Calophyllum inophyllum Oil as Novel, Green and Low Cost Ester-Based Drilling Fluids

by

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12295

A dissertation submitted in partial fulfillment of

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CERTIFICATION OF APPROVAL

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Approved by,

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(Dr. Sonny Irawan)

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TRONOH, PERAK

JUNE 2012

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 acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

(Irshad Fajar, ID: 12295)

ABSTRACT

In oil and gas drilling operation, the drilling fluid performance dictates the successfulness of the project. The oil based mud (OBM) is proven to drill faster and results in lesser wellbore problems compared to water based mud (WBM). Yet, the usage of OBM is prohibited by regulation due to its environmental impact. The drilled cuttings are not allowed to be discharged in the environment. Thus, the usage of plant derived ingredient base oil in drilling mud using *Calophyllum* methyl ester might be a breakthrough in drilling fluid industry.

Calophyllum inophyllum oil is naturally organic, has similar physical properties with mineral oil base, and economic due to its non-edibility, hence very suitable as base fluid. *Calophyllum* oil is extracted from *Calophyllum inophyllum* plant, or in Malaysia, it is called 'Bintangor'. With wide distribution from east Africa, southern coastal India, Indonesia and Australia, *Calophyllum inophyllum* plant is a potential sustainable resource of base oil supply for drilling mud. The crude *calophyllum* oil is processed through esterification and transesterification process, to reduce its viscosity. As result, *calophyllum* methyl ester is produced which then be used as base fluid for ester-based drilling fluids.

The purpose of this research is to investigate the possibility of calophyllum methyl ester base fluid to be used as alternatives to replace conventional mineral oil base fluid for synthetic-based mud (SBM) application.

The scopes of this project are limited to studying the characteristic and botanical description of *Calophyllum inophyllum* plant as the source of calophyllum methyl ester base oil, formulating the calophyllum methyl ester based mud, testing the drilling mud performance and emulsion stability; and to conduct economic analysis prior to the application. The rheological behavior study of the lime and organophilic clay concentration effect, mud density changes, temperature changes and oil water ratio change were investigated. Filtration characteristic of the designed mud system was investigated as well.

The eco-toxicological properties test was carried out to check the impact of the mud towards the environment. *Poecilia reticulate* (Guppy fish) is used as fresh-water

organism indicator for this test. A comparison study about the rheological behavior of calophyllum methyl ester-based mud was conducted with palm methyl ester and common mineral oil-based mud to observe its mud compatibility and performance. Advantages and cost analysis of using calophyllum methyl ester-based mud to the application are studied and analyzed.

Rheological properties of calophyllum based mud were tested to fulfill requirement by the specification, as result, formulation of IF-6 mud sample with the value of Plastic Viscosity (PV) 39 and 53 cp, before and after hot rolling test, respectively with the range of specification of 45-55 cp after hot rolled. The Yield Point (YP) is 9 and 15 lb/100ft^2, before and after hot rolling test, respectively with specification range of 15-25 lb/100ft^2, and Gel Strength (GS) 10 minutes, 12 and 9 lb/100ft^2, before and after hot rolling test, respectively, with specification range of 20-40 lb/100ft^2. It showed that the rheological properties of calophyllum based mud had been fulfilled for PV and YP specification, yet for GS; it needs to be modified by adding the gel additives more. For the emulsion stability, IF-6 had been fulfilled the requirement of emulsion stability standard which is more than 600 Volts, the value of IF-6 Emulsion Stability (ES) is 940 Volts after subjected to hot rolling. In addition the results of rheological behavior study with different variation were supportive to previous research study and theory. In conclusion, calophyllum ester-based mud is compatible with the mud additives and technically acceptable with the given rheological specifications. Calophyllum ester based mud was also environmentally friendly with the concentration below 10,000 ppm for the freshwater environment based on the environmental test results.

This paper is consisted of five (5) sections. Introduction part contains background information, problem statement, objectives and scope of study of this project. Then, in literature review part contains the botanical description, production system and the physical-chemical properties of *Calophyllum inophyllum.* Methodology part contains procedural steps to achieve project objectives. Then, it is followed by results and discussion about the feasibility of *calophyllum* oil as ester-based drilling fluid applications, and; lastly conclusion and recommendations for overall project.

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CHAPTER 1

INTRODUCTION

1.1. Background Information

Drilling fluid is a circulating fluid used in rotary drilling to perform various important functions required in drilling operations. In order to have effective and smooth drilling operations, drilling fluid design are needed to be optimized.

Oil based drilling fluids are more preferred in highly technical and challenging well drilling operations, mainly, because it creates lesser borehole problems than water based mud. This is due to non-polar nature of the base oils used in formulating oil based mud, they are essentially non-reactive with troublesome formations such as shale, gypsum, anhydrate etc[1]. The advantages of oil based mud over water based mud includes; providing good wellbore stability, good lubrications which leads to faster rate of penetration, better thermal stability, lesser dilution volume, low formation damage, fewer hole problems, reduced risk of pipe stuck due to differential sticking, clay swelling and shale sloughing events; reduced overall cost due to reduced fishing operations and Non Productive Time (NPT).

1.2. Problem Statement

As the facts that, oil based mud performs better than water based mud, but unfortunately it is highly toxic due to its high level of aromatic content. Thus, the drilled cuttings and its oil retention are prohibited to be discharged into environment. Industry currently uses mineral oil based mud (e.g. sarapar 147 and saraline 185V) with lower toxic level and aromatic content, but due to poor biodegradability and stricter environment regulations, this type of mud cannot be used in certain geographical locations.

As for today, in many parts of the world such as USA, United Kingdom, Holland, Norway, Nigeria and Australia, the use of diesel and mineral oil based mud in offshore operations is restricted or banned because of their toxicity and bio-accumulation. A typical well may produce between 1000 and 1500 tons of cuttings. With average oil retention of 15%, around 150-225 tons of oil from discharged drilling fluid into the sea for each drilled well[2], thus creating major environmental hazard as high toxic oils pollute waters and perish marine life creatures. The usage of oil based mud in drilling can pollute and impact ecological of marine life and onshore life due to its toxic cuttings and low biodegradability. The mineral oils and most petrochemical synthetic based fluids are being pollution and ecological disturbance of marine life due to low biodegradability, especially in anaerobic conditions, high toxicity due to the presence of aromatic compounds and its severe impact on the sea floor sediments, underground water and food chain. All of these bad impacts are of course would be unwanted.

The other problems came up when the requirement of environmentally friendly oil based mud or synthetic based mud simultaneously increasing as the number of wells keeps increasing. There will be shortage of supply in catering the ‘green’ drilling fluid base demand all over the world. The existed palm ester-based drilling fluid were not enough to supply the entire operations. Moreover, palm oil are also used for human consumption, that makes the price is expensive and become not economical to be used as drilling fluid base. The alternatives, thus, are always needed to replace palm methyl ester based mud. The alternatives oil base which fulfills the criteria of technical acceptability, environmental acceptability and low cost / economic hence are still questionable.

1.3. Significant of Project

The alternatives oil base for environmentally friendly, readily biodegradable and technically qualified mud with low cost / economic manner to currently used oil based mud and mineral oil based mud systems are required to be researched. The plant derived oil and ester based mud are the most suitable candidate to fulfill those requirements due to its natural components, thus, pose little to no danger to aquatic or terrestrial, offshore or onshore environments[1]. Besides, the previous research done by Peresich et al, showed that ester based mud derived from palm oil had successfully tried in Norwegian offshore drilling operation. The result was technically and environmentally acceptable.

However, observing that palm oil is also used for human consumption (e.g. frying oil), hence, there will be competition between the usage as drilling mud base oil and as food stock. Observing this condition, previous studies has been conducted by Kania, et al, showed that there is possible alternative to change palm oil with other plant derived oil, such as jatropha oil[3]. This study showed that the physical properties of jatropha methyl ester has similarity with the mineral oil base, even the rheological properties is stable at high temperature environment, so it is suitable as drilling fluid base[3].

Yet, consider the abundant requirement of drilling fluid base, some alternatives besides jatropha oil are highly demanded by industry to fulfill drilling mud stock used in the drilling operation. Thus, the research on other alternative should be performed. The suitable candidate of plant derived oil used as fluid based mud is predicted to be Calophyllum oil derived from *Calophyllum inophyllum* plant. Based on previous research conducted by Aningditya, et al, Calophyllum oil has similar properties with palm oil[4]. With no competition with food consumption and also abundant development and distribution of this plant, the oil extracted from Calophyllum seed can be promising for drilling fluid base candidate.

Prior to the drilling fluids properties stability during expected actual wellbore temperature and pressure changes, the estimation of the fluid rheological properties modeling under down-hole conditions is necessary to be investigated. All types of esters, including Calophyllum methyl ester, might undergo hydrolysis and revert back to the acid and alcohol in the presence of reserve alkalinity at high temperature[5]. The high viscosity also can be problematic for drilling operations, such as formation damage. Thus, the studies on the optimum mud formulation are investigated in this study to obtain the acceptable viscosity level and stable emulsion without hydrolysis occurrence under high temperature.

In this study, *Calophyllum* crude oil, initially, is converted to methyl ester *Calophyllum* using esterification and transesterification process to reduce its kinematic viscosity. The usage of *Calophyllum* methyl ester feasibility as drilling fluid base fluid is, hence, investigated.

1.4. Objectives

1.4.1. To formulate an environmentally friendly and stable invert emulsion of Calophyllum methyl ester-based mud derived from *Calophyllum inophyllum* crude oil under high temperature high pressure condition.

1.4.2. To measure rheological properties, filtration characteristics and emulsion stability of Calophyllum ester-based mud under high temperature high pressure conditions and compare it with palm methyl ester based mud and mineral oil based mud (OBM).

1.4.3. To measure eco-toxicological properties of Calophyllum methyl ester-based mud towards fresh-water fish (Guppy fish/ *Poecilia reticulate*) by Fish Acute Toxicity Test LC_{50} according to the EPA standard.

1.5. Methodology

1.5.1. Equipments Required

- Performance test: mud balance, weight indicator, mud mixer, viscometer, filter press unit, HPHT filtration loss unit, pH meter, electrical stability probes unit and hot rolling test unit.
- Environmental Assessments: aquarium and compressor.

1.5.2. Materials Required

- Drilling Fluid Development:
  - Base Fluid: *Calophyllum inophyllum* methyl ester and Brine
  - Additives: organophilic clay, emulsifier, lime, fluid loss additives and barite.
- Environmental Assessments: *Poecilia reticulate* (Guppy fish)

1.5.3. Experimental Condition

- Mud Performance test is conducted in normal API temperature of 120^{o}F (49^{o}C) and High-Pressure-High-Temperature (HPHT) environment. For aging test, developed mud is tested in temperature of 250^{o} F for 16 hours.

1.6. Scope of Study

The preliminary study which is carried out in this project is limited to development of *Calophyllum* methyl ester based mud. The technical performance criteria, which is mud rheological properties and mud filtration properties are tested with given specific range of temperature, mud additives concentration, mud weight and oil water ratio. Then, to compare the rheological properties with palm methyl ester and mineral oil based mud that is already field tested. The emulsion stability of the drilling mud system is also tested to observe the emulsion strength after hot rolled. The environmental test impact is carried out to observe its eco-toxicological properties towards freshwater environment. Lastly, the cost analysis and advantages of Calophyllum methyl ester is studied.

1.7. Relevancy of Project

Environmentally friendly drilling mud base oil is greatly required since the strict environmental legislation set by the environment regulator (i.e. Environmental Protection Agency) becoming more and more stringent. As explained earlier, in many countries, nowadays, especially in Europe operations, the usage of diesel / oil based mud and mineral oil based mud had been banned. If the usage of oil based mud still applied, the penalties / fine will be imposed. Thus, the relevancy of calophyllum ester based drilling mud is very relevant to the drilling fluid industry and oil and gas industry operations in this century onwards.

1.8. Feasibility of Project

Calophyllum ester based mud was feasible to be made and rheologically modified in university laboratory and facilities. The rheological properties measurement tests, HPHT filtration tests, emulsion stability test, environmental test and cost analysis were also feasible to be completed on time within the schedule. The cost analysis on the feasibility of calophyllum ester based mud application to be used in the real operation for the economic wise were found to be economic.

The economic feasibility was concluded based on the abundant resource of calophyllum oil as the source of oil base fluid and the low cost production cost. In addition, the economic feasibility was supported by the fact that calophyllum methyl ester has no competition in human consumption (non-edible), unlike palm oil methyl ester which is also used as frying oil and human food stock.

CHAPTER 2
LITERATURE REVIEW AND THEORY

2.1. Drilling Fluid Overview

There are two primary types of drilling muds which also called drilling fluids in use today: water based drilling muds (WBM), and non-aqueous drilling muds (NADM)[4]. Water based mud, used in most offshore drilling operations in US waters, consist of fresh or salt water containing barite, clay or an organic polymer, and various inorganic salts and organic additives to modify the physical properties of the mud. In NADM, the continuous phase is a mineral oil or synthetic hydrocarbon, usually emulsified with brine, and containing barite, organophilic clays or polymers, and various additives.

2.1.1. Drilling Fluid Functions

The functions of these drilling fluids are consisted as below[4].

- Counteracting formation pressure. The column of drilling mud in the drill pipe and annulus provides a hydraulic head (weight) that counteracts the pressure in the formations being drilled, preventing formation fluids from flooding the well bore, causing a blowout.
- Removing cuttings from the borehole. Jets of drilling mud exiting from the drill bit carry drill cuttings away from the bit, preventing it from clogging, and convey them to the surface.
- Suspending solids. The drilling mud must have sufficient viscosity to suspend cuttings and weighting agent when drilling stops, as during addition of new lengths of drill pipe. This function is particularly important during drilling of horizontal wells.
- Cooling and lubricating the drill string and bit. Friction of the rotating drill string heats the drill pipe and, particularly the bit. Circulating drilling mud cools the drill string and lubricates it where contact is made with the formation, at the drill bit, and in curved sections of deviated or horizontal wells.
- Protecting, supporting, and stabilizing the borehole wall. Some types of formation minerals, particularly shales, are sensitive to water and swell and slough off the

side walls into the well bore during drilling, decreasing wellbore quality. Water based mud may contain additives to minimize shale swelling.

- Protecting permeable zones from damage. Special drilling mud additives build a filter cake a low-permeability layer of packed solids) on the walls of the well, preventing drilling mud loss into permeable formations, which may damage the formation and increase drilling costs.
- Supporting part of the weight of the drill string. The steel drill pipe is supported in the hole in proportion to the volume and density of the drilling mud displaced by the pipe in the annulus.

2.2. Rheological Properties and Filtration Characteristics Measurement Parameters

Rheology is defined as physics of the flow and the deformation of matter. The rheological properties of drilling fluids describe the flow characteristics of a drilling fluid and how these characteristics affect movement of the fluid[5]. The usage to measure mud rheological properties is to design or to formulate the desired drilling mud system that is required in particular drilling section. Drilling mud system can be designed according to wellbore problem or field challenges that occurred. There are several performance tests parameters in drilling fluid preparation, as below[5, 21].

2.2.1. Mud Weight or Mud Density

It is defined as the mass per unit volume of a drilling fluid, synonymous with mud density. Weight is reported in lbm/gal (also known as ppg), kg/m^3 or g/cm^3 (also called specific gravity or SG), lb/ft^3 or in hydrostatic gradient, $lb/in^2/ft$ (psi/ft) or pptf (psi/1000 ft).

2.2.2. Plastic Viscosity (PV)

It is a parameter of the Bingham plastic rheological model. PV is the slope of the shear stress/shear rate line above the yield point as shown in figure 1. PV represents the viscosity of a mud when extrapolated to infinite shear rate on the basis of the mathematics of the Bingham model. A low PV indicates that the mud is capable of drilling rapidly because of the low viscosity of mud exiting at the bit.

