EVALUATION OF PALM OIL BASED FATTY ACID FOR SYNTHESIS OF EMULSIFIER FOR DRILLING FLUIDS

BY

MUHAMMAD AFIQ BIN RAHIM

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BANDAR SERI ISKANDAR

31750 TRONOH

PERAK DARUL RIDZUAN

CERTIFICATION OF APPROVAL

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Muhammad Afiq bin Rahim

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Approved by,

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MS. RAJA RAJESWARY SUPPIAH

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
January 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.

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MUHAMMAD AFIQ BIN RAHIM

ABSTRACT

Achieving greater oilfield efficiency and productivity depends on wellsite operations that cost-effectively maximize recovery of oil and gas reserves, while minimizing the impact on the environment. In recent years, environmental issue on drilling operations always had been hot news. Drilling fluids that discharged to the environment give a big impact to marine life and the Mother Nature. The conventional drilling fluids used are not environmental friendly. Although the drilling fluids comply with certain rules and regulations, their increasing price has been a great concern. In order to tackle the issue, this project proposes the invention of a new emulsifier, which is a plant based to be incorporated into drilling fluids. The nature of plant and vegetable which is non-polluting and non-toxic will provide a better solution to overcome the environmental problem. Besides that, this vegetable based emulsifier is cheap and readily available. This new emulsifier is expected to enhance the lubricating properties and provides better stability at various drilling conditions.

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CHAPTER 1: INTRODUCTION

1.1 Project Background

Emulsifier is one of the essential components in drilling fluids during drilling operations. Emulsifier and other parts of drilling fluids such as base mud, weighting agent and additive are used to transport cuttings from the bore hole to the surface. Besides that, it is also used to cool, lubricate and support part of the weight of the drill bit and drill pipe. Nowadays, plant oil or vegetable oil is widely used to formulate the emulsifier according to their characteristics. It is found that the vegetable oil and palm oil which are biodegradable are possible to provide satisfactory high performance as a functional fluid in drilling process. The main thing is they are environmental friendly and good lubricants.

There have been several environmental issues on the hike lately due to consequences from drilling fluids. Among them are the spillages of large amount of lubricant to the environment. This result in environmental pollution due to non-biodegradable lubricants accumulated in the ecosystem. Therefore, the investigation on biodegradable synthetic base emulsifier extracted from palm oil will tend to reduce the environmental pollution.

1.2 Problems Statement

1. Current emulsifiers used in drilling fluids for drilling operations are mainly synthetic polymer. These synthetic polymers are extracted from pine wood and were added with some chemicals. The major problems in using this emulsifier are their low oxidative stability; relatively low viscosity and tendencies to solidify at low operating temperature as manifested by relatively high pour point.
2. Most emulsifier derivatives are bought from foreign countries. That's make the price of emulsifier is more expensive. Charges of these emulsifiers increase the

expenditure of operations onsite and low revenue will be gained. By producing local emulsifier, cost of drilling operation can be reduce and also enhance the revenue of productions.

3. Palm tree is one of the Malaysia's natural resources. It contains fatty acid which is the main component of emulsifier. However, no performance studies have been carried out. Through this study, palm tree can be commercialized for potential use of emulsifier and enhance the growth of Malaysia's economy.

1.3 Objectives

1. To obtain new derivatives and polyfunctional compounds acceptable as a high performance drilling mud.
2. To obtain optimum synthesis method by changing certain reaction parameters for the synthesis of a polyfunctional compound i.e. temperature of reaction, pressure and types of catalyst.
3. To develop palm oil based fatty acid emulsifier that is environment friendly.
4. To incorporate the available information into a screening-level ecological risk assessment for discharge of SBF cuttings to deep offshore waters.

1.4 Scope of Study

Scope of studies for this research is on mud properties which are to observe a new derivative and polyfunctional compounds acceptable as a high performance drilling fluids. Palm oil will be used as main component in this study. Basically, several laboratory analyses will be conducted to obtain the optimum synthesis method. Various parameters such as temperature of reaction, pressure and types of catalyst will be analyzed by mixing it with drilling fluid to obtain the best result.

CHAPTER 2: LITERATURE REVIEW AND THEORY

2.1 Drilling Fluid

Drilling fluid is one of most important components used in drilling operations. In oil and gas industry, drilling fluid is often called as drilling mud. The term of drilling fluid can be referred to as any number of liquids, gaseous fluids and mixture of fluids that used in the rotary drilling process while drill the borehole in the earth. The functions of drilling fluid can be divided into two groups which are major functions and minor functions:

Major functions:

1. Drilled-cuttings removal – beneath the drill bit and from the annular space around the drill string.
2. Containment of subsurface formation fluid pressures.
3. Hole stabilization – prior to casing/cementing.

Minor functions:

1. Cooling and lubrication of drill string and drill bit
2. Suspension of desired solids and ease of removal of undesired solids
3. Reduction in weight of casing string and drill string while suspended in wellbore
4. Aid in formation evaluation
5. Cleaning of drill bit.

Generally, most of the drilling problems are related to the drilling fluid that being used. Drilling Engineer is the person who is responsible to estimate the parameters of drilling fluid during drilling operations. The failure estimation of drilling fluid in the operations may lead to costly drilling problems. Hence, the proper selection of drilling fluid is vital to provide a beneficial operation. Below are the benefits of the proper selection of drilling fluid:

1. Minimum formation damage
2. Minimum corrosion effects on drilling equipment
3. Enhancement in rate of penetration (ROP)
4. Minimum environmental impact
5. Enhancement in safety issues.
6. Reduction in friction pressure losses.

2.2 Emulsion

Emulsion drilling fluids is an emerging new technology in the drilling process. It is very useful in order to enhance the performance of drilling in oil and gas industry. The emulsion drilling fluids was firstly introduced in June 1943 at Shell Carribean Petroleum Company's well MG-470 in the Mene Grande field of Western Venezuela [1]. After that, it has been developed and widely used in the drilling operations. Until now, it is one of the vital components in drilling fluids.

According to Schlumberger oil glossary, emulsion is defined as a dispersion of one immiscible liquid into another through the use of a chemical that reduces the interfacial tension between the two liquids to achieve stability [2]. Emulsion is not only used in oil and gas industry but it is also has been used in other industries such as food and cosmetics industries. There are six advantages of using emulsion drilling fluids:

1. Lower fluid losses
2. Holes are closer to gauge
3. Greater drilling speed
4. Lower drill pipe torque
5. Better lubrication
6. Easier control.

There are two types of emulsion which is oil-in-water (O/W) and water-in-oil (W/O). Usually in oil and gas terms, oil-in-water (O/W) emulsion is called 'emulsion mud' and water-in-oil (W/O) is called 'invert emulsion mud'. These two types of emulsions are different in the functionality but same way on the process making. Based on Bancroft's rule, emulsion type depends more on the nature of the emulsifying agent compared to the relative proportions of oil or water present and also compared to the methodology of preparing emulsion. In the oil-in-water emulsion, emulsifying agents are more soluble in water than in oil and in the water-in-oil emulsion, emulsifying agents are more soluble in oil compared to water. Hence, the phase in which an emulsifier is more soluble constitutes the continuous phase. The emulsion types can be tested by dilution test, conductivity test, dye-solubility test, refractive index measurement and also filter paper test.

2.2.1 Oil-in-water emulsion (O/W)

Oil-in-water emulsion is the condition which oil is present as the dispersion phase or minor substance and water is present as the dispersion medium or the major substance (continuous phase). This process is dependent on the type of emulsifier, and not the oil-to-water ratio. According to the research by Eric Dickinson, oil-in-water emulsions are stabilized by two main types of molecular emulsifying agents: small-molecule surfactants and water-soluble polymers. The stabilizing layer influenced the crystallization behavior of the dispersed phase and the kinetics of mass transport between droplets [3]. There are research works that been conducted found that the existence of an empirical relationship between the drilling rates obtained the faster drilling rates through use of oil-in-water emulsion drilling fluids, when the surface tension of the filtrate has a reduced value [4].

