EFFECT OF NANO MATERIAL (GRAPHENE) IN OIL BASED DRILLING MUD

by

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Petroleum Engineering

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Dissertation submitted in partial fulfilment of
the requirements for the
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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
Petroleum Engineering Programme
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BACHELOR OF ENGINEERING (Hons)
(PETROLEUM ENGINEERING)

Approved by,

____

(Raja Rajeswary Suppiah)

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
September 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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TAUFIQ BIN AHMAD

ABSTRACT

Current experience shows, it is often impossible to fulfill certain functional tasks that are essential in challenging drilling and production environments using conventional macro and micro type fluid additives due to their inadequate physical, mechanical, chemical, thermal and environmental characteristics. Hence, the industry is looking for physically small, chemically and thermally stable for designing smart fluids to use virtually in all areas of oil and gas exploration. Due to totally different and highly enhanced physio-chemical, electrical, thermal, hydrodynamic properties and interaction potential of nanomaterials compared to their parent materials, the nanos are considered to be the most promising material of choice for smart fluid design for oil and gas field application. This project will describe the formulation and preliminary test results of graphene enhancement as an additional nano- additive for drilling fluids. This project is basically to test whether graphene material enhancement can give a significant improvement to the conventional oil based drilling fluid it terms of its rheological properties and performance.

ACKNOWLEDGEMENT

In completion of this Final Year Project, I would like to express my deepest gratitude to all parties that involved in making this project as a meaningful and great achievement for me. Throughout the project timeframe, I have learned a lot of new things and gained so much experience that will be a huge value for me in the future.

First and foremost, I would like to thank UTP's Petroleum Engineering Department for providing me guidance to complete the project and in the same time arranging the project activities for me. With their strong support and advice, I was able to plan and conduct my project very well.

Special thanks should go to my Final Year Project supervisor, Miss Raja Rajeswary Suppiah, who becomes the most important person to teach and guide me in conducting this project. His strong support and willingness to share his knowledge and wide expertise has given me a very important experience in this project study.

Besides that, I would like to thank the lab assistants who had helped me to complete the project especially in conducting the laboratory work. I truly appreciate their help and their sharing of information in order for me to execute the experiment well.

Finally, my deepest appreciation goes to my family and my friends in UTP for their continuous support and encouragement which have enabled me to do my best for this project. I hope that after this project completion, all the findings and knowledge that I have shared through this dissertation would be useful for everyone in the study field.

TABLE OF CONTENT

ABBREVIATIONS AND NOMENCLATURES

OBM	-	Oil Based Mud
SBM	-	Synthetic Based Mud
WBM	-	Water Based Mud
HTHP	-	High Temperature High Pressure
HS	-	Hydrogen Sulfide
CO	-	Carbon Dioxide
N	-	Nitrogen
YP	-	Yield Point
MW	-	Mud Weight
RPM	-	Revolution Per Minutes
API	-	American Petroleum Institute

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CHAPTER 1

INTRODUCTION

Drilling mud is an important element in the drilling operation. Nowadays, service companies invest millions of dollars in research and development purposes in producing higher quality of drilling mud.

1.1 Background of Study

Drilling operation is becoming increasingly more difficult and expensive. Drilling fluid stability and performance are still problematic in deeper and high-temperature high-pressure formations (HPHT). Nano materials have attractive properties for applications in drilling fluid enhancement where heat transfer, drag reduction, formation consolidation, gel formation, wettability alteration and corrosive control are of interest.

In this research, the evaluation to find the effects of graphene enhancement in OBM will provide new information for whether nano material (graphene) gives a significant improvement to the conventional OBM. Prior to the limited resources, *Sarapar 147* is selected as control OBM in the experiment. In the other hand, nano material that is selected for the research purposes is *Graphene*, which is provided by Platinum NanoChem Sdn. Bhd.

1.2 Problem Statement

Application of oil base mud basically only in high temperature and high pressure well due to environmental issue and also the cost of it usage. There are many types of oil base mud that commercially being used in oil and gas industry and some of it were different in name depending on the producing company. These types of oil base mud not just only different in the name but also the performance in term of mud rheology for HTHP wells. Thus, enhancement oil base mud by introducing nano material can increase its quality and performance compare to conventional mud.

There a few implementations of nano materials in drilling mud recently, but none of them are using graphene material. The effects of graphene enhancement in drilling mud never been tested before. Since its nano material, the enhancement of graphene in drilling mud most probably can give significant changes towards the performance of the drilling mud.

1.3 Project Objectives

The first objective of this research is to establish an evaluation and comparison on the performance of conventional oil based mud with graphene enhancement oil based mud and its suitability in HTHP wells. Generally, the evaluations are based on the mud rheology properties such as yield point, gel strength, viscosity, mud weight, HTHP fluid loss, emulsion stability and so on to compare the performance in elevated pressure and temperature.

The second objective of the project is to set a new platform for testing the application of graphene in drilling mud application in general performance. This graphene material will be added into the oil based mud as an extra additive with all other additives for conventional oil based mud remains the same. Maybe in the future, if the result shows a significant improvement in the drilling mud performance, graphene enhancement can be tested specifically as fluid loss reducer additive for example.

1.4 Scopes of Project

Scope of studies for this research is on mud properties in general for both oil based mud with and without graphene enhancement. Basically, the result will be on the mud rheology characteristics; viscosities, gel strength, yield point, HTHP fluid loss, mud weight, and emulsion stability. It is required for all these data on mud rheology to determine the path of this research and investigate the effects of graphene enhancement in oil based mud. Basically, there are two part of comparison. The first part is the mud rheology characteristics before hot rolling process and for the second part comparison after the hot rolling process. The rheological behavior is to indicate the performance of drilling fluid in hole cleaning and hole erosion, suspension of drill cutting, hydraulic calculation, fluid loss, and requirement of drilling fluid treatment in HTHP wells. The viscosity is focusing on plastics viscosity to indicate the drilled cuttings suspension and hole cleaning abilities under dynamic condition.

1.5 Feasibility Analysis

Feasibility analysis is a guidance to identify the possible risks that would be gained if the project is approved. First, is important to be familiar with the functional area. In order to meet the objectives of the project, the author must know well about the scope of studies so that the author can gather all the data correctly within timeframe. The author also must know the procedure of lab testing correctly to get better result. Next, it is about familiarity with technology which means the author must know well about the technology is used. Considering the scopes of studies only focusing on mud properties, it can be done in UTP laboratory and the author believes that the timeframe to complete the research is enough within one semester. For organizational feasibility, the author already get help from one of oil and gas company in consulting to lead this research as it has the commercial value that the company is looking forward to it.