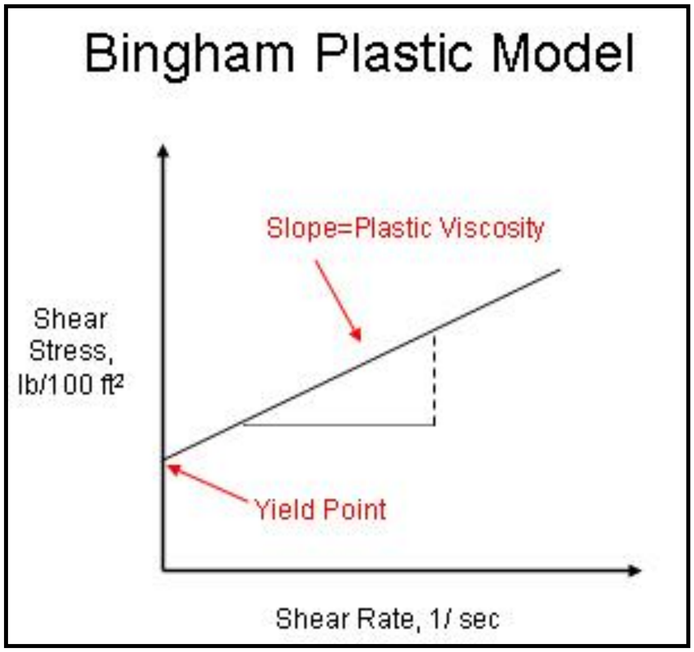

Figure 1 Bingham Plastic Model to Describe PV and YP[5]

PV (in cP) is measured by taking the difference between the dial readings taken at the two highest speeds of 600 rpm and 300 rpm. For instance, PV can be expressed as below.

PV = θ600 - θ300

If temperature is a factor, then the mud sample should be tested at 120° F, with the mud in a heating cup.

Plastic Viscosity (PV) is also known as resistance of fluid to flow. Any increase in solid content in drilling mud as Barite, drill solid, lost circulation material, etc will result in higher the plastic viscosity. In order to lower the PV, the solid content must be reduced that can be achieved by using solid control equipment and/or diluting drilling mud with base fluid. With increasing temperate while drilling deeper, the plastic viscosity of the drilling mud will decrease because the viscosity of the base fluid decreases.

Normally, the higher mud weight, the higher PV will be. However, if there is an increasing trend of PV without mud weight change, it means that there is an increase in ultra-fine drill solid content in the mud system. In oil base mud, the emulsified water in oil base drilling fluid will act like a solid, and it will increase the plastic viscosity drastically. Several impacts of PV on drilling operation are as follows.

- Equivalent Circulating Density (ECD) – The higher PV, the higher ECD will be.
- Surge and Swab Pressure – The PV has the same effect as ECD. If the PV increases, surge and swab pressure will increase.
- Differential Sticking – A chance for differential sticking will increase, especially in water base mud, when the plastic viscosity increases because of increases in solid content.
- Rate of Penetration (ROP) – The ROP will be directly affected by the plastic viscosity. Drilling with the high PV drilling mud will result in slower rate of drilling, thus lower PV result in faster rate of drilling.

2.2.3. Yield Point (YP)

It is a parameter of the Bingham plastic rheological model. Yield Point is the yield stress extrapolated to a shear rate of zero. As shown in figure 1, a Bingham plastic fluid plot as a straight line on a shear rate (x-axis) versus shear stress (y-axis) plot, in which YP is the zero-shear-rate intercept. (PV is the slope of the line). Yield Point is calculated from 300- and 600-rpm viscometer dial readings by subtracting PV from the 300-rpm dial reading, as follow.

$$\mathbf{YP = \theta 300 - PV}$$

Yield Point is used to evaluate the ability of a mud to lift cuttings out of the annulus. A high YP implies a non-Newtonian fluid, one that carries cuttings better than a fluid of similar density but lower YP. YP is lowered by adding deflocculant to a clay-based mud and increased by adding freshly dispersed clay or a flocculant, such as lime. YP is measured in unit of $lbf/100ft^2$.

When plastic viscosity rises, this is usually an indication that the solids control equipment are running inefficiently. Ideally, the yield point should be just high enough to suspend the cuttings as they are circulated up the annulus.

Yield Point is characterized also as resistance of initial flow of fluid or the stress required in order to move the fluid. The YP indicates the ability of the drilling mud to carry cuttings to surface. Moreover, frictional pressure loss is directly related to the

YP. Higher YP will result in higher pressure loss while drilling mud is being circulated. In oil-based mud, the causes of increasing in YP are listed below.

- Drill solid – the higher content of drill solid, the higher YP will be.
- Treatment CO2 in the mud with lime (CaO) – The lime (CaO) will chemically react with CO2 to form Calcium Carbonate (CaCO3) which will increase the YP.
- Low temperature – in the oil base system, the low temperate will increase the viscosity and the YP.

Operational impacts of the YP are as follows.

- Equivalent Circulating Density (ECD) – The ECD typically increases when the YP increases.
- Hole Cleaning – in drilling a large diameter hole, the YP in the drilling mud must be high in order to help hole cleaning efficiency.

2.2.4. Gel Strength (GS)

Gel Strength (GS) is defined as the shear stress measured at low shear rate after a mud has been set in static condition for a period of time (10 seconds and 10 minutes in the standard API procedure, although measurements after 30 minutes or 16 hours may also be made). The unit is $lbf/100ft^2$.

There are two readings for gel strengths, 10 second and 10 minute with the speed of the viscometer set at 3 rpm. The fluid must have remained static prior to each test, and the highest peak reading will be reported. The gel strength quantifies the thixotropic behaviour of a fluid; its ability to have strength when static, in order to suspend cuttings, and flow when put under enough force. Ideally the two values of gel strength should be close rather than progressively far apart. It demonstrates the ability of the drilling mud to suspend drill solid and weighting material when circulation is ceased.

The 3-rpm dial reading will be recorded after stirring the drilling fluid at 600 rpm from a viscometer. Normally, the first reading is noted after the mud is in a static condition for 10 second. The second reading and the third reading will be 10 minutes static conditions.

The reason of the 10 minute-reading will indicate whether the mud will greatly form the gel during an extensive static period or not. If the mud has the high gel strength, it will create high pump pressure in order to break circulation after the mud is static for long time. Furthermore, increasing in a trend of 10-minute gel strength indicates a build up of ultra fine solid. Therefore, the mud must be treated by adding chemicals or diluting with fresh base fluid.

The following causes will result in the high gel strength in the water base mud.

- Bacteria
- Drill solid
- Salt
- Chemical contamination as lime, gypsum, cement, and anhydrite
- Acid gases as Carbon Dioxide (CO2), and Hydrogen Sulphide (H2S)

For an oil base drilling fluid, there are several points that will cause the high gel strength in the mud system as follows.

- Over treatment with organic gelling material
- Build up of fine solid particles in the mud

The impacts of the gel strength are as follows;

- Cutting suspension ability – Low get strength drilling mud will not be able to efficiently suspend cuttings; therefore, the cutting will quickly drop once pumps are shut down. This can lead to several problems such as stuck pipe, hole pack off, and accumulation of cutting beds.
- Barite sag – The barite sag issue mostly occurred because of low gel strength drilling fluid. Hence, the mud weight in the hole will not be constant. The lower mud weight will be observed at the shallow depth but the heavier mud weight will be noticed at the deeper section of the well. This situation could possibly lead to a

well control incident because of insufficient mud weight to balance formation pressure at the shallow section of the wellbore.

- Break circulation pressure – High progressive gel strength fluid will lead to high pump pressure required to break circulation. Once high pumping pressure is applied, it could lead to break formation and results in lost circulation issue.

2.2.5. Fluid Loss and Filter Cake

Fluid loss is defined as the amount of water expelled from the drilling mud under particular pressure and temperature during some time (30 minutes). Fluid loss is measured in ml/30 minutes at 100 psi (for API test) or 500 psi and Bottom Hole Temperature (BHT) (200^o F) for high temperature/ high pressure (HTHP). Filter cake is measured in 1/32".

The fluid loss gives a representation of the fluids interaction with the well bore under simulated pressure and temperature conditions. Ideally the fluid should form a thin, flexible, impermeable layer (filter cake) against the wall and prevent fluid (filtrate) from entering the rock and reacting with the formations. A mud system with a low value of filtrate loss cause minimum swelling of clays and minimum formation damage. The filter cake should be in the region of 1 to 2 /32" and should never be greater than 3/32", even in an HPHT test with WBM[5].

2.2.6. pH Test

The term pH is used to express the concentration of hydrogen of hydrogen ions in an aqueous solution. pH is defined by **$pH = \log [H^+]$**, Where $[H^+]$ is the hydrogen ion concentration in moles per liter. At room temperature, the *ion product constant of water, Kw*, has a value of 1.0×10^{-14} mol/L. thus, for water[11]:

$[H^+]$	pH	[OH]	pOH
1.0×10^{0}	0.00	1.0×10^{-14}	14.00
1.0×10^{-1}	1.00	1.0×10^{-13}	13.00
1.0×10^{-2}	2.00	1.0×10^{-12}	12.00
1.0×10^{-3}	3.00	1.0×10^{-11}	11.00
1.0×10^{-4}	4.00	1.0×10^{-10}	10.00

1.0×10^{-5}	5.00	1.0×10^{-9}	9.00
1.0×10^{-6}	6.00	1.0×10^{-8}	8.00
1.0×10^{-7}	7.00	1.0×10^{-7}	7.00
1.0×10^{-8}	8.00	1.0×10^{-6}	6.00
1.0×10^{-9}	9.00	1.0×10^{-5}	5.00
1.0×10^{-10}	10.00	1.0×10^{-4}	4.00
1.0×10^{-11}	11.00	1.0×10^{-3}	3.00
1.0×10^{-12}	12.00	1.0×10^{-2}	2.00
1.0×10^{-13}	13.00	1.0×10^{-1}	1.00
1.0×10^{-14}	14.00	1.0×10^{0}	0.00

Table 1 Relations between pH, $[H^+]$ and $[OH^-]$ in water solutions[11]

$$\mathbf{H_2O = H^+ + OH^-}$$

$$\mathbf{Kw = [H^+][OH^-] = 1.0 \times 10^{-14}}$$

For pure water, $[H^+] = [OH^-] = 1x10^{-7}$, and the pH is equal to 7, since in any aqueous solution the product $[H^+][OH^-]$ must remain constant, an increase in $[H^+] > [OH^-]$ is said to be acidic, and a solution in which $[OH^-] > [H^+]$ is said to be alkaline. The relation between pH, $[H^+]$, and $[OH^-]$ is summarized in Table 1.

The pH of a fluid can be determined using either a special pH paper or a pH meter. The pH paper is impregnated with dyes that exhibit different colors when exposed to solutions of varying pH. The pH is determined by placing a short strip of the paper on the surface of the sample. After the color of the test paper stabilizes, the color of the upper side of the paper, which has not contacted the mud, is compared with a standard color chart provided with the test paper. When saltwater muds are used, caution should be exercised when using pH paper. The solutions present may cause the paper to produce erroneous values[11].

The pH meter is an instrument that determines the pH of an aqueous solution by measuring the electro potential generated between a special glass electrode and a reference electrode. The electromotive force (EMF) generated across the specially formulated glass membrane has been found empirically to be almost linear with the pH of the solution. The pH meter must be calibrated using buffered solutions off known pH[15].

2.2.7. Electrical Stability (ES) Test

It is a test for oil-base and synthetic-base muds that indicates the emulsion and oil-wetting qualities (stability) of the sample. The electric stability is read as an voltage reading from 0 to 2000 volts, which indicates the easiness of electric current to pass the mixture of liquid which results in the higher value will indicate that the emulsion is stable or more compacted, on the contrary if the voltage level is low, it indicates the emulsion stability is weak or not stable. The API standard for good emulsion of synthetic based mud (SBM) or oil based mud (OBM) is above 400 volts of ES value. Below this value, it is said that the emulsion is not good and the mud is not recommended to be used in drilling circulation system in drilling operations.

2.3. Synthetic Base Fluids

2.3.1. Synthetic Hydrocarbons

- **Linear Alpha Olefins (LAO)**

Linear Alpha Olefins (LAO) or Normal Alpha Olefins (NAO) are olefins or alkenes with a chemical formula C_xH_{2x}, distinguished from other mono-olefins with a similar molecular formula by linearity of the hydrocarbon chain and the position of the double bond at the primary or alpha position.linear alpha olefins are commonly manufactured by two main routes: oligomerization of ethylene and by Fischer-Tropsch synthesis followed by purification There are seven commercial processes which oligomerize ethylene to linear alpha olefins. Five of these processes produce wide distributions of linear alpha olefins[7].

- **Polyalphaolefins (PAO)**

The PAO-based material which has been used for drilling is manufactured by the catalytic polymerization of linear alpha-olefins such as 1-octene or 1-decene. Control over chemical structure by the adjustment of the reaction parameters in the polymerization process and selection of starting alpha-olefins[6].

- **Internal Olefins (IO)**

Linear internal olefins were also manufactured by chlorination/dehydrochlorination of linear paraffins. Internal olefins are used as synthetic drilling fluid base for high value, primarily off-shore synthetic drilling fluids. The preferred materials for the synthetic drilling fluid application are linear internal olefins, which are primarily made by isomerizing linear alpha-olefins to an internal position. The higher internal olefins appear to form a more lubricious layer at the metal surface and are recognized as better lubricants[7].

2.3.2. Esters

Esters are synthesized from fatty acids and alcohols. Fatty acid is derived from vegetable oils. Proper selection on the hydrocarbon chain length will minimize viscosity. Ester has general structure as adjacent figure. To form an ester, an alcohol is reacted with fatty acid, under acidic condition using acid catalyst[6].

2.3.3. Ethers

Ethers are a class of organic compounds that contain an ether group — an oxygen atom connected to two alkyl or aryl groups — of general formula R–O–R'. Ethers can be prepared in the laboratory in several different ways. Ethers are also categorized as a broad range of materials which are usually synthesized from alcohols[1].

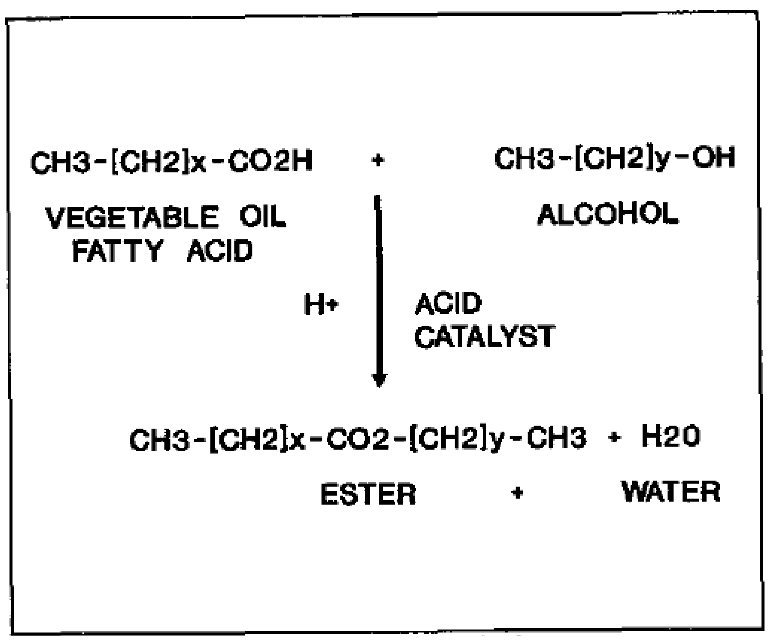

Figure 2 Schematic process of the ester synthesis[1]

2.4. Previous Study on Ester-Based Drilling Fluid

Based on the research conducted by Peresich, et al., a number of complex esters with suitable properties to formulate a stable invert emulsion drilling mud were identified. In the laboratory experiments conducted by European Community (EC) and SFT protocols, these esters were found to be of very low toxicity and readily biodegradable aerobically and anaerobically. In terms of technical competencies, a field trial was conducted in Norwegian offshore field; the performance was superior to WBM and comparable to OBM used in close offset wells. It was reported that this ester-based drilling mud derived from palm oil showed significant cost savings and technical advantages are possible if the operators are able to achieve OBM drilling performance. The additional good point from Ester-Based Mud (EBM) used is that is without environmental penalties associated with the use of OBM formulated with mineral oil[8].

In addition, the study by Yassin, et al., indicate that palm oil ester have the acceptable base oil properties to be used in drilling fluids. Palm ester base fluid has potential as an acceptable substitute for diesel oil in the formulation of oil mud and is compatible with market oil mud additives at that time[9]. The emulsion stability, rheological control and filtration control properties are easily maintained in the palm diesel oil mud systems. All of these research indicates that the usage of drilling mud using ester-based fluids which derived from plant are potentially technically succeed and more environmentally friendly.

The usage of other ester type that is already researched is Jatropha fatty acid methyl ester (FAME) base drilling mud that is conducted by Kania, et al. The study indicates that the usage of Jatropha methyl ester as ester-based drilling fluid is acceptable in terms of performance and physical properties. Comparison study of Jatropha FAME-based mud with synthetic and palm FAME based mud showed that Jatropha FAME mud posses superior drilling fluid characteristics that had stable PV and desirable YP and gel strength properties[3].