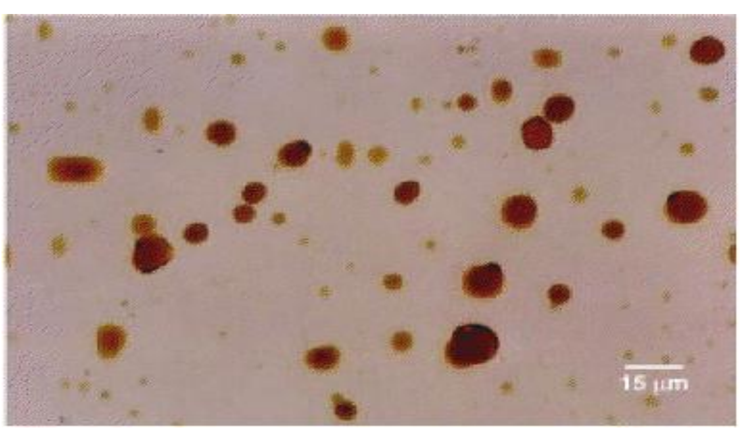


Figure 1: Photomicrographic of an oil-in water emulsion

2.2.2 Water-in-oil emulsion (W/O)

Water-in-oil emulsion is the condition which water is present as the dispersion phase or minor substance and oil is present as the dispersion medium or the major substance (continuous phase). This process is dependent on the type of emulsifier, and not the water-to-oil ratio. Formulating water-in-oil emulsion is more difficult compare to oil-in-water emulsion. People often said that emulsion must be water-in-oil emulsions when the water phase is added to the oil phase. It is not true because the amount of water and oil is basically same whether it is oil-in-water or water-in-oil. As a rule of thumb, the amount of water is always approximately 60%-80%. The type of emulsifier will determine the type of emulsion. Water-to-oil emulsion drilling fluid is used to avoid the formation damage arising from the use of aqueous and oil-in-water emulsion drilling fluids and yet retain advantages of the oil-in-water and the drilling fluids whose fluid phase consist entirely of oil. However, water-in-oil emulsion drilling fluids is frequently unstable and tend to break or to revert to oil-in-water emulsions [5]. Besides that, water-in-oil emulsion drilling fluids also can be used as the treatment of oil-producing formations.

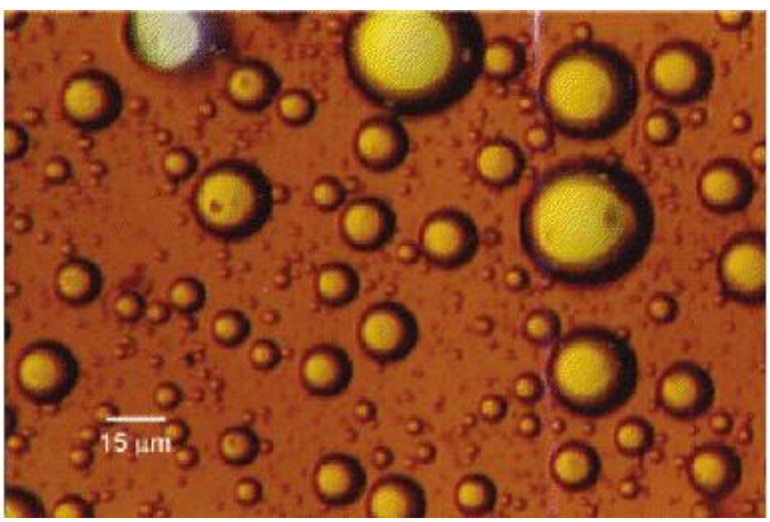
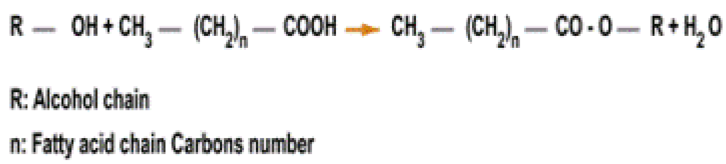


Figure 2:Photomicrographic of a water-in-oil emulsion

2.3 Emulsifier

Emulsifier could be classified as a substance or agent of chemical that stabilize a physical emulsion once it is formed by increasing the kinetic energy. It is important to have emulsifier in the emulsion formulation because these emulsifier agents will determine whether the emulsion is an oil-in-water emulsion drilling fluids or water-in-oil emulsion drilling fluids.



Molecular structure of emulsifier

Fundamentally, emulsifier will be added in the oil based mud or synthetic based mud. It is to prevent the water droplets from coalescing and settling out of the formation. Besides that, it is also permits water originally present in the rock destroyed by the bit to emulsify easily [6].

Emulsifier does works by standing on the boundary between the continuous oil phase and the water droplet or solid particles. Then, emulsifier molecules preferentially are an “oil wet” to the face of the wellbore, drill cuttings and all steel surfaces. There are three main functions of emulsifier in emulsions drilling fluids:

1. Imparting weak gel strength to oil mud’s because gel strength is necessary for suspension of weighting materials
2. Emulsification of any water picked up during drilling operations
3. Controlling the tightness of any water emulsion resulting from water contamination, thus, controlling the fluid loss in the wellbore

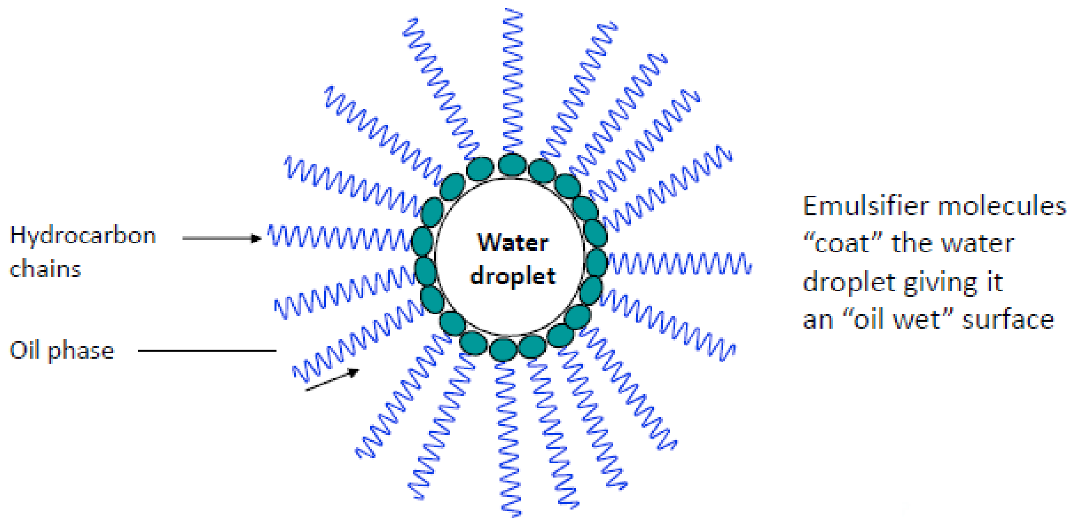


Figure 3: Emulsifier molecules

2.4 Based Mud

Many types of drilling fluids have been used in the oil and gas industry. Main type includes air, water and oil based drilling fluids. Water based mud are the most commonly used drilling fluids. Water based mud is the mud in which water is the continuous phase. It consists of four basic phases. There are active colloidal solids, water, inner solids and also the chemicals. In water based mud, clay is also added to increase density, viscosity, gel strength and yield point, and also to decrease fluid loss.

There are two types of water based mud; non-inhibitive water based mud and inhibitive water based mud. The non-inhibitive water based mud contains of spud mud, clear water mud, bentonite-treated mud, and lignite / lignosulfonate mud. The non-inhibitive water based mud is least expensive and are easy to maintain but their applications ceases when it encounter high-temperature formations, dispersive formations or formations that may contain certain contaminants such as H_2S. Hence, some researcher has found the inhibitive water based mud. This type of mud is form by the controlled contamination of clay/water mud by electrolytes or deflocculants. The function is to resistant subsequent contaminants encountered during the drilling operation [6].

Although water based mud is widely used but the disadvantages do exist. So, to overcome the problem, oil based mud is used. Oil based mud is defined as a system which have oil as the continuous phase. Typically, diesel oil, mineral oil and low toxicity mineral oil is the most selected oil [7]. Because of water present in the formation, a chemical emulsifier must be added to avoid the water droplets from coalescing and settling out of emulsion. The most common applications for oil mud are drilling in deep hot formations (>300 degree F), drilling formations containing H_2S and CO_2, corrosion control, drilling weak formations or subnormal pore pressure and preventing or freeing stuck pipe.