1.6 Relevancy of Project

The application of nano-technology in oil and gas industry, in drilling mud especially creates total evolution in order to create higher success rate in drilling wells. So, in order to make it reality, this project can be a platform to test whether graphene enhancement as a nano material can improve conventional drilling mud or not. This might be the first step to create more studies and research on this extra additive to create better performance of drilling mud. The question is how significant this graphene enhancement performance compared to the conventional drilling mud. So, this research basically to fill up the purposes of evaluating this new extra additive to observe its performance before applying it into the real operations

For this project, the author is applying his theoretical and practical knowledge in petroleum engineering to evaluate base oil performance and to analyze the substances in this. The basic principle involved range from drilling fluid study, well completion and production, thermodynamics, facilities engineering and management of drilling fluid. Hence, this study seems to be fit as a platform for the author in applying his petroleum engineering knowledge and skills. The outcome of this research is deemed crucial towards providing future technology in drilling fluid application.

CHAPTER 2

LITERATURE REVIEW

2.1 Nano Materials

2.1.1 Nano Definition

Nanofluids for oil and gas field applications are defined as drilling, completion, stimulation or any other fluids used in the exploration and exploitation of oil and gas that contain at least one additive with particles size in the range of 1-100 nanometers. Physically a nano sized particle has a dimension that is thousand millionth of a meter. 100 nanometer fibers or particles have diameters that are about 800 to 1000 times smaller than the diameter of a human hair and roughly equal to the width of 10 hydrogen atoms (Saeid et al. 2006). Based on the number of nano-sized additives in the fluid, these fluids can be classified as simple nano-fluid and advanced nano-fluid. Nano-fluids with one nano-sized additive are defined as simple nanofluids and nanofluids with more than one-sized additive are defined as advanced nanofluids. From functional points of view, a nanomaterial could be single functional or multifunctional. The significantly higher functional ability with a reduction in overall fluid cost in spite of high cost individual additive is to be one of the characteristic features of nano-based smart fluids. [16]

2.1.1 Nano-Particles

Nanotechnology application can revolutionalise the additive characteristics and behavior by turning particle properties to meet certain operational, environmental, and technical requirements. Hence, the nanotechnological research leading to create some custom made nanoparticles could be a promising step change research for smart fluid development for different industrial applications. These particles are ultra fines in nature, usually larger than an atom cluster but smaller than ordinary micro particles and this have very specific area with enormous area of interactions. Due to nano-scale particle dimension, the nano-type fluid additives have both external as well as internal inhibition potential, require a very low additive concentration and thus expected to provide superior fluid properties at a drastically reduced additive concentration.

Nano-particles with high thermal stability and affinity to acid gases such as H2S and CO2 will help meet the technical challenges of sour gas environment, deep and geothermal drilling and this expected to complete a well economically and development of nontoxic, environment friendly and biodegradable nano-particle based drilling fluids are expected to meet the current as well as the future environmental norms and regulations for drilling and production in deep water and sensitive environments. [16]

2.1.3 Graphene Enhancement Material

Graphene, as a single layer of graphite, has become the subject of much research interest for its unique materials properties. Among other interesting features, a pristine graphene monolayer has a theoretical Connolly surface area of 2965 m2/g and has been shown to form a membrane impermeable even to helium gas. Graphene might make a good candidate as a pore-plugging filter in oil-drilling fluids. Graphite Oxide was first synthesized by Brody in the 19th century by carefully reacting natural graphite with oxidizing agents in a solution of oxidizing acids. [15]

Dispersed GO flakes can be shifted out of solution and pressed in order to make a strong paper-like material, which results from a robust tile-like interlocking of the flakes. This could be beneficial for making a thin impermeable film to prevent fluid loss in the wellbore. More importantly, the nanometer thickness of the GO flakes could also result in much thinner filter cakes than those obtained using clay-based materials. The thickness of a wellbore's filter cake is directly and strongly correlated to the differential torque needed to rotate the pipe during drilling, to the drilling time, and to drilling costs. GO solutions also exhibited greater shear thinning and higher temperature stability compared to clay-based fluid-loss additives, demonstrating potential for high-temperature well applications. [15]

2.2 Drilling Fluid

Drilling fluid is used in the rotary drilling process to clean the rock fragments from beneath the bit and carry them to the surface, exert sufficient hydrostatic pressure against subsurface formations to prevent formation fluids from flowing into the well, keep the newly drilled borehole open until steel casing can be cemented in the hole, and cool and lubricate the rotating drillstring and bit. In addition to serving these functions, the drilling fluid should not have properties detrimental to the use of planned formation evaluation techniques, cause any adverse effects upon the formation penetrated, or cause any corrosion of the drilling equipment and subsurface tubulars.

The main factors governing the selection of drilling fluids are the types of formations to be drilled, the range of the temperature, strength, permeability, and pore fluid pressure exhibit by the formations, the formation evaluation procedure used, the water quality available, and ecological and environmental considerations. However to a large extent, the drilling fluid usage that yields the expendable cost usually determined by trial and error. Water-based mud is most commonly used in drilling fluids. But it is restricted to the area with lower temperature and lower pressure due to the inconsistency mud rheology in high temperature. Oil-based muds are generally is limited to drilling extremely hot formations or formations that are affected adversely by water-based muds due to its costs and require more stringent pollution control procedures than water-based muds. The use of gases as drilling fluids is so rare and is limited to areas where the formations are competent and impermeable.[2] Gas or liquid mixtures can be used when only a few formations capable of producing water at significant rates are encountered.[2]

2.3 Base Oil

Hydrocarbon oils are the continuous phase in oil-base fluids. They are non-polar, low-surface tension liquids and interact only weakly with mineral solids. This characteristic is the basis for the use of oil-base fluids as non-reactive, inert drilling fluids. The most commonly used oils today are synthetics where certain environmental regulations prevail, low-aromatic-content, and low-toxicity mineral oils.

Crude oil has been used in the past but finds little application in today"s modern day oil-mud drilling fluids. Crude is relatively cheap, often available, but may need topping to minimize flammability since a flash point greater than 180°F (82°C) [3,4,5] is advised. Crude contains native asphaltenes and resins which can interfere with other additives.

Diesel oil is a moderately-priced, commonly available distillate which contains none of the native asphaltenes or resins in crude and is the most commonly used oil for mixing oil-base fluids.[7] The aromatics in diesel oil can swell rubber gaskets, seals, and pipe rubbers, however, an aniline point greater than 140°F (60°C) (the higher the aniline point, in general, the lower the concentration of aromatics)[4] is recommended.