2.5. Emulsion Stability Issue on Ester-Based Drilling Fluids

According to Patel, et al., esters undergo hydrolysis at high pH and temperature conditions. While undergoing hydrolysis, the fatty acid esters used in conventional drilling fluid applications produce fatty acids and alcohols, like initial products before undergo esterification and transesterification process. Under drilling and in the presence of lime, they produce the calcium salt of the fatty acid. These calcium soaps of fatty acids will affect the rheological properties of drilling muds severely. The rate of hydrolysis will depend on pH and temperature conditions. However, the amount of lime used as reserve alkalinity in the mud system is sufficient enough to cause the hydrolysis of ester-based drilling fluid.

Based on the experiment conducted by Patel, et al., the presence of lime concentration of 3 to 4 lb/bbl in the mud system will cause hydrolysis in the ester based mud at the temperature exceeding 200^{o}F (93^{o}C)[10]. The hydrolysis was proofed by the presence of alcohol byproducts in the mud system after hot rolling test. Thus, in order to have more stable emulsion invert system, the usage of 3 to 4 lb/bbl of lime is likely to be avoided. Instead, using lower or higher lime concentration will be beneficial to the emulsion stability.

2.6. Botanical Description of *Calophyllum inophyllum*

Calophyllum inophyllum is included in *Calophyllum* family which has wide distribution location around the world. This plant has different names in each place, like bintangor in Malaysia, hitaullo in Maluku island (Indonesia), nyamplung in Java island (Indonesia), bintangur in Sumatera island (Indonesia), poon in India and in United Kingdom, it is known as Alexandria laurel, tamanu, pannay tree, also sweet scented calophyllum[11]. *Calophyllum inophyllum* taxonomy according to Heyne (1987) is as Table 2 below[12].

Classification	Taxonomy Name
Division	Spermatophyta
Sub Division	Angiospermae
Class	Dicotyledone
Order	Guttiferales
Family	Guttiferae
Genus	Calophyllum

Species	*C. inophyllum*
Binomial Name	*Calophyllum inophyllum* L.
General Name in Malaysia	Bintangor

Table 2 Taxonomy Classification of *Calophyllum inophyllum L*[12]

Calophyllum inophyllum tree is medium-sized plant with the height of 8-20 meter and sometimes 30-35 meter[11]. Height of non-branched stem is up to 21 meter with diameter up to 0.8 meter. The stem is grayish and white in color with lateral branching style. The root is root-riding, round-shaped and brownish (Martawijaya et al, 2005). The leave is oval-shaped with pointed end, thick and dark flashy greenish in color without any visible fur. The flower is blooming at the corner end of axilla, without any branch. The fruit is bullet-shaped with pointed end; with the length of 25-50 mm. external fruit skin is greenish when it still with the tree and changed to yellowish and brownish when it is cooked. The fruit flesh is thin and as time goes by; the flesh will be wrinkled, friable and finally be peeled off; inside the flesh, there is yellowish seed[12].

Figure 3 Tree, stem, flower, leaves, fruit and seed of *Calophyllum inophyllum*[13]

Calophyllum inophyllum seed is thick, hard and round-shaped; relatively large sized with diameter of 2.5 – 4 cm. The seed flesh is thin and the dried seed can be kept for 1 month. The seed core contains yellowish and brownish oil. *Calophyllum inophyllum* seed has oil content from 71.4% up to 75%. According to Heyne (1987), seed core contains 3.3% water and 71.4% oil. The fresh seed contains 55% oil while dried seed contain more oil up to 70.5%. Morphology of *Calophyllum inophyllum* is shown in figure 3.

2.7. *Calophyllum inophyllum* Distribution

Generally, *Calophyllum inophyllum* live in coastal area, beach area and forest area in lowland. However, this plant can be well lived in medium-height land. This plant has high tolerance towards various kinds of soil, sand, mud and even degraded soil. According to Martawijaya et al (1981), *Calophyllum inophyllum* live in tropical forest with 1000-5000 mm rainfall, at the swampy soil near shore up to, dry soil with elevated hill with the height of 800 m above sea level. Environmental growth condition of *Calophyllum inophyllum* is shown in Table 3 as below[12].

No	Parameter	Suitable Environmental Condition
1	Climate	Medium temperature up to wet and not suitable in very cold temperature
	Height	0-800 m above sea level
	Rainfall	1000-5000 mm
	Dry season period	5 months
	Maximum temperature	37^0 C
	Minimum temperature	12^0 C
	Average temperature	33^0 C
2	Soil Description	Well grown in sandy soil with sufficient rainfall, highly tolerance in clay, rocky soil, shallow soil and saline soil.
	Soil texture	Tolerance in sandy soil, sandy loams dan sandy clay loams.
	Soil drainage	Tolerance in bad soil drainage.
	Acidity	pH 4,0-7,4

3	Tolerance in extreme condition	Tolerance in saline water, heavy wind and dryness
	Dry Season	Tolerance in dry season for 5 months
	Sun light	Suitable to live in full sun light exposure and can live well in shady area as well.
	Freezing condition	Not tolerance with freezing condition
	Waterlogging	Tolerance in waterlogging/swampy area

Table 3 *Calophyllum inophyllum* Environmental Growth Condition[11]

Calophyllum inophyllum have wide distribution area in Indonesia, started from Sumatera island (West Sumatera, Riau, Jambi, South Sumatera, Lampung), Java island (along south coastal area), Kalimantan (West Kalimantan and Central Kalimantan), Sulawesi, Maluku, East Nusa Tenggara up to Papua island. It is also widely distributed from east Africa, southern coastal India to Indonesia and Australia. According to Indonesia Ministry of Forest (2008), image interpretation by Landsat7 Enhanced Thematic Mapper Plus (ETM+) of Indonesia Satellite in 2003, showed that *Calophyllum inophyllum* tree was observed in area of 480,000 hectares (60% in forest area)[12].

Calophyllum inophyllum live in swampy soil near the shore, dry soil and alluvial soil at hills with elevation of 100-150 m above sea level[12]. *Calophyllum inophyllum* can live at flatted topography (lowland) range up to hilly area with rainfall range up to 2959 mm (Martawijaya et al. 2005; Rostiwati, 2007). The map of *Calophyllum inophyllum* distribution in Indonesia is shown at Figure 4 below.

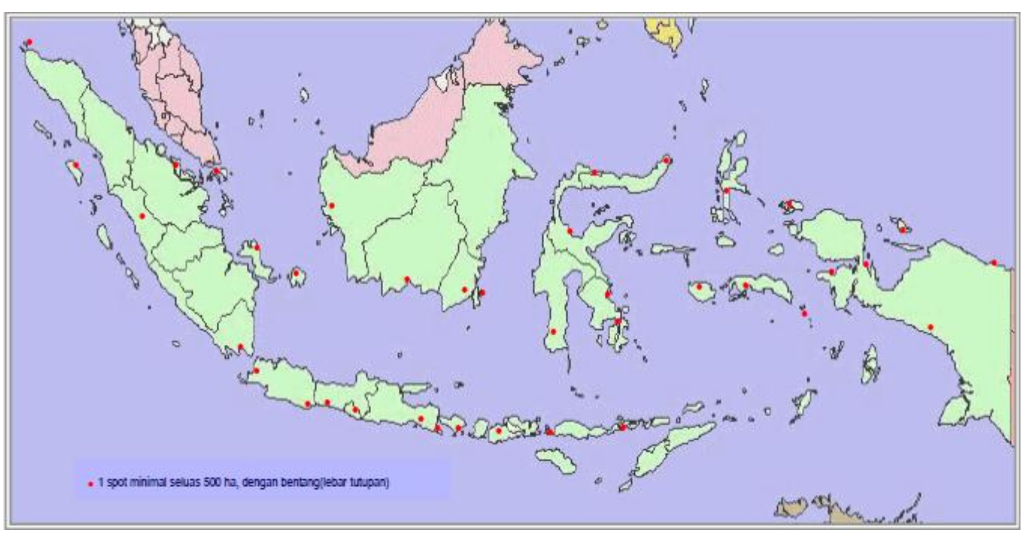

Figure 4 *Calophyllum inophyllum* Distribution in Indonesia[13]

Calophyllum inophyllum seed annual production can be up to 20 ton/hectare. *Calophyllum inophyllum* seed contains 55% oil at fresh core seed and 70.5% oil at dried core seed (Heyne, 1987), and 75% oil according to Dweek and Meadows (2002). According to Soerawidjaja (2001), *Calophyllum inophyllum* core seed contains 40-73% oil. According to Friday and Okano (2006), one (1) tree of *Calophyllum inophyllum* can produce 100 kg fruit/year and *Calophyllum inophyllum* oil for 5 kg. If with 3 x 3.5 m^2 planting distance, every tree produces 30 kg seed or 5.1 kg oil, thus in one (1) hectare of *Calophyllum inophyllum* crop, 26,973 kg of seeds or 4,585 kg of *Calophyllum inophyllum* oil can be produced[12].

According to Mahfudz (2008), if 3 years old *Calophyllum inophyllum* plant is able to produce fruits and if in 1 branch of *Calophyllum inophyllum* produce 1 kg of fruit with assumed 100 branches per tree, then in one (1) *Calophyllum inophyllum* tree can produce 100 kg *Calophyllum inophyllum* fruit or 100 tons of *Calophyllum inophyllum* fruit in 1 hectare area of crop with 3 x 3 m^2 crop distance. If methyl ester/biodiesel production required to use 2% of *Calophyllum inophyllum* oil, then in 1 hectare of *Calophyllum inophyllum* crop, 2,200 liters of *Calophyllum inophyllum* methyl ester will be produced or equal to 4,400 liters of kerosene[12].

2.8. *Calophyllum inophyllum* Crude Oil Description

Calophyllum inophyllum crude oil is brown greenish in color, viscous, sharp smell and poisonous. *Calophyllum inophyllum* crude oil has high saturated fatty acid content such as oleat acid and other components like fatty alcohol, sterol, xanthone, cuomarin derivatives, calophyllic, calophyllic, isophthalate, kapelierat, pseudobrasilat acid and triterpenoat with the content of 0,5-2% which can be used as medicine[12]. According to Debaut et al, (2005), fatty acid structures of *Calophyllum inophyllum* crude oil are shown in table 4 as below[12].

No	Analysis	Unit	Result
1	Water	%	0,25
2	Density	G/ml	0,944
3	Viscosity	Cp	21,97
4	Acid number	mg KOH/g	59,94

5	Free Fatty Acid	%	29,53
6	Sapofinication Number	mg KOH/g	198,1
7	Iodine number	Mg/g	86,42

Table 4 *Calophyllum inophyllum* Crude Oil Characteristic[12]

Calophyllum inophyllum crude oil which is produced from pressing process generally has high free fatty acid content up to 30%, thus in order to be made as biodiesel/methyl ester, it has to be given specific treatment such as degumming, esterification and transesterification process. Fatty acid composition of *Calophyllum inophyllum* crude oil compared with other vegetable oil is shown in Table 5 as below[12].

Component	*Calophyllum inophyllum* Oil	*Jatropha curcas* Oil	CPO	Soya Bean Oil
Miristat acid	0,09	-	0,7	0,1
palmitat acid	15,89	11,9	39,2	10,2
stearat acid	12,30	5,2	4,6	3,8
oleat acid	48,49	29,9	41,4	22,8
linoleat acid	20,70	46,1	10,5	51,0
Lonolenat acid	0,27	4,7	0,3	6,8
arachidat acid	0,94	-		0,28
erukat acid	0,72	-		0,2

Table 5 Fatty acid composition of *Calophyllum inophyllum* crude oil compared with other vegetable oils[12]

2.9. Production and Application of *Calophyllum inophyllum* Methyl Ester

In order to process fresh/crude *Calophyllum* oil become *Calophyllum* methyl ester, there are chemical process steps to do. The procedural steps are as follow[13].

1. Separation of *Calophyllum inophyllum* seed flesh with its shell.
2. Steam the seed without its shell for 2 hours.
3. Pressing the seed to extract the *calophyllum oil*.
4. Degumming is carried out to let phosphate acid settled in 1% concentration.
5. Esterification process of degummed *Calophyllum* oil using acid catalyst HCL 1% for 1 hour.
6. Transesterification process reaction as Figure 4, using methanol with NaOH catalyst 1% for 1 hour, as result *Calophyllum* methyl ester.

7. Wash the processed methyl ester produced from previous step with distilled water until Free Fatty Acid (FFA) content is below 0.5%.
8. Ensure produced *Calophyllum* methyl ester has been fulfilled ASTM standards.

The reaction of transesterification and esterification is shown in figure 5. The fresh *Calophyllum* oil and methyl ester *Calophyllum* which is the last products of esterification-transesterification process is shown in figure 6.

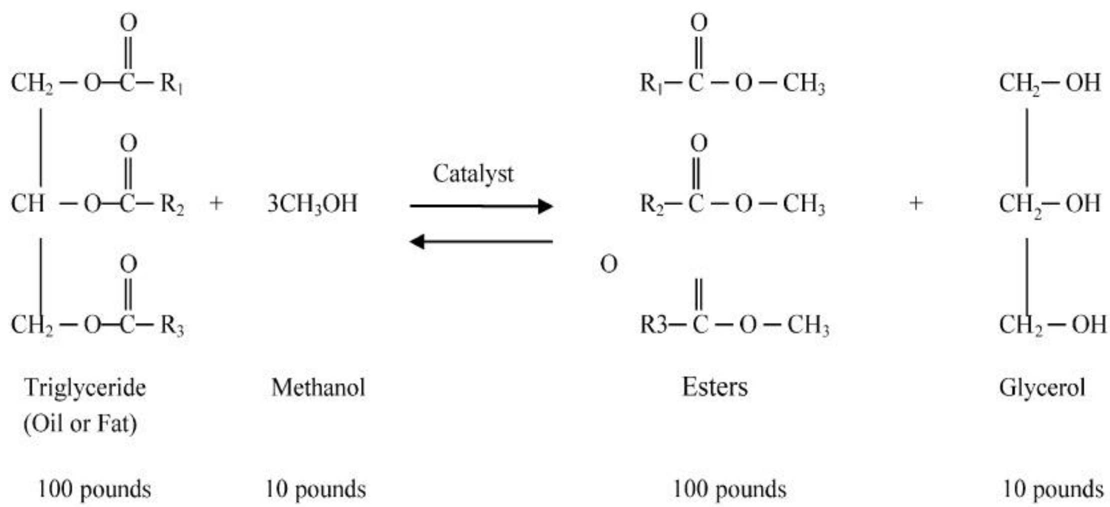

Figure 5 Equation of transesterification reaction[14].

Figure 6 *Calophyllum* fresh oil (left) and *Calophyllum* methyl ester (right) [13]

The procedures to produce methyl ester or biodiesel as explained above are summarized in figure 7. The collection of calophyllum oil, esterification process and refining the oils to the final methyl ester product are well summarized in figure 7 below.