Moreover, according to the present of water in the oil based mud, the invert emulsion has been introduced. Invert emulsion change the major phase of formation from oil to water. By adding solvents and other surfactant washes, it will convert the oil-wet solids of the filter into water-wet solid. The water-wet solids in the filter cake are necessary so that the subsequent acid wash can attack the particles of the mud cake and remove them prior to production [8]. The problem is surfactant in invert drilling fluid system can complicate the cleaning process in open hole completion operations.

The main disadvantage of using oil based mud is environmental pollution. Oil based mud have a problem in disposal of the oil-contaminated drill cuttings, high toxicity liquids and not readily biodegradable by microorganism. These properties of oil based mud is abuse the environmental guidelines. Hence, the strict country will avoid of using this based mud for drilling operations.

CATEGORY	CONCENTRATION (LC50)
Non-toxic	> 100,000 mg L-1
Almost Non-toxic	10,000 - 100,000 mg L-1
Slightly toxic	1,000 - 10,000 mg L-1
Moderately toxic	100 - 1,000 mg L-1
Toxic	1-100 mg L-1
Very toxic	< 1 mg L-1

Table 1: Toxicity rating classification system using DoIR

The inventor then produced a better environmental based mud to replace the oil based mud which is synthetic based drilling fluids. Synthetic based drilling fluid is a new class of drilling fluids that are particularly useful for deepwater and deviated hole drilling. This new based mud is combination of technical advantage of oil based mud and water based mud. The continuous phase of synthetic drilling fluids is a well-characterized synthetic organic compound. Salt brine usually is dispersed in the synthetic phase to form an emulsion. Synthetic based drilling fluids contain the same metal as water based mud [9].

Even though the synthetic based drilling fluids is better environmental friendly compare with oil based mud but still it has an environmental pollution problems. Usually, bulk synthetic based drilling fluids are not discharged into the ocean. However, during drilling operations, the cuttings will be discharged to the ocean. It will give pollution to the marine habits. The main environmental concern resulting from the discharge of synthetic based drilling fluids cuttings to deep offshore waters is that the cuttings may accumulate on the sea floor and adversely affect the benthic communities living. Adverse effects on benthic communities may occur as a result of the toxicity of synthetic based drilling fluids cuttings ingredients, organic enrichment of sediments from biodegradation of organic matter in the synthetic based drilling fluids cutting, direct smothering of benthic fauna by the accumulation of cuttings solids on the sea floor, and alteration of sediment texture and physical/chemical properties, rendering the sediments less suitable for some species and more suitable for others [9].

In order to overcome the environmental problems and enhance the quality of based mud, many researches have been done. The idea is using vegetable or plant oil as the based lubricant. Hence, there are some researches carried out on the plant oil or vegetable oil based lubricants. According to the early work done by Sevim Z. Erhan, Brajendra K. Sharma and Joseph M. Perez on oxidation and low temperature stability of vegetable oil-based lubricants, they targeted to improve the oxidation and cold flow behavior using a synergistic combination of additives in high-oleic vegetable oil blended with synthetic fluid. The additives used converts the hydroperoxides formed during the oxidation process to non-radical products, thus preventing chain propagation. This leads for the vegetable oil to easily tumble and glide over one another resulting in better fluidity of the total matrix.

Along with the advantages, there have traditionally been a number of technical concerns with the performance of vegetable oil-based lubricants. The two main issues are operational temperature limitations and oxidative instability. In cold weather, vegetable oils have the tendency to solidify more readily than do petroleum products, giving them a higher pour point (the temperature at which the lubricant can still be poured). In conditions of extreme heat, they are subject to oxidation, a chemical reaction that causes the oil to break down and reduces its functioning capacity. In addressing these concerns, researchers have developed a number of plant-based lubricants that meet or exceed the performance expectations of petroleum lubricants. Current approaches include reformulating additives, chemically modifying the vegetable base oil and genetically modifying the oilseed crop.

Related to the vegetables oil issue, Yeong Shoot Kian and his friends in their paper have proposed of using by-product of polyol ester based palm olein to be used as lubricant for hydraulic fluids. Palm olein is a liquid fraction obtained when the crude palm oil is fractionated and refined into the solid and liquid fractions. In general, palm oil contents higher saturation fatty acid. Hence, fractionation is performed in order to reduce the contents of saturation fatty acid. Normally, palm olein has higher Iodine Value (IV). Iodine Value is used as an indication of the degree of unsaturation. They target in the invention was to produce palm olein having higher IV, high in tocopherol and tocotrienol content (800-1500ppm), low in free fatty acid (FFA) (<0.5%), high oleic (46%) and low linoeic and linolenic (16%) content. According to the target, this lubricant is suitable to be used only in tropical region such as Malaysia, Indonesia, Brazil or any other countries which have the same climate.

From the combination between the natural oil and the polyol ester, it results in better properties due to the synergetic effect. One of the advantages is that the resultant oil has a lower pour point and better oxidative stability compare with the natural vegetable oil. Besides using the polyol ester, other polyols also can be used such as neopentyl glycol, trimethylol propane and dipentaerythritol. It must be tested at different molar ration. Perhaps, it could give a better outcome.

Another research related with palm based polyol esters was done by (Yunus et al., 2002) with the title “A Simple Capillary Column GC Method for Analysis of Palm Oil Based Polyol Esters”. Polyol esters (PE) are products of transesterification of fatty acids (FA) or FA esters with a polyol such as trimethylolpropane (TMP). PE is the base oil for various lubricants. The analysis of palm oil polyol esters can be done using capillary column GC. The polyol esters were esterified from palm oil methyl esters (POME) and palm kernel oil methyl esters (PKOME) with trimethylolpropane [2-ethyl-2- (hydroxymethyl)-1,3-propanediol; TMP] to produce the base oil for lubricant production.

Analysis was performed using a high-temperature capillary column, SGE HT5 operated at a temperature gradient of 6 °C/min starting from 80-340 °C. This procedure provided a complete separation of reaction product: TMP, methyl ester, monoesters (ME), diesters (DE), and trimesters (TE). This separation can be achieved through TLC separation of ME and DE spots. GC analysis could be used to monitor the progress of the transesterification reaction. The proposed GC procedure described has several advantages, which allowed simultaneous detection of all compound involved in a reaction, which are ME, DE, TE, methyl esters and TMP.

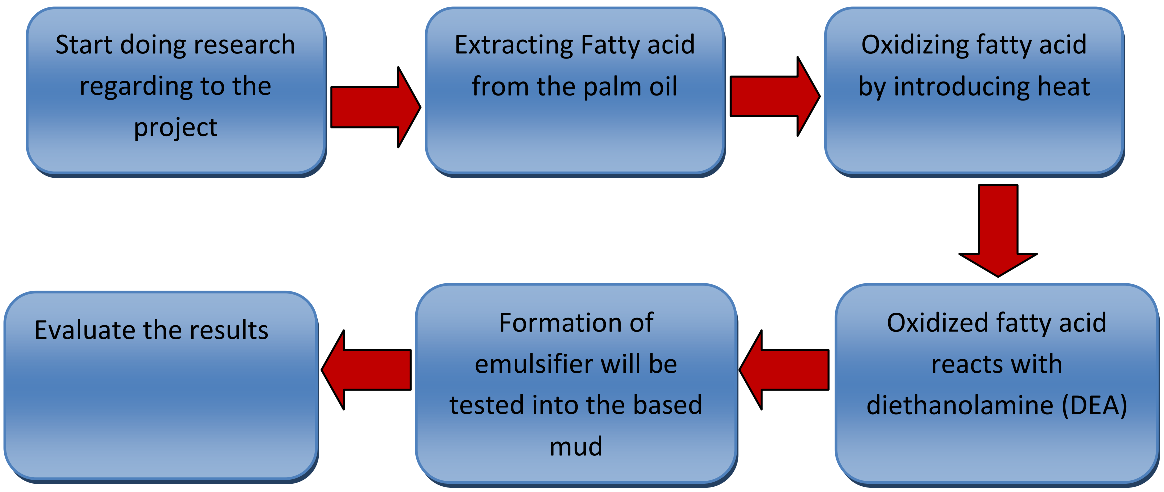
Moreover, Ismail Ab Raman and his team have done an analysis on changing the co-surfactant in palm oil-based microemulsion for practical used. Microemulsion is thermodynamically stable solution, low in viscosity and small in the droplet size (<100 nm). Compare to the standard emulsion which consist of higher viscosity, bigger droplet size (>5µm) and are only kinetically stable, the microemulsion is much better. In the invention, they used glycerol derivative to replace the conventional co-surfactant which is 1,2-hexanediol. The glycerol derivative contains of 55%-60% glycerol *mono*-tert-butyl ether and 30%-35% unreacted glycerol. In their study found that with using of 10%-15% (w/w) glycerol *mono*-tert-butyl ether (≥60% diol), a palm oil-based microemusion could be form.