Synthetic-base drilling fluids (SBF) use a synthetic type material as the continuous phase. Synthetics are the preferred oil in offshore drilling operations where environmental regulations prohibit the discharge of cuttings and/or whole mud to the sea. Unlike mineral and diesel oils which are distilled from crude oils, synthetic type materials are usually polymerized from ethylene. Since the synthetics are pure products made from ethylene, they contain no aromatics, thereby lowering the toxicity level normally associated with aromatic compounds. Like hydrocarbon oils, the synthetic type materials are more viscous than water.[7]

2.4 OBMs Basic Chemistry

Oil mud require special to ensure that the emulsion is extremely stable and can withstand conditions of high temperature and contaminants. Every single product must be dispersible in the external oil phase. [3,4,7,8]

Emulsifying systems	Calcium soaps are the primary emulsifier in oil muds. These are made in the mud by reaction of lime and long-chain fatty acids. Soap emulsions are strong emulsifying agents but may take reaction time before emulsion is actually formed. Thus secondary emulsifiers are used: they consist in very powerful oil-wetting chemicals which generally do not form emulsions but wet solids before the emulsion is formed. Also used to prevent from any water intrusion.
Lime	Lime is essential in OBMs. It neutralizes fatty acids in the fluid, stabilizes the emulsion when present in excess, and controls alkalinity. In the field, it also neutralizes acid gases such as H S and CO .
HTHP fluid loss reducer	Many types of chemicals can be used as fluid loss control agents. They are usually organophilic lignites (amine-treated lignites), Gilsonite or Asphalt derivatives, or specialpolymers (polyacrylates)
Wetting agents	Supplemental additives to quickly and effectively oil-wet solids that becomes water-wet.
Chemicals to control rheology	Additives that build the viscosity of the mud. Bentonite, hectorite or attapulgite, treated with amine to make them oil-dispersible, are commonly used organophilic gellants. When their properties are reduced by high temperature, polymeric viscosifiers are added.
Weighting agents	Used to increase the density of the oil mud. The most commonly used are Calcite (MW up to 10.8 ppg), Barite (MW up to 21 ppg), and Hematite (MW up to 24 ppg).

Table 1: OBM Basic Chemistry

CHAPTER 3

METHODOLOGY

3.1 Project Planning

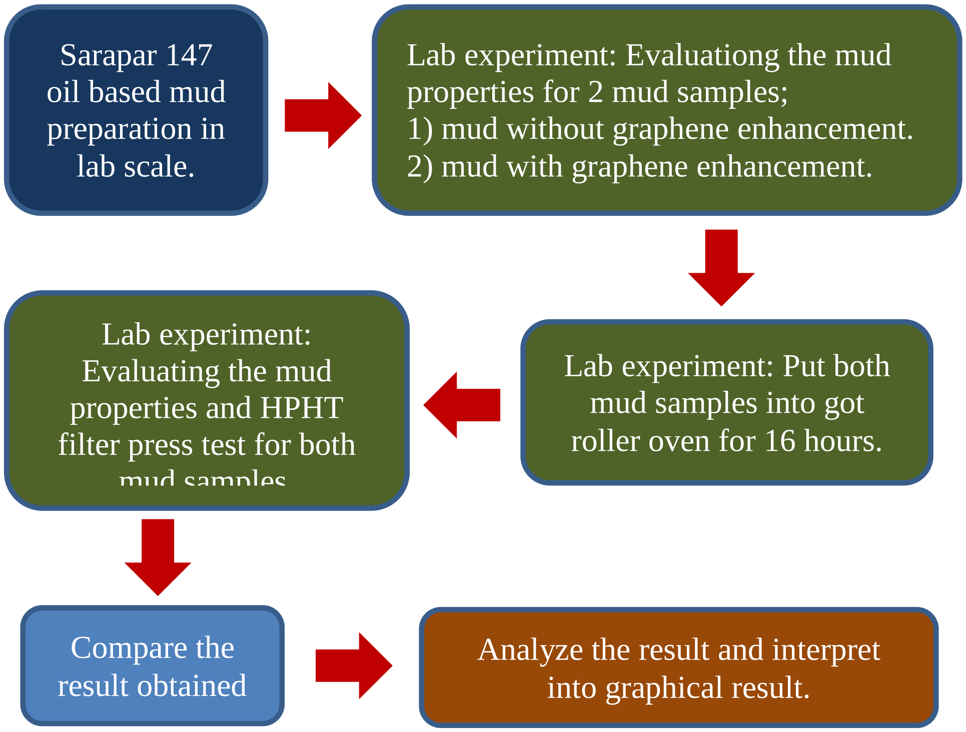

Figure 1: Project Planning Flow

1. Before proceeding with the research, preparation of oil-based mud by using Sarapar 146 as control oil base is necessary in the process.
2 The next step is to proceed with mud rheology lab testing for with and without graphene enhancement oil based mud to compare the result as its performance.
3 Then, perform experiment on the drilling mud in constant high temperature and high pressure using hot roller oven for 16 hours approximately to evaluate the performance in application of HTHP wells. [*see Appendix*].
4 Perform mud rheology lab testing once again as well as HPHT filter press test and record the result.
5 All the data from the lab testing will be gathered to analyze and evaluate as a milestone to proceed with the study on the effects of graphene enhancement in the oil base mud.

3.2 Experimental Procedures

The initial process is to prepare the drilling fluid to run the test. Then, there will be three stages of lab experiments that consists of experiments for analyzing the differences of rheology properties of conventional mud (without graphene enhancement) and graphene enhancement mud before hot rolling process, both mud samples undergoes hot rolling process for 16 hours, experiments for evaluating the performance of oil-based mud properties after hot rolling process and evaluate the performance of HPHT filter press result.

3.3 Procedure of OBM Preparation

The addition of components in their proper sequence when initial mixing an oil mud, will optimize the performance of each product. The order of addition as below is the most common procedure for the preparation of oil-based muds, though each mud system may require some modifications of the procedure. Basically, this is the basic procedure for lab scale:

1) Add the required quantity of base oil to the mixing vessel [*see Appendix*].
2) Add the primary and secondary emulsifiers as required.
3) Add the organoclay gallant as required.
4) Add filtration control additives
5) Add lime in excess.
6) Add required amount of brine.
7) Add graphene 1 wt% materials [*this step only for OBM with graphene sample*].
8) Mix for a long time to ensure a good emulsion is formed.
9) Add weighting material as required for the desired density.

3.4 Experiment for Analyzing Rheology Properties of Mud Samples

3.4.1 Mud Viscosity Test

Theory:

Viscosity of a fluid is defined as its resistance to flow and is measured as the ratio of the shearing stress to the shearing strain. The desired viscosity is influenced by several factors, including mud density, hole size, pumping rate, drilling rate, pressure system and requirements, and hole problems. Two types of fluid characterizations are There are two types of measurement which are for field measurement and laboratory measurement.

Equipment: The FANN Model 35A Viscometer (for laboratory measurement).