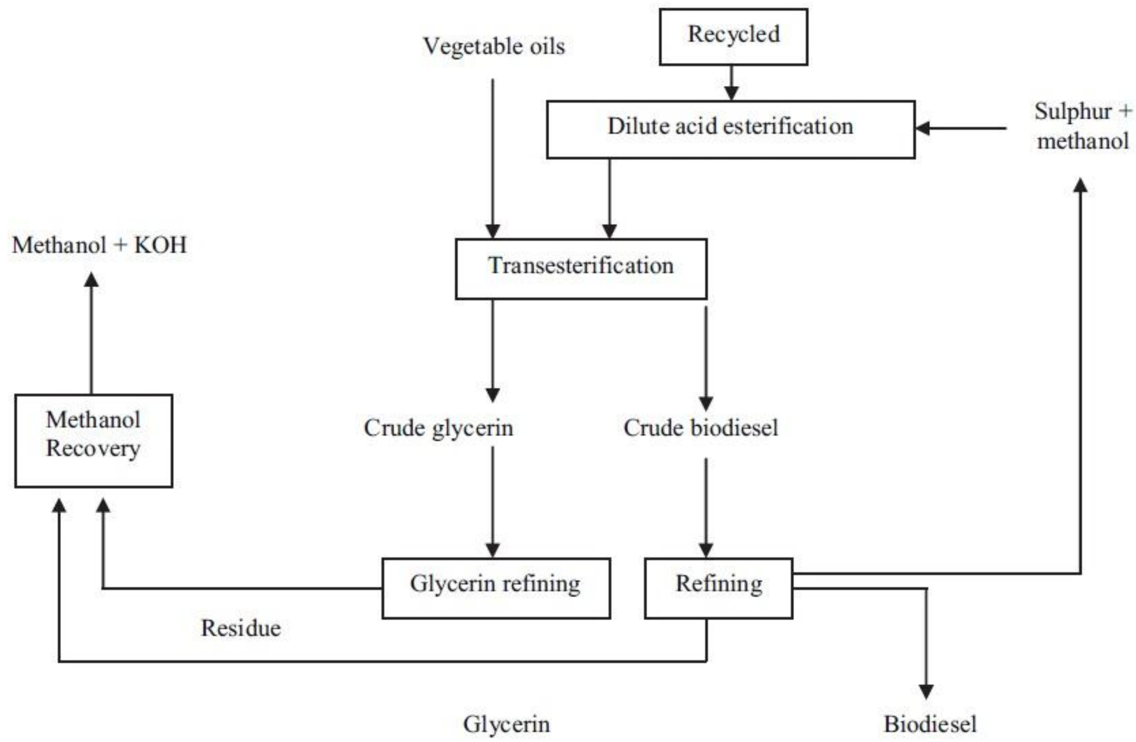

Figure 7 Basic schemes for biodiesel production[14]

In order to develop the mass production of calophyllum methyl ester, the first step is to locate the possible *Calophyllum inophyllum* plants locations or else create the plantation of *Calophyllum inophyllum.* After that is to cultivate the fruits and later the seeds of Calophyllum. The cultivated and collected seeds are peeled, screened and dried. The calophyllum seeds are containing approximately 71.4 – 75% of oil content, which are good if the extracted oil could be maximized.

After screened and dried, the dried seeds are pressed in the pressing unit machine to extract the calophyllum crude oil. The color of crude oil is relatively black to dark brownish. The extracted calophyllum crude oil then is degummed to remove any impurities which not associated with the calophyllum oil substances. After that calophyllum crude oil will undergo esterification and transesterification process to reduce its high viscosity by mixing it with alcohol and catalyst. After washing and purification process, methyl ester of calophyllum inophyllum oil could be produced with following the standard of ASTM and API of base oil used for synthetic based mud application, as shown in table 6. These processes are shown in figure 8 as below.

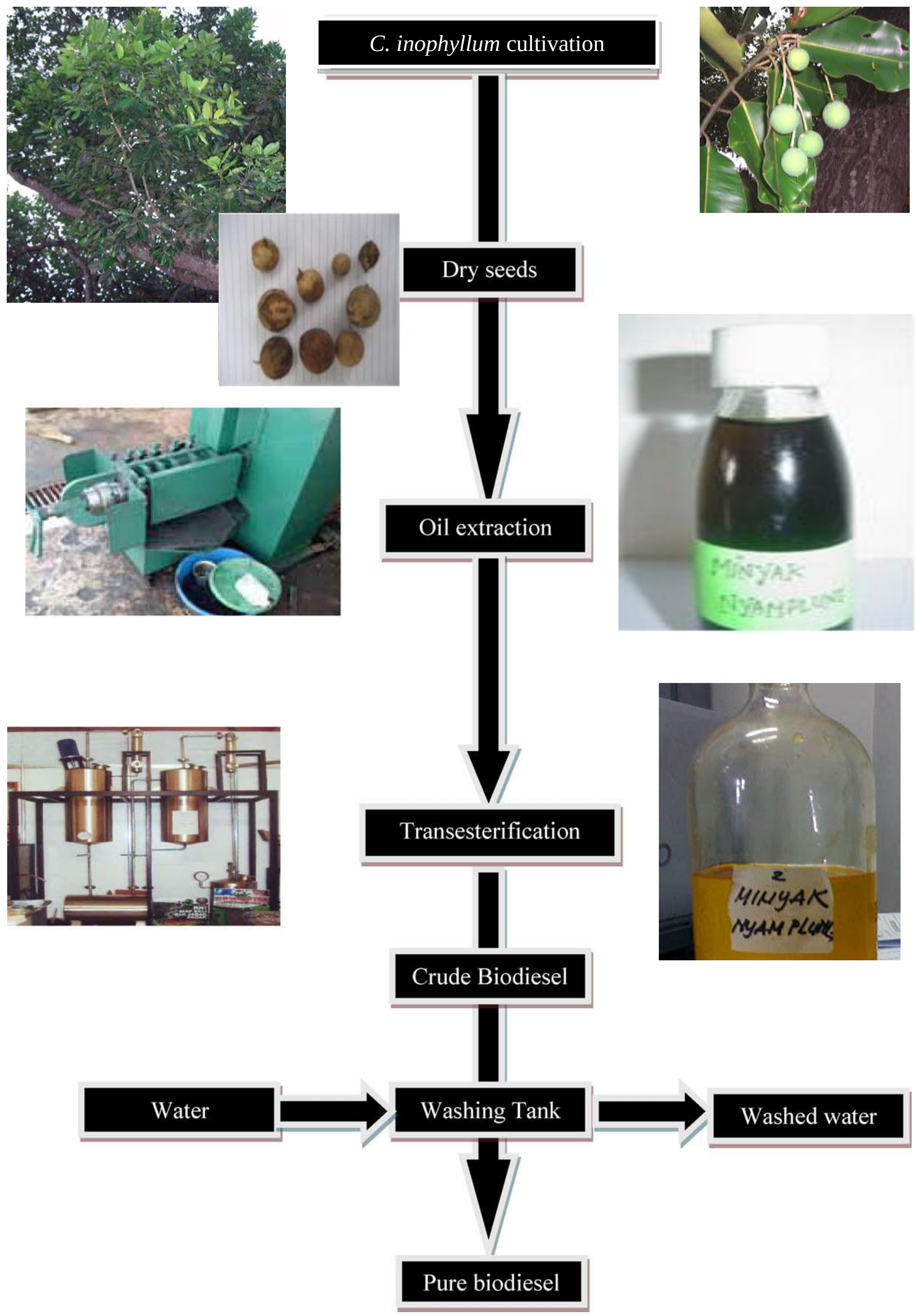

Figure 8 *Calophyllum inophyllum* biodiesel (methyl ester) production chain[14]

2.10. Chemical and Physical Characteristics of *Calophyllum inophyllum* Methyl Ester

Physical-chemical characteristic of methyl ester derived from *Calophyllum* inophyllum is shown in table 6 as below[12].

No	Parameter	Unit	Method	Value	*Calophyllum* Methyl Ester
1	Density at 40^0C	Kg/m^3	ASTM D 1298	850-890	888,6
2	Kinematic Viscosity at 40^0C	mm^2/s	ASTM D445	2,3-6,0	7,724
3	Cetane Number	-	ASTM D 613	Min 51	51,9
4	Flash Point	^{0}C	ASTM D 93	Min 100	151
5	Cloud Point	^{0}C	ASTM D 2500	Max 18	38
6	Copper strip corrosion (3 h at 50^0C)	-	ASTM D 130	Max 3	1 b
7	Carbon Residue	% mass	ASTM D 4530	Max 0,05 Max 0,3	- 0,434
8	Water and sediment	% volume	ASTM D 1796	Max 0,05	0
9	Distillation Temperature 90 %	^{0} C	ASTM D 1160	Max 360	340
10	Sulfated Ash	% mass	ASTM D 874	Max 0,02	0,026
11	Sulphur	ppm-m	ASTM D 1266	Max 100	16
12	Phosporus	ppm-m	ASTM D 1091	Max 10	0,223
13	Acid Number	mg-KOH/ gram	AOCS Cd 3d-63	Max 0,8	0,96
14	Total glycerol	% mass	AOCS Ca 14-56	Max 0,24	0,232
15	Alkil ester content	% mass	SNI 04-7182-2006	Min 96,5	96,99
16	Iodine number	% mass	AOCS Cd 1-25	Max 115	85

Table 6 Physical-chemical characteristic of *Calophyllum inophyllum* methyl ester with ASTM D 6751-3[12]

2.11. Use of Synthetic Based Mud (SBM) to Solve Drilling Problems

Synthetic based mud (SBM) systems are formulated as emulsion in which the synthetic liquid forms the continuous phase while brine serves as the dispersed phase. The solids in the mud system and the shale formations are primarily exposed to the synthetic liquid and not to the aqueous phase. It is this basic characteristic that allows synthetic based drilling fluids to successfully drill difficult wells. Because the cuttings do not readily disperse into the mud, the system does not need large dilution volumes to control solids. The need to dispose of whole mud is eliminated and only the mud that is retained on the cuttings is discharged. Additionally, the fluids will drill near gauge holes thus reducing the volume of cuttings generated. To date, there have been three synthetic based mud systems used in the field. All three synthetic based muds can be formulated with a variety of mud weight and flow properties[7].

2.12. Synthetic Based Mud (SBM) System Stability

Synthetic Based Mud (SBM), also known as pseudo-oil-based muds or inert drilling fluids, have promise as environmentally acceptable alternatives to conventional oil based mud (OBM), because their base fluids (non petroleum organic compound) are thought to be readily biodegradable. Laboratory and simulated seabed studies indicate that, in most cases, SBM do indeed degrade in seawater faster than OBM. At the same time, SBM generally appear to perform as well as OBM in the field. However, the unique chemistry of synthetic fluids imparts special properties to SBM that can lead to some operational differences.

Two key properties that can affect the upper limits of temperature, mud density and O/W ratio are viscosity and emulsion stability. SBM have higher viscosity than OBM at low to moderate temperature, but observations in the field indicate they also thin more rapidly with increasing temperature. Emulsion stability of SBM versus OBM is understood even less, especially at elevated temperatures. Since SBM, like OBM, are generally formulated as invert emulsion (water-in-oil) muds, it may be more difficult to achieve high stability of SBM at high temperature if the enhanced degradability of SBM is somehow associated with their increased dispersibility in seawater[17].

The questions are raised whether it is possible to formulate SBM that have acceptable emulsion stability and can adequately suspend cuttings and weighting material at elevated temperatures, yet are pumpable and degradable in seawater at low temperatures[17]. This can be managed by appropriate components and quantity of each component in drilling fluid system. In addition, there was previous study conducted by Patel, et al. that showed that the addition of lime can destabilize the mud system due to hydrolysis of ester. The study resulted that the presence of 3 to 4 lb/bbl of lime in ester based mud, the ester hydrolyzed severely during aging condition, which was confirmed with the presence of alcohol by products in the mud after hot rolling test was performed.

For *Calophyllum inophyllum* methyl ester based drilling mud system, External phase or dominant part is *Calophyllum inophyllum* methyl ester and the internal phase is brine. The drilling fluid component is as shown in Table 7.

Component	Description
External phase	*Calophyllum* methyl ester
Viscosifier	Organophilic clay
Emulsifier	Polyamide (Primary & Secondary)
Internal Phase	25% $CaCl_2$
Fluid Loss Additives	Gilsonite
Weighting Agent	Barite
Corrosion Control Agent	Lime

Table 7 Drilling Fluid Component

2.13. Vegetable Oil Based Mud Advantages over Conventional OBM and WBM

Vegetable oil based mud are more biodegradable and less toxic than most oil based mud. Thus, discharging the drilled cuttings to the sea is permitted with some allowable discharge standards. Hence, minimizing transportation and processing cost of drilled cuttings to upland disposal. Comparing with water based mud, many significant technical advantages of vegetable oil based mud utilization over water based mud such as wellbore stability, thermal stability, better lubrication which leads to faster rate of penetration, less Non Productive Time, less risk of pipe stuck due to wellbore problems that usually occurred in water based mud drilling, less overall cost due to less NPT and less fishing operations.

CHAPTER 3

METHODOLOGY

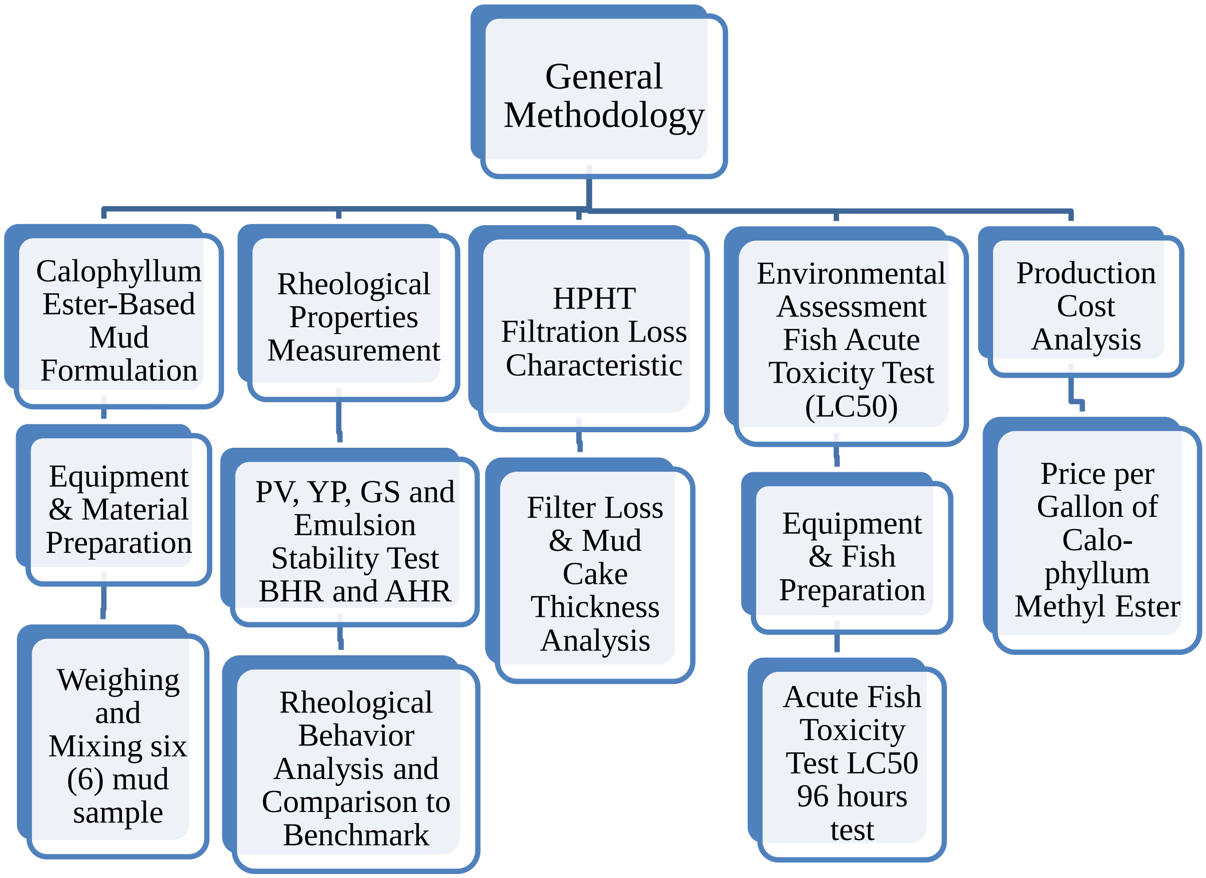

Figure 9 Process Methodology

3.1. Formulations of Drilling Mud Using *Calophyllum* Methyl Ester

In this study, drilling mud components are made from the following ingredients: Calophyllum methyl ester that was purchased from ‘Koperasi Tani Jarak Lestari’, Cilacap, Indonesia; primary emulsifier, secondary emulsifier, organophilic clay, fluid loss control, lime, calcium chloride and water for brine; and barite.

Calophyllum methyl ester act as base fluid for the EBM, primary emulsifier improves emulsion and thermal stability under high temperature and contamination, secondary emulsifier act as additional emulsion stabilizer and as second wetting agent. Organophilic clay act as viscosifier and to improve filter cake quality for reduced fluid loss, it increase carrying capacity and hole cleaning characteristics of the fluid. Fluid loss control additive is used to reduce HPHT fluid loss. Lime is added to activate the emulsifiers, increase pH, increase corrosion resistance, to increase alkalinity of the mud system. Brine is added to add viscosity and promote dehydration of shales in the formation being drilled. Brine is emulsified inside the oil base fluid as dispersed phase to create invert emulsion system. Then, lastly, barite is added as weighting agent to increase mud density.

All drilling fluid ingredients were mixed using FANN multi mixer for some time. Timing for mixing and order procedures are described in table 8 below[3].

