Flow diagram process flow on making the biodiesel from jatropha Oil

#### 2.3.1. Laboratory scale

In lab scale the reactant that used is from methanol or ethanol and for the catalyst is from KOH and NaOH type of catalyst. When methanol is used the ratio between triglyceride is 10:1 and the catalyst used is KOH with 1% concentration, depend on the volume oil that want to produce.

### FORMATION DAMAGE

The critical parameter determining well productivity is the condition of the near the wellbore region. Formation damage is problem encountered in almost every week development program and production. The nature and thickness of the filter cake deposited on the borehole wall will influence the potential for differential pressure. The permeability reduction will reduce the natural productivity due to the imposition of an extra pressure drop as the fluids flow to the wellbore. The extra pressure drop has to be compensated for either by a reduce pressure drop across the choke by a small production rate. Figure 1 illustrates the resulting producing pressure profile due to formation damage compared to the equivalent pressure profile for undamaged well



 $\Delta P_d = Extra pressure drop due to formation damage$ 

Figure 1: The effect of formation damage on well inflow pressure profile

### 2.5 PERMEABILITY

Permeability in the earth sciences (commonly symbolized as  $\kappa$ , or k) is a measure of the ability of a material (typically, a rock or unconsolidated material) to transmit fluids. It is of great importance in determining the flow characteristics of hydrocarbons in oil and gas reservoirs, and of groundwater in aquifers. There are several factors that influence the permeability of a soil or the core: the viscosity of its water which is slightly influenced by temperature, size and shape of the soil particles, degree of a material. The void ratio is the ratio of volume of voids to volume of solids. However, for a given soil, permeability is inversely proportional to soil density. The more tightly materials particle are packed, the tendency for the material to allow water to flow through it is reduced.

Permeability needs to be measured, either directly using Darcy's Law or through estimation using empirically derived formulas. A common unit for permeability is the Darcy (D) or more commonly the millidarcy (mD) (1 darcy 10-12m<sup>2</sup>). Permeability is part of the proportionality constant in Darcy's law which relates discharge (flow rate) and fluid physical properties (e.g. viscosity), to a pressure gradient applied to the porous media. The proportionality constant specifically for the flow of water through a porous media is the hydraulic conductivity; permeability is a portion of this, and is a property of the porous media only, not the fluid. In naturally occurring materials, it ranges over many orders of magnitude.

For a rock to be considered as an exploitable hydrocarbon reservoir without stimulation, its permeability must be greater than approximately 100 mD (depending on the nature of the hydrocarbon - gas reservoirs with lower permeabilities are still exploitable because of the lower viscosity of gas with respect to oil). Rocks with permeabilities significantly lower than 100 mD can form efficient seals. Uconsolidated sands may have permeabilities of over 5000 mD.

#### 2.6 POROSITY

Porosity is the ratio of void space to the bulk volume of rock containing that void space. Porosity is a measure of the void spaces in a material or percentage of pore volume in a volume of rock and is measured as a fraction, between 0-1 or a percent between 0-100%. The term porosity is used in multiple fields including manufacturing, earth sciences and construction. Used in geology, hydrogeology, soil science and building science, porosity of a porous medium (such as rock or sediment) describes the void space in the material, where the void may contain for example air or water. It is the proportion of the non-solid volume of material and is defined by ratio:

$$\emptyset = \frac{V_p}{V_m}$$

Where  $V_p$  is the non-solid volume (pores and liquid) and  $V_m$  si the total volume material, including the solid and non solid parts. The porosity of a rock, or sendimentary layer, is an important consideration when attempting to evaluate the potential volume water of hydrocarbons it may contain. A value for porosity can alternatively calculated from the bulk density and particle density.

Porosity is directly related to hydraulic conductivity; for two similar sandy aquifer, the one with higher porosity will typically have a higher hydraulic conductivity (more open area for the flow of water), but there are many complications also have very high porosities (due to the structured nature of clay minerals), which means clays can hold large volume of water per volume of bulk material, but they do not release water very quickly. Well sorted (grains of approximately all one size) materials have higher porosity than similarly sized poorly sorted materials (where smaller particles fill gaps between larger particles).

### 2.7 PROBLEM ASSOCIATED WITH FORMATION DAMAGE

The eak-off of filtrate into the formation can substantially reduce the permeability the near wellbore region by a number of mechanism ; most importantly clay swelling particle migration, solid invasion and geochemical transformation.

### 2.7.1 Clay Swelling

Clay minerals which are in small size and plate structural, have larger surface area and therefore tend to react with injected fluids. The clay commonly associated with productivity impairment in smectile, illite, kaolinite, chlorite and mixed layers. These clay minerals are sensitive to composition, pH and ionic strength of the surrounding water which will hydrate and swell many times of its normal size and reduce the radius of flow in a pore, which it is located and facilities the migration of particle by weakening the internal bond strength holding the particle together. Smectite is considered to be the most potentially damaging of these clay groups; illite and kaolinite, while considered to be non-swelling are hydratable and contribute to the migration of fines within the formation.

### 2.7.2 Fines Migration

Fines migration has been identified as a major course of permeability impairment in porous media. The fine particles may either come directly from the drilling mud or released by the invading filtrate from the pore walls. Many different types of migratable fines including the non-expanding authigenic clay minerals (smectile, kaolinite and chlorates), expanding authigenic clay minerals (montmorillonite), quartz and carbonates were identified earlier. These fines loosely adhere to the pore walls and are released due to colloidal and hydrodynamic forces exerted by the invading fluids. The released fines in addition to the invaded solids, if present in sufficient quantities in the flowing fluid, plug the pores throats therefore reduce the permeability.

