POTENTIAL SEPIOLITE AS A SUBSTITUTE MATERIAL FOR DRILLING FLUID

By

NUR ZAFIRAH BINTI MAT RAZALI

DISSERTATION

Submitted to the Petroleum Engineering Programme
In Partial Fulfillment of the Requirements
for the Degree
Bachelor of Engineering (Hons)
(Petroleum Engineering)

Universiti Teknologi Petronas
Bandar Seri Iskandar
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CERTIFICATION OF ORIGINALITY

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

NUR ZAFIRAH BINTI MAT RAZALI

ABSTRACT

In this study, rheological behavior of Sepiolite as a drilling fluid are investigated and comparison study between rheological properties of Bentonite with Sepiolite has been made to ensure optimum value of Sepiolite rheological properties. In high salt contamination and high temperature well, the utilization of Bentonite mud system lead to undesirable rheological performance. Hence, process of removing drilling cuttings is disturbed; unacceptable fluid losses and optimum equivalent circulation density (ECD) can not be achieved.

This research is focus on rheological properties of Sepiolite at various temperatures and brine concentrations for instances at 150^0F and 250^oF of 16 hours hot rolled. Semi saturated and saturated brine of 5 lb and 10 lb potassium chloride are formulated in Sepiolite mud system. Furthermore, contradistinction analysis between Bentonite and Sepiolite are analyzed to confirm the efficacious of Sepiolite performance.

In order to design an effective drilling fluid system, fresh water is mixed with potassium chloride to obtain a brine solution. Soda ash which is hardness material is added in the solution and mixed for duration of 1 minute. Next, caustic soda is circulated into the solution for about 5 minute. The function of this chemical is to adjust pH value between 9.0 and 9.5. After that, HYDRO PAC-LV is added and mixed for 5 minutes followed by Bentonite. Last but not least, barite is added for 20 minutes as a weighting agent.

In conclusion, Sepiolite mud system performs better than Sabah and India Bentonite. Based on the pattern achieved from laboratory experiment, Sepiolite gives best rheological values followed by Sabah Bentonite and India Bentonite. Nevertheless, Sepiolite mud system exhibits highest fluid loss compare to the other Bentonite. This situation can be treated by using higher performance of fluid loss reducer.

ACKNOWLEDGEMENT

First and foremost, I would like to give thanks to the Almighty who made all things possible. I also would like to express my utmost gratitude to my helpful supervisor namely Dr Sonny Irawan who have greatly helped me in giving opinions, suggestions and advices as well as his continuous encouragement without whose knowledge and assistance this study would not have been successful. He inspired me greatly to work in this project. His determination to motivate me contributed tremendously to my project.

Upon my request to the Geosciences & Petroleum Engineering lab executive, I have been given special access to drilling fluid laboratory during semester break in the month of December. Hence, I would like to acknowledge and extend my heartfelt gratitude to those who helped set up the laboratory equipments that allowed me to run my experiment which without them, I could not have written this thesis. They are Amirul Qhalis B Abu Rashid and Ahmad Shahrul B Ahmad.

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NOMENCLATURE

μp: plastic viscosity

τ: Shear stress

τo: yield stress

k: power law consistency index

n: power law flow behavior index

Wwt: The weight of the moisture specimen with tare

Wdt: The weight of the dried specimen with tare

Wt: The weight of the container

LL: The soil's liquid limit

N: The number of blows until the soil groove closed

MC: The soil's water content

NM: The soil's natural moisture content, in percent

PL: The calculated plastic limit

PI: The calculated plasticity index

CHAPTER 1

INTRODUCTION

1.1 BACKGROUND OF PROJECT WORK

The employment of Bentonite mud system result in unacceptable rheological properties in high salt contamination and high temperature environment. Drilling fluid circulation is disturbed, unusual high fluid losses and viscosities, as well as ridiculous gel strengths values are the major problems that are commonly associated with high temperature high salinity well. Therefore, it is challenging to formulate a drilling fluid operating sufficiently notably in temperature above 250 $^{\circ}$F. Apart from that, physical and chemical properties of Bentonite mud system are changed due to this condition.

Previous studies on utilization of Bentonite suggest that, hydration competency of current Bentonite is reduced at high water salinity condition. The present of salt have become main interference for hydration to occur. On the other hand, Sepiolite mineral are not affected by these condition. This is due to its chemical and physical structure. Alternatively, Sepiolite which is a magnesium silicate with fibrous texture can extends performance of drilling fluid in both conditions. Although there might be a little change in Sepiolite structure due to high temperature, Sepiolite is stable up to 500°F. Previous study indicate that Sepiolite based drilling fluid is superior to the Bentonite based drilling fluids in terms of both rheological and fluid loss properties under elevated temperature and pressure conditions, particularly at high salt concentrations [1]. This paper is an attempt to propose Sepiolite mud system as a replacement for Bentonite mud system. The evaluation of Sepiolite is based on rheological properties of Sepiolite. After acquire necessary data, comparative study between Sepiolite and Bentonite is conducted to ensure performance of Sepiolite as a drilling fluid.

1.2 PROBLEM STATEMENT

In general, performance of Bentonite mud system is less effective at high temperature well because it can easily be decomposed due to Bentonite flocculation that lead to unfavorable rheological properties. According to former study, flocculation of Bentonite occurs when the borehole temperature is greater than 347°F [1]. Rheological properties values skyrocketed even though the clay is treated with additives. As a result, high pump pressure is required to initiate the flow of drilling fluid. Circulation breaks, abnormally high fluid losses and unacceptable high gel strength happened. In order to overcome this matter, expensive mud additives should be added, expertise must be hired not to mention the damage caused to the well. As such, drilling cost will be increased as problems arise.

Apart from that, Clay has ion exchange properties due to isomorphous substitution. Adsorption of cations produces sheets with net negative charge. These is unaffected by pH changes. Therefore, Bentonite easily swells in water. Nevertheless, mass action by high concentration of salt restrains Bentonite hydration that contributes to borehole instability. Thus, a fibrous clay mineral called attapulgite may be applied when the water salinity is too high as Bentonite replacement. However, attapulgite is unable to give good performance in high temperature environments even though they can perform best at high salinity condition. Normally, high salinity environment is also high temperature environment. Therefore, the application of attapulgite is limited. This requires the search for substitute clays. In addition, flow properties of Bentonite suspensions vary particularly under bottom-hole condition. Flow behavior at high temperature depicts entirely different flow behavior at low temperature even though it's the same Bentonite. It is crucial to have drilling fluid capable of sustain variety of borehole condition. Furthermore, the usage of Sepiolite mineral is neglected regardless it's important.