Mud materials	**Mix 1 lab bbl on Hamilton mixer**		
	Order	**Speed**	**Time**
Base fluid	1	High	-
Primary emulsifier	2	High	2
Wetting agent and secondary emulsifier	3	High	2
Organophilic clay	4	High	5
Fluid loss control	5	High	2
Lime ($Ca(OH)_2$)	6	High	2
Water	7	High	15
Calcium Chloride ($CaCl_2$)			
Barite ($BaSO_4$)	8	High	2
	Additional time		30
	Total time, minutes		60

Table 8 Time Duration and Order of Mixing Ingredient[3]

The concentration of each mud additives are formulated based on trial in lab experiment. In real field use, mud formulation must adhere to wellbore and formation condition. The calophyllum methyl ester-based mud formulation samples and its concentration are shown in table 9 below.

Components (lb/bbl)	Concentration (lb/bbl)					
	IF-1	IF-2	IF-3	IF-4	IF-5	IF-6
CME	212.2	204.4	191.9	214.8	215.3	203.1
Primary Emulsifier	2	2	2	2	2	3.3

Secondary Emulsifier	4	4	4	4	4	6.7
Organophilic Clay	0.5	0.5	0.5	0.5	1	1
Fluid Loss Additives	8	8	8	8	8	8
Lime ($Ca(OH)_2$)	2	2	2	6	2	5
$CaCl_2$ Brine 25%	80.80	55.00	32.40	81.80	82.00	77.40
Barite	110.6	249.3	389.3	103.1	105.8	157.7
MW (ppg)	10	12.5	15	10	10	11
Oil Water Ratio	80:20	85:15	90:10	80:20	80:20	80:20

Table 9 Calophyllum methyl ester-based mud formulation samples designed for performance test, stability test and comparison study

The complete procedures of Calophyllum methyl ester based mud including the formulations and rheological properties measurement are described as below.

1. Obtain all materials required, consisted of *Calophyllum* methyl ester, brine and mud additives.
2. Prepare tools and equipment consisted of mud balance, weight indicator, mud mixer, viscometer, HPHT filter press unit, pH meter, electrical stability probes unit, hot rolling test unit.
3. Prior to mix the drilling mud system using multi mixer, fill the mixing cup with the required volume of *callophyllum* methyl ester, Add primary emulsifier to base oil to emulsify for 2 minutes.
4. Add secondary emulsifier immediately and mix about 2 minutes.
5. Add filtration control additives at the required weight as fluid loss control and mix for 2 minutes.
6. Lime is added to system is sheared for 2 minutes.
7. Prepare water and calcium chloride ($CaCl_{2)}$ 25% to formulate brine and weigh in weight balance to determine the quantity needed. Brine is poured slowly and mixed for 15 minutes.
8. Weighing material (barite) is added to the mixture to desired mud weight (to be calculated) and then mix until mixing time is over.

9. Mix another 30 minutes to obtain well mixed mixture and stir for some minutes before switching off the mixer.
10. Record all the data of mud performance experiments includes: rheological properties results (PV, YP and GS) for API at 120^oF (49^oC) using FANN viscometer, Electrical Stability (ES) using ES meter and mud pH using pH meter.
11. Conduct hot rolling test (250^oF for 16 hours) using hot rolling unit.
12. Record all the data of mud performance experiments includes: rheological properties results (PV, YP and GS), Electrical Stability (ES), pH meter of mud.
13. Create conclusion of all results and report all the recorded data into representative graph.
14. As result, *Calophyllum* methyl ester based mud is developed, mud performance is tested and mud stability is tested using electrical stability test.

3.2 Rheological Properties and Filtration Characteristic Measurement

3.2.1 Rheological Properties Measurement

- **Mud Weight or Mud Density**

Apparatus: The mud balance is the instrument generally used for mud weight determinations. The mud balance is designed such that the mud cup, at one end of the beam, is balanced by a fixed counterweight at the other end and a sliding rider free to move along a graduated scale. A level-bubble is mounted on the beam to allow for accurate balancing. The mud balance unit is shown in figure 10.

Procedures

1. The instrument base should be set on a flat, level surface.
2. Measure the temperature of the mud and record on the Drilling Mud Report form.
3. Fill the clean, dry cup with mud to be tested; put the cap on the filled mud cup and rotate the cap until it is firmly seated. Insure that some of the mud is expelled through the hole in the cap in order to free any trapped air or gas (see Appendix D of API RP 13B-1 for Air Removal).
4. Holding cap firmly on mud cup (with cap hole covered), wash or wipe the outside of the cup clean and dry.

5. Place the beam on the base support and balance it by moving the rider along the graduated scale. Balance is achieved when the bubble is under the center line.
6. Read the mud weight at edge of the rider toward the mud cup. Make appropriate corrections when a range extender is used.
7. The instrument should be calibrated frequently with fresh water. Fresh water should give a reading of 8.3 lb/gal or 62.3 lb/ft^3 (1000 kg/m^3) at 70°F (21°C). If it does not, adjust the balancing screw or the amount of lead shot in the well at the end of the graduated arm as required.

Additives: Increasing mud density should only be done with additions of a weight material, e.g. barites, hematite or acid soluble, calcium carbonate, and not through build up of drilled solids. Decreasing mud density should only be done by dilution and acceptable solids control practices.

Below are some useful formulae for calculating changes in the fluid volume or density as a result of addition of solids or dilution[5]:

Weight increase using barites

$$X = 1470*(W2 - W1) / (35 - W2) \quad (7.1)$$

Volume increase using Barites

$$V = 100*(W2 - W1) / (35 - W2) \quad (7.2)$$

Where,

X = No of 100 lbs sacks per 100 bbls of mud

V = No of bbls increase per 100 bbls of mud

W1 = Initial mud weight (ppg)

W2 = Desired mud weight (ppg)

Figure 10 Mud Balance Unit

- **Plastic Viscosity (PV)**

Apparatus: Viscometer or rheometer is a device used to measure the viscosity, yield point and gel strength of mud, a sample of mud is placed in a slurry cup and rotation of a sleeve in the mud gives readings which can be mathematically converted into plastic viscosity (PV) and yield point (YP), direct reading viscometer is shown in figure 11. Multi-speed rheometers are recommended whenever possible since readings can be obtained at 600, 300, 200, 100, 6 and 3 rpm. PV (in cP) is measured by taking the difference between the dial readings taken at the two highest speeds of 600 rpm and 300 rpm. For instance, PV can be expressed as below.

$$\mathbf{PV = \theta600 - \theta300}$$

If temperature is a factor, then the mud sample should be tested at 120^{o} F, with the mud in a heating cup[5].

- **Yield Point (YP)**

Apparatus: Same equipment as used for measurement of plastic viscosity. Yield Point (YP) is calculated from the following:

$$\mathbf{YP = \theta300 - PV}$$

When plastic viscosity rises, this is usually an indication that the solids control equipment are running inefficiently. Ideally, the yield point should be just high enough to suspend the cuttings as they are circulated up the annulus[5].

- **Gel Strength (GS)**

Apparatus: Six speed viscometer (600, 300, 200, 100, 6 and 3 rpm). There are two readings for gel strengths, **10 second** and **10 minute** with the speed of the viscometer set at **3 rpm**. The fluid must have remained static prior to each test, and the highest peak reading will be reported. The gel strength quantifies the thixotropic behaviour of a fluid; its ability to have strength when static, in order to suspend cuttings, and flow when put under enough force. Ideally the two values of gel strength should be close rather than progressively far apart[5].

Procedures for Obtaining Plastic Viscosity, Yield Point and Gel Strength Using Viscometer

1. Place a sample of the drilling fluid in a thermostatically controlled viscometer cup. Leave enough empty volume in the cup for the displacement of the viscometer bob and sleeve. The bob and sleeve will displace approximately 100 cm3 of drilling fluid. Immerse the rotor sleeve exactly to the scribed line. Measurements in the field should be made with minimum delay from the time of sampling. Test should be made at either 120 ±2°F (50 ±l°C) or 150 ±2°F (65 ±l°C).
2. Heat or cool the sample to the selected temperature. Intermittent or constant shear at the 600 rpm speed should be used to stir the sample while heating or cooling to obtain a uniform sample temperature. After the cup temperature has reached the selected temperature, immerse the thermometer into the sample and continue stirring until the sample reaches the selected temperature. Record the temperature of the sample.
3. With the sleeve rotating at 600 rpm, wait for dial reading to reach a steady value (the time required is dependent on the mud characteristics). Record the dial reading for 600 rpm.
4. Shift to 300 rpm and wait for dial reading to reach steady value. Record the dial reading for 300 rpm.
5. Stir drilling fluid sample for 10 seconds at high speed.
6. Allow mud to stand undisturbed for 10 seconds. Slowly and steadily turn the hand-wheel in the direction to produce a positive dial reading. The maximum reading is the initial gel strength. For instruments having a 3-rpm speed, the maximum reading attained after rotation at 3 rpm is the initial gel strength. Record the initial gel strength (10 sec gel) in lb/100 ft^2 (Pa).

Figure 11 Direct indicating viscometer

7. Re-stir the mud at high speed for 10 seconds and then allow the mud to stand undisturbed for 10 minutes. Repeat the measurements as in step number 6 and report the maximum reading as 10 minute gel in lb/100 ft^2 (Pa).

3.2.2 HPHT Filtration Test

Measurement protocols are based on the API recommended practice procedure 13B-2[3]. Both tests work on filling a cell with drilling fluid, and sealing it shut. Inside the cell is a filter paper that has been placed between the mud and the aperture in the cell. Pressure is applied to the cell which forces the mud and solids through the filter paper. The solids accumulating on the filter paper form a filter cake and the filtrate passing through the paper is collected in a graduated cylinder. The mud in the cell is pressurized for 30 minutes and the fluid or filtrate is collected and measured. The filter paper is also collected, washed, then examined and the deposited filter cake is measured. HPHT tests with the cell put under heat are usually carried out on wells where the temperature is greater than 200° F. the HPHT filter press unit is shown in figure 12. Here are the equipments and complete step by step procedures of filtration test.

Equipments

The high-temperature/high-pressure filter press apparatus consists of:

1. A filter cell to contain working pressures up to 1300 psi (8970 kPa) at temperature.
2. A pressurized gas source, such as carbon dioxide or nitrogen with regulators.
3. A heating system to heat to 350°F (177°C).
4. A high-pressure filtrate collection vessel maintained at proper back pressure (see Table 2) to avoid flashing or evaporation of the filtrate.
5. The filter cell contains a thermometer well. It is fitted with a removable end, a filter-media support and with oil-resistant seals. Valve stems on each end of the cell can be opened or closed during the test.
6. Filter medium: Whatman No. 50 or S&S 576, or equivalent filter paper.
7. Timer: to measure 30-minute interval. Mechanical or electronic.
8. Thermometer: to measure up to 500°F (260°C) with 5-inch (12.5 cm) or longer stem.

9. Receivers: 10 cm^3 and 20 cm^3 long, slender graduated cylinders, as used for oil, water and solids content. (See Section 6 and Figure 15).
10. Receiver (optional): 25 cm^3 glass graduated cylinder (TC).
11. Field Mixer: cup type, to operate at 10, 1000 to 15,000 rpm.
12. Ruler: to measure filter cake thickness in 1/32-inch increments.

Procedures

1. Place the thermometer in the well of the heating jacket. Preheat the jacket to approximately 10^{o}F (6°C) above the desired test temperature. Adjust the thermostat to the desired test temperature.
2. Stir the drilling fluid sample for 5 minutes using the field mixer. Pour the fluid sample into the filter cell, leaving at least 1 inch (2.5 cm) space in the cell, to allow for fluid expansion. Install the filter paper in the cell.
3. Complete the assembly of the filter cell. With upper and lower valve stems closed, place the cell in the heating jacket. Transfer the thermometer from the heating jacket into the well of the filter cell.
4. Connect the high-pressure filtrate collection vessel onto the lower valve stem and lock it in place.
5. Connect the pressure-regulated gas source to the upper valve. Connect a similar gas source to the filtrate collection vessel and lock these connections in place.
6. While keeping the two valve stems closed, adjust the upper pressure regulator to a pressure 100 psi (690 kPa) higher than the "Minimum Back Pressure" value. Next, set the lower regulator to the pressure "Minimum Back Pressure". Maintain this pressure until the test temperature is reached.
7. When the sample reaches the selected test temperature, open the lower valve stem and immediately increase the pressure on the upper regulator to 500 psi (3450 kPa) higher than the back pressure. This will start the filtration process. Start the timer. Maintain the test temperature to within ±5°F (±3°C) during the test. If the back pressure rises above the selected back pressure during the test, cautiously draw off and collect a portion of the filtrate to reduce the back pressure.
8. Collect the filtrate in the long, slender graduated glass cylinder (or graduated cylinder). Read the 30-minute total (water plus oil) filtrate volumes. Also read

volumes of solid and water phases, if present. The collected filtrate volume was doubled to correct it to the area of the API filtration test.

9. Immediately after collecting the 30-minute filtrate, close the upper and lower valve stems to contain the pressure. Following the manufacturer's detailed instructions, bleed pressure off the regulators and hoses, then disconnect the gas pressurization system. Remove the cell from the heating jacket and allow cell to cool to below 125°F (52°C). Keep the cell upright during cooling, depressurization and disassembly.
10. Bleed pressure from the filter cell by slowly opening the upper valve stem. Avoid spraying drilling fluid as gas exits stem. Carefully disassemble the cell. Be sure no pressure is trapped before dislodging the cap.
11. Pour the liquid from the cell.
12. Remove the filter cake on the paper. Measure the filter cake thickness at its center.

Figure 12 HPHT Filter Press Unit

3.3 Emulsion Stability Test

Emulsion stability test indicates the emulsion and oil-wetting qualities (stability) of the sample. The test is performed by inserting the ES probe into a cup of 120°F [48.9°C] mud and pushing a test button. The ES meter automatically applies an increasing voltage (from 0 to 2000 volts) across an electrode gap in the probe. Maximum voltage that the mud will sustain across the gap before conducting current is displayed as the ES voltage. The modern ES meter has sine-wave circuitry, whereas older meters used square-wave circuits. The ES sine-wave design and meaning of ES readings have been studied and were found to relate to an oil mud's oil-wetting of solids and to stability of the emulsion droplets in a complex fashion, however not yet understood[16]. The emulsion stability test unit is shown in figure 13. Here are step by step procedures to measure ES value of the invert emulsion system or calophyllum methyl ester based mud system.

Procedures

1. Inspect the electrode probe and cable for evidence of damage.
2. Ensure that the entire electrode gap is free of deposits and the connector to the instrument is clean and dry.
3. Disconnect the electrode probe (if possible) and run a voltage ramp test, following instructions in the ES Meter operating manual. If the meter is working properly, the ES reading should reach the maximum voltage permitted by the instrument.
4. Reconnect the electrode probe to the ES Meter and repeat the voltage ramp in air. Again, the ES reading should reach the maximum permitted voltage; if it does not, the electrode probe and connector may need to be re-cleaned or replaced.
5. Repeat the voltage ramp test with the electrode probe in tap water. The ES reading should not exceed 3 volts. If ES does exceed 3 volts, re-clean the electrode probe or replace it.
6. Check the accuracy of the ES meter with the standard resistors and/or Zener diodes. The ES readings should fall within 2.5% (combined uncertainty of meter and resistor/diodes) of the expected values. If any of the ES readings fall outside this range, the instrument should be returned to the supplier for adjustment or repair.
7. Verify daily equipment calibration/performance.
8. Place the oil mud sample, which has been screened through a 12-mesh screen or a Marsh funnel, in a viscometer cup maintained at 120 ±5ºF (50 ±2ºC). Record the mud temperature on the Drilling Mud Report Form.
9. Clean the electrode probe body thoroughly by wiping with a clean paper towel. Pass the towel through the electrode gap a few times. Swirl the electrode probe in the base oil used to formulate the mud. If the base oil is not available, another oil or a mild solvent (such as isopropanol) is acceptable. Clean and dry the electrode probe as before.
10. Hand-stir the 120°F sample with the electrode probe for approximately 10 sec to ensure that the composition and temperature of the mud are uniform. Position the electrode probe so that it does not touch the bottom or sides of the container, and be sure that the electrode surfaces are completely covered by the sample.

11. Begin the voltage ramp. Follow the procedure described in the ES meter operating manual. Do not move the electrode probe during the voltage ramp.
12. At the conclusion of the ramp test, note the ES value displayed on the readout device.
13. Repeat step 9 through 11 with the same mud sample. The two ES values should not differ by more than 5%. If they differ by more than 5%, check the meter or electrode probe for malfunction.
14. Record the average of the two ES measurements.