## 2.7.3 Solid Invasion

The major cause of damage during the drilling is due to the invasion of the mud solids. During drilling the bridging mud solids, whose ranges in size varies from larger to slightly smaller than the pore openings of the rock, build up on the wellbore to form a low permeability filter cake. Particles which are smaller than the pre opening flow in the formation along with the mud filtrate. These solids eventually form internal bridges at pore restrictions between internal minerals grains. The colloidal particles in the drilling fluid that might migrate and block flow channels include clay, cuttings, weighing agents, various polymers and lost circulation agents. Much of the filter cake will be removed through the action of a drill bit and circulating mud, but most of the internally bridged solids may be trapped. These solids create a skin effect around the wellbore, resulting a significant formation pressure drop. If these particles are not flused out completely when the well is put on production, they may block the near wellbore region. The invasion of these colloidal particles in turn can be controlled by adding enough bridging particles of the right size to the drilling fluid. The type of damage (internal or external) as well as the bridging process are functions of the particle or particle or pore ratio, their shape, fluid velocity, particle concentration and charges on the particle and the pore walls.

## 2.7.4 Geochemical Transformation

The majority of formation damage problems arise from fluid and the rock interactions. Damage results due to adverse chemical reactions between the introduced fluid either with the reservoir fluids or with the rock mineralogy. For example differences in chemistry between the invading filtrate and the reservoir fluids could lead to adverse reactions which result in the formation of organic (paraffin and asphaltense) and inorganic (CaCo, BaSO<sub>4</sub>, SrSO<sub>4</sub> and FeCO<sub>3</sub>) scales. Numerous chemical additive such as emulsifiers, corrosion inhibitors, oxygen scavengers, alkalinity control agent and bactericide, normally used in various oil field operations can interact adversely with the wettability from either water – wet to oil – wet or vice verse. Such a change could reduce the effective hydrocarbon permeability in the invaded near wellbore regions the reservoir.

Several parameters such as temperature, fluid velocity, fluid composition, pH of the solution, charges on the particle and pore well and rock mineralogy are important variables that affect these mechanisms. Often, there is simultaneous occurrence of the processes whose relative contribution escalates the formation damage problem. Both water based and oil based mud reported to cause damage to various extent. Due higher solid concentration the solid invasion problem is expected to be higher in oil based mud. To minimize damage there are several specifications a mud should specify. Those include low fluid loss, low concentration of fine particles, able to form a thin and impermeable filter cake, proper mud weight and rheology and non-damaging filtrate.

# CHAPTER 3 METHODOLOGY

## **3.1 GENERAL FLOW CHART**



## **3.2 PROCEDURE IDENTIFICATION**

Masikewish and Bennion (1999) classified the effort necessary for fluid testiong and design to be used in the formation damage tests into six steps;

- 1. Identification of the fluid and rock characteristics
- 2. Speculation of the potential formation damage mechanism
- 3. Verification and quantification of the pertinent formation damage mechanism by various tests.
- 4. Investigation of the potential formation damage mitigation techniques.
- 5. Development of the effective bridging systems to minimize and/or avoid fluids and fines migration into porous media.
- 6. Testing of candidate fluids for optimal selection.

The selection of suitable fluids is important to simulate the exact borehole conditions. It is important to design the same fluids as specified in API standard. For this experiment, the first to third procedure is taking as consideration for formation damage tests using FDS-800-10000. It is recommended however that the implementation of the above procedures into the testing program in order to improve the analysis of the formation damage. Figure 2 illustrates the methodology flowchart of this research project.



**Figure 2: Methodology Flowchart** 

#### **3.3 SAMPLE PREPARATION**

#### 3.3.1. Jatropha Oil Preparation

### 3.3.1.1 Jatropha seeds preparation

Jatropha curcas seeds must be dehulled and ground in order to increase efficiency of the extraction. By using blender, jatropha curcas seeds were ground to particle size 500µm. To obtain homogeneous particle and accurately particle size of those jatropha seeds, sieve trays were used which arranged from 100µm to 500µm size to sieve the jatropha seeds.

After sieving process, it was then placed in the oven to release the moisture content. The oven was set at  $70^{\circ}$ C in 48 hours. The moisture content of the seeds was measured using Mettler Toledo moisture analyzer. The seed were placed in the sample pan until its entire surface homogeneously covered by the seeds. Prior to start calculating the sample's moisture content, ramp time which was 3 minutes must be set then followed by setting the desired temperature which  $100^{\circ}$ C. The reading of the moisture content started when the temperature reached  $100^{\circ}$ C and it was hold for 30 minutes.

#### 3.3.1.2 Oil extraction

There are at least 2 processes to extract oil from the seeds namely mechanical extraction and chemical extraction. Prior to oil extraction the jatropha seeds have been dried in the oven at 70°C to reduce its moisture content. Using mechanical method to extract the oil, either whole seeds or kernels or both of it can be fed into the process. Meanwhile, only kernels can be applied into the process if chemical extraction is used.

#### Mechanical Expeller

To extract oil from the seed mechanically, either a manual ram press or an engine driven screw press can be used. These mechanical expellers can extract the oil from the seed achieve 60 - 65% and 75 - 80% of the available oil using the manual ram press and engine driven screw press, respectively (Henning RK, 2000).

## **Chemical Extraction**

The oil can be extracted chemically. The most common chemical used for extracting oil from the seeds is hexane since it can extract the oil up to 99% of the total amount of the oil available in the seed (W.M.J. Achten et al., 2008). On the other hand, this solvent extraction will only be economical if it is applied in large-scale production of biodiesel which more than 50 tons per day (T. Adriaans, 2006 and W.M.J. Achten et al., 2008) and J. Van Gerpen et al. [56] recommends to use solvent extraction if the mass flow rate is more than 300,000 kg/day.