1.3 SIGNIFICANT OF PROJECT

Sepiolite mud system performs effectively at high temperature well because its chemical structure hardly deteriorated. Consequently, this mud system presents good rheological properties. Hence, drilling cost could be optimized. In addition, Sepiolite mineral give positive response toward salinity concentration. Sepiolite can adsorb water on its external surface that initiates swell and hydration.Although there could be temperature dependent changes in crystalline structure, Sepiolite are stable at temperatures up to 800 ^{O}F. [3] This is pleasurable condition because strong borehole stability can be achieved. As seen, study on rheological properties of Sepiolite is important to minimize drilling problem, reduce cost and offer good drilling fluid performance. Other than that, comparison study between Bentonite mud system and Sepiolite mud system is significant to ensure Sepiolite mud system performs better than Bentonite mud system. The results can be utilized as a significant reference for future recommendation.

1.4 OBJECTIVES

The objectives of this project are as follows:

1. To determine rheological properties of Sepiolite as a drilling fluid.
2. To compare rheological properties of Sepiolite with Bentonite as a drilling fluid.

1.5 SCOPE OF STUDIES

The scope of study is minimized to determine the rheological properties of Sepiolite at different temperatures and brine concentrations. Temperature variations are at $150^{0}F$ and $250^{0}F$ of 16 hours hot rolled. There are 2 different solutions as base fluids which are semi saturated and saturated brine. The type of salt used is Sodium Chloride (KCl). Semi saturated solution is added with 5 lb KCl while saturated solution is added with 10 lb KCl. Standard product of Bentonite will be taken from India and Sabah. API RP-13B standard procedures are employed during the laboratory work. Rheological properties are based on Plastic Viscosity (PV), Yield Point (YP) and Gel Strength.

Plastic Viscosity of Sepiolite mud system is determined to analyze viscosity of mud. It is preferable to have PV value as low as possible. Size, surface area and concentration of solids give great influence to PV value. Small solid size will result in large surface area of solid. As a result, high PV value is obtained. Therefore, it is important to control the speed of mixer. On the other hand, YP is the indication of the mud to suspend solid at dynamic condition. In contrary, 10 minutes and 10 seconds gel strength is the indication of the mud to suspend solid at static condition in the standard API procedure, even though measurements after 30 minutes or 16 hours may also be made. The examples of static conditions are tripping and cementing. Gel strength must behave in thixotropic manner so that they perform gel structure. All these tests will be run for the purpose of this study.

1.6 THE RELEVANCY OF THE PROJECT

Worldwide demand for energy keeps on increasing at rates never before seen. The demand is encompassing geographically and influences all energy sources. Projections of continued growth in hydrocarbon demand are driving the oil and gas industry to explore new or under-explored areas. As the search for petroleum becomes more extreme in terms of depths, pressures, and temperatures, the relevance of Sepiolite mud system as a drilling solution is highly unquestionable.

Sepiolite mud performs best in high temperature high salinity environment. Furthermore, this mud can be utilized in geothermal well. Sepiolite mineral that possess stable crystalline structure over Bentonite is one of the main reasons for higher performance drilling fluid compare to Bentonite.

The proposed study is highly relevant in Asia context, considering limited research had been done in this study. Consequently, Sepiolite mineral can be commercialized in drilling fluid application. This paper is aiming to examine potential Sepiolite as substitute material in drilling fluid.

1.7 FEASIBILITY STUDY

The Gantt chart prepared serves of how this study evolves and move through the end of project. Measurement of Sepiolite rheological properties start from early November 2010 till the end of January 2011. Long time consumption is due to try and error process to find the best formulation of mud. Other than that, the mud is hot rolled for 16 hours. Repetitive laboratory experiment had been taken into account so that accurate result will be produced. Documentation of results finished in March 2011.

CHAPTER 2

THEORY AND LITERATURE REVIEW

2.1 THEORY

Rheology fluids can be categorized as Newtonian or Non-Newtonian fluid depending on their response to shearing. Shear stress is proportional to the shear rate for a Newtonian fluid. Most drilling muds are non-Newtonian fluids, with viscosity decreasing as shear rate increases, and correspond more closely to one of the other three models which are Power Law Model, Bingham Plastic Model and Herschel-Buckley Model. For Bingham Plastic Model, Power Law and Herschel-Buckley Model, the shear rates are as follows [6]:

Bingham plastic: $$\tau = \tau_0 + \mu_p \dot{\gamma}$$

Power Law: $$\tau = K\dot{\gamma}^n .$$

Herschel-Buckley: $$\tau = \tau_0 + K\dot{\gamma}^n$$

Where the μp is the plastic viscosity, τ is the shear stress, τo is the yield stress, K is the power law consistency index and n is the power law flow behavior index. An excellent case can be made for generalizing the Bingham plastic model to a Herschel-Buckley. [6]

Mud rheology is adjusted with additives or dilution to meet the needs of the operation. In water-base fluids, water quality plays an important role in how additives perform. Temperature affects behavior and interactions of the water, clay, polymers and solids in a mud. [6]

The rheological properties of drilling fluid are determined using a motor driven Fann 6 Speed Viscometer. Since the offshore rig usually operate of 60 hertz, the viscometer motor runs at the same electrical cycles (60 herts) as the rig power. Drilling fluid is located in the annular space between two concentric cylinders. Constant rotational velocity is applied by the outer cylinder or rotor sleeve that produces a torque on the inner cylinder namely bob. Consequently, the dial attached to the bob will detect bob displacement. This procedure follows by API 13RB for field testing water based drilling fluids.

Among the most important dial reading in mud rheology is the 6 rpm reading. The equations below are for rheological properties. Normally, mud programs will define a range for the 6 rpm reading. This will lead the other indicators of rheological properties such as plastic viscosity, yield point and initial gel strengths become a function to meet this low end specification. Research proves that the initial gel strength should be more or less the same as the 6 rpm reading.

If the value of 10 minute gel strength higher than 10 second gel strength, it depicts an increasing trend and a widening divergence which are a good indicator of colloidal solids build up that may not be detected by solids analysis. This is resulted due to solids percent may remain as the actual size of the particles that lead to decrease of surface area as degradation occurs.

Gel strength behave in thixotrapic behavior can be defined as when a mud form gelled structures when stagnant and liquefying while sheared. The specific gel strength of drilling fluid is denoted as low-flat, progressive flat ad high-flat. The low-flat is most desirable curve meanwhile progressive and high-flat is undesirable.