This new invention gives a lot of advantages towards the industry. It could cut a big amount of money because of the price of glycerol tert-butyl ether is much cheaper. In addition, the new microemulsion provides a good cleaning performance, longer shiny effect and also can be added with oil-soluble active ingredients which can enhance the value-addition of products.

Figure 4: Palm-microemulsion liquid cleaner

CHAPTER 3: Methodology

3.1 Research Methodology



Project methodology

1. Before initiating any project regarding the emulsifiers of drilling fluids in drilling operations, the first step taken is to analyze the previous research regarding emulsifier which had done by several researchers. This is to improve understanding about the characteristics of the emulsifier.
2. Experiment of producing new emulsifiers will start by extracting the fatty acid from the palm oil. Fatty acid is the main components of emulsifiers.
3. The fatty acid then will be modified to suite the application of emulsifier in based mud.
4. The next step is oxidizing the fatty acid by using the special equipment.
5. After oxidizing process completed, the fatty acid will be synthesized by reacting with DEA. Catalyst will be added to the emulsifier only when it required.
6. Lastly, the performance of the new emulsifier will be tested to several types of based mud and comparison will be made to conclude the best results. Further study or clarification can be done depending on the result obtained from the experiments conducted.

3.2 Project Activities

3.2.1 Emulsifier Formation Test

Objective: To overcome a new emulsifier of the drilling fluids by using palm oil based fatty acid.

Theory: Emulsifier is a chemical substance/agent that stabilizes an emulsion by increasing its kinetic stability.

Procedure:

1) Place some amount of the extracted fatty acid into the reactor.
2) Do oxidize the fatty acid by introducing the heat.
3) Change the temperature of reactor to produce various results.
4) React the oxidation fatty acid with diethanolamine (DEA).
5) If necessary add catalyst to obtain better formation.

3.2.2 Mud Density Test

Objective: To determine density of the drilling fluids.

Theory: The density of the drilling fluids must be controlled to provide adequate hydrostatic head to prevent influx of formation fluids.

Procedure:

1) Instrument base must be set on a flat level surface.
2) Measure and record the mud temperature.
3) Fill the mud cup with the mud to be tested. Gently tap the cup to encourage any entrapped gas to break out.
4) Replace cap and rotate until it is firmly seated, ensuring some of the mud is expelled through the hole on top, to free any trapped gas.
5) Holding cap firmly (with cap hole covered with thumb) wipe the outside of the cup until it is clean and dry.

6) Place the beam on the base support and balance it by using the rider along the graduated scale. Balance is achieved when the bubble is directly under the centre line.

3.2.3 Funnel viscosity Test

Objective: To measure plastic viscosity, yield point and gel strength of the drilling fluids.

Theory: The viscosity is defined as its resistance to flow. It is important because in drilling operation it influenced several factor such as mud density, pumping rate and pressure system.

Procedure:

1) Cover the orifice with a finger and pour a freshly agitated fluid sample through the screen into the clean, dry and upright funnel until the liquid level reaches the bottom of the screen.
2) Quickly remove the finger and measure the time required for the fluid to fill the receiving vessel to the one quart (946 ml).
3) Report the result to the nearest second as Marsh Funnel viscosity and the temperature to the nearest degree.

3.2.4 Rheology Test

Objective: To determine single or multi-point viscosity.

Theory: Viscosity is measured as the shearing stress to the rate of shearing strain. There are two types of fluid characterized which is Newtonian fluid and Non-Newtonian fluid.

Procedure:

1) Stir the sample at 600 rpm while the sample is heating, or cooling, to 120˚ F. Ensure the dial reading has stabilized at this speed before noting the result and proceeding to the 300, 200, 100, 6 and 3 RPM speeds.

2) Having taken the 3-RPM reading stir the sample at 600 RPM for 30 secs before taking the 10-second gel at 3 rpm.

3) Restir the sample at 600 rpm for 30 seconds and leave undisturbed for 10 minutes, ensuring the temperature stays at 120˚ F. Take the 10 minute gel reading at 3 rpm.

3.2.5 Retort Analysis Test

Objective: To determine quantity of solid and liquid of mud from the evaporation of weighted sample mud.

Theory: Knowledge of liquid and solid content in mud is essential for good control of mud properties. By this testing, poor performance of mud can identify and can be solve by provide a treatment.

Procedure:

1) Ensure retort assembly to be used is clean and dry. It is vital that all traces trof previously retorted solids are removed from the retort cup to guarantee 50 ml of fluid is actually retorted. Remove all traces of previously used steel wool. Water can be retained in steel wool when the upper retort body is washed / cleaned. Failure to change the steel wool can result in inaccurate measurements, as this extraneous water will become included in the total water content.

2) Weight the clean and dry retort cup and lid on the triple beam balance.

3) Add the mud, which has been allowed to cool to ambient temperature, to the retort cup, gently tap the cup to remove any air bubbles and place the lid with a rotational movement to obtain a proper fit. Be sure an excess of fluid flows out of the hole in the lid.

4) Carefully clean the cup and lid of excess fluid and reweigh on the triple beam balance. The retort mud weight SG is determined as the difference between the empty and full weights, in grams, divided by 50 (the volume of mud).

5) Pack the retort body with new steel wool, apply Never– Seez, to the threads and assemble top and bottom parts. Ensure that the two parts are fully screwed together. If it is not possible to fully screw together the two parts, it will be necessary to clean the

threads and repeat the above steps. Failure to get a good seal could result in leakage that will lead to an inaccurate result.

6) Attach the condenser and place the retort assembly in the heating jacket and close the insulating lid.

7) Place clean, dry liquid receiver below condenser outlet and turn on heating jacket.

8) The temperature control should be adjusted so that the retort cell glows dull red at the end of the distillation and the final drops coming out of the retort should be observed to be black (the heavy fraction) and sink through the clear oil to the meniscus. Ultimately smoke will emerge from the retort and the distillation is only complete when the smoke stops.

3.2.6 HTHP Filtration Test

Objective: To testing drilling fluids at elevated temperature and pressure.

Theory: Filtration control is essential to prevent differential sticking in the formation which minimization of formation damage.

Procedure:

1) To standardise this test the following procedure must be adhered to. Backpressure is applied during the test to avoid filtrate evaporation.

2) Allow the heating jacket to reach the required temperature.

3) Check out all the "O" rings on the HPHT bomb and lid. Change out any damaged rings. The rings to be checked are the four small stem "O" rings, which tend to pick up cuts and grooves with time, and the two large "O" rings, one in the lid and one in the cell. The large "O" rings should have a rounded profile and be free from dirt.

4) Tighten the bottom valve stem, taking care not to over tighten, and fill the cell to about 0.5 inch from the rim.

5) Place a filter paper on the rim and put the lid on the cell. Ensure the lid stem is open while doing this to avoid damaging the filter paper.

6) Tighten the six studs in the bomb and close the lid stem.

7) Place the bomb in the heating jacket with the lid facing downwards. Rotate the bomb until it seats on the locking pin.

8) Place a CO_2 cartridge in each regulator and tighten up the retainers.

9) Place the top regulator on the stem and engage the locking pin. Close the bleed off valve and turn the regulator clockwise until 100 psi is showing on the gauge.

10) Repeat the process with the bottom regulator.

11) Turn the top valve stem 1/4 to 1/2 turn anti clockwise to pressure up the cell to 100 psi.

12) When the cell reaches the required test temperature open the bottom stem (1/2 turn) and then increase the pressure on the top regulator to 600 psi over +/- 20 seconds.

13) Commence the test. The test should be carried out as soon as the bomb reaches the test temperature. Leaving the cell for long periods at high temperatures will produce unreliable results.

14) If the pressure on the bottom regulator increases significantly above 100 psi bleed off some of the filtrate into the graduated cylinder.