The oil content of the jatropha seeds was determined by soxhlet extractor. Either 20 grams or 30 grams of jatropha seeds were accurately weighed and placed in the thimble. Meanwhile, hexane with purity 99.9% which ordered from Merck was measured for 140 ml and put inside of the round bottom flask. Then, extraction time started after the equipment had been set up and reached at desired temperature, boiling point of hexane (68°C). To get more accurate data, this process was varied from 2 hours to 6 hours with increment 2. This set up equipment can be seen as figure



**Figure 3: Soxhlet Extraction Process** 

Basically the flow for producing the oil from jatropha seeds will be summarized as flow chart below:



#### **3.3.2.** Core and Mud Sample Preparation

## 3.3.2.1 Core Sample Preparation

There are simple mechanisms of preparing core sample for this research. Unfortunately for this experiment the core that used had been used before. But, there is not the problem since for this research, the result will compare the change of porosity and permeability of the sample core. The core for this research is obtained from the field. Based on the observation the core still in good condition, then its still can be used for the research. The tests should be conduct to measure the initial porosity and permeability of the sample cores. This test is important to make sure that the core still can be used when testing.

The cores were already in 1.5 inch in diameter as suit to the holder size for formation damage test in one foot long. The core is cut to 1 inch length so that the purpose to minimizing the time taken for the flow to pass-through. The cores were trimmed to make sure the flat surface of the cores. The end of the surface of the cores were also been cleaned with the fine flour produced during trimming. The lubricant of the coolant used is the tap water to preserve the state of properties of the samples. Then, the cores were dried in the oven at 100°C overnight to ensure there is no water trapped inside the core.

It is fairly enough to assume that the core that used is same characteristics in the reservoir of the field, since the sample cores were obtained from that area. These condition brought to the conclusion that the rock characteristics of the lithology would be similar in close depth range. The sample cores that using for this research as shown below:

### **3.3.2.2 Mud Sample Preparation**



## 3.4 PHYSICAL AND CHEMICAL ANALYSIS FOR THE JATROPHA OIL

## 3.4.1 Acid value determination

According to American Standard for Testing Material (ASTM D 974–06), acid number is defined as the quantity of base, expressed in milligrams of potassium hydroxide per gram of sample that is required to titrate a sample to a specified end point. The formula how to calculate the acid number based on ASTM D 974-06 is presented as follows :

Acid number, mg of KOH/g = 
$$[(A - B)M X 56.1]/W$$
 (1)

where :

A = KOH solution required for titration of the sample, ml

- B = KOH solution required for titration of the blank, ml
- M = Molarity of the KOH solution
- W = Sample used, g

Procedure to determine the acid number was referred to ASTM D 974-06. 20 g sample was dissolved in a mixture of toluene and isopropyl alcohol containing a small amount of water. Without delay, the resulting single-phase solution was then titrated at a temperature below 30°C with standard alcoholic base to the end point indicated by the colour change. To obtain more accurate data, determination of the acid number was randomly carried out in triplicates with deviation 0.08

## **3.4.2 Physical Properties Analysis**

There are many parameters to determine the physical analysis of the oil. For this experiment the parameters is chosen based on the effectiveness of the properties to the drilling fluids.

Properites	METHOD ROOT
Flash Point	ASTM D93
Aromatics	AMS 140.31
Viscosity	ASTM D445
Density	ASTM D1298
Pour Point	ASTM D97
Sulphur Content	ASTM D3120

Table 1: The properties measured and the method used for each properties

## 3.5 RHEOLOGY CHARACTERIZATION

The rheology test is conducted based on the Recommended Practice Standard Procedure for Field testing Oil-Based Drilling Fluids. (API Recommended Practice 13-B2)



## 3.5.1 Mud Weight (Density) Test.

A mud balance is the usual device on the rig for measuring mud density. A mud balance is a beam balance. The balance beam has a small cup on one end that holds a precise fraction of a gallon, cubic meter, or cubic foot of mud. The beam (arm) has a sliding weight. The arm rests on a fulcrum. A level-bubble on the beams tells the operator when the beam is balanced. A graduated scale on the arms shows mud weight in ppg and  $lb/ft^3$ 



Figure 4: Mud balance

#### 3.5.2 Viscosity and Gel Strength Tests

A direct indicating-viscometer measure gel strength, plastic viscosity and yield point. Gel strength is a measure of the fluid's ability to temporarily gel(become semisolid) when at rest. Plastic viscosity is a fluid's resistance to flow because of friction. Yield point is a fluid's resistance nto flow because attraction between clay particles.

The direct-indicating viscometer consist of 2 cylinders, one inside the other, that rotate by means of a motor or a hand crandk. The mud sample sits between the 2 cylinders. Rotating the outer cylinder (the rotor sleeve) turns the mud, which transfer torque to inner cylinder (the rotor sleeve) turns the mud which transfer s torque to the inner cylinder, or bob. A spring restrains the movement of the bob, and dial indicate moves.



Figure 5: Viscometer

## 3.4.3 Filtration Lost Test, Low Pressure Low Temperature (LPLT)

Measurement of the filtration behavior and the filter cake characterization of an oil-based drilling fluids are fundamental to the treatment and control of a drilling fluid, as are the characteristics of the filtrate, such as oil, water or emulsion content.