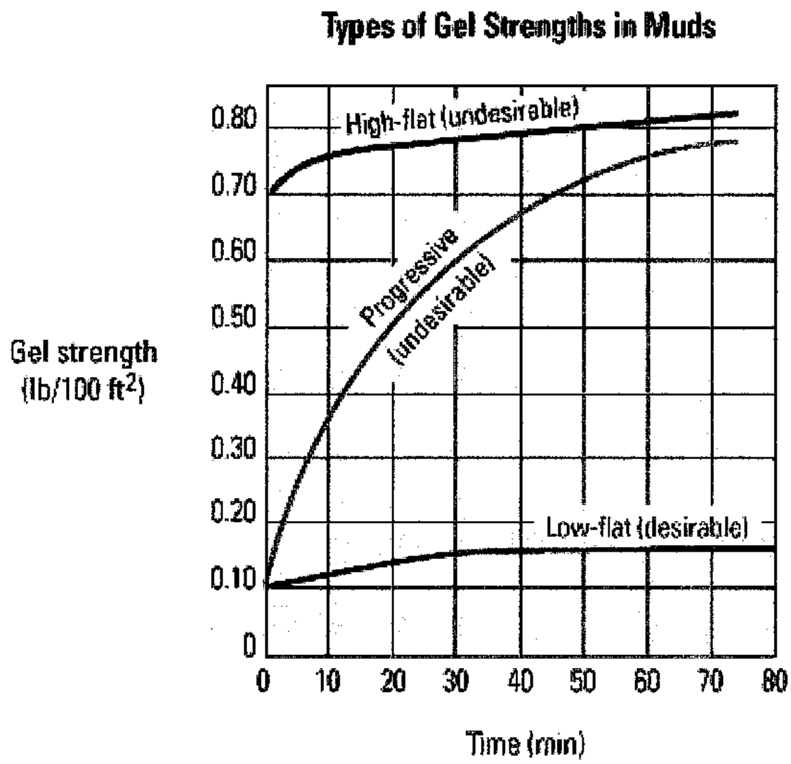


Figure 1: X-Y plot of gel strengths [12]

The equations related to mud rheological properties are listed below:

Apparent Viscosity (AV) in Centipoises (cps) = 600 reading ÷ 2
Plastic Viscosity (PV) in Centipoises (cps) = 600 reading - 300 reading
Yield Point (YP) in lb/100 ft2 = 300 reading – PV
Yield Stress = 2 x 3 reading = 6 reading
Gels in lb/100 ft^2 = as per 10 sec & 10 min reading

All these equations above are obtained from API RP-13B Standard procedures.

Although there could be temperature dependent changes in crystalline structure, Sepiolite is stable at temperatures up to 800°F [1]. Refer to figure 1 and 2 in appendix section. As we can see, the crystalline structure in figure 1 is very tightly bonded to each other compare to figure 2 where their crystalline structure is loose between each other. In conclusion, almost all drilling fluid sample contain Sepiolite mineral, is able to provide viscosity, This is utmost important to achieve safe drilling operations with optimal cost needed for the life of the well.

Several tests are involved to determine the quality of mud either it can be used in drilling operation or not. There are:

1. **Methylene Blue Test (MBT)** is a measure of cation exchange capacity (CEC). (CEC) is a measure of the potential reactivity of the clay components

CEC (meq/100g) = mls of methylene blue solution x 1.95 Meq = milliequivalent

2. **Moisture Content (MC)** is calculated with the following formula:

$$MC = 100\% * \frac{W_{wt} - W_{dt}}{W_{dt} - W_t}$$

This test tells us something about the type, size and therefore the active surface areas of solids in the mud.

3. The multi-point **Liquid Limit (LL)** is defined as the moisture value corresponding to the intersection of a regression line of blow counts and moisture values with a vertical line extending from 25 blows.

$$LL = MC * \left(\frac{N}{25}\right)^{0.121}$$

4. The **Liquidity Index (LI)** is calculated as:

$$LI = \frac{NM - PL}{PI}$$

All these equations above are obtained from ASTM D 2487 and ASTM D 3282.

2.2 LITERATURE REVIEW

KCL Polimer Mud, seawater gel mud and Sepiolite mud were tested. It was difficult to evaluate the effect of these three drilling mud to the borehole stability from the result of experiment [4]. Sepiolite mud gives best rheological values if the liquid phase contains no salt. However, acceptable viscosities are still achieved even if liquid phase is fully saturated with NaCl at ambient conditions. [1] The yield of the material from this deposit is dependant on the percentage of Sepiolite present in the sample and the grind size. [9] This show that Sepiolite capable of providing borehole stability, gives best rheological properties and fluid loss control in fresh or saturated brine.

The effects of mixing speed, the mixing time, and the grain size on rheological and filtration properties for the Bentonite based mud have small effect at room condition and can easily be ignored. In contrary, Sepiolite mud has entirely different behavior at room condition. Mixing time and mixing speed influence viscosities and filtration properties due to grain size. If grain size is reduced, more surface area is exposed that lead to increase in plastic viscosity and yield point value. Because these clays are relatively unaffected by electrolytes, their viscosity is retained whereas bentonites flocculate and lose their high viscosity [5]. Bentonite and sepiolite particles showed Bingham plastic behavior and highly thixotropic flow according to their swelling properties in water. However, the mixture of them showed Newtonian flow and almost non-thixotropic behavior. Bentonite has adequate rheological and adsorption properties to be made as a drilling fluid except at high temperatures due to the increasing flocculation. Sepiolite also has adequate rheological properties but can be stable at high temperatures. However, sepiolite has poor fluid loss control and sensitive to contamination. Therefore, the mixture should have the adequate properties as a drilling fluid. [1]

The atterberg test results for the sepiolite showed a liquid limit of about 500%, a plastic limit of about 200% and a corresponding plasticity index of 300%. The activity coefficient of sepiolite was calculated to be 3. Since clays with activity coefficients > 3 classify as active clay. The maximum dry density and optimum water content of the sepiolite were obtained as 0.7 Mg/m^3 and 86% respectively. [10] The Cation Exchange Capacity (CEC) of sepiolite, using barium chloride method, was determined to be 10 meq/100g soils, and the specific surface area obtained using EGME method was 370 m^2/g. The high SSA and low CEC are typical characteristics for sepiolite and palygorskite which is in contrast with other clay mineral [10] The sepiolite tested showed that after four days, 32% swelling was obtained. The high water holding capacity of the sepiolite is about one order of magnitude larger than the illitic sample. Sepiolite has a very low amount of alumina in comparison to palygorskite and other clay minerals. It has highest silica ion concentration. Thus, it is not expected that sepiolite will directly affect ettringite development in post stabilization failure. [10]