Figure 13 Emulsion Stability Test Unit

3.4. Environmental Impact Test with Fish Acute Toxicity Test LC_{50}

This test is based on the United States Environmental Protection Agency (EPA) '*Ecological Effects Test Guidelines*' for fish acute toxicity test for freshwater environment[18]. This test is aimed to observe any mortality and abnormalities of fish in the freshwater environment after being exposed with contaminants, which in this study; Calophyllum methyl ester-based mud (IF-6) is used. The standard indicator freshwater fish that was used is Guppy fish (*Poecilia reticulate*). Fishes are three months old (juvenile) and evenly placed in aquarium for male and female fish.

The test was closely monitored for 96 hours, every 24, 48, 72 and 96-hour with three contaminants concentrations (5,000, 10,000 and 20,000 ppm). The parameters that are measured and observed were the mortality number, pH and temperature, after being exposed with IF-6 mud sample. The additional parameters that are observed were swimming behavior (erratic/normal), excitability increment, loss of reflex, discoloration event and change of physical/abnormalities. The equipments and fish acute toxicity LC_{50} test are shown in figure 14. Here are step by step procedures to conduct acute fish toxicity test based on the EPA (Environmental Protection Agency) standards.

Procedures

1. Obtain developed mud after experiments: *Calophyllum inophyllum* methyl ester based drilling mud IF-6 as the contaminant.
2. Prepare quantity of drilling mud to be exposed to aquatic organism (*Danio rerio* /zebra fish). The concentration was 5,000 ppm (10 ml), 10,000 (20 ml) ppm and 20,000 (40 ml) ppm. With total water volume of 2 liter per aquarium.
3. Prepare aquatic organism Guppy fish (*Poecilia reticulate*) inside aquarium filled with fresh water simulating fresh water environment equipped with air compressor. There were three (3) tested aquariums and one (1) control aquarium.
4. Record all initial data (parameters) of initial time, initial pH & temperature of water, the organism physical appearance, swimming ability and number of samples live.
5. Parameters which are observed and measured were mortality number due to exposed IF-6 mud system in 96 hours using LC_{50} standard. Below 50% population dead categorized as environmentally acceptable, above 50% died categorized as not acceptable. Additional parameter: water pH, water temperature, survival rate & abnormal behaviour (swimming ability & physical appearance).
6. Record all data continuously every 0, 6, 24, 48, 72 and 96 hours.
7. Analyze and conclude the results with the outcome of mortality numbers with respective contaminant's concentrations. Record pH, quality of water and temperature of the water as well.
8. Observe and record any abnormalities and physical changes found on the fish (swimming manner erratic or normal, excitability increment, loss of reflex, discoloration event on the fish and any physical changes or appearances on the fish).
9. Tabulate all the results in table with respective contaminant's concentration.

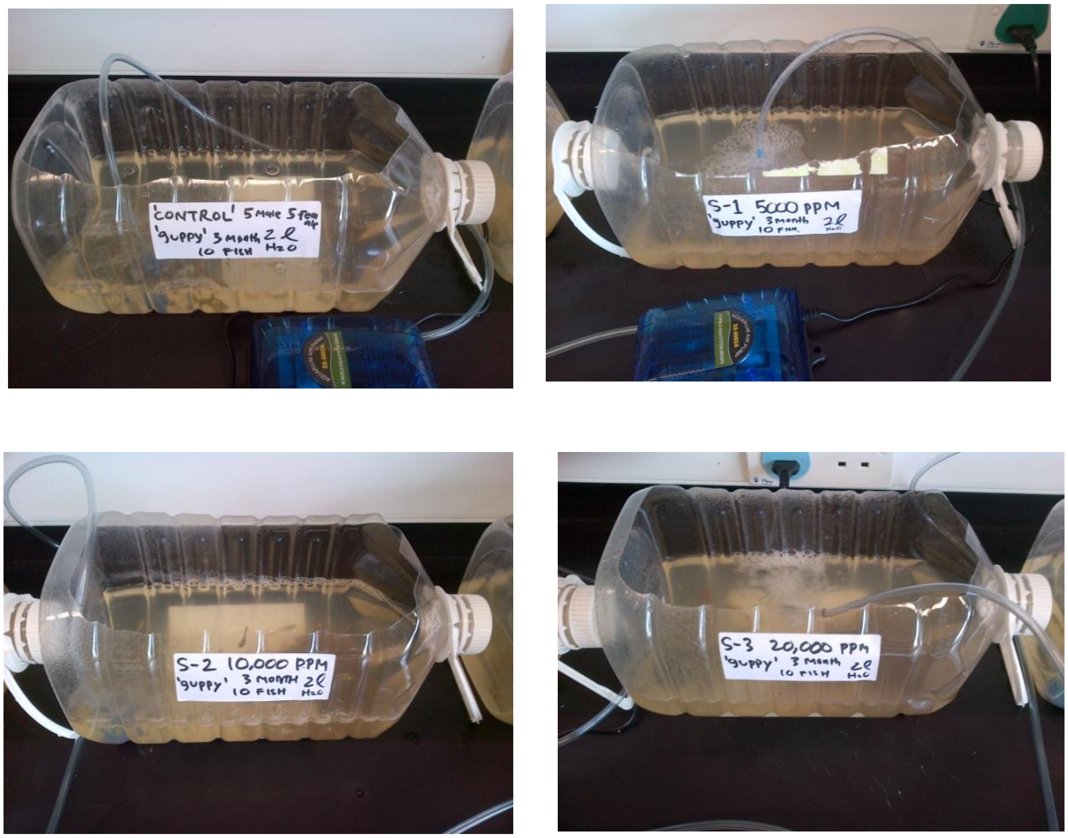

Figure 14 Fish Acute Toxicity 96 hours LC_{50} Test

3.5. Equipments and Materials Required

3.5.1. Equipments Required

- Mud Development: mud balance, weight indicator, mud mixer, marsh funnel, viscometer, filter press unit, HPHT filter press unit, pH meter, electrical stability probes unit, hot rolling test unit.
- Environmental Assessments: aquarium and air compressor

3.5.2. Materials Required

- Drilling Fluid Development:
  - Base Fluid: *Calophyllum inophyllum* methyl ester and Brine
  - Additives: organophilic clay, emulsifier, lime, fluid loss additives, barite, calcium chloride.
  - Environmental assessments: IF-6 mud sample, conditioned freshwater environment and Guppy fish (*Poecilia reticulate*).

3.6. Key Milestone and Schedule of Project Activities

Activities	2011				2012				
	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May
Topic Selection and Preliminary Research									
Preliminary Literature Review and Theory Analysis									
Oral Defense Presentation									
Procurement of Calophyllum Methyl Ester Base Oil from Koperasi Tani Jarak Lestari, Indonesia									
Calophyllum Methyl Ester Based Mud Formulations Based on API RP 13B									
Rheological Properties Measurements of Calophyllum Methyl Ester Based Mud Based on API 13-B Protocols									
Filtration Characteristics of Calophyllum Methyl Ester Based Mud Based on API 13-B Protocols									
Environmental Assessment: Fish Acute Toxicity LC50 for 96 hours Based on EPA Standard for Fish Acute Toxicity Test									
Production Cost Analysis									
Milestone	**2011**				**2012**				
	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May
Topic Selected and Completion of Feasibility Study									
Completion of Theory Formulation and Literature Review									
Calophyllum Methyl Ester Obtained									
Completion of Calophyllum Methyl Ester Based Mud Formulations based on API RP 13-B									
Completion of Rheological Properties and HPHT Filtration Characteristics Measurements Based on API RP 13-B									
Documentation of Rheological Properties and Filtration Characteristics Results									
Completion of Analysis on Rheological Behavior Studies									

Completion of Comparison Study of Mud Rheological Specifications with Benchmark Mineral Oil Based Mud and Palm Methyl Ester Based Mud									
Documentation of Environmental Assessment Results of Fish Acute Toxicity LC50 Test									
Production Cost Report									

Table 10 Key milestone and schedule of project activities

CHAPTER 4

RESULTS AND DISCUSSION

4.1. Rheological Behavior of Calophyllum Methyl Ester-Based Mud

During this project, *Calophyllum* methyl ester (CME) based drilling mud was formulated and tested for the performance and its emulsion stability under several conditions. The variation of conditions were comprised of lime concentration (high and low), wide range of density and oil/ water ratio, variation on organophilic clay concentration and variation on temperature.

There were five (5) mud samples for mud rheological behavior test under different conditions said. The effect of lime concentration (low and high), mud density and oil water ratio changes, temperature changes (120^{o}F and 250^{o}F) were analyzed and determined. In addition, the emulsion stability test was performed to analyze the stability of the invert emulsion under wide range of conditions mentioned.

4.1.1. Effect of Lime Concentration

Two concentration of lime (2.0 and 6.0 lb/bbl) were tested in IF-1 and IF-4 mud samples. The concentration given was to check the effect of low and high concentration of lime to the rheological properties and the hydrolysis occurrence of the mud system before hot rolling (BHR) and after hot rolling (AHR). The mud formulation of IF-1 and IF-4 is previously shown in Table 9.

The rheological properties measurement result is tabulated in Table 11 below, it indicates that in IF-1 and IF-4, the PV increase significantly from 39 to 41 cP after subjected lime concentration addition from 2 to 6 lb/bbl before hot rolling, with all component concentration remained the same. This indicates that lime addition flocculated or activated the viscosifier (organophilic clay) thus, increased PV. After hot rolling at 250^{o}F, in IF-1 the PV decreased significantly from 39 to 29 cP which indicates unstable viscosity, yet, in IF-4 PV increased slightly from 41 to 45 cp after hot rolled, which means more stable emulsion.

It can be concluded that more lime concentration will enhance the stability of rheological properties BHR and AHR. This is due to flocculation effect was more retained in IF-4 (6 lb/bbl) causing the IF-4 was more stable during thermal degradation with stronger emulsion bond. IF-4 which has more lime concentration showed more flat and stable rheology in PV and YP BHR and AHR than those in IF-1 with lower lime concentration.

As for YP, adding lime concentration from 2 to 6 lb/bbl clearly increased the carrying capacity or YP of the mud system in IF-4 compared to IF-1 before hot rolling. This was due to lime function as the flocculant activator. In emulsion stability result, the emulsion was reported stable as shown in figure 15. In general, it can be stated that the calophyllum ester based mud had shown compatibility with the mud additive by showing typical manner in rheological properties, in this case, lime.

There were no hydrolysis occurrence, as there was no alcohol and acid separated from the mixture observed after hot rolled. These findings obviously supported previous study by Patel, et al. which explains that in the presence of 3 – 4 lb/bbl of lime, ester-based fluid might undergo hydrolysis and become solid during aging test.

Parameter	IF-1		IF-4	
	BHR	AHR	BHR	AHR
Plastic Viscosity (cP)	39	29	41	45
Yield Point (lb/100 ft^2)	3	12	18	16
Gel Strength 10-Sec (lb/100 ft^2)	8	7	12	9
Gel Strength 10-Min (lb//100 ft^2)	8	8	15	11
Electrical Stability (Volts)	725	1999	618	958
pH $[H^+]$	9.43	5.38	11.48	5.76

Table 11 Rheological properties result of IF-1 and IF-4 based on lime concentration addition effect study

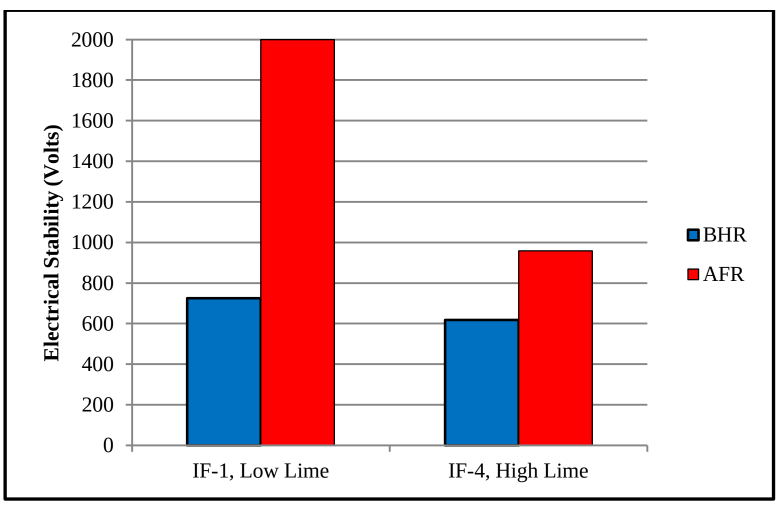

Figure 15 Emulsion Stability Result BHR and AHR of IF-1 and IF-4

4.1.2. Effect of Mud Density and Oil Water Ratio Change

There were three mud density and oil water ratio tested, 10 ppg with 80:20 OWR, 12.5 ppg with 85:15 OWR and 15 ppg with 90:10 OWR, respectively for IF-1, IF-2 and IF-3, as shown in mud formulation at Table 9. 15 ppg mud weight was formulated to observe the capability of the base oil to withstand the respective high density mud. The mud weight or solid content was increased as the oil water ratio increased.

The result as shown in Table 12 indicates that IF-2 and IF-3 rheological properties were stable BHR and AHR as shown in figure 16. In IF-2 and IF-3, PV value BHR and AHR showed slight change which only differ 4 and 2 cp for IF-2 and IF-3, respectively. The slight change indicates the strong thermal and rheological stability of mud in maintaining the rheological properties for IF-2 and IF-3. In IF-1, PV decreased significantly from 39 to 29 cP and YP increased significantly from 3 to 12 lb/100 ft^2 there were big changes in the value BHR and AHR which was indication of thermal instability, this is due to less viscosifier additives and lime additives, results in instability of emulsions especially in AHR conditions. Gel Strength (GS) value was 8 and 8 lb/100 ft^2 BHR and AHR which was stated as stable in maintaining the suspended cutting. Emulsion Stability test was reported stable for IF-1, IF-2 and IF-3, with the ES value of more than 400 Volts BHR and AHR. Based on results, IF-2 and IF-3 showed that calophyllum methyl ester based muds were able to maintain the rheological properties in flat rheological manner in various mud weight and oil water ratio.

Parameter	IF-1		IF-2		IF-3	
	BHR	AHR	BHR	AHR	BHR	AHR
PV, cP	39	29	32	36	40	38
YP, (lb/100 ft^2)	3	12	9	11	11	9
GS-10-Sec (lb/100 ft^2)	8	7	7	8	9	7
GS-10-Min (lb/100 ft^2)	8	8	8	88	10	9
ES, Volts	725	1999	1223	1999	1943	1999
pH [H^+]	9.43	5.38	9.78	5.11	11.52	6.14

Table 12 Rheological properties result of IF-1, IF-2 and IF-3 based on mud density and OWR change effect study

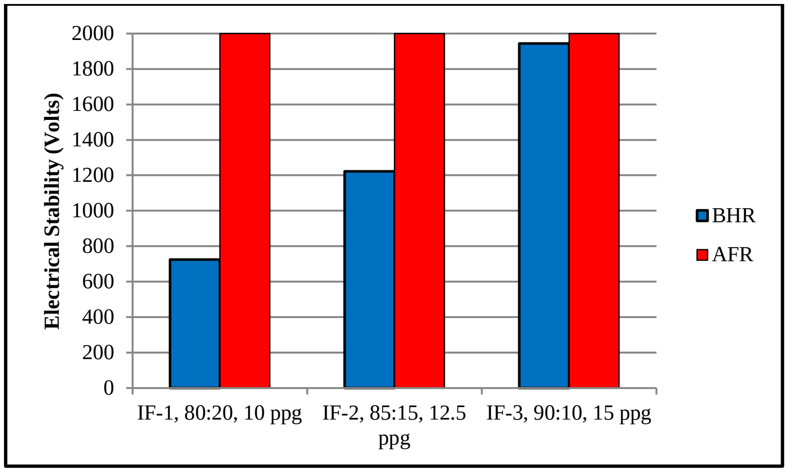

Figure 16 Emulsion Stability Result BHR and AHR of IF-1, IF-2 & IF-3

4.1.3. Effect of Organophilic Clay Concentration

Two different organophilic clay concentrations were tested to IF-1 and IF-5, 0.5 and 1 lb/bbl, respectively. The rheological behavior result as shown in Table 13 indicates that the additional 0.5 lb/bbl in IF-5 compared to IF-1 had increased its YP value BHR. The YP increased significantly for both IF-1 and IF-5 AHR. This finding supported previous study[16] conducted by Ghalambor, et al. which explains that the addition of organophilic clay will linearly increase the ability of cutting lifting or YP in the mud system up to 10 lb/bbl concentration[19].