The LPLT filter press must have a filter area of 4520 to 4640 square millimeters, which is a diameter 75.86 to 78.6 mm. the filer press gasket is the determining factor of the filter area. It is recommended that the filter press gasket used be tested by a conical gauge tat has the maximum (78.6 mm) and the minimum (75.86 mm) marked on it. Any filter press gasket found out of these ranges will be discarded



Figure 6: Low Pressure Low Temperature filter press

### 3.6 FORMATION DAMAGE EXPERIMENT SETUP

The design of the apparatus for testing of core samples with fluids varies with specific objective and applications. Typical testing systems include core holders, fluid reservoir, pumps, flow meters, and fluid sample collectors, control systems for flow, pressure and temperature, and data acquisition system. As described by Doane (1999), Figure **\*\*** indicates the current reservoir condition leak-off evaluation system. This system was designed for core testing at near in-situ conditions. The leak-off evaluation system contains 2 back pressure systems which controls the back pressure to whatever gas supplied. In this study, the back pressure system I sued to simulate the differential pressure in place of the pressure transducers. The leak-off evaluation system is similar to FDS-800-10000 which is used for this study. Several additional functions are added to increase the advantage of the FDS-800-10000 such as the ISCO metering pump and overburden pump. The pressure limit of the overburden as well as the back-pressure

system of FDS-800-10000 us 10,000 psi which is much highest that the apparatus Doane et al worked on. The previous leak-off evaluation system has a pressure limit of 2000 psi.

Formation damage measurement were made with 2 different drilling fluids. Each permitted the determination of the core permeability before and after exposure to the fluids. One of the drilling fluids will use Sarapar oil as a oil base and for the second is used Jatropha oil as a oil base. Pressure taps along the core holder is maintained by constantly applied the pump flowrate of 5 mil/min intervals allow direct spatial resolution of the permeability impairment during and after filtration. Before used, cores were vacuum saturated with brine 30000 ppm simulating connate water saturation from the reservoir. The initial permeability was measured by directly incjected sarapar simulating the reservoir oil. The core is assumed to be in reservoir condition with residual water saturation. Figure 7 shows the flowchart of the procedure:



Figure 7 : Experimental flowchart for Formation Damage Control

The core sample prepared should be loaded into the core holder through the sleeve provided. The domes are attached at the ends after fillings up the spaces within the sleeve with spacers which are hollowed cylindrical metal blocks. The spacers prevent the sleeve from rupture under overburden pressure acting upon the sleeve. The core sample and holder are mounted horizontally for the analysis. The overburden pressure is gradually increased until it reached the desired pressure, which in this experiment is 2000 psia. This represents the reservoir pressure also known as the confining pressure. When the overburden pressure is achieved the flow of the test fluid is started using the ISCO pump. The pump injects the test fluid at a specific rate in ml/min. specific valves are opened manually to commence.

The formation damage test system s turned on for at east 1 hour to warm up the electronics. As the electronic circuits are warming up, the electronic gauges are calibrated to show the true zero. The valves C2 and C3 are opened to allow brine flow from accumulator. Valves 26 and 27 are opened to allow fluid flow into the core samples. All other valves, A1,A2,A3,B1,B2,B3,C1,28 and 29 are closed. The confining pressure, which simulates the reservoir pressure. Water is flowed into the core holder and pressurized to 2000 psi. the overburden pressure is confined by closing the overburden pressure valve. Next, the dome back pressure is set. The inlet BPR is set at 0 psi. the system is ready for experiment.

Test nonemator	Core samples		
Test parameter	1	2	
Overburden pressure	2000	2000	
ISCO pump flowrate (ml/min)	5	5	
ISCO Pump Pressure (psia)	500	500	
Inlet BPR pressure (psia)	500	500	
Outlet BPR pressure (psia)	0	0	

At the Smart Series software, the system is set to 'Online'. All communications must be good before any further step is taken. The flowrate of the ISCO pump and safety pressure is set at 1 ml perminute and 1000 psi respectively. The flowrate of the ISCO pump is set at 1 ml per minute to allow the brine to flow through the core sample. The safety pressure

is to ensure that the brine fluid flows into the core without restrictions. Logging mode on the Smart Series software is et to auto and the intervals is set to every one minute. The ISCO pump is activated by pressing the 'START' button on the software. The pressure gauges are monitored closely and adjusted accordingly. The logging for results is stopped after reading has stabilized. This could take at least 1 hour.

The experiment test system is divided into 3 parts: test system setup, processes and termination of experiment. The processes section is where logging of permeability reading is taken. The processes are named as Process 1-Brine (normal flow), Process 2-Drilling Fluid (normal flow) and Process 3- Brine (reverse flow). Figure 8 shows the flowchart of formation damage testing procedures.



Figure 8: Flowchart for Formation Damage testing Procedures.

### **CHAPTER 4**

## **RESULTS AND DISCUSSION**

## 4.1 FREE FATTY ACID TEST

For the free fatty acid test the standard methodology had used. The method is from AOCS Official Method Aa 6-38 (Revised 2001). The test include some calculation to calculate the free fatty acids content in the jatopha oil. This test is very important to make sure that the jatropha is not too viscous and meet requirement of API standard.

There are three repeated test for this experiment. Table below show the result of the test using this method.

	Rice Toris wild Value
First test	6.773
Second test	6.908
Third Test	6.514
Average with standard deviation	6.753+-0.193 mg of KOH/g.

Table 2: Value for free fatty acids in Jatropha Oil

The value for free fatty acids for the oil is high. Than, some additives is need in order to reduce the viscosity. High fatty acids will produce the soap when react with the additive especially with the water.

# 4.2 PHYSICAL PROPERTIES ANALYSIS OF JATROPHA AND SARAPAR OIL

	altanis o si su galetti Nga su sakara parte		Name and the second
		Saaparen	
Flash Point , °C	240	120	Improved Safety
Aromatics, WT%	N/A	<0.01	Less Dermatitis & Rubber Swelling
Viscosity (at 25°C) , mm2/s	45.2	5	Drilling Speed
Density (at 25°C), g/cm3	0.98	0.77	Mud Weight Effect
Pour Point, oC	-2.5	-16.11	Winter Performance
Sulphur Content, ppm	<1	10	

Table 3: Physical properties for Jatropha and Sarapar Oil

The table showed the physical properties of the jatropha and sarapar oil with the mineral oil advantage of the properties. From the table jatropha oil has three advantages than the sarapar oil which is as below:

## i) Flash Point

Jatropha oil has higher flash point than sarapar oil. This will increase the safety of the oil used during the drilling because the depth of the bore will increase as long as with the bore temperature.