15) After 30 minutes, close the top and bottom valve stems. Slack off the regulator on the bottom collection vessel. Bleed off the filtrate into the graduated cylinder. Disconnect bottom collection vessel, fully open the bleed off valve and tip any residual filtrate into the graduated cylinder.

16) Bleed the pressure off the top regulator.

17) Disconnect the top regulator and remove the bomb from the heating jacket, allowing it to cool in a safe place.

18) When the bomb has cooled bleed off the trapped pressure by slowly opening the top valve with the bomb in an upright position. With the residual pressure bled off invert the cell, loosen the six studs and remove the lid.

19) Examine the filter paper and report the thickness in 32nds of an inch**.** Comments about the quality of the cake should be noted in the comments section of the mud report i.e. tough rubbery etc.

20) Thoroughly clean the bomb and stems in preparation for the next test.

21) **Do not** preheat the bomb by resting on the heating jacket.

3.2.7 Emulsion Test:

Objective: To indicate the stability and type of emulsion in mud.

Procedure:

1) Before placing the probe in the mud, it is essential to test the meter in air. The reading should go off scale and the display start flashing. If the meter does not go off scale, it is an indication that the probe is shorting out due to an accumulation of detritus between the two prongs. It is clear that the probe can short out before the end point of the mud is reached and an erroneous reading will result. The probe should be carefully cleaned and retested in air to ensure that it now goes off scale before testing the mud.

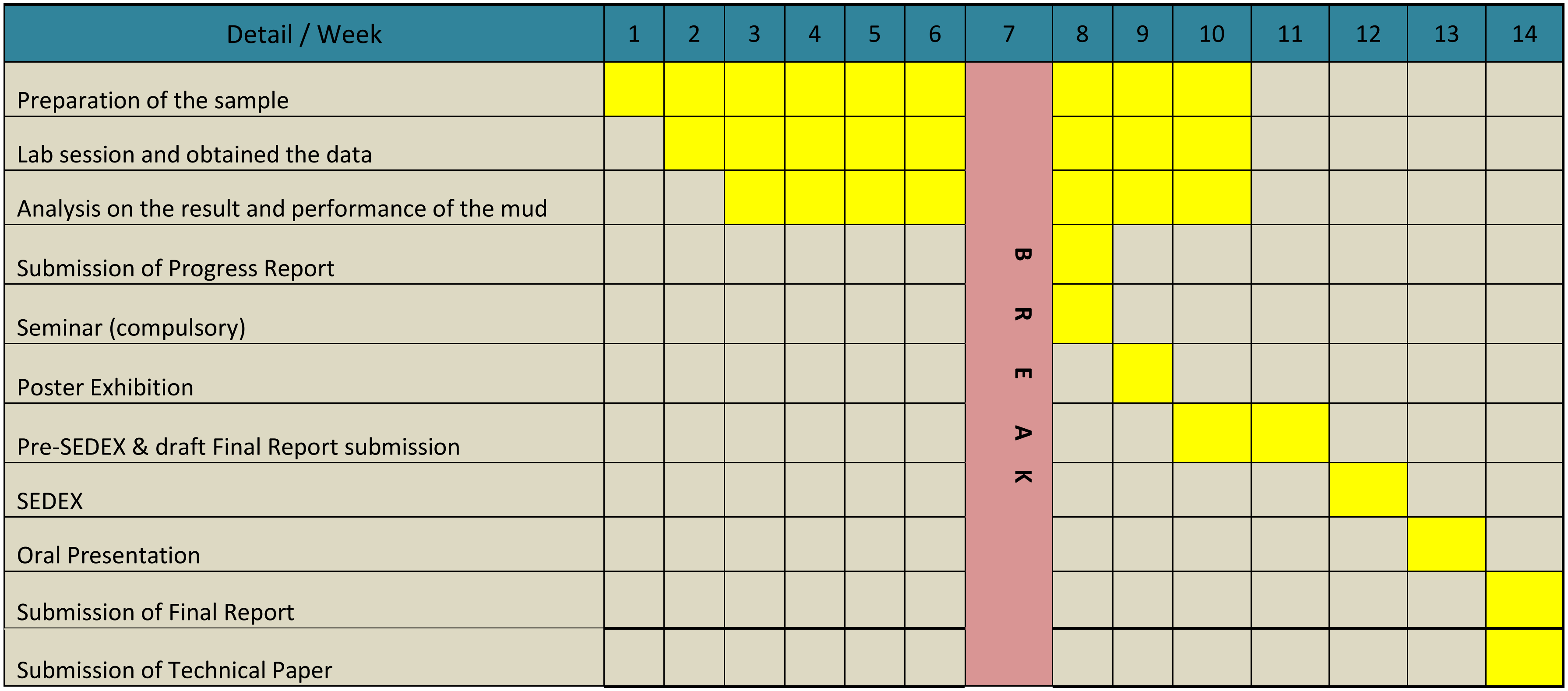
2) Place the clean and checked probe in the sample at 120˚ F and use it to stir the fluid to ensure homogeneity. Position the probe so it does not touch the bottom or sides of the heated cup, ensuring the tip of the electrode is completely immersed.

3) Press the button to initiate the voltage ramp, holding the probe still until the end point is reached and a steady reading is seen in the digital display. Note the reading.

4) Repeat the test. The two ES values should be within 5% and anything greater would indicate a problem with the equipment.

5) The result is the average of the two readings.

3.3 Gantt Chart



CHAPTER 4: RESULT AND DISCUSSION

The experiments were initiated by preparing the new sample of emulsifier. This sample was prepared by reacting palm oil based fatty acid with diethanolamine (DEA) to produce a new type of emulsifier. In this experiment, the palm oil based fatty acid with carbon chain C_8-C_{18} has been used as raw material and DEA has been used as the additive agent. 500 ml of palm oil based fatty acid has been added with 50 ml of DEA in a container of reactor. Then, the container is heated until it reaches 100 degree Celcius. During the heating process, the liquid is always be stirred to make sure the reaction is consistent. After that, air has been purged into the container. The temperature is maintained and the liquid is always be stirred. This process is continued for 30 minutes. The new emulsifier from palm oil based fatty acid is in the figures below.

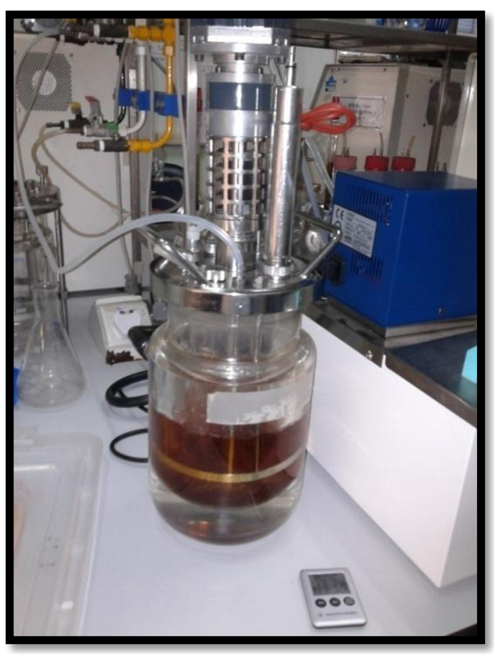


Figure 5: Modified bioreactor

Figure 6: Oxidation process of palm oil based fatty acid by using reactor

Figure 7: New emulsifier from palm oil based fatty acid

Although the sample of new emulsifier is obtained, it is still not followed the planning. As the planning before, the new emulsifier must be produced same like the conventional emulsifier in term of parameters, carbon chain, process, etc. But according to the some restriction, there are some modification has been made. For examples, the carbon chains preferred to use before is C_{22}-C_{38} but it is difficult to find it because basically for standard carbon chains for palm oil fatty acid that produced to industry is C_8-C_{18}. It will take a long time and high cost if still wants to use that carbon chains.

Besides that, the heating temperature of reaction also been reduced due to the limitation of the reactor. For this experiment, the modified bioreactor has been used to replace the chemical reactor. This reactor can only support the heating temperature until 100 degree Celcius because it using water as the heating medium. According to the research done, heating temperature of complete reaction for fatty acid is 157 degree Celcius and above. The experiment was continued by using temperature 100 degree Celcius even though the result will be degraded or not good like conventional emulsifier.