Most sepiolite fibers have a diameter below the resolution limit of the light microscope Thus, the analysis of clays, soils and dusts for the presence of sepiolite may require the use of both X-ray diffraction and electron microscopy. The crystalline of sepiolite samples may vary considerably, but the strongest line at 1.21 nm in an X-ray powder diffraction pattern is best suited for its identification [11]. Single fibres may be visualized and characterized by means of transmission or scanning electron microscopy. Selected area electron diffraction or X-ray microanalysis for the characteristic magnesium: silicon ratio can confirm the identity of sepiolite particles [12]. Sepiolite and sodium sepiolite (loughlinite) have been classed with palygorskite among the hormitic clays [8]. Sepiolite may have been described geologically only in 1758, but it has been used in a nearly pure form for many hundreds of years in the Mediterranean basin for carving pipes and making pottery. [10] in 1847 E.F. Glocker first used the name 'sepiolite' for the minerai called 'Meerschaum' by C.E. Werner in 1788 and 'ecume de mer' by RJ. Hauy in 1801. [11]

The clay mineral Sepiolite belongs to a group of magnesium silicate with a fibrous texture whose idealized formula can be written as $Si_{12}Mg_8O_{32}.nH_2O$. Structural characteristics and physical and chemical properties make Sepiolite mineral unique in the clay mineral family. The Atterberg limits are a basic measure of the nature of a fine-grained soil. It involves liquid limit (LL), plastic limit (PL), plastic index (PI), moisture limit (MI), moisture content (MC), and ignition loss (IL).

Standard procedure for liquid and plastic limit based on ASTM D4318-84. These two limits depend on moisture content of the sample. The liquid limit is the moisture content where the clay changes from plastic to liquid state. Meanwhile, plastic limit is the moisture content at which a ball of clay when rolled to a diameter of 1/8 inch. Plasticity index is the different between liquid and plastic limit. Since qualitative mineral content can be analyzed, moisture adsorption (MA), moisture content (MC), and ignition loss (IL) can be determined. MA is the percentage of water loss when clay from saturated atmosphere is dried in an oven at 105oC. MA is used to figure out mineralogy nature of clay.

MC is the percentage of water loss when clay from saturated atmosphere is dried in an oven at 105oC. The ignition loss is the percentage of weight loss when dried clay at 105oC is fired to 1000oC in the furnace [4].

Table 1: Summary of mineralogy composition

SAMPLES	Bulk mineralogy	Clay mineralogy	% of clay minerals in whole sample	d(060)	Biscaye index	Kubler index
BEMG	Phyllosilicates (94%) – Quartz (2%) – Feldespars[a] (3%) – Calcite (1%)	Smectite (95%)	Smectite (89%)	1.525 Å	0.86	0.26° 2θ
		Illite (5%)	Illite (<5%)			
BEAL	Phyllosilicates (95%) – Quartz (traces) – Calcite (traces) – Plagioclase (2%) – Cristobalite (3%)	Smectite (100%)	Smectite (95%)	1.490 Å	0.90	–
SEPI	Phyllosilicates (97%) – Calcite (3%)	Sepiolite (100%)	Sepiolite (97%)	–	–	–
SABA	Phyllosilicates (52%) – Quartz (18%) – Calcite (15%) – Dolomite (11%) – Plagioclase (4%) Traces of random mixed-layers illite-smectite	Smectite (41%)	Smectite (22%)	1.520 Å 1.490 Å	0.30	0.27° 2θ
		Illite (38%)	Illite (19%)			
		Kaolinite (21%)	Kaolinite (11%)			
EGRIS	Phyllosilicates (85%) – Quartz (10%) – K-feldspar (3%) – Plagioclase (2%)	Smectite (12%)	Smectite (10%)	1.498 Å	0.53	0.22° 2θ
		Illite (39%)	Illite (33%)			
		Kaolinite (49%)	Kaolinite (42%)			
CMO	Muscovite (32%) – Quartz (33%) – Calcite (26%) – K-feldspar (6%) – Plagioclase (3%) Traces of Analcime	–	–	–	–	–

[a] Including K-feldspar and plagioclase in the same proportion.

Based on the table 1 above, treatment at 550 °C revealed a shift in reflection to 9.9 Å, which was consistent with a structural collapse process in a mineral with the interlayer space occupied by hydrated exchangeable. cations. All the identified features pointed to a trioctahedral smectite as the prevailing mineral of the sample [2]. The measurement of the Biscaye crystallinity index reaching a value of 0.86 indicates a good ordering of the smectite. On the other hand, the nearly 15 Å spacing of the d(001) reflection of the smectite, in a sample not saturated with cations, suggests that the interlayer space was mainly occupied by divalent cations. The subordinated clay mineral showed no changes in its basal d-spacing after application of the above described treatments, remaining around 9.9 Å, which identify this mineral as illite. Its crystallinity value according to the Kubler index is 0.26° 2θ [2].

CHAPTER 3

METHODOLOGY

A number of laboratory experiments are designed to discover the potential use of Sepiolite derived from clay mineral in drilling fluid application. The research methodology will be as follows:

1. Measurement of rheological properties (plastic viscosity, yield point and gel strength) of drilling fluid using Fann 35 Viscometer based on API standard procedure.
2. Comparison study of Sepiolite mineral with Bentonite.
3. Documentation of result.

3.1 EQUIPMENTS

Figures below show the equipment used for this study as well as their functions.

EQUIPMENT	FUNCTION	EQUIPMENT	FUNCTION
 Viscometer	To check mud rheology Consist of 6 dial reading	 Roller Oven	Simulated circulating conditions which is rotatable to support aging cell
 Electronic Balance	Measure chemical weight	 Mixer	Mix drilling fluid

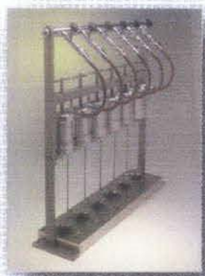
EQUIPMENT	FUNCTION	EQUIPMENT	FUNCTION
 Aging Cell	Mud is subjected in this cell during hot rolled	 API Filter Press	Carried out at ambient temperature and with 100 psi of differential pressure.

Figure 2: Equipment and its function [API Specification 13A, 1995]

The proposed drilling fluid formulation for semi saturated and saturated brine is as follows:

Table 2: Drilling fluid formulation

MUD	1	2	3	1	2	3	Mixing Order	Period (Minutes)	Function
Fresh water	319.73			317.70			1		Base
Potassium Chloride	5.00			10.00			2	2	Brine
soda ash	0.50			0.26			3	1	Hardness
Caustic soda	0.25			0.27			4	1	pH
HYDRO-PAC LV	4.00			5.10			5	5	Bridging Agent
Sabah Bentonite	28.34			28.16			6	5	Viscosifier
India Bentonite		28.34			28.16		7	5	Viscosifier
Sepiolite			28.34			28.16	8	5	Viscosifier
DRILL-BAR	62.42			58.68			9	20	Weighting Agent

All formulations were hot rolled at 150^{0}F and 250^{o}F for duration of 16 hours.