Procedure:

<u>The FANN Model 35A Viscometer</u>: [*see Appendix*]

1) Place a recently agitated sample in the cup, tilt back the upper housing of the viscometer, locate the cup under the sleeve (the pins on the bottom of the cup fit into the holes in the base plate), and lower the upper housing to its normal position.
2) Turn the knurled knob between the rear support posts to raise or lower the rotor sleeve until it is immersed in the sample to the scribed line.
3) Stir the sample for about 5 seconds at 600 RPM, and then select the RPM desired for the best.
4) Wait for the dial reading to stabilize.
5) Record the dial reading and RPM.

3.4.2 Gel Strength Test

Theory:

The gel strength is a function of the inter-particle forces. An initial 10 seconds gel and 10 minutes gel strength measurement give an indication of the amount of gelation that will occur after circulation ceased and the mud remains static.

Equipment: The FANN Model 35A Viscometer.

Procedure:

1) Stir a sample at 600 RPM for about 15 seconds.
2) Turn the RPM knob to the STOP position
3) Wait the desired rest time (normally 10 seconds or 10 minutes).
4) Switch the RPM knob to the GEL position.
5) Record the maximum deflection of the dial before the Gel breaks, as the Gel strength in lb/100 ft^2.

3.4.3 Yield Strength Test

Theory:

This is the measure of the electro-chemical or attractive forces in the mud under flow (dynamic) conditions. These forces depend on surface properties of the mud solids, volume concentration of the solids, and electrical environment of the solids. The yield point of the mud reflects its ability to carry drilled cuttings out of the hole.

Equipment: The FANN Model 35A Viscometer.

Procedure:

By means of the viscometer calculations procedure, determine the Apparent and Plastic Viscosities, Yield Point and initial 10 seconds and final 10 minutes Gel Strength parameters.

Yield Point (YP) = 300 RPM – Plastic Viscosity.

3.4.4 Emulsion Stability Test

Theory:

Water present in an oil mud is in the form of the emulsion. A chemical emulsifier must be added to prevent the water droplets from coalescing and settling out of the emulsion.

Equipment: The Electric Stability kit. [*see Appendix*]

Procedure:

1) Before placing the probe in the mud, it is essential to test the meter in air.
2) 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.
3) 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.
4) 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.
5) Repeat the test. The two ES values should be within 5% and anything greater would indicate a problem with the equipment.
6) The result is the average of the two readings

3.4.5 HPHT Fluid Loss Test

Theory:

Filtration control is one of the primary characteristics of a drilling fluid and fulfils a variety of functions from the prevention of differential sticking to minimisation of formation damage. The loss of liquid from a mud due to filtration is controlled by the filter cake formed of the solid constituents in the drilling fluid. The test in lab consists of measuring the volume of liquid forced through the mud cake into the formation drilled in 30 minutes period under given pressure and temperature using a standard size of cell. This test tends to be run at temperatures that reflect expected bottom hole temperatures and thus the procedures are for temperatures up to 300˚ F.

Equipment: HTHP Filter Press [*see Appendix*]

Procedure:

1) Turn on heated jacket at the mains and insert a thermometer into the jacket and leave to preheat to the desired temperature.
2) Check out all the "O" rings on the HPHT bomb and lid.
3) With stem valve closed on bottom of cell, fill up cell with mud to within 0.5" of the „O" ring groove, to allow for thermal expansion.
4) Insert filter paper into the cell followed by the bottom cell plate assembly over the filter paper and twist to align with the safety locking lugs. Ensure the lid stem is open while doing this to avoid damaging the filter paper.
5) Tighten the 6 grub screws evenly using the Allan key provided.
6) Ensure all stem valves are tightly closed.
7) Invert cell and place in filtration mounted heated jacket assembly. Rotate the bomb until it seats on the locking pin. Insert a thermometer into the HTHP cell.
8) Place a CO or N cartridge in each regulator and tighten up the retainers.
9) Place the pressure unit on top valve and lock into place using a locking pin. Lock the bottom pressure unit to the bottom valve into place, again ensuring that locking pin is inserted.

10) Apply 100 psi to both ends of the HTHP cell with the valves still closed.
11) Open the top valve by turning 1/4 to 1/2 anticlockwise to apply 100 psi to the mud while heating to prevent the mud from boiling prior to reaching the target temperature. The time for heating the mud sample to the target temperature should not exceed 60 minutes.
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.
14) If the pressure on the bottom regulator increases significantly above 100 psi bleed off some of the filtrate into the graduated cylinder.
15) Collect the filtrate for 30 minutes maintaining the temperature to within ± 5˚ F.
16) Once the test has finished close the top and bottom valves and shut off the pressure supply from the regulators. Bleed the lines using the relief valves provided.
17) Allow filtrate to cool for 30 minutes and then draw off into a graduated 20 ml measuring cylinder and read volume. SAVE the filtrate for ionic analysis.
18) CAUTION - the cell still contains 500 psi pressure, so cool cell to room temperature ideally in a water bath or alternative safe place and then bleeds off the pressure slowly by opening the valves.
19) Disassemble the cell and discard mud into mud waste container only. Save filter paper handling with care and wash filter cake with a gentle stream of distilled water.
20) Measure and report the thickness of the cake to the nearest 1/32” (0.8 mm). Report any other observations, such as texture, colour, hardness, flexibility etc.

3.3 Project Gantt Chart/Key Milestone

<u>Final Year Project 1:</u>

<table>
<tr><th>No</th><th>Detail/Week</th><th>1</th><th>2</th><th>3</th><th>4</th><th>5</th><th>6</th><th>7</th><th>8</th><th>9</th><th>10</th><th>11</th><th>12</th><th>13</th><th>14</th></tr>
<tr><td>1</td><td>Selection of Project Topic: Resistivity Modeling of Shaly Sand Formation in High Water Salinity</td><td style="background:#4BACC6"></td><td style="background:#4BACC6"></td><td></td><td></td><td></td><td></td><td rowspan="7">Mid Sem Break</td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></tr>
<tr><td>2</td><td>Preliminary Research Work: Research on literatures related to the topic</td><td></td><td style="background:#4BACC6"></td><td style="background:#4BACC6"></td><td style="background:#4BACC6"></td><td style="background:#4BACC6"></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></tr>
<tr><td>3</td><td>Submission of Extended Proposal</td><td></td><td></td><td></td><td></td><td></td><td>●</td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></tr>
<tr><td>4</td><td>Proposal Defense (Oral Presentation)</td><td></td><td></td><td></td><td></td><td></td><td></td><td style="background:#4BACC6"></td><td style="background:#4BACC6"></td><td></td><td></td><td></td><td></td><td></td></tr>
<tr><td>5</td><td>Project work continues: Further investigation on the project and do modification if necessary</td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td style="background:#4BACC6"></td><td style="background:#4BACC6"></td><td style="background:#4BACC6"></td><td></td><td></td></tr>
<tr><td>6</td><td>Submission of Interim Draft Report</td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td>●</td><td></td></tr>
<tr><td>7</td><td>Submission of Interim Report</td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td>●</td></tr>
</table>