After producing the new sample of emulsifier, the experiment then continued by testing the new emulsifier into mud. The objective of this testing is to evaluate the performance of mud drilling fluids by using this sample as emulsifier. Some others SCOMI's products also been added into the mud to provided the requisite concentrations. The products that used in the test were tabulated as below:

Product Name	Function
SARALINE 185V, SARAPAR 147 & ESCAID	Base oil
CONFI-GEL HT	High temperature viscosifier
CONFIMUL-P	Primary Emulsifier
CONFIMUL-S	Secondary Emulsifier
CONFI-TROL XHT	Very high temperature fluid loss control additive
Lime	Alkalinity buffer
Barite	Weighting agent

Table 2: SCOMI's Products

The first test was conducted by using SCOMI's products which are CONFIMUL-P and CONFIMUL-S. These emulsifiers will be the main emulsifiers to compare with the new sample of emulsifier later. CONFIMUL-P and CONFIMUL-S is common emulsifiers that SCOMI used in the formulation of mud drilling fluids. These emulsifiers have been used in normal condition and also in high temperature and high pressure condition. For this test, the emulsifiers have been tested in high temperature and high pressure condition (300 degree F/ 500 Psi). To obtain the mud density needed which is 11.5 lb/gallon, the emulsifiers has been added in the formulation as shown in the Table 3. The concentrations of chemicals in this test have been made constant to make an easy comparison. The fluids properties specifications of high pressure and high temperature condition were determined based on the laboratory request that usually asked by the clients. The specifications are shown on Table 4.

Products	Sample W	Sample X	Sample Y	Base Sample	Sample Z
Base oil -SARAPAR 147(g)	155.03	154.79	154.66	157.31	156.18
CONFI-MUL P (g)	0.00	3.00	0.00	3.00	0.00
CONFI-MUL S (g)	9.00	0.00	0.00	9.00	9.00
Palm oil based fatty acid emulsifier (g)	3.00	9.00	12.00	0.00	3.00
CONFI-GEL XHT (g)	8.50	8.50	8.50	8.50	4.00
CONFI-TROL XHT (g)	8.00	8.00	8.00	8.00	8.00
LIME (g)	8.00	8.00	8.00	8.00	8.00
Drillwater(g)	67.11	67.01	66.95	67.23	67.61
CaCl2 (g)	26.85	26.80	26.78	26.89	27.04
DRILL-BAR-barite (g)	197.70	198.10	198.30	195.26	200.36

Table 3: Mud Formulation

Fluid Properties	Specification
Mud density, SG	11.5
6 rpm dial reading	8-16
Yield point, $lb/100ft^2$	15-25
10 sec gel strength, $lb/100ft^2$	8-18
10 min gel strength, $lb/100ft^2$	15-30
HTHP @ 300°F, 500 psi, ml/30min	<4
Electrical stability, volt	>400

Table 4: Fluids properties specifications

After preparing the base sample of mud, the next testing is to evaluate the function of new emulsifier in the drilling fluids. Basically in common test, SCOMI has used two types of emulsifier which are CONFIMUL-P and CONFIMUL-S. CONFIMUL-P is function as primary emulsifier and CONFIMUL-S as secondary emulsifier. Three samples with different formulation have been prepared for the testing. Sample W, the new emulsifier has been tested as primary emulsifier; second sample (Sample X), the new emulsifier has been tested as secondary emulsifier and Sample Y, the new emulsifier has been tested as primary and secondary emulsifier. The formulation has shown in Table 3.

The results of these three samples then were compared with the base sample. The first specification that been compared is emulsion stability test. This purpose of this test is to measure the ability of the emulsifier to stabilize the emulsion in the mud drilling fluids. The results were shown in the chart below:

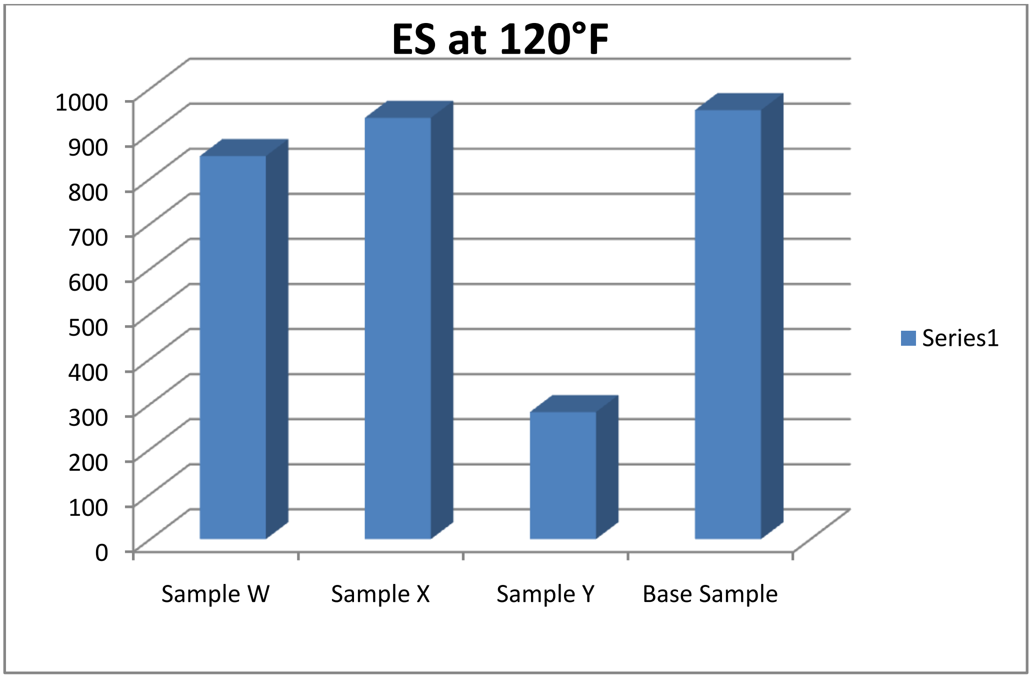


Chart 1:Emulsion Stability

The results show that formulation of Sample Y is not suitable for mud drilling fluids because the ES reading is below the specification. Sample W and X show good emulsion stability but not good as base sample. Then, the testing were proceeds to rheology test and fluid loss filtration test. For Sample X, the rheology test could not be done because the mud had solidified like a gel. Even though the mud had been mix in the mixer to liquefied to liquid in 1 hour, but after 10 minutes later it has solidified again. The picture of Sample X is shown in Figure 8, 9 and 10.

Figure 8: Sample X after hot rolled

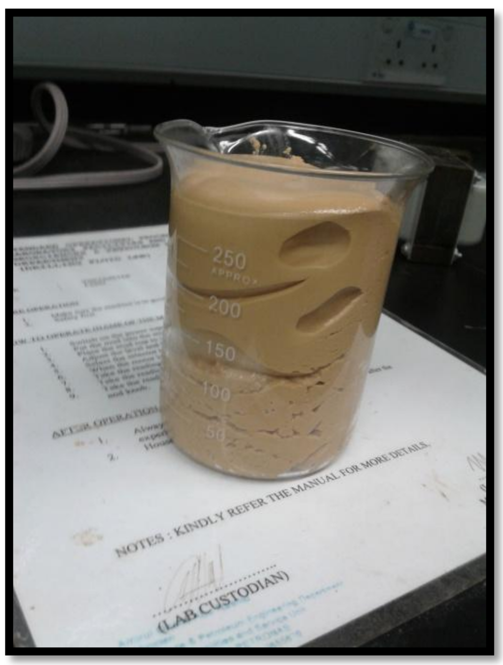


Figure 9: Sample X after 10 minutes (i)

Figure 10: Sample X after 10 minutes (ii)

The rheology test then proceeds by using Sample W. Based on the result, the parameter of plastic viscosity (PV), yield point (YP) and gel strength are too high and exceed the specification given. Hence, it could not be used in the fields. The results show that the mud formulation of base sample is not suitable to use for new emulsifier. Some modifications of mud formulations have been made to obtain the better results. The mud formulation is shown in Table 3.

The results of new mud formulation sample (Sample Z) are tabulated in table 5. The results are then used to compare with Sample W and base sample.