#### ii) Aromatics

Jatropha oil didn't have any aromatics amount because the oil is from the chain of the organic components. It will give a 'green' impact to the environment and will not give a big impact or harm to the area of the drilling activities if there are blowout happened.

iii) Sulphur Content

Sulphur is naturally present in small quantities in petroleum and coal. SO2 emissions contribute to the formation of secondary inorganic aerosol gases, fine particles which are harmful to human health. Jatropha oil has very small of sulphur content compared with the sarapar oil. It will let give small impact to the operator at the rig when dealing with this oil

The viscosity and density of the jatropha oil didn't show the good result. Because the viscosity of jatropha oil is too high and the density is low. The high viscosity of the jatropha oil will give a big impact to the mud. The mud is expected to be too viscous and specific additives need to be mix with the solution to reduce the viscosity of the mud. Density of jatropha oil is lower than the water. Than, it is expected the weighting material like barite should be used to increased the density of the oil.

For the 4 season country like Euro and US, the pour point characteristics should also be part of the fluid. Jatropha oil has higher pour point than sarapar which is not very suitable enough when drilling during winter season.

### **4.3 DRILLING FLUID TEST**

## 4.3.1 Rheology Characterization

So far, there are 5 experiment had been conducted to find the best rheology for the mud. The rheology part for all the experiment will be focused on the PV,YP and GS value only. For the first experiment, the methodology to prepare the mud is by following the method performed by Scomi Oiltools company. But for this experiment, the oil will substitute from sarapar oil with the jatropha oil. This experiment was failed due to the super highly viscous of the mud. The cause stll cannot find yet at this moment. The additives that used for this experiment as below:

Sarapar (Substitute Jatropha Oil) (ml)	242.0
Versamui (ppb)	5.0
Versacoat (ppb)	2.0
Lime (ppb)	5.0
Drill Water (Substitute as a Distilled water) (ml)	60.6
Calcium Chloride (ppb)	15.0
Visplus (ppb)	6.0
Versatrol (ppb)	5.0
Barite (ppb)	170.0

For the next experiment, the experiment was conducted by taking out some additives which is Visplus and Versatrol.

For the second experiment, the Visplus and Versatrol additives was taken out and the others are maintain.

Sarapar (Substitute Jatropha Oil) (ml)	242.0
Versamul (ppb)	5.0
Versacoat (ppb)	2.0
Lime (ppb)	5.0
Drill Water (Substitute as a Distilled water) (ml)	60.6
Calcium Chloride (ppb)	15.0
Barite (ppb)	170.0

The experiment also was failed due to very high viscous of the mud. Than, literatures review had been done with all the function of the additives to know what is the function and the effect of the additives to the mud. Decision was made to restart all the procedure of adding all the additives to the mud. Try and error method will be done to achieve that. The third experiment, the basic jatropha oil with the distilled water will be added to the mud with the ratio 70:30. All the mixing are being mixed with duration for 30 minute. The rheology of the mud as followed:



Kneology	
Mud weight (ppg)	7.25
600rpm	81
300rpm	55
PV(cP)	26
YP(ib/ft2)	29
Gel Strength (10sec) (lb/ft2)	70
Gel Strength (10min) (lb/ft2)	82

From the table we can see that the mud weight is low but the PV, YP and the GS are hugh. Some barite need to added to the mud in order to increase the mud weight.



For the fourth experiment, the barite with 100g was added with the solution that contained jatropha oil and the distilled water with 70:30 ratio. The rheology for the solution as below:

Rheology

Mud weight (ppg)	9.2
600rpm	231
300rpm	132
PV(cP)	99
YP(lb/ft2)	33
Gel Strength (10sec) (lb/ft2)	226
Gel Strength (10min) (lb/ft2)	245

The results show that the mud weight, PV, YP and GS value was increasing from the previous experiment.

Percent of increased from the fourth experiment : Mud weight = 26% PV = 280% YP = 13.7% GS (10 sec) = 222.8%

## GS (10 minute) = 198%

The barite effect to much with PV and GS value. But the value of the mud weight is still low. This experiment has to get the mud weight value about 10 ppg and above.

For the fifth experiment, the barite was increased by 200g. It was added with the solution that contained jatropha oil and the distilled water with 70:30 ratio. The rheology for the solution as below:



#### **Rheology**

Mud weight (ppg)	10.7
600rpm	270
300rpm	156
PV(cP)	114
YP(lb/ft2)	42
Gel Strength (10sec) (lb/ft2)	286
Gel Strength (10min) (lb/ft2)	299

The results show that the mud weight, PV, YP and GS value was increasing from the previous experiment.

Percent of increased from the fifth experiment :

Mud weight = 16% PV = 15% YP = 27% GS (10 sec) = 26% GS (10 minute) = 22%

The barite did not affect much the PV,YP and GS value. But it has increased the mud weight to 10 ppg.

From the observation for all the experiment, the plastic viscosity, yield point and gel strength of the mud is still at the high value. Some additives need to be searched to reduce the value.

From the 3 test above, it showed that the PV value is giving the very impact to the Mud Rheology. Than, another additive has to find to reduce the PV value of the mud. Thus, the rheology test again repeated by using the same method but, at this time the experiment used the Kerosin or Thinner to reduce the viscosity of the mud as been normally practice at the rig.