3.2. KRAMAT PULAI SEPIOLITE

In this research, Sepiolite used was taken from Kramat Pulai, Perak. It occurs as a white fibrous mineral in the forms of stringers, vein and films along irregular fractures in the marble host rock from an abandoned quarry. The following other properties of Sepiolite are as shown in the table below:

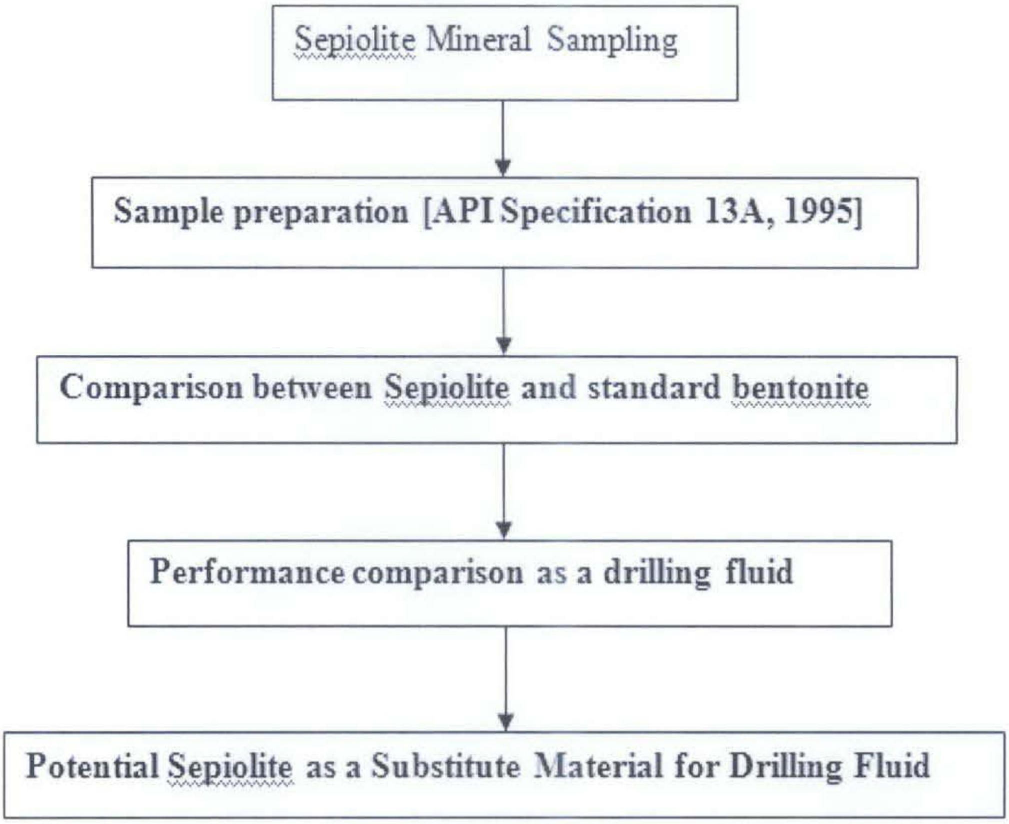


Figure 3: Project analysis study

3.3 CHEMICAL COMPOSITION

These figures below represent chemical composition of Sepiolite sample in comparison with bentonite sample [2]

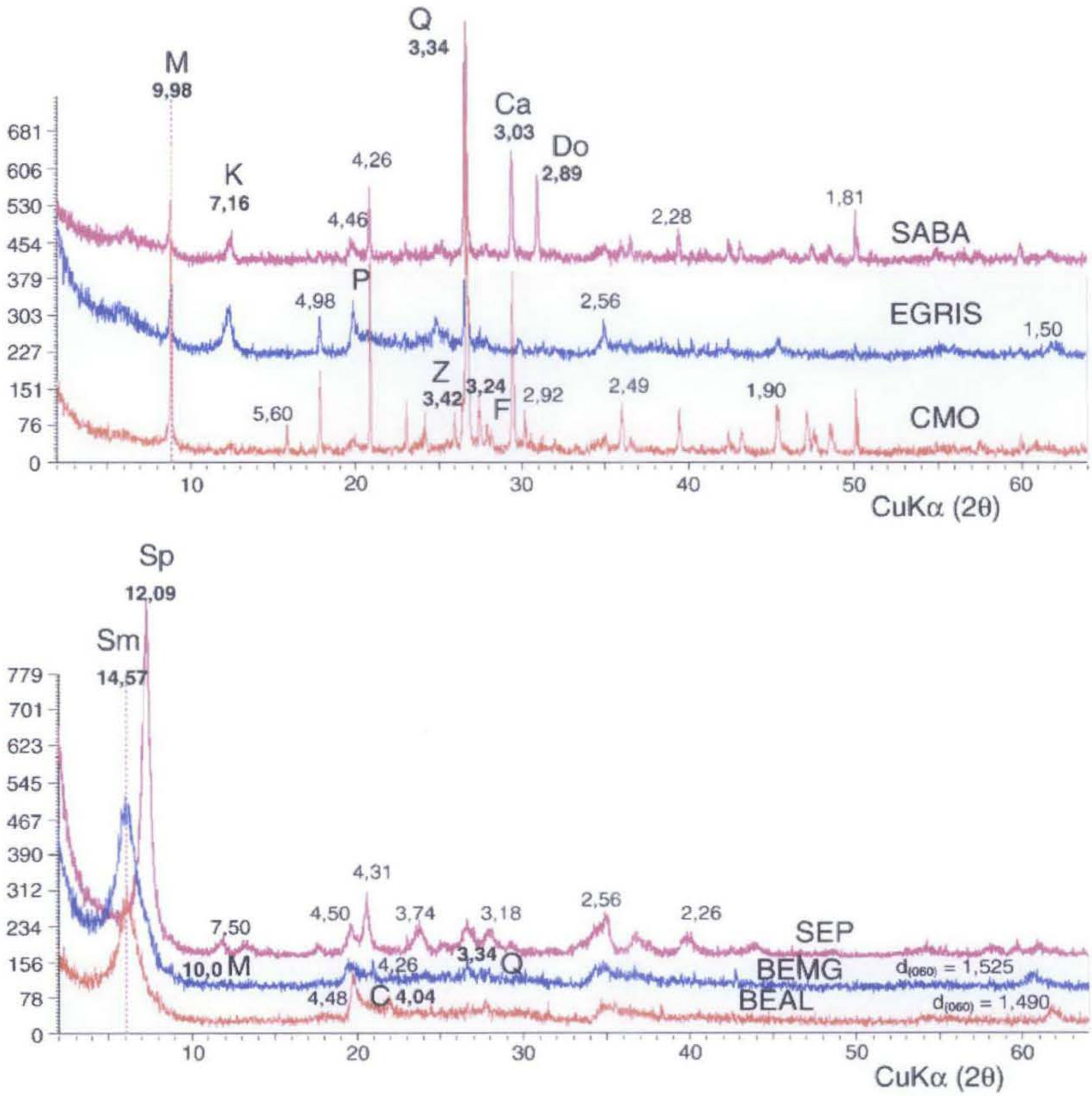


Figure 4: XRD random powder patterns of studied bulk samples showing the main reflections (Å) and minerals identified. M. Mica. K. Kaolinite. P. Phyllosilicates. Z. Zeolites (analcime). Q. Quartz. F. Feldspars. Ca. Calcite. Do. Dolomite. C. Cristobalite. Sm. S