After hot rolling at 50C for 16h	SPEC	Sample W	Base Sample	Sample Z
Mud density, lb/gal		11.5	11.5	11.5
Rheological properties		120 °F	120 °F	120 °F
600 RPM		357	84	96
300 RPM		318	59	65
200 RPM		295	46	52
100 RPM		283	24	37
6 RPM	>10@150	158	18	19
3 RPM		125	15	17
PV, cP	<30	39	25	31
YP, lb/100 ft^2	15-25@ 120	279	34	34
Gel 10 sec, lb/100 ft^2	8-18	110	17	19
Gel 10 min, lb/100 ft^2	15-30	128	22	30
HTHP, cc/30min at 300F	<4	3.6	4	3.2
ES, volts at 120 °F	> 400	850	952	644

Table 5: Results after hot rolled

The plastic viscosity is calculated by using equation:

$$Plastic\ viscocity, PV = Reading(600rpm) - Reading(300rpm)$$

And Yield Point is calculated by using equation:

$$Yield\ Point, YP = Reading\ (300rpm) - Plastic\ Viscosity(PV)$$

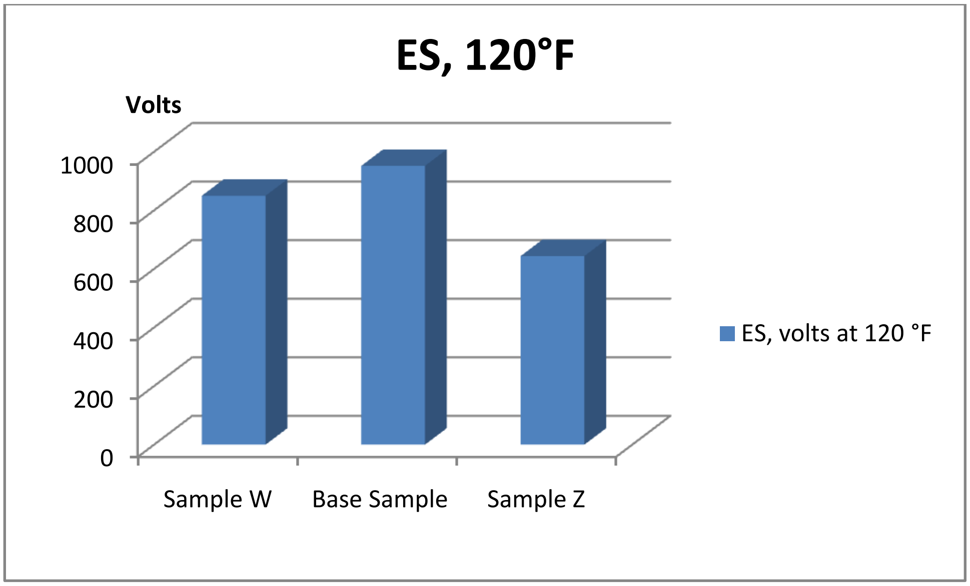


Chart 2: Emulsion Stability

ES value shows the emulsion stability of the mud. The higher the reading, the better results emulsion stability of the mud. From the test, base sample is the most stable emulsion compares with Sample W and Sample Z. However, all of these samples could be used in field in terms of emulsion stability because they were exceeded the fluid properties specifications.

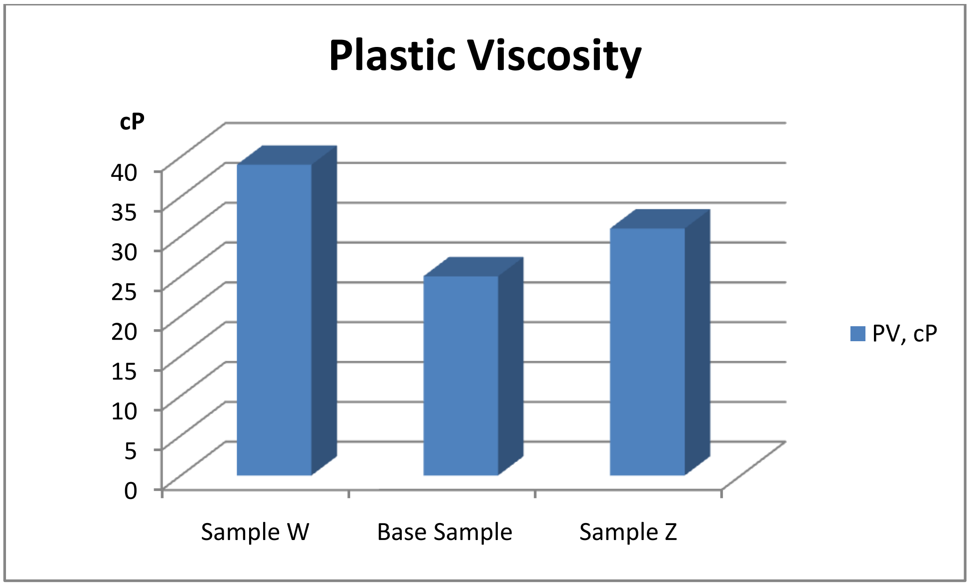


Chart 3: Plastic Viscosity

PV represents the viscosity of a mud. A low PV indicates that the mud is capable of drilling rapidly and high PV is caused by a viscous base fluid and by excess colloidal solids. According to the test, base sample shows a good PV result. Even though Sample Z is exceeded the specification, it still could be considered to accept because the exceeded value is not too much. But, Sample W could not be accepted because the PV is too high and out of the specifications.

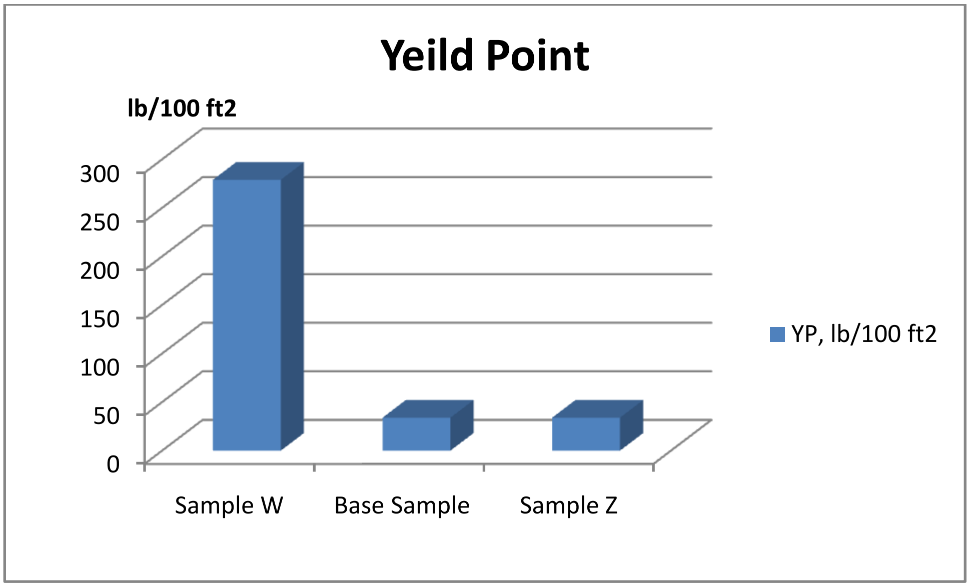


Chart 4: Yield Point

YP 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. Sample W is totally out of specification because the value of YP is too high. Base Sample and Sample Y also not in the range of specification. Some alteration should be done on the formulation of mud in order to get the better results.