For the fourth experiment, the test used mixing of thinner to substitute the water as the solution for the mud. The percent mixing of the solution consist of 70:30 of oil and thinner respectively.



#### Rheology

Mud weight (ppg)	7.5
600rpm	29
300rpm	14
PV(cP)	15
YP(lb/ft2)	1
Gel Strength (10sec) (lb/ft2)	28
Gel Strength (10min) (lb/ft2)	28

From the rheology we can see that the PV and all the viscosity part is reduces but at the same time the mud weight of the mud is reduce due to the thinner is the one of diemulsifier. From the API standard (for education) the value is at par of the standard. But again the mud weight is still lower than API standard.

The test again repeated with the same amount ratio of thinner and the oil but at this time the barite is included to increase the mud weight. 50 g of the barite is to be mix with the solution.



Rheology	
Mud weight (ppg)	9
600rpm	40
300rpm	22
PV(cP)	18
YP(lb/ft2)	4
Gel Strength (10sec) (lb/ft2)	41
Gei Strength (10min) (lb/ft2)	38

From the data, showed that the effect from the barite is increased the mud weight of the mud, but at the same time, the value of PV, YP and GS are also increasing.

Percent of increase from the previous experiment:

Mud Weight = 20% PV = 20% YP = 300% GS (10 SEC) = 46% GS (10 MIN) = 36%



The test is optimize by try to put more of the barite to increase the mud weight to 10 ppg. About 100 g of barite is added with the basic solution (70% oil and 30% thinner).

## Rheology

Mud weight (ppg)	9.2
600rpm	53
300rpm	29
PV(cP)	24
YP(lb/ft2)	5
Gel Strength (10sec) (lb/ft2)	55
Gel Strength (10min) (lb/ft2)	53

From the data is showed that the increased the barite is not effect much on the oil and thinner solution. The mud weight increased about 2%. Increasing the barite is more effect

to the viscosity value especially the PV and the GS, the increased percent is about 33% and 30% respectively.

From the all the experiment, the parameter only include the mud weight and the viscosity only. But the filtration lost study is not include as the parameter. Than, from the literature review, there are commonly 2 additives that basically used by the operator at the rig to increase lower down the filtration lost of the mud into the bore. The 2 additives that basically used by the operator are Visplus and Vesatrol.

For next test, the parameter for the filtration lost is also include as the rheology part. The additives for the mud is fixed but the other 2 additives are also included for the test. The amount for the 2 filtration lost additives are 12g and 10 g for Visplus and Vesatrol respectively.



## Rheology

Mud weight (ppg)	8.4
600rpm	54
300rpm	28
PV(cP)	26

YP(lb/ft2)	2
Gel Strength (10sec) (lb/ft2)	55
Gel Strength (10min) (lb/ft2)	56
Filtration Lost(30min) (ml)	45

From the data it showed that the filtration lost for this mud is quite high, its about 45ml within 30 minutes. It showed that the thinner is act as the diemulsifier for the mud. It mean that the thinner act as the surfactant that unclump and disettle out from each other of the mud and create the easiness of the liquid to flow out pass through the solid in the mud.

The next test is including the water and reducing the thinner to the mud while at the same time maintaining the other additives. Water is expect can act as the emulsifier for the oil, which means that by adding the water can reduce the filtration lost happened to the mud.



## **Rheology**

Mud weight (ppg)	8.4
600rpm	129
300rpm	69
PV(cP)	60
YP(lb/ft2)	9
Gel Strength (10sec) (lb/ft2)	126
Gel Strength (10min) (lb/ft2)	132
Filtration Lost(30min)(ml)	8

From the rheology, it shows that the filtration lost for the mud is decreasing to 82%. It prove that water can act as a emulsifier for this solution. But at the same time water also can increase the PV value for the mud by 53%, thus increased viscosity of the mud.

An optimization is trying to do with the mud by increasing the additives for the filtration lost. For that purpose, the next test is to increased the amount of the Versatrol and Visplus to double. The filtration lost is expected to decrease more from the previous test.



## Rheology

Mud weight (ppg)	9
600rpm	130
300rpm	70
PV(cP)	60
YP(lb/ft2)	10
Gel Strength (10sec) (lb/ft2)	128
Gel Strength (10min) (lb/ft2)	135
Filtration Lost(30min)(ml)	6

From the test is show that the filtration lost for the this mud is reducing to 25% as expected. But the percent of reducing is not give much impact to the mud. So, the previous amount of the filtration additive should be enough.

From all the test conducted to found the optimize solution for the mud, the next step that would cover the formation damage test will be using the mud that consist of:

	Amount	Function
Jatropha Oil (ml)	280	
Thinner (ml)	45	To reduce viscosity
Water(ml)	35	Emulsifier
Barite(g)	50	To increase the density
Visplus(g)	6	Eiltration lost additives
Vesatrol(g)	5	Fill alon lost additives

Table 4: The additives and the function of it to the Jatropha Oil Base Mud

For formation damage test the experiment will conducted by comparing the performance of the Jatropha Oil Base Mud with the Sarapar Oil Base Mud that commonly used in the real field. The additives for the Sarpar Oil Base Mud as below:



1 V	• -
YP	6
GS(10SEC)	7
GS(10MIN)	14
Filtration Lost(30min)	4.4

The rheology from the mud shows that it is better that the Jatropha Oil Base Mud. Its expected to give a small damage to the core during the formation damage test comparing using the Jatropha Oil Base Mud. The expectation base because the fluid is commonly used for drilling in many areas and its prove work.