The BEMG sample which is bentonite sample showed a high percentage of phyllosilicates in the total fraction (N90%) with characteristic reflections at 14.6 Å, 4.55 Å, 2.58 Å and 1.525 Å. The last d-spacing, which corresponds to d (060) reflection, clearly shows that the phyllosilicates are trioctahedral. The presence of other minerals was very low, namely limited to traces of quartz, calcite and feldspars [2].

The study of the clay fraction revealed the existence of two minerals, which, after the appropriate treatments, led to infer the existence of a predominant swelling phase and a highly subordinated non-swelling phase. Thus, the study of the Mg2+ and K+ saturated oriented aggregates revealed d-spacing with very intense reflections at 15 Å and 12.5 Å, respectively. Treatment with ethylene glycol caused this reflection to shift to 17.0 Å, which revealed a 2:1 swelling phyllosilicate as shown in figure 5 [2].

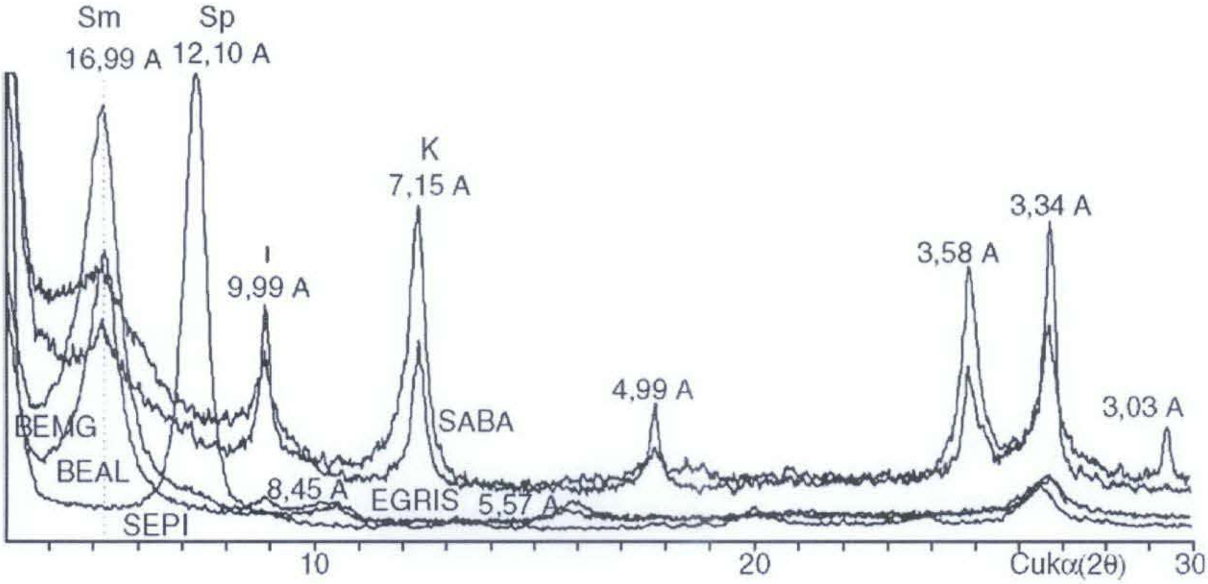


Figure 5: XRD traces from clay fraction oriented samples, previously Mg2+-saturated and solvated in ethylene glycol atmosphere. Sm. Smectite. Sp. Sepiolite. I. Illite. K. Kaolinite [2]

3.4 RAW SAMPLE PREPARATION

Analysis of raw sample preparation, which included the field sampling, drying process and sample sieving, were analyzed.

3.4.1 Field Sampling

Raw samples were collected from this area, Kramat Pulai Mine, Pulai, Kinta District, Perak, Malaysia as shown in figure 6.

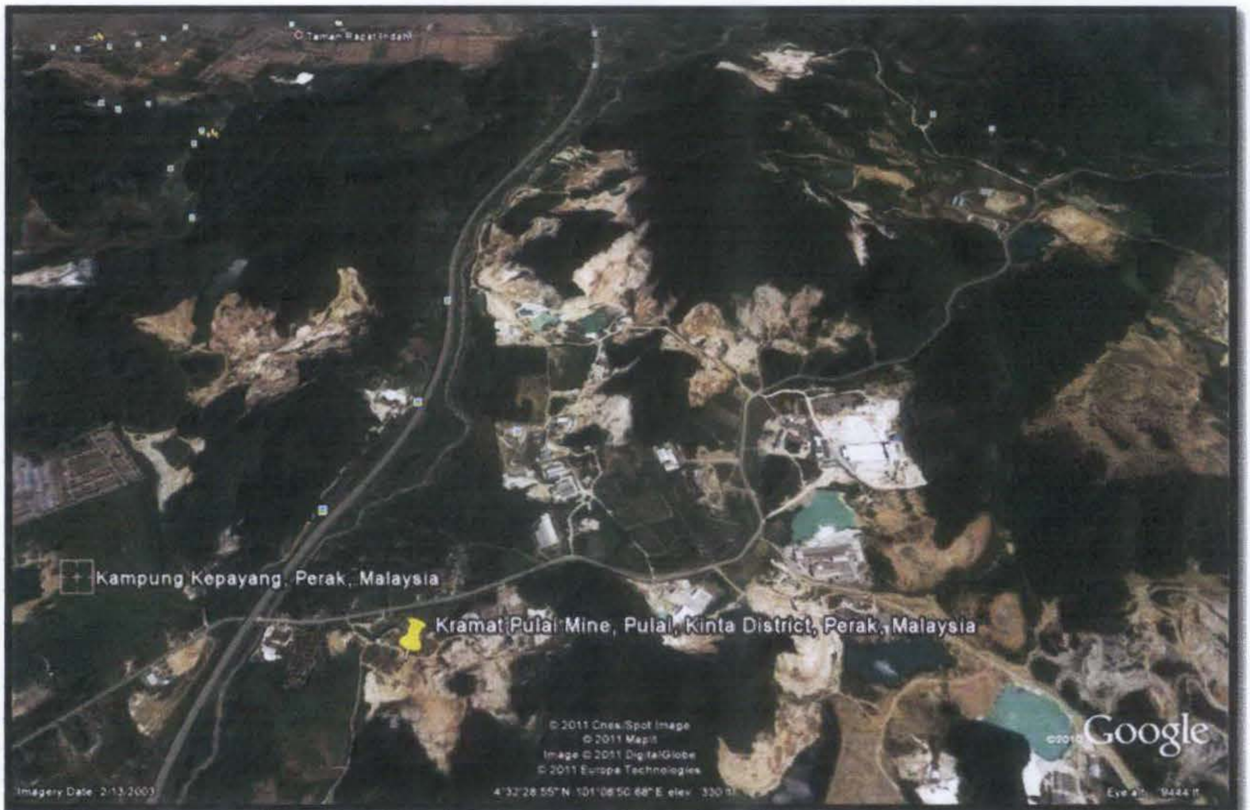


Figure 6: Location of, Kramat Pulai Mine, Pulai, Kinta District, Perak, Malaysia.