Table 2: Project Gantt Chart for FYP 1

Final Year Project 2:

No	Detail/Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	**Project Work Continues:** Part 1: Preparing the core samples							**Mid Sem Break**							
2	Submission of Progress Report								●						
3	**Project Work Continues:** Part 2: Resistivity Measurement														
4	Pre-SEDEX										●				
5	Submission of Draft Report											●			
6	Submission of Dissertation (soft bound)												●		
7	Submission of Technical Paper												●		
8	Oral Presentation													●	
9	Submission of Project Dissertation (Hard Bound)														●

Table 3: Project Gantt Chart for FYP 2

3.4 List of Materials

1) Base Oil - Sarapar 147
2) Additive :
    - CONFI-MUL P
    - CONFI-MUL S
    - CONFI-GEL
    - CONFI-TROL XHT
    - LIME
3) Brine :
    - Drillwater
    - CaCL2
4) Weighting agent :
    - DRILL-BAR
5) Additional additive :
    - Graphene Material

3.5 List of Apparatus and Equipment

1) Baroid Multimixer
2) The FANN Model 35A Viscometer
3) HPHT Filter Press
4) Hot Roller Oven
5) Electric Stability Kit
6) Stop Watch

CHAPTER 4

RESULT AND DISCUSSION

In this experiment, the performance of graphene enhancement as an additional additive was observed by comparing mud properties in Sarapar 146 oil based. This graphene material was supplied by Platinum NanoChem Sdn. Bhd. *Sarapar 146* was used as the control case study for oil base mud type in order to investigate the performance of graphene enhancement in conventional oil based mud. Using the basic mud formulation 11.5lb/gal at 75/25 oil water ratio OBM formulation, the formulation for base oil were determined by using this ingredient:

Base oil	Sarapar 147
Primary emulsifier	Confi-Mul P
Secondary emulsifier	Confi-Mul S
Viscosifier	Confi-Gel
Fluid loss reducer	Confi-Trol XHT
Alkalinity source	Lime
Brine	Fresh water
	CaCl
Weighting agent	Drill Bar

Table 4: Elements In Basic OBM Formulation

Emulsifier, viscosifier, fluid loss reducer, and alkalinity source were made as constant elements to generate 11.5lb/gal at 75/25 oil water ratio OBM formulation for Sarapar 147 base oil. Quantities for these elements in mixing procedure for base oil were the same, and using some calculation, quantities of base oil, brine and weighting agent can be determined.

PRODUCT	DESCRIPTION	QUANTITY (gram)
CONFI-MUL P	Primary emulsifier	3.00
CONFI-MUL S	Secondary emulsifier	9.00
CONFI-GEL	Viscosifier	8.50
CONFI-TROL XHT / CONFI-	Fluid loss control	8.00
LIME	Alkalinity source	8.00

Table 5: Constant Elements In OBM Formulation

Formulation for *Sarapar 147* and *Sarapar 147 + Graphene* samples as below :

Products	Sarapar 147
Base oil	159.66
CONFI-MUL P	3.00
CONFI-MUL S	9.00
CONFI-GEL	8.50
CONFI-TROL XHT	8.00
LIME	8.00
Drillwater	67.37
CaCl2	26.95
DRILL-BAR	192.71
Graphene	0.00

Table 6: Formulation for Sarapar 147 mud sample

Products	Sarapar 147 + Graphene
Base oil	157.31
CONFI-MUL P	3.00
CONFI-MUL S	9.00
CONFI-GEL	8.50
CONFI-TROL XHT	8.00
LIME	8.00
Drillwater	67.23
CaCl2	26.89
DRILL-BAR	195.26
Graphene	4.88

Table 7: Formulation for Sarapar 147 + Graphene mud sample

There were two part of mud rheology test. For the first part, the test was conducted after the mixing process completed. Result from this part was called as initially result. For the second part, the rheological properties of the mud samples have been tested once more after the mud been introduced in elevated high temperature and high pressure in hot rolling oven. In order to simulate for real applications of HPHT wells, the temperature and pressure were set to 300°F and 500 psi respectively. Then mud samples were leave in the hot rolling oven for 16 hours. Result from this part was called after ageing process result.

The results of evaluation the performance of graphene enhancement towards conventional *Sarpar 147* OBM consists of two main subjects, initial result right after mixing procedure, and after ageing result.

4.1 Initial Properties:

Properties (Initial)	SPEC	Sarapar 147	Sarapar 147 + Graphene
Mud density, lb/gal		11.5	11.5
Rheological properties		120 °F	120 °F
600 RPM		75	79
300 RPM		47	49
200 RPM		37	36
100 RPM		25	25
6 RPM	>10@150	9	8
3 RPM		7	7
PV, cP		28	30
YP, lb/100 ft^2	15-20@ 120	47	49
Gel 10 sec, lb/100 ft^2		10	10
Gel 10 min, lb/100 ft^2		14	16
ES, volts at 120 °F	> 400	420	475

Table 8: Initial result after mixing procedure

4.2 After Ageing Properties:

AHR @ 300F for 16h	SPEC	Sarapar 147	Sarapar 147 + Graphene
Mud density, lb/gal		11.5	11.5
Rheological properties		120 °F	120 °F
600 RPM		90	125
300 RPM		51	74
200 RPM		38	55
100 RPM		24	34
6 RPM	>10@150	8	9
3 RPM		6	6
PV, cP	<30	39	51
YP, lb/100 ft^2	15-25@ 120	51	74
Gel 10 sec, lb/100 ft^2	8-18	10	8
Gel 10 min, lb/100 ft^2	15-30	20	17
HTHP, cc/30min at 300F	<4	5.2	3
ES, volts at 120 °F	> 400	329	433

Table 9: After ageing result

4.3 Result Analysis:

1) Plastic Viscosity

Plastic Viscosity	Sarapar 147	Sarapar 147 + Graphene
Initial	28	30
AHR @ 300F for 16h	39	51