The addition of gel additives or organophilic clay increased YP and GS simultaneously, due to increased gel content inside the mud system which flocculate the mud solid particles and bond it together. Thus, besides the mud carrying capacity increased, the hole cleaning ability was also increased. The mud system with 1 lb/bbl (IF-5) of organophilic clay was found to be more stable than 0.5 lb/bbl mud (IF-1) before hot rolling, as shown in figure 17. After hot rolled, higher organophilic clay concentration mud (IF-5) was less stable than IF-1. However, both mud samples showed good emulsion stability as ES value is more than 400 Volts as per API standard for ester-based mud.

Parameter	IF-1		IF-5	
	BHR	AHR	BHR	AHR
Plastic Viscosity (cP)	39	29	38	30
Yield Point (lb/100 ft^2)	3	12	4	15

Gel Strength 10-Sec (lb/100 ft^2)	8	7	9	5
Gel Strength 10-Min (lb//100 ft^2)	8	8	9	5
Electrical Stability (Volts)	725	1999	817	938
pH [H^+]	9.43	5.38	11.08	5.3

Table 13 Rheological properties result of IF-1 and IF-5 based on organophilic clay concentration effect study

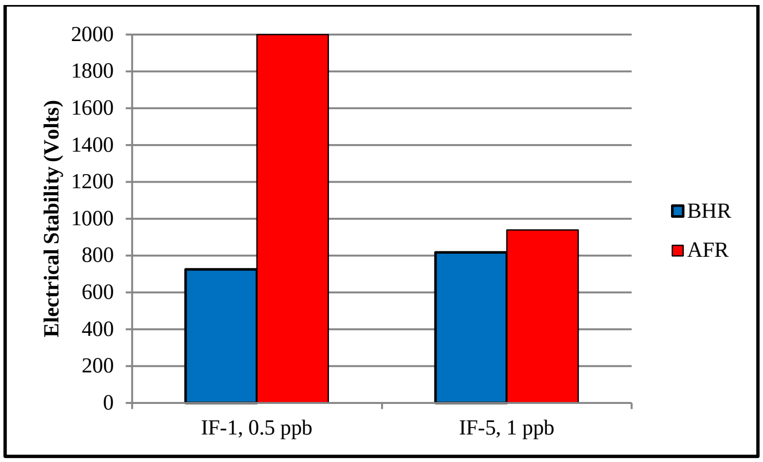

Figure 17 Emulsion Stability Result BHR and AHR of IF-1 & IF-5

4.1.4. Effect of Temperature Change on Emulsion Stability of Calophyllum Methyl Ester-Based Mud

As shown in Table 12 to 14, the mud rheological properties result, it indicates that generally all mud samples were reported stable after being subjected to heat aging test or hot rolling test BHR and AHR. As the electrical stability (ES) test had shown the ES level more than 400 Volts, as shown in figure 8 to 10. This finding implies that before and after hot rolling test, most of the Calophyllum methyl ester-based muds are stable in terms of emulsion. This is due to the good formulation and correct concentration given for the mud additives into the all six (6) mud samples. It also can be stated that the mud compatibility issues were not found and observed.

Concerning about the rheological properties changes after subjected to heat aging test, one of the formulation was found to be unstable (IF-1) yet, the rest of the samples were generally had slightly changes of its rheological properties as shown in Table 12 to 14. The instability which was observed in IF-1 was due to the incorrect or not precise formulation concentration of the lime and organophilic clay, there were not enough of both additives which led to the inflocculated mud solid particles and the low emulsion stability value compared to other IF-2, IF-3, IF-4 and IF-6 mud samples.

4.2. Filtration Characteristics of Calophyllum Methyl Ester-Based Mud

HPHT (High Pressure High Temperature) Filtration Loss test was carried out to all mud samples after being subjected to hot rolling test for 16 hours at 250^{o}F. The tests were carried out for 30 minutes under temperature of 250^{o}F and pressure of 500 psi.

The results of filtration characteristic of all calophyllum ester based mud sample are shown in Table 14. It indicates that most of the mud sample were failed to fulfill the requirement of maximum filtration fluid loss according to API which is 15 ml. this is due to lack of fluid loss additives given in the mud systems. Based on the result, IF-1 and IF-6 were the only mud sample which fulfills the requirement of API with fluid loss of 14.0 and 10.0 ml, respectively, as shown in figure 18.

Based on the observation towards the presence of water in the filtrate, all mud samples filtrate were reported to have no water in the filtrates. Based on this finding, it is concluded that the emulsions were very stable towards thermal degradation and high pressure conditions (HPHT). Thus, the water, alcohol and acids did not separate from the invert emulsion system.

Based on the mud cake measurement result, IF-1, IF-4, IF-5 and IF-6 were determined to have good mud cake layer. However, IF-2 had shown very thin mud cake, almost no mud cake detected. This was due to the dispersed solid content inside IF-2 was very low in the particle size and the solid content was extremely flocculated and lumped inside the filtration cup. The flocculated barite and solid particles were found to not mingle well with liquid phase and balled. As for the IF-3, the mud cake was thick, as shown in table 12, this is due to the large solid content as IF-3 has 90:10 oil water ratio and 15.0 ppg, thus very dense and large amount of solid content. The other reason of separation of barite was due to less lime additives to create better emulsions stability, since the lime was just 2 lb/bbl. More lime concentration and mud cake thickness reducer additive, need to be added to reduce the thickness of mud cake and improve the dispersion of solid content inside the dominant phase of invert emulsion.

Parameter	IF-1	IF-2	IF-3	IF-4	IF-5	IF-6
HTHP @500 psi, cc	14.0	20.0	54.6	44.0	21.0	10.0
Cake Thickness (in 32 inch)	1.392	0.0096	8.704	1.952	0.848	0.736

Table 14 HPHT Filtration Loss Characteristics Results

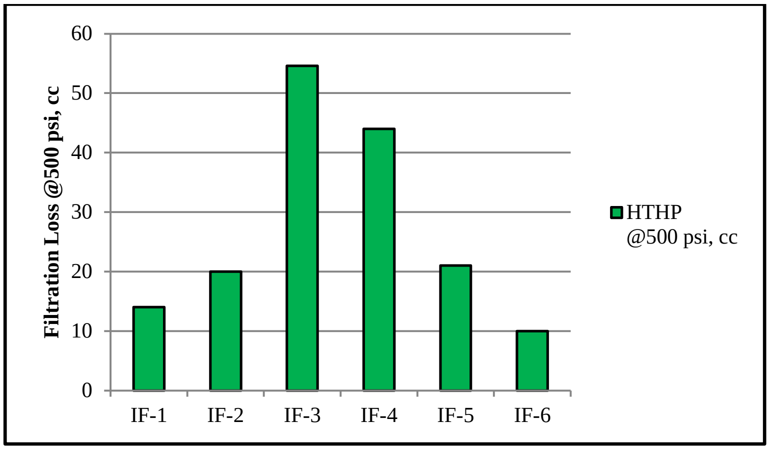

Figure 18 HPHT Filtration Loss at 500 psi

4.3. Eco-toxicological Properties of Calophyllum Methyl Ester-Based Mud with Acute Toxicity Test LC_{50}

Acute toxicity test was conducted, using IF-6 mud samples as the contaminant. Freshwater indicator fish was used as the environmental impact of the discharged mud system to the freshwater environment. Guppy fish (*Poecilia reticulate*) was used. The age of fish was juvenile 3 months old, male and female species. Three (3) concentration (5,000, 10,000 and 20,000 ppm) of IF-6 were exposed to three aquarium equipped with fresh water and ten (10) Guppy fishes each. The initial condition of fishes was ensured to be in the good and healthy conditions. 96 hours acute fish toxicity test was performed according to EPA standard.

The toxicity level result, as shown in Table 15 to 17, shown that the calophyllum methyl ester based mud were environmentally friendly with the concentration below 10,000 ppm due to less than 50% mortality observed. Level of pH in the water has slightly affected by the discharged mud with minimum tolerable increment.

For experiment-1 with 5,000 ppm contaminant, at 72-h, it was observed that one fish out of 10 fishes had erratic swimming behavior. At the end of 96-h test, the mortality is one (1) fish as shown in Table 15. The excitability increment, erratic swimming behavior and loss of reflex were detected at 96-h. Discoloration and changes of physical were observed to be normal during 96-h test. In addition, the contaminant caused severe invisibility level of the water.

Parameters	Experiment-1 (5,000 ppm)					
Hours	0	6	24	48	72	96
pH	6.34	6.58	6.45	6.48	6.69	5.93
T (^oC)	22.0	21.9	22.1	22.0	22.0	22.0
Mortality (n)	0	0	0	0	0	1
Swimming (Erratic/Normal) (E/N)	N	N	N	N	E 1 fish	E
Excitability Increment	No	No	No	No	No	Yes
Loss of Reflex	No	No	No	No	No	Yes
Discoloration	No	No	No	No	No	No
Change of Physical	No	No	No	No	No	No

Table 15 Acute Toxicity Test Result for 5,000 ppm concentration

For experiment-2 with 10,000 ppm contaminant, it was observed that nine (9) out of ten (10) fishes still alive at 96-h observation as shown in Table 16. However, erratic swimming behavior and loss of reflex were detected. In addition, the water clearness was reduced, the water color turned to darker and there was little foam at the surface of water. At the end of 96-h test, the fishes were very passive and loss of reflex.

Parameters	Experiment-2 (10,000 ppm)					
Hours	0	6	24	48	72	96
pH	6.30	6.85	6.86	7.03	7.42	5.31
T (^oC)	22.0	21.7	22.2	22.5	22.5	21.9
Mortality (n)	0	0	0	0	0	1
Swimming (Erratic/Normal) (E/N)	N	N	N	N	N	E
Excitability Increment	No	No	No	No	No	No
Loss of Reflex	No	No	No	No	No	Yes

Discoloration	No	No	No	No	No	No
Change of Physical	No	No	No	No	No	No

Table 16 Acute Toxicity Test Result for 10,000 ppm concentration

For experiment-3 with 20,000 ppm contaminant, all ten (10) fishes were confirmed died at 96-h observation as shown in Table 17. The fish movements were very passive and start dying at 72-h observation. In addition, the water visibility is limited, water color turned to darker, some of the solids were suspended at the water surface and the water clearness level is very low. Loss of reflex were detected at 72-h.

Parameters	Experiment-3 (20,000 ppm)					
Hours	0	6	24	48	72	96
pH	6.19	6.87	6.59	6.67	6.98	5.71
T (^{o}C)	22.0	21.5	22.0	22.0	22.0	21.8
Mortality (n)	0	0	0	0	0	10
Swimming (Erratic/Normal) (E/N)	N	N	N	N	N	E
Excitability Increment	No	No	No	No	Yes	Yes
Loss of Reflex	No	No	No	No	No	Yes
Discoloration	No	No	No	No	No	No
Change of Physical	No	No	No	No	No	No

Table 17 Acute Toxicity Test Result for 20,000 ppm concentration

Based on three acute toxicity test result above, it can be concluded that calophyllum methyl ester based mud was found to be environmentally friendly below 10,000 ppm to discharge to the freshwater environment. According to the limit of mortality specified by EPA standards about Lethal Concentration (LC50)[15], the allowable mortality should be no more than 50% of the population. Hence, the experiment-1 and experiment-2 were passed this requirement. However, at experiment-3 with 20,000 ppm IF-6 contaminants, the fish mortality level is maximum, thus more than 50% of the population were died. Thus, the concentration of 20,000 ppm must not be used in the drilling operations.

Further comprehensive research on the optimum concentration shall be discharged to the freshwater environment should be conducted for future improvements.

4.4. Comparison Study of Calophyllum Methyl Ester-Based Mud with Palm Oil Methyl Ester-Based Mud, Sarapar 147 and Saraline 185V Mineral Oil-Based Mud

Mud formulation for rheological properties study comparison is shown in Table 18. Calophyllum methyl ester based mud (IF-6) was formulated with the same additive concentrations, mud weight and oil water ratio (11 ppg and 80:20). The comparison study is aimed to check the compatibility of the mud additive with calophyllum methyl ester base oil and to check the similarity of rheological properties with the benchmark chosen as the technical performance assessment.

The specifications of the mud rheological properties and the mud rheological properties for sarapar 147, saraline 185V and palm FAME-based mud were obtained from the previous study[3] on Jatropha methyl ester as base oil done by Kania, et al. The rheological properties specifications range were specified by the Scomi Oiltools. Sdn. Bhd, drilling fluid company in Malaysia, as the case studies for this oil/synthetic based mud comparison study.

The rheological properties were compared with other oil/synthetic based mud including Sarapar 147, Saraline 185V and Palm Fatty Acid Methyl Ester (FAME) mud as mentioned before. The rheological properties results BHR and AHR are shown in Table 19.

For rheological properties results, formulation of IF-6 mud sample with the value of Plastic Viscosity (PV) 39 and 53 cp, BHR and AHR, respectively with the range of specification of 45-55 cp AHR condition. The Yield Point (YP) is 9 and 15 lb/100ft^2, BHR and AHR, respectively with specification range of 15-25 lb/100ft^2 AHR condition, and Gel Strength (GS) 10 minutes, 12 and 9 lb/100ft^2, BHR and AHR, respectively, with specification range of 20-40 lb/100ft^2 AHR condition. It showed that the rheological properties of calophyllum based mud had been fulfilled for PV and YP specification standard, yet for GS; it needs to be modified by adding the gel additives more since it was still too low. For the emulsion stability, IF-6 had been fulfilled the requirement of emulsion stability standard which is more than 600 Volts, the value of IF-6 Emulsion Stability (ES) is 940 Volts after being subjected to hot rolling test.

The result showed that calophyllum methyl ester based mud has met the rheological properties specifications standard of PV and YP compared to other benchmark oil based mud as shown in figure 19 and 20. For GS, as shown in figure 21, IF-6 has failed to meet the requirement, as it was too low, this condition was occurred to the other oil based mud benchmarks as well. However, by adding gel additives or more viscosifiers to strengthen gel characteristic properties, the GS could be improved significantly. The emulsion stability specification requirement was met by IF-6 with the value of 940 Volts; and the ES value was comparable as other oil based mud as shown in figure 22.

Components	**Concentration (lb/bbl)**			
	Callophyllum Methyl Ester Based mud (IF-6)	**Sarapar 147 mud**	**Saraline 185V mud**	**Palm FAME mud**
Calophyllum Methyl Ester	203.1	-	-	-
Sarapar 147	-	163.1	-	-
Saraline 185V	-	-	163.1	-
Palm FAME	-	-	-	191.7
Emulsifier	10	10	10	10
Liquid fluid loss control	-	2.5	2.5	2.5
Organo clay	1	1	1	1
Fluid loss control	8	8	8	8
Lime	5	5	5	5
$CaCl_2$ brine 35%	77.40	85.1	85	87.2
Barite	157.7	187.2	186.6	156.7

Table 18 IF-6 Mud Formulation for Comparison Study with other benchmarks

In conclusion, based on the result of rheological properties measurement test, it can be concluded that IF-6 or calophyllum methyl ester based mud were comparable with other benchmark base oil (Sarapar 147 mud, Saraline 185V mud and Palm methyl ester mud). The compatibility with other mud additives was achieved based on the good emulsion stability and stable and flat rheological properties result BHR and AHR. The technical performance was comparable with minimum difference with other benchmark mud system especially palm FAME based mud. IF-6 was found to be successfully achieved rheological properties specification standard given by the real field data requirement.

In addition, the flexibility in changing or modifying the rheological properties was found to be possible and applicable in calophyllum methyl ester based mud. Hence, there is no doubt that calophyllum methyl ester base oil is suitable candidate for the synthetic base oil alternatives.