## **4.4 POROSITY MEASUREMENT**

For this experiment program, two cores used for testing is labeled as 1 and 2 and each representing single test. The labeling of the cores is as follows

Core sample	Fluid testing
Core 1	Sarapar Oil Base Mud
Core 2	Jatropha Oil Base Mud

Table 5: Core sample labeling

Porosity is scalar measurement by the ratio of pore volume to the bulk volume of the porous media. Faruk Civan (2000) suggested the reservoir porosity ranges from 1.5% to 50%

Sample	Effective Co		
	Porosity(%)		
Core 1	21.06		
Core 2	24.06		

Table 6: The percentage of effective core porosity

As suggested by Civan, the core samples tested in this experiment meet the requirement of average porosity os 20%. From table 6 for core no 2 the permeability of the core is expected more than core no 1. The expectation are based on the fact that porosity do not have interrelation to the permeability yet can be one of the criteria for early assumption

#### **4.5 FORMATION DAMAGE TEST**

Formation damage is determined difference in initial and final permeability. The initial permeability of the core is different between each other, because this test will use the used clean core for the formation damage test. The core had been cut into two inches lng for testing purposes.

The results from the tests were then plotted to clearly view the behavior or the impact of the mud onto the permeability of the core. By convention of the dependent variable, elapsed time defines the x-axis and the independent variables such as permeability and percentage of reduction defines the y-axis. The experiment is conducted for 30 minute for every testing. The core sample exposed to 500 psi differential pressure at vertical conditions. At higher differential pressure, the migration of solid particles proportional to the differential pressure. Generally, greater solid invasion causes severe blockage of pore throats that induces greater reduction in permeability of the core sample.



Figure 9: Permeability change using Sarapar Oil Base Mud with respect to time

Figure 9 show the permeability change plot before and after Sarapar Oil Base Mud was injected to the core. Brine was injected to get the initial permeability. During this process the results show the constant permeability of the core average 57.75 mD showing the

core has average permeability. The core was than injected with the Sarapar Oil Base Mud to see the impact of the mud to the core. After that the core is backflow with the brine to demonstrate the reservoir condition during the production where brine replacing the water produce and leaving with residual oil inside the core sample. During this process the permeability show zero value (see figure 10) which mean the brine cannot pass through the core during backflow process. It is because of the mud cake that created during the mud injection give a support to the core to hold the brine from pass through. Than the brine again was direct injected through the core and the permeability was slightly reduce to 23.4 mD. In real drilling, its mean that the fluid just can flow direct to the mud and the oil from the reservoir can't pass through the mud. The impact give the good characteristics for the fluid to use it as drilling fluid.



Figure 10: Permeability of the core during backflow injection



Figure 11: Permeability percentage reduction of Core 1

From figure 11 show the plot for percentage reduction calculated by dividing the change of the core permeability to the initial of the permeability. The percentage reduction shows the slightly increase in permeability (58%) reduction of the core with respect to time. This effect show that the Sarapar Oil Base Mud does not give significant damage and considerably good fluid.



Figure 12: Permeability change using Jatropha Oil Base Mud with respect to time

Figure 12 shows the permeability plot before and after Jatropha Oil Base Mud injection. The graph show that the high and constant permeability around 67.23 mD of the core before injecting with the mud. However, the final permeability of the core show the extremely decreasing the permeability of the core after direct inject it with the brine. The average value of the core after injected is 0.00 mD. Its goes same happened when using Sarapar Oil Base Mud during the backflow injection, (see figure 13) the permeability of the core is almost none or zero. It shows that the fluid cannot pass through neither direct nor backflow of the core. This happened because the characteristics of the mud that very high in viscosity that had blocked all the porous medium in the core. As a result the fluid cannot pass through the core even after the period of time.



Figure 13: Permeability of the core during backflow injection



Figure 14: Permeability percentage reduction of Core 2

From figure 14 shows the plot for percentage reduction calculated by dividing the change to initial permeability. The percentage reduction shows the huge increase in permeability reduction of the core with respect to time. This effect show that the Jatropha Oil Base Mud does give significant damage and considerably not a good drilling fluid.

Sample	Permeabilit	Permeability(mD)		Damage Ratio
	Initial	Final		
Core 1	57.75	23.4	58%	31.13
Core 2	67.23	0.00	100%	67.23

Table 7: Data for the Formation Damage test

## **CHAPTER 5**

### **CONCLUSIONS AND RECOMMENDATIONS**

## 3.4 CONCLUSION

- Free fatty acids for the material used for this experiment which is the Jatropha Oil is very high. This lead to the increasing of the viscosity of the fluid. Free fatty acids is represent the amount of the oleic acids in the material or the fluids. For this experiment, the amount of free fatty acids in the fluids is 6.753+-0.193 mg of KOH/g.
- 2. For the physical properties of the jatropha oil, it show a better properties compared with the sarapar oil that commonly used as a drilling fluid. Jatropha oil has the advantages compared with the sarapar oil in term of flash point, amount of aromatics and sulphur contents. The characteristics for the higher flash point make the jatropha oil more safer to used compared with sarapar oil.
- 3. However, the main problem that facing by the jatropha oil is the viscosity of the oil. It more viscous than sarapar oil that can lead to the higher value of viscosity for the rheology for the oil base mud.
- 4. It proves that when rheology test had been conducted to find the optimum additives for the jatropha oil, the optimum solution is hard to find. Several tests and experiments had to conduct to find the right amount of additives for the mud. It is because, from the experiments, it's found that the there is trade of in order to reduced the viscosity and the mud weight for the mud.
- 5. Generally, for the Sarapar Oil Base Mud, it has perfect in rheology characteristics compared with the Jatropha Oil Base Mud at the same temperature and pressure

(ambient condition). These characteristics represent the better performance of the Sarapar Oil compared with the Jatropha Oil.