Table 10: Comparison of Plastic Viscosity

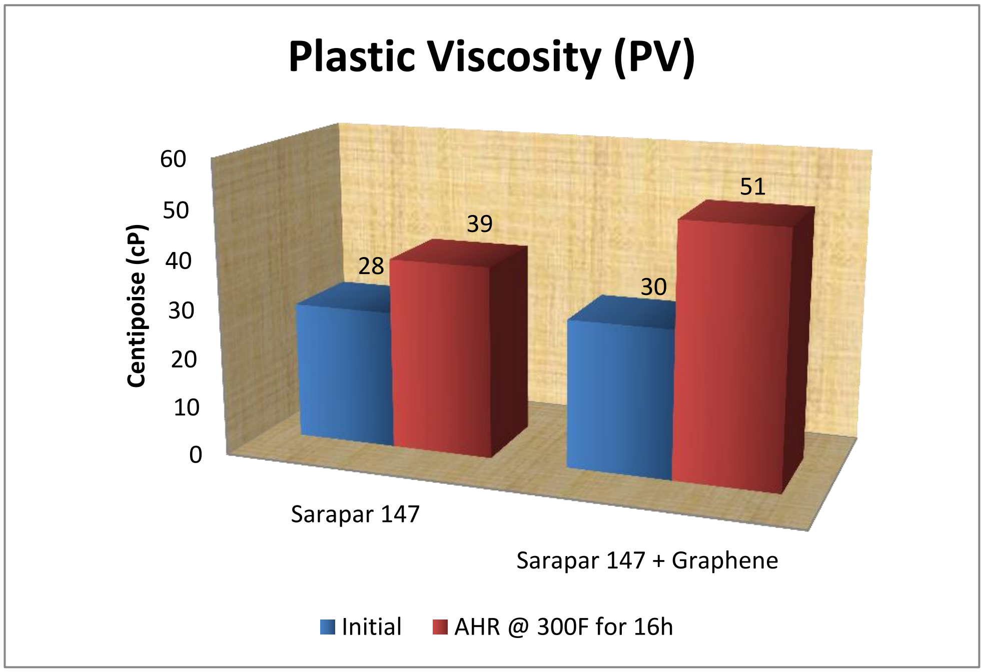

Figure 2: Plastic Viscosity bar chart for both mud samples

Plastic viscosity represents the viscosity of a mud. It indicates the amount of solid in the mud. It indicates the amount of solid in the mud. A low plastic viscosity indicates that the mud is capable of drilling rapidly and high plastic viscosity is caused by a viscous base fluid and by excess colloidal solids.. From the results, it shows that *Sarapar 147* have the lowest plastic viscosity compare to *Sarapar 147 + Graphene*. Comparing the performance between these two mud samples, *Sarapar 147* seems having good plastic viscosity than *Sarapar 147 + Graphene.*

2) Yield Point

Yield Point	Sarapar 147	Sarapar 147 + Graphene
Initial	19	12
AHR @ 300F for 16h	19	23

Table 11: Comparison of Yield Point

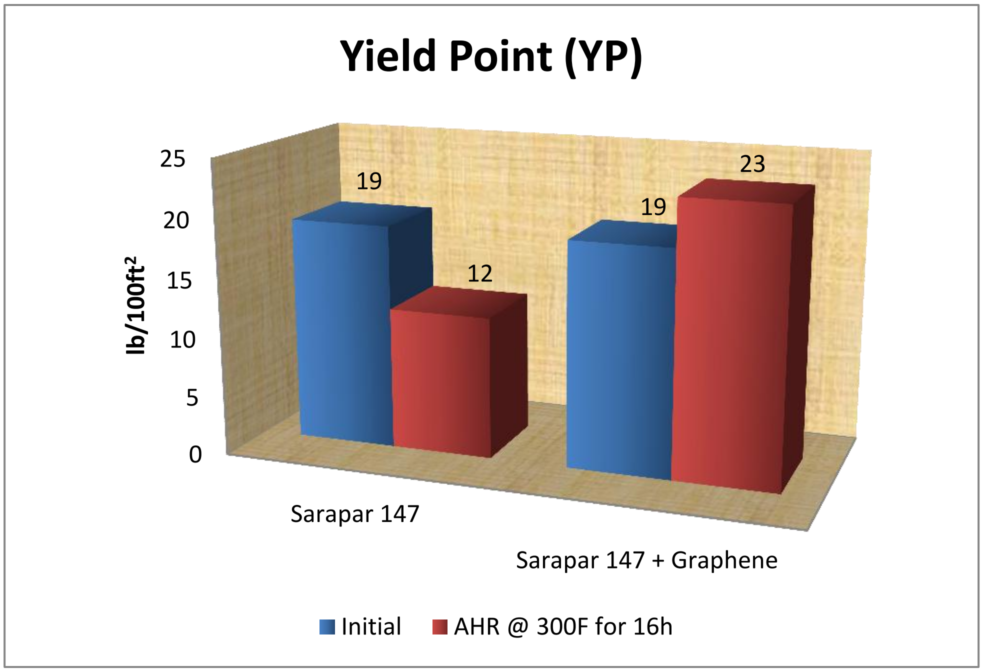

Figure 3: Yield Point bar chart for both mud samples

Yield point indicates the ability of mud to suspend and lift cutting out from the annulus. It's also indicates how much pressure needed for the pump to start circulate cuttings from wellbore to the surface. For HTHP conditions, yield point specification to be reach for mud to work best is in range 15-35. It shows that *Sarapar 147 + Graphene* perform better in lifting the cuttings in HPHT wells.

3) Gel Strength

Gel Strength for 10 mins	Sarapar 147	Sarapar 147 + Graphene
Initial	14	16
AHR @ 300F for 16h	20	17

Table 12: Comparison of gel strength for 10 mins

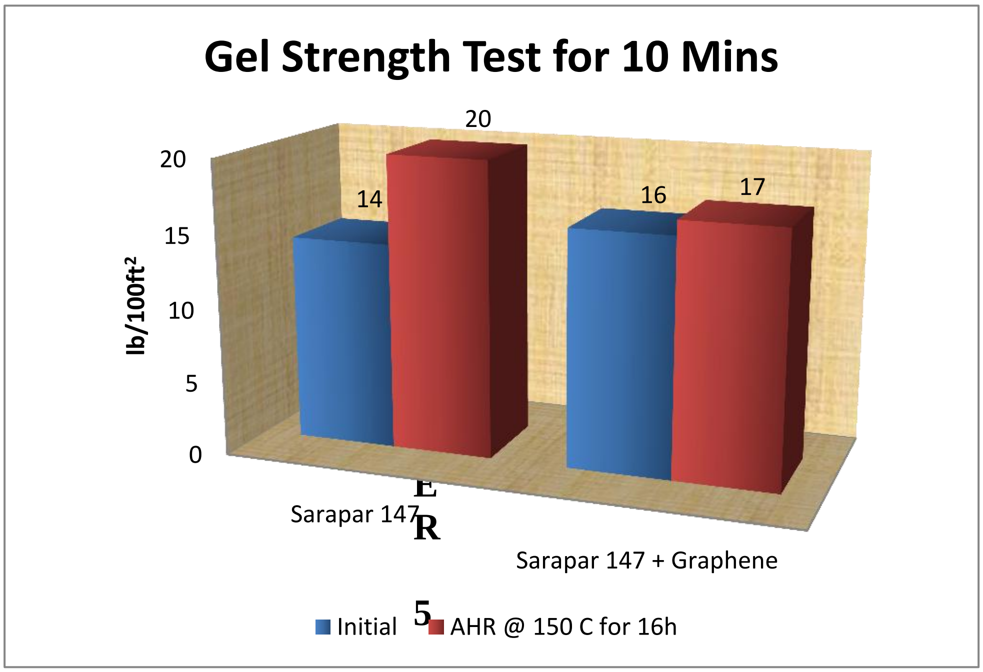

Figure 4: Gel strength for 10 mins bar chart for both mud samples

Gel Strength for 10 secs	Sarapar 147	Sarapar 147 + Graphene
Initial	10	10
AHR @ 300F for 16h	10	8