Rheological Properties	**Specification Range**	**Callophyllum M. E. based mud (IF-6)**	**Sarapar 147 mud**	**Saraline 185V mud**	**Palm FAME mud**
Before Hot Rolling @120°F					
PV, cP		39	15	12	36
YP, lb/100ft^2		9	2	2	5
Gel 10 sec		11	3	2	7
Gel 10 min		12	4	3	9
ES (Volt)		880	1468	1333	1380
After Hot Rolling @200°F for 16 hours					
PV, cP	45- 55	**53**	13	13	47
YP, lb/100ft^2	15 - 25	**15**	3	2	30
Gel 10 sec	10 - 20	**8**	3	2	15
Gel 10 min	20- 40	**9**	10	7	18
ES (Volt)	>600	**940**	897	980	1380

Table 19 Rheological properties results of comparison study of calophyllum methyl ester-based mud (IF-6) with other benchmarks ester-based mud

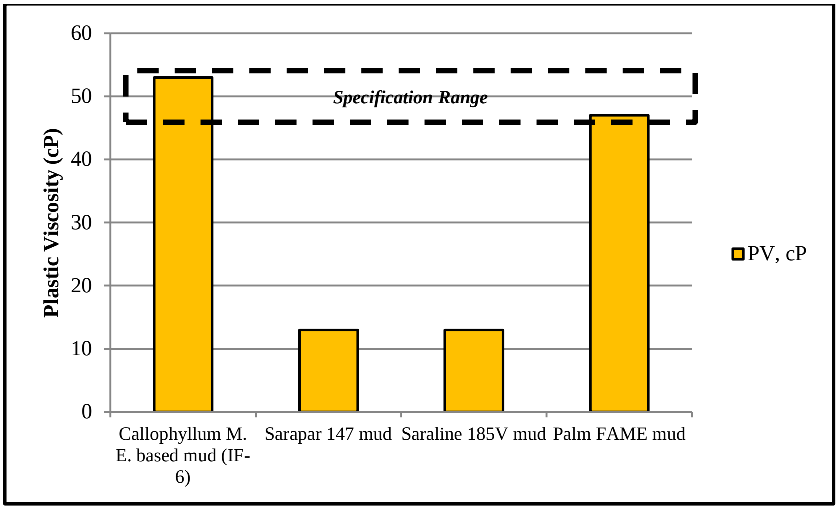

Figure 19 Plastic Viscosity of IF-6 compared with other benchmarks

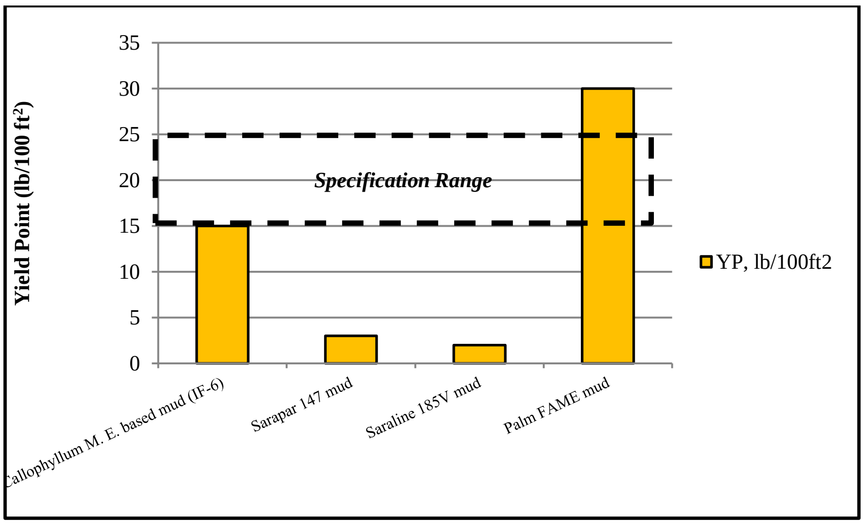

Figure 20 Yield Point of IF-6 compared with other benchmarks

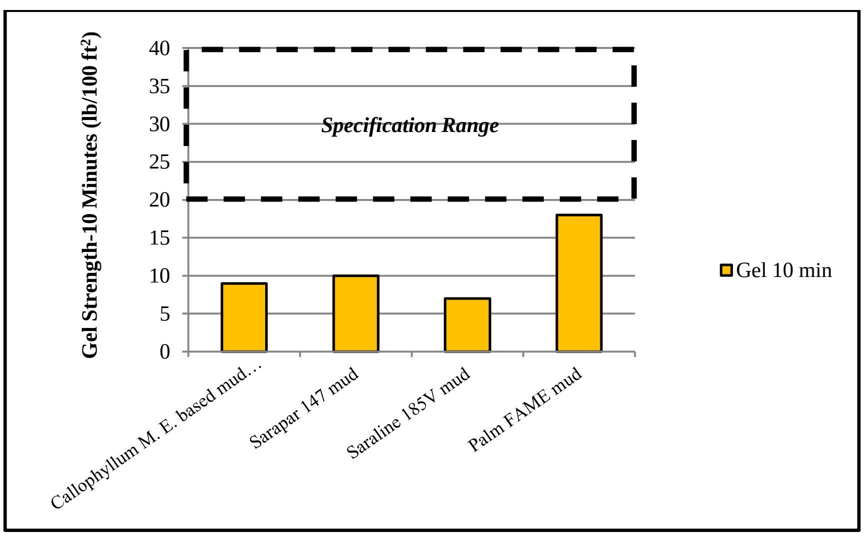

Figure 21 Gel Strength-10-Min of IF-6 compared with other benchmarks

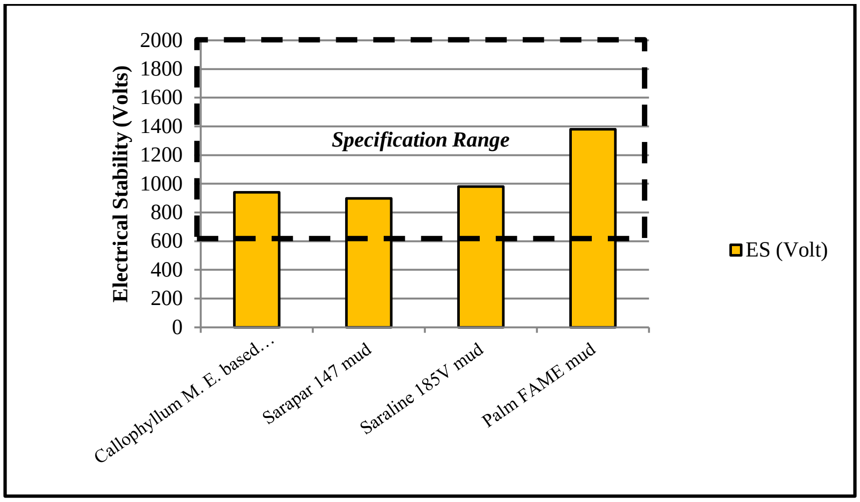

Figure 22 Emulsion Stability of IF-6 compared with other benchmarks

4.5. Advantages of Calophyllum Methyl Ester-Based Drilling Fluids

As base fluid of drilling fluid system, calophyllum methyl ester has many advantages over other type of base fluid. The advantages of calophyllum methyl ester are summarized below.

- Based on the acute toxicity test conducted in this study, Calophyllum methyl ester is considered as acceptable and environmentally friendly towards the freshwater indicator fish during 96-h test.
- Calophyllum methyl ester is readily biodegradable since its natural source of oil is from plant (*Calophyllum inophyllum L*.).
- Compared to other plant derived ester base fluid, Calophyllum oil as the source of its methyl ester can be extracted from the seed until 75% of the total oil content. Thus, the more oil supply that can be extracted, the more methyl ester can be produced as base fluid for drilling fluid.
- Calophyllum oil has abundant and renewable source of supply. Unlike petroleum derive base oil, the source of calophyllum oil are sustainable and can be adjustably produced accordingly to cater the demand of the base oil supply. In addition, as petroleum has been depleted, the usage of alternative source of base oil is preferred.
- The usage of calophyllum methyl ester will enhance drilling cost per feet as it can drill faster than water based mud; reduce the formation damage and more importantly less wellbore problems.

- Elimination of water for dilution as the WBM application to reduce the mud weight, thus reduced the cost.
- Has better thermal stability under high temperature well than WBM.
- Lower total drilling cost, subjected to count the overall cost. Lowered drilling cost due to faster drilling days because of lesser wellbore problems, lesser Non Productive Time (NPT) and lesser fishing requirement.
- As technical performance, based on this study, Calophyllum methyl ester based mud is proven to be comparable with other benchmark (palm and mineral oil based mud). Calophyllum methyl ester based mud is considered successful in terms of drilling mud rheological properties, fulfilling the specification given and mud additives compatibility.

4.6. Cost Analysis of Calophyllum Methyl Ester Base Fluid

The production cost detail of calophyllum methyl ester production is shown in Table 20. The total production cost for calophyllum methyl ester base fluid is 2,703 USD/month with 8.33 tons of calophyllum dried seeds processed every month. Total production of calophyllum methyl ester is 9,639 liter per month, thus the production cost of the methyl ester is 0.28 USD/liter or 1.06 USD/Gallon. The retail price in the market for calophyllum methyl ester is 0.33 USD/liter or 1.25 USD/gallon.

Variable Cost	Unit/Month	Cost/Month (USD)
Dried Calophyllum Seeds	9 tons	981
Extracted Crude Oil	6,426 Liter	-
Methanol & Ethanol	3213 Liter	1,189
Fuel for Generator	120 Liter	58.86
Water + Electric Supply	-	10.9
Labor	-	408.75
Additional Services	-	54.5
Total Production Cost	-	2,703
Total Methyl Ester Production	9639 Liter	
Cost per Liter	0.28 USD/Liter	
Cost per Gallon	1.06 USD/Gallon	

Table 20 Production cost of calophyllum methyl ester

The cost of the mud additives is shown in Table 21 for drilling mud cost calculation according to various synthetic based mud formulation compositions[3]. The comparison study on various base fluid prices is shown in Table 22. It is clear that calophyllum-base fluid is the lowest cost compared to other benchmark base oil with the 1.25 USD/gallon retail price.

Additives	Price (USD/lb)
Emulsifier	$ 0.72
Organophilic clay	$ 0.85
Fluid loss control	$ 1.12
Lime (Ca(OH)2)	$ 0.05
Calcium chloride (94%)	$ 0.16
Barite (BaSO4)	$ 0.05

Table 21 Drilling fluid additives cost[3]

Fluid Type	Price (USD/gallon)
Calophyllum M.E	$ 1.25
Jatropha FAME[3]	$ 1.85
Palm FAME[3]	$ 2.30
Synthetic oil[3]	$ 4.71
Diesel oil[3]	$ 3.58

Table 22 Base oil price comparison

CHAPTER 5
CONCLUSION

Sustainability and Feasibility Calophyllum inophyllum Ester Based Mud

- *Calophyllum inophyllum L, plant* as sustainable source of calophyllum oil, are widely distributed all over the world and are suitable as the source of alternative base oil stock supply for synthetic-based drilling mud.
- The production of calophyllum oil could be increased or decreased to follow the demand of base oil for drilling fluid in the future market.
- The production cost of the methyl ester is 0.28 USD/liter or 1.06 USD/Gallon. With that, Calophyllum methyl ester base fluid is the lowest cost compared with other benchmark base fluid in the market with the retail price in the market for calophyllum methyl ester is 0.33 USD/liter or 1.25 USD/gallon.

Rheological Behavior Study of Calophyllum Methyl Ester Based Mud

- On the rheological behavior study, calopyllum methyl ester based mud result showed that the rheological properties can be adjusted and modified to fulfill the requirement of the rheological properties specification given.
- In lime concentration addition effect, After hot rolling at 250^{o}F, in IF-1 the PV decreased significantly from 39 to 29 cP which indicates unstable viscosity, yet, in IF-4 PV increased slightly from 41 to 45 cp after hot rolled, which means more stable emulsion. It can be concluded that more lime concentration will enhance the stability of rheological properties BHR and AHR. This is due to flocculation effect was more retained in IF-4 (6 lb/bbl) causing the IF-4 was more stable during thermal degradation with stronger emulsion bond. IF-4 which has more lime concentration showed more flat and stable rheology in PV and YP BHR and AHR than those in IF-1 with lower lime concentration.
- The hydrolysis did not occur in the presence of lime in all mud sample after being subjected to heat aging test as there was no alcohol and acid observed at the surface of the sample. It showed good thermal stability of the emulsion of the mud system.
- In variation of mud weight and oil water ratio effect, IF-2 and IF-3 rheological properties were stable BHR and AHR. PV value BHR and AHR showed slight change which only differ 4 and 2 cp for IF-2 and IF-3, respectively. The slight change

indicates the strong thermal and rheological stability of mud in maintaining the rheological properties for IF-2 and IF-3.

- The mud system showed acceptable and stable rheological properties before and after being subjected to heat aging test under mud density up to 15 ppg and it can accommodate well of oil water ratio range tested. The higher mud density is possible to be applied for future use.
- In the case of organophilic clay concentration addition effect, the addition of gel additives or organophilic clay increased YP and GS simultaneously, due to increased gel content inside the mud system which flocculate the mud solid particles and bond it together. Thus, besides the mud carrying capacity increased, the hole cleaning ability was also increased. The mud system with 1 lb/bbl (IF-5) of organophilic clay was found to be more stable than 0.5 lb/bbl mud (IF-1) before hot rolling, as shown in figure 10. After hot rolled, higher organophilic clay concentration mud (IF-5) was less stable than IF-1. However, both mud samples showed good emulsion stability as ES value is more than 400 Volts as per API standard for ester-based mud.
- In high temperature and high pressure test/hot rolling test effect, the mud rheological properties result indicates that generally all mud samples were reported stable after being subjected to hot rolling test BHR and AHR. As the electrical stability (ES) test had shown the ES level more than 400 Volts, as shown in figure 8 to 10. This finding implies that before and after hot rolling test, most of the Calophyllum methyl ester-based muds are stable in terms of emulsion. This is due to the good formulation and correct concentration given for the mud additives into the all six (6) mud samples. It also can be stated that the mud compatibility issues were not found and observed.
- Calophyllum methyl ester based mud was proven to have good compatibility with common mud additives in field applications, as it showed good ES value and responsive rheological properties values towards the concentration change of mud additives.

Filtration Characteristics of Calophyllum Methyl Ester Based Mud

- In HPHT filtration loss characteristics, the results indicate that most of the mud sample were failed to fulfill the requirement of maximum filtration fluid loss according to API which is 15 ml. this is due to lack of fluid loss additives given in the mud systems. Based on the result, IF-1 and IF-6 were the only mud sample which fulfills the requirement of API with fluid loss of 14.0 and 10.0 ml, respectively.

- Based on the observation towards the presence of water in the filtrate prior to post-HPHT filtration test, all mud samples filtrate were reported to have no water in the filtrates. Based on this finding, it is concluded that the emulsions were very stable towards thermal degradation and high pressure conditions (HPHT). Thus, the water, alcohol and acids did not separate from the invert emulsion system.

Comparison Study of Calophyllum Methyl Ester Based Mud with the Benchmarks

- Calophyllum methyl ester based drilling fluid was proven to be technically comparable as other benchmark (Sarapar 147, Saraline 185V and Palm FAME drilling mud system) as shown in the rheological properties result, formulation of IF-6 mud sample with the value of Plastic Viscosity (PV) 39 and 53 cp, before and after hot rolling test, respectively with the range of specification of 45-55 cp after hot rolled. The Yield Point (YP) is 9 and 15 lb/100ft^2, before and after hot rolling test, respectively with specification range of 15-25 lb/100ft^2, and Gel Strength (GS) 10 minutes, 12 and 9 lb/100ft^2, before and after hot rolling test, respectively, with specification range of 20-40 lb/100ft^2.
- The rheological properties of calophyllum based mud had been fulfilled for PV and YP specification, yet for GS; it needs to be modified by adding the gel additives more. For the emulsion stability, IF-6 had been fulfilled the requirement of emulsion stability standard which is more than 600 Volts, the value of IF-6 Emulsion Stability (ES) is 940 Volts after subjected to hot rolling.

Environmental Assessment Test

- With the stricter government regulation on the drilling mud environmental impact, calophyllum methyl ester based mud can be alternative solutions to reduce the environmental impact.
- Calophyllum methyl ester based mud was proven to be environmentally acceptable for freshwater environment based on the acute toxicity test (LC50) result with the concentration below 10,000 ppm.

CHAPTER 6
RECOMMENDATIONS

- For future works, there is need to add more fluid loss control additive, since the filter loss is high on several mud samples (more than 15 ml).
- The addition of organophilic clay concentration affects exponential increment on yield point of the mud system. It can be used to increase capability to lift cuttings.
- Future works are required to be conducted to find the best modification of mud formulation to solve the existing imperfect rheological properties.
- Further and more detailed research on the cost and economic analysis are needed to be conducted to observe in more detail from production of calophyllum methyl ester until the application of it to be used as base oil in drilling fluid.
- The future work shall consider others plant derived ingredients in order to fulfill the demand of drilling mud based from vegetable oils. It might be derived from various sources of plants, such as corn, soy bean, rapeseed, etc.
- In order to be more environmentally friendly, mud additives that are used shall be from organic materials as well, future research is recommended to search for environmentally friendly mud additives to suit the wellbore condition in drilling operations.

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