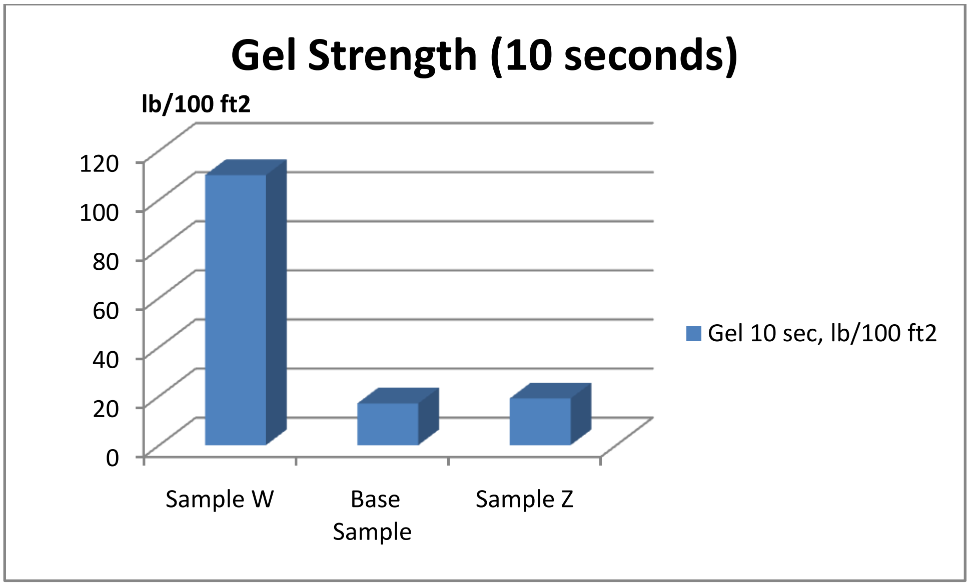


Chart 5: Gel Strength (10 seconds)

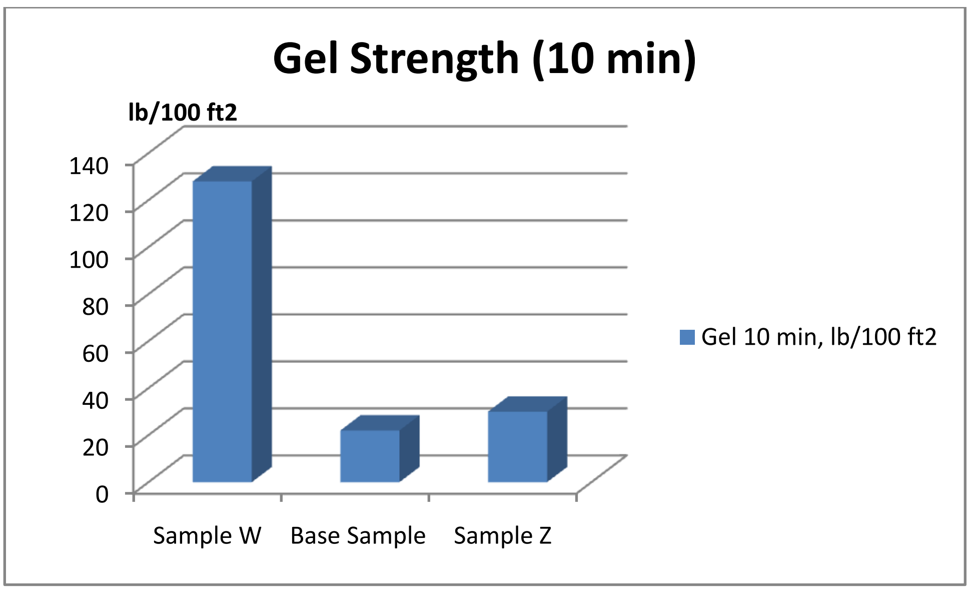


Chart 6: Gel Strength (10 Minutes)

Gel strength is a measure of the ability of a colloidal dispersion to develop and retain a gel form, based on its resistance to shear. In this test, barite is used as colloidal clays and adding to drilling fluids to increase its gel strength. The shear stress measured at low shear rate after a mud has set quiescently for a period of time (10 sec and 10 min in the standard API procedure). According to the test, Base Sample give a best result compare Sample W and Sample Z. The results of Sample W are too high and could not be used in the field.

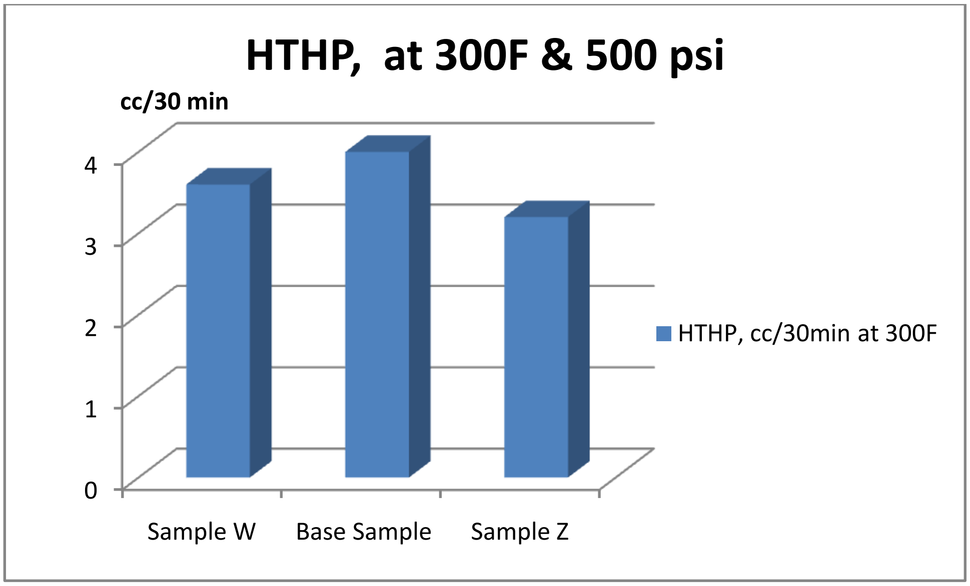


Chart 7: Fluid Loss Filtration in HTHP

The purpose of HTHP filtrate test is to measure the quantity of fluid loss from the mud during the high temperature and high pressure conditions. The less quantity of the fluid gained, the better the result. From the test, Sample Z shows the best results of filtrate compare to Sample W and base sample.

Based on the overall results, base sample and Sample Z could be used as the mud drilling fluids in the fields. These samples had followed the specification given. It shows that palm oil based fatty acid also could be used as emulsifier in drilling fluids but further study should be done to enhance the performance of emulsifier. Changing in carbon chains and synthesis process are much recommended to enhance the emulsifier performances.

CHAPTER 5: CONCLUSION AND RECOMMENDATION

5.1 Conclusion

. Based on the research done, plant-based oil could be the best options on making the new emulsifier. The results of mud performance in the charts show that the palm oil based fatty acid is suitable to use as emulsifier in the mud drilling fluids. Even though the results of new emulsifier not good as conventional emulsifier, but it had shown that it could be one of the best emulsifier in the future. Hence, it is good to make a further study on new emulsifier from palm oil based fatty acid in order to obtain the same or better performance with low cost and more environmental friendly.

Upon improvement of their disadvantageous characteristics, palm oil based fatty acid could be a good emulsifier for drilling fluids in drilling operations. Palm oil has significant criteria which could replace the conventional emulsifier. It provides a good lubricity to the fluids, better weighting agents, low toxicity and good biodegradability.

In other hands, palm oil based fatty acid emulsifier gives a solution to the environmental pollution problems from the conventional drilling fluids. In term of economical, this new emulsifier would give a cheaper cost with better performance. Thus, company could optimize their income by reducing the cost.

5.2 Recommendation

Study on the vegetable plant behavior will give a huge beneficial to the country and also the environment. As we know, vegetable plant have a lot of significant contents that not be recovered yet. By doing a detail research in vegetable plant, we could acquire a lot of benefit. It is easy to obtain in short time. For example, there are many lubricant product made from the vegetable source have been commercialized.

Besides that, the use of vegetable plant will enhance the income of the country. As example, Malaysia is one of the biggest exporters for palm oil in the world. They should optimize the usage of each component in the palm oil to make a huge profit. A lot of research should be done to make this suggestion become beneficial.

Thus, I recommend increasing the research about this natural source to optimize the usefulness of this source. Besides of using the palm oil, we also can try to use other vegetable oil that could be suitable for our requirement.

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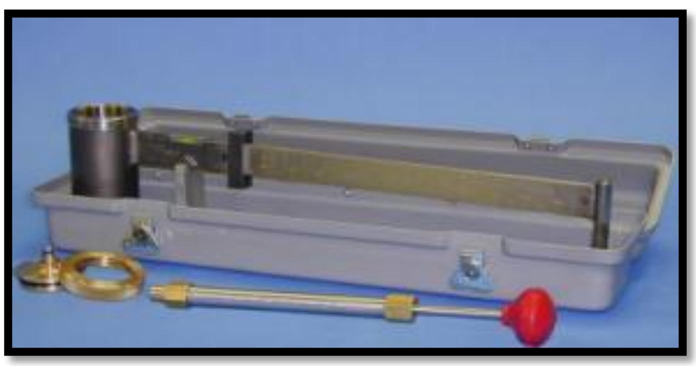
APPENDICES

List of Equipments:

Mixer

Fan 35 Viscometer



Mud Weight

Retort

HTHP Filtrate