Table 13: Comparison of gel strength for 10 secs

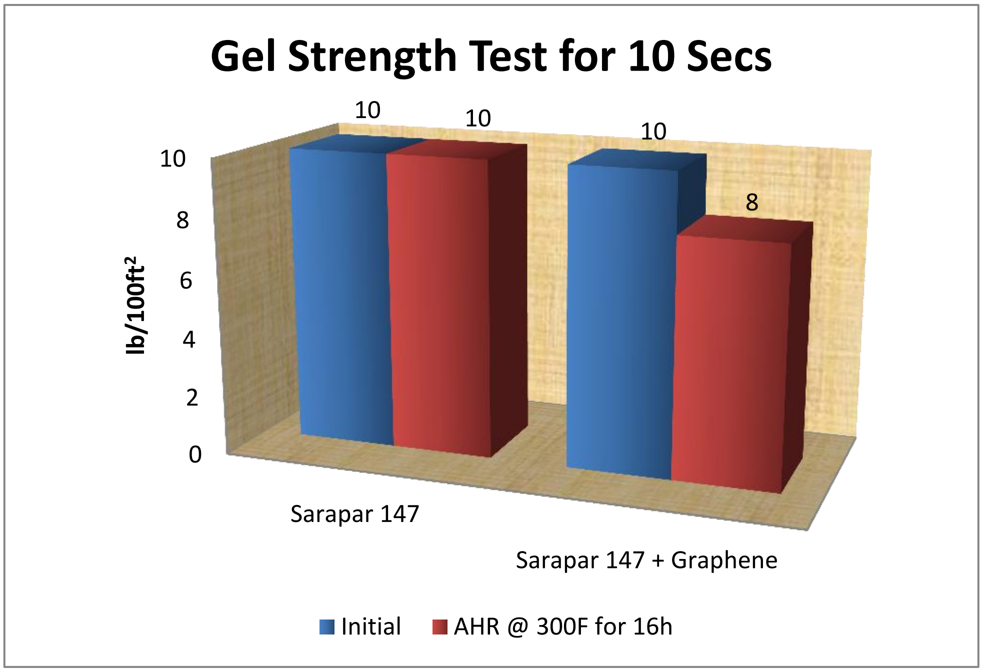

Figure 5: Gel strength for 10 secs bar chart for both mud samples

The gel strength or shear strength of a drilling mud determines its ability to hold solids in suspension and retain its gel form. In this study, barite was used as colloidal clay. CONFI-GEL also being used as organophilic hectorite clay to give the gel strength and also act as viscosifier. In this case, both mud samples meets the specifications which are for 10 seconds gel strength, the range are 8-18 lb/100 ft² and for 10 minutes gel strength, the range are 15-30 lb/100 ft². In term of stability, *Sarapar 147 + Graphene* show slightly better compare to *Sarapar 147* because it initial and after ageing result shows consistent value.

4) Emulsion Stability

Emulsion Stability	Sarapar 147	Sarapar 147 + Graphene
Initial	420	475
AHR @ 300F for 16h	329	433

Table 14: Comparison of Emulsion Stability value

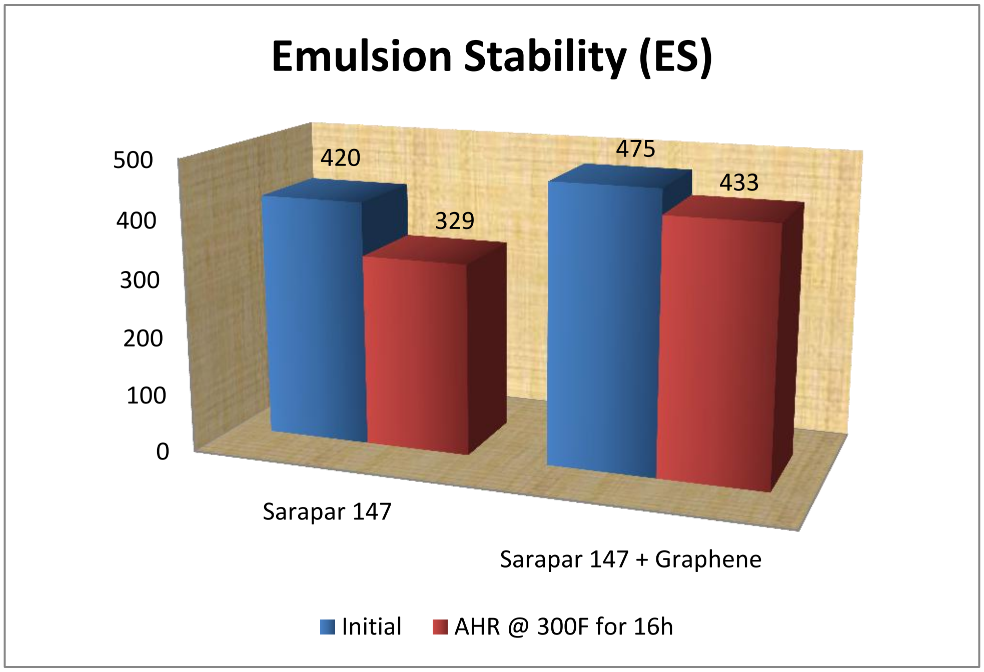

Figure 6: Emulsion Stability bar chart for both mud samples

The higher the emulsion stability value, the better is the mud. *Sarapar 147 + Graphene* shows promising result as the emulsion stability still mantain higher than 400 after hot rolling process. Applying for real cases that *Sarapar 147 + Graphene* have stabilize emulsion in HTHP wells. This proved that graphene enhancement can improve the emulsion stability for the drilling mud.