The depth of sampling was taken 0.5 meter. The detail procedures for sampling are as follows:

1. Auguring was carried out along a square grid of 200 meters x 200 meters spacing by using the Dormer hand auger to collect the raw samples.
2. The samples were collected and placed inside a plastic bag of 30 centimeters x 20 centimeters size and sealed properly to avoid any moisture loss from the raw sample.
3. The plastic bags with raw sample were put into a pail and close the cover properly.

3.4.2 Drying Process

Since the raw sample from the field are still high contaminations and high moisture content, it must be corrected to get accurate result. The procedure of raw sample preparation is as follows:

1. Hammer was used to crush the wet raw samples from the field.
2. Those samples was placed in an oven for duration of four hours at temperature of 55°C until it reached moisture content less than 10%.
3. The dried raw sample was transferred to the grinding machine and crushed till it transform to powder (kept in container to maintain the value of moisture content to be lower than 10%)
4. The raw sample powder was sieved to 75 μm

3.4.3 Performance Test as a Drilling Mud Material

The sepiolite and standard product of bentonite samples have undergo a series of testing to determine its capability as a drilling mud material in accordance to the standard requirements of API 13A Section 4 specifications (API Specification 13A, 1995).

3.4.4 Rheology Test

1. Placed the mud sample in a viscometer cup.
2. 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.
3. Having taken the 3-rpm reading, stirs the sample at 600 rpm for 30 seconds before taking the 10-second gel at 3 rpm.
4. Re-stir the sample at 600 rpm for 30 seconds and leave undisturbed for 10 minutes, ensuring the temperature stays at 120 °F (48.9 °C).
5. Take the 10 minute gel reading at 3 rpm.
6. The significant of this test is to find out the value of plastic viscosity, yield point and gel strength at 10 seconds and 10 minutes.

CHAPTER 4

RESULTS & DISCUSSION

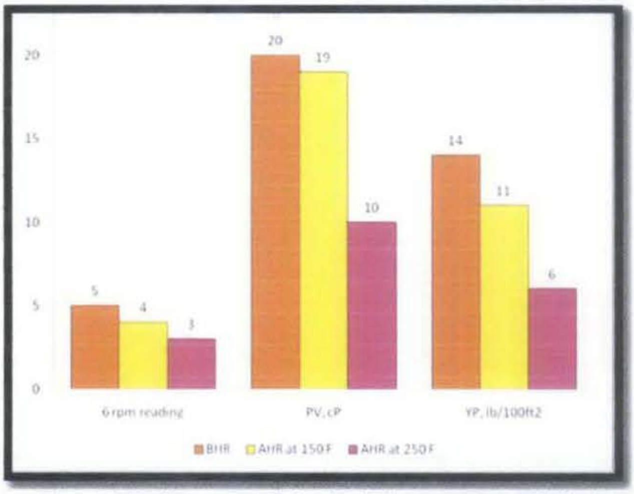


Figure 7: Performance of rheological properties, 10 lb/gal WBM, KCL concentration of 5 lb with Sabah Bentonite

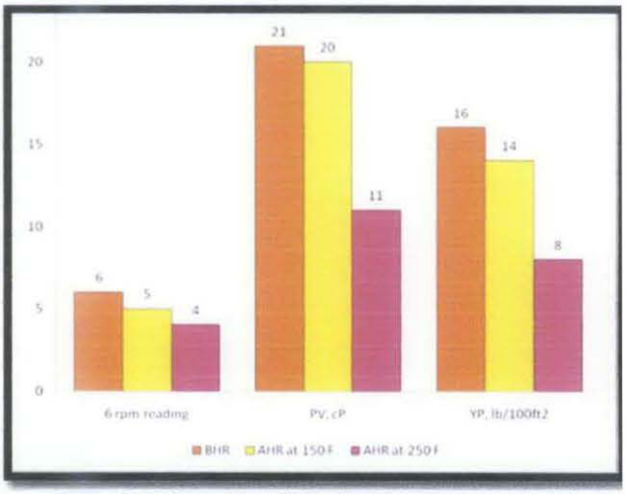


Figure 8: Performance of rheological properties, 10 lb/gal WBM, KCL concentration of 5 lb with India Bentonite

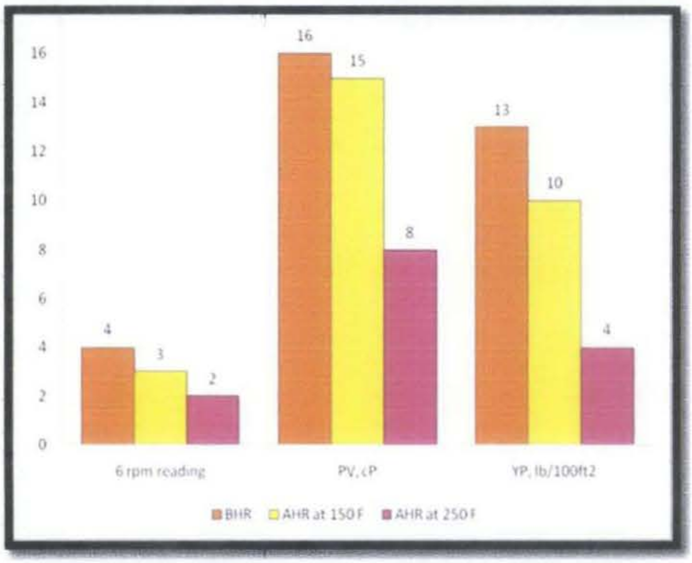


Figure 7: Performance of rheological properties, 10 lb/gal WBM, KCL concentration of 5 lb with Sepiolite

This section analyses the results of the experiment. The experiment went as expected with no unusual events that would have introduced error. The pattern of 6 rpm dial reading, PV and YP is decreasing in value after subjected to 16 hours hot rolled at 150 ^{0}F and 250 ^{0}F (Refer to figure 7, 8, and 9). This sequence can be observed through out the 3 types of drilling fluid graph. The 6 rpm dials reading is the indicator of mud gel strength. The reading of 6 rpm is getting lower after exposed to higher temperature. Therefore, the ability of the mud to suspend drilling cutting and perform gel structure at high temperature is reduced. The reason behind this phenomenon is because the product inside the mud failed to behave base on their function. Other than that, this is due to water viscosity is reduced when exposed to high temperature. So, plastic viscosity decreases proportionally.