6. During formation damage testing using Sarapar Oil Base Mud, the result show the damage occur for the core is about 58%. It explained that the ability of the core to flow the fluid is decreasing to 58% after injection with the mud. Compared with the Jatropha Oil Base Mud, the mud gives totally damage (100%) to the permeability of the core. It is because of the properties of the mud that has higher viscosity and will lead to the affect of the permeability of the core.

### 3.5 **RECOMMENDATIONS**

Since Jatropha Oil give totally damage to the permeability, the oil can use for the dead zone area, which is not production zone. It's because the mud cake that created by the mud will hold the bore to prevent any liquid to flow in or out through the core.

For important and meaningful formation damage characterization, laboratory core flow tests should be conducted under certain conditions:

- 1. Samples of actual fluids and formation rocks and all potential rock fluid interactions should be considered. This will exhibits the effect of wetting conditions of the grain surface with the fluids or solid invasion
- 2. Laboratory tests should view the specific and real conditions of the specific field operations especially on the temperature and pressure of certain selected reservoir to show the real effects of the fluids to the specific reservoir, including drilling, completion, stimulation and present and future enhance oil recovery technique

For future analysis, it is recommended to further this research into these parts:

- 1 The conditions of the test should be various possible temperature and pressure conditions, from Low Pressure and Low Temperature (LPLT) to High Pressure and High Temperature (HPHT) conditions so that it will identified, at certain pressure and temperature, the effect will change and comes to the most suitable and optimum conditions.
- 2 The purity of the additives such as barite should be identify the purification to ensure that exact composition of the barite and will not affect the rheology characteristics of the mud.
- 3 Specific and special additives should be used to treat Jatropha Oil Base Mud in order to reduce the viscosity and at the same time to maintained the mud weight of the mud. The organic sulphate additives can be used as good additives for the next tests.

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# APPENDICES

APPENDIX I Tabulated data for Formation Damage Test

Elapsed	Γ	[			Damage
Time	Initial Perm	Backflow	Final perm	Damage Ratio	percent
1	58.314	0	27.183	31.131	53.38512193
2	58.314	0	27.968	30.346	52.03896148
3	58.314	0	27.133	31.181	53.47086463
4	58.314	0	27.57	30.744	52.7214734
5	58.314	0	27.722	30.592	52.46081558
6	58.314	0	25.956	32.358	55.48924787
7	58.314	0	24.705	33.609	57.6345303
8	58.314	0	23.527	34.787	59.65462839
9	57.366	0	22.497	34.869	60.78339086
10	58.314	0	21.81	36.504	62.59903282
11	58.314	0	22.055	36.259	62.17889358
12	57.366	0	22.031	35.335	61.59571872
13	57.366	0	22.57	34.796	60.65613778
14	57.366	0	22.816	34.55	60.22731235
15	57.366	0	22.914	34.452	60.05647945
16	58.314	0	22.595	35.719	61.25287238
17	58.314	0	22.325	35.989	61.71588298
18	57.366	0	22.129	35.237	61.42488582
19	57.52	0	22.57	34.95	60.76147427
20	57.366	0	22.742	34.624	60.35630861
21	<b>57.36</b> 6	0	23.846	33.52	58.43182373
22	57.366	0	24.435	32.931	57.40508315
23	57.366	0	24.582	32.784	57.1488338
24	57.366	0	24.165	33.201	57.87574521
25	57.366	0	23.871	33.495	58.38824391
26	57.366	0	23.674	33.692	58.7316529
27	57.366	0	23.478	33.888	59.07331869
28	57.366	0	23.331	34.035	59.32956804
29	57.366	0	23.38	33.986	59.24415159
30	57.366	0	23.552	33.814	58.94432242
Average	57.750	0	23.97106667	33.77926667	58.501

Data Collected for Formation Damage test using Jatropha Oil Base Mud (core1)

Damage percentage using Sarapar Oil Base Mud is 100%

Elapsed	Initial		Final		Damage
time	Permeability	Backflow	Permeability	Damage Ratio	percent (%)
1	61.231	0	0.001	61.23	100.00
2	61.312	0	0.001	61.311	100.00
3	61.451	0	0.001	61.45	100.00
4	61.231	0	0.001	61.23	100.00
5	62.012	0	0.000	62.012	100.00
6	62.099	0	0.001	62.098	100.00
7	62.124	0	0.001	62.123	100.00
8	62.579	0	0.001	62.578	100.00
9	62.165	0	0.002	62.163	100.00
10	62.687	0	0.003	62.684	100.00
11	62.598	0	0.003	62.595	100.00
12	63.575	0	0.004	63.571	99.99
13	66.392	0	0.004	66.388	99.99
14	65.868	0	0.003	65.865	100.00
15	67.347	0	0.004	67.343	99.99
16	68.304	0	0.003	68.301	100.00
17	68.445	0	0.002	68.443	100.00
18	67.741	0	0.004	67.737	99.99
19	69.257	0	0.003	69.254	100.00
20	69.085	0	0.004	69.081	99.99
21	69.085	0	0.003	69.082	100.00
22	68.454	0	0.004	68.45	99.99
23	70.424	0	0.005	70.419	99.99
24	70.366	0	0.007	70.359	99.99
25	73.389	0	0.006	73.383	99.99
26	73.42	0	0.009	73.411	99.99
27	73.42	0	0.008	73.412	99.99
28	76.26	0	0.007	76.253	99.99
29	76.831	0	0.006	76.825	99.99
30	78.001	0	0.007	77.994	99.99
Average	67.238	0	0.004	67.23483333	99.99

Data Collected for Formation Damage test using Jatropha Oil Base Mud (core2)

Damage percentage using Jatropha Oil Base Mud is 100%