5) HPHT Fluid Loss

HPHT Fluid Loss (cc/30min @ 300F)	Sarapar 147	Sarapar 147 + Graphene
AHR @ 300F for 16h	5.2	3

Table 15: Comparison of Fluid Loss value

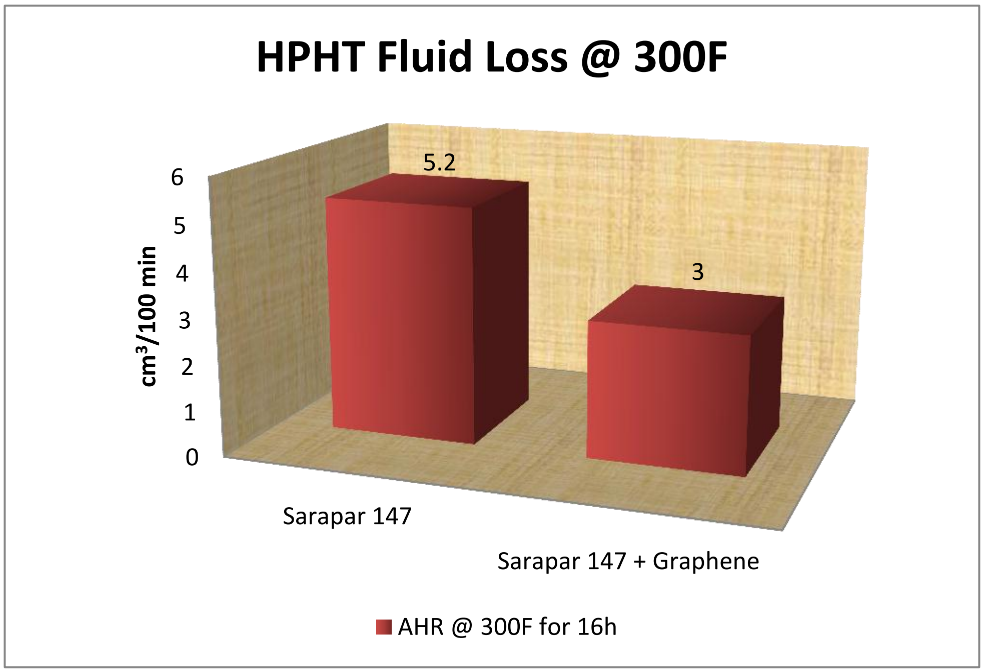

Figure 7: Fluid Loss bar chart for both mud samples

HTHP fluid loss test is designed to measure the mud ability to prevent fluid loss during mud circulation in the high temperature and high pressure wells. Mud will considered as good mud if it has minimal fluid loss into the formation. The results show that the significant less fluid loss was *Sarapar 147 + Graphene*. Meanwhile for *Sarapar 147*, it seems the fluid loss rate much higher for HPHT wells.

4.4 Discussion

Graphene is one type of nano material that was believed to have significant characteristic to improve conventional drilling performance, especially in HPHT wells mud application. Recently, there were studies that have been conducted to test the effect of nano material as a fluid loss additive to the drilling mud. However, for graphene material, it is still new and has not tested before with drilling mud. This project was the first in any time, tested the effect of graphene material towards the performance of drilling mud.

In any drilling mud formulation, mud rheology need meet requirement of industry standard specification that being used by most company in oil and gas industry. Thus, in accordance this specification, if mud formulation meets the range of these specifications, it will be considered as good mud and ready to be used in real time applications.

Mud Properties	Specification
Mud density, SG	1.2
6 rpm dial reding	8-16
Yield point, lb/100ft^2	15-35
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 16: Industry Standard Specification after Ageing Process

Based on the result that have been analyzed, it show that *Sarapar 147 + Graphene* only good in few rheological properties as a drilling mud. It perform very poor for PV test since the result show its PV was very high compared to *Sarapar 147*. For YP, Gel strength and ES test, *Sarapar 147 + Graphene* shows slightly better compare to *Sarapar 147*. The most significant improvement that can be seen was in HPHT fluid loss test. Sarapar 147 + Graphene show better performance. This shows that graphene material have promising tendency to become fluid loss additive for drilling mud.

Results from this project were affected by a two main factors. First factor was the type of graphene that have been used in this experiment. Based on discussions with the company that supply the graphene material, it comes out that the graphene that have been used was unfunctionalized graphene. According to them, the unfunctionalized graphene need to go through some chemical processes in order to make it functionalize and ready to mix with any fluid. The functionlized graphene is believed can give more significant modification towards the desired fluid properties.

Another factor was in order to mix graphene with drilling mud, it needs to dissolved first in a medium fluid (HEC), before mix with drilling mud. Since HEC was not available, graphene material has directly mix into the drilling mud without medium fluid. This cause graphene material not totally dissolve in the drilling mud and some particles remain suspend. These maybe gave some effects towards the performance in drilling mud as shown in the results part of this project.

CHAPTER 5

CONCLUSION

Nowadays, deep water wells have been explored due to its high production rate. Unfortunately, the risks also will be higher rather than shallow water wells. In most cases, drilling fluids that will be used in drilling extremely hot wells is oil based mud. Today, we already have new invention to explore the ability of nano material enhancement in order to improve the performance of oil base drilling fluids. Eventually, there still no experiment has been done to test the effect of graphene as a nano material enhancement towards drilling fluids. Form this research, information on comparison based on the performance of the Sarapar 147 as conventional oil based mud and Sarapar 147 + Graphene as oil based mud with nano enhancement in term of rheology properties and behaviors in high temperature can help to identify which properties that graphene enhancement can give significant improve. Eventhough there are few factors affecting the outcome of the result which have been neglected, the result that was obtained however was still deemed to be of significance.

RECOMMENDATIONS

For recommendations, for further study, all the factors should be taken into account in order to get more accurate and significance result. Since the graphene enhancement shows an improvement in fluid loss test, it can be tested specifically as fluid loss additive to further the experiment. With this, we can observe how significant graphene enhancement improvement towards fluid loss test compares to conventional fluid loss additives. Furthermore, economic analysis also should be done in order to prove the applicability and practicality of this new technology in drilling mud for real operation environment.

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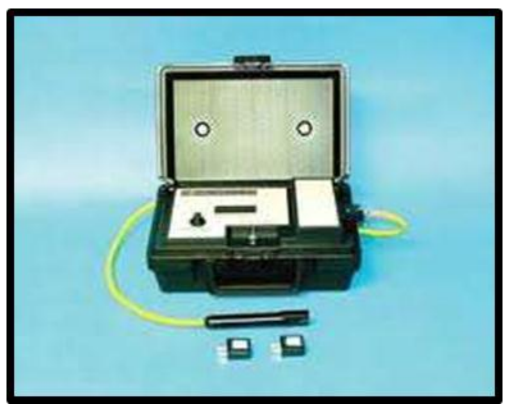
APPENDIX

	
Baroid Multimixer	**The FANN Model 35A Viscometer**
	
HPHT Filter Press	**Electrick Stability Kit**