Even though the entire PV value pattern is decreasing, the value of Sepiolite is still lower compare to Sabah and India Bentonite. This is favorable condition because if Sepiolite is subjected to greater temperature, it still can survive the extreme condition unlike Sabah and India Bentonite. Yield point is a function of concentration of mud solids and their surface charges and possibilities which affect inter particle forces. Dispersants and deflocculating adsorbed on the mud particles. This action changes the chemical behavior of the surfaces and therefore affects the inter particle forces, resulting in viscosity and yield point reductions.

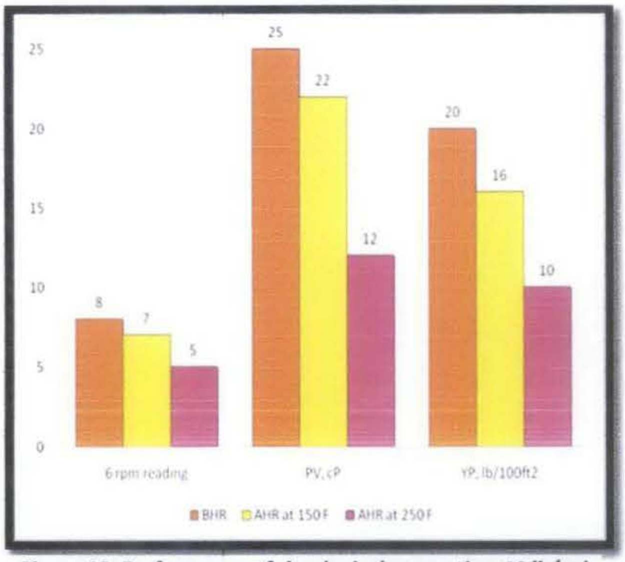


Figure 10: Performance of rheological properties, 10 lb/gal WBM, KCL concentration of 10 lb with Sabah Bentonite

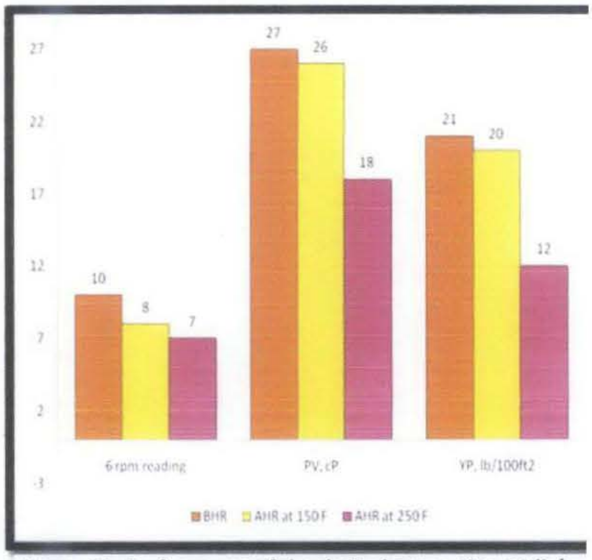


Figure 11: Performance of rheological properties, 10 lb/ga WBM, KCL concentration of 10 lb with India Bentonite

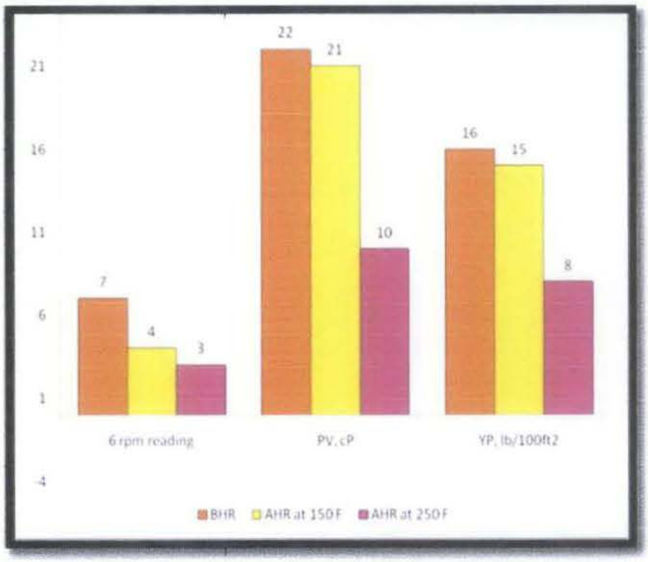


Figure 12: Performance of rheological properties, 10 lb/gal WBM, KCL concentration of 10 lb with Sepiolite

= Before Hot Rolled

= After Hot Rolled 150^{0}F

= After Hot Rolled 150^{0}F

Rheological properties of10 lb/gal WBM, KCl concentration of 10 lb performance is summarized in the graph above. Viscosity value for each mud is higher compare to 5 lb KCl concentration mud. This is due to more solid concentration available inside the mud compare to 10 lb/gal WBM, KCl concentration of 5 lb. Since PV is a function of solid concentration, the PV value for 10 lb/gal WBM, KCl concentration of 10 lb with increasing of solid concentration. The same principle applies for 6 rpm dial reading and YP value.

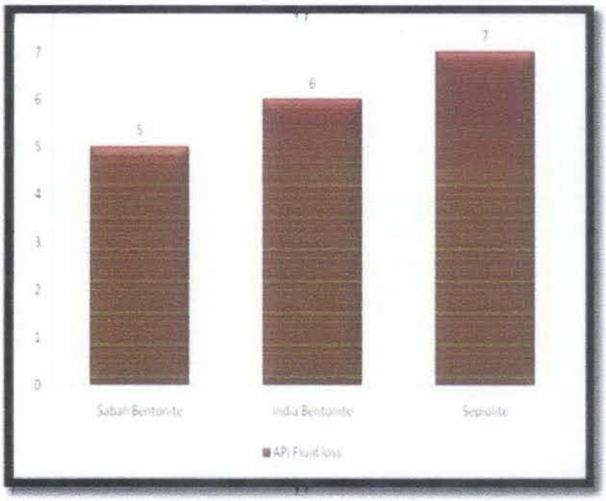


Figure 13: Performance of API fluid loss, 10lb/gal WBM, KCL concentration of 10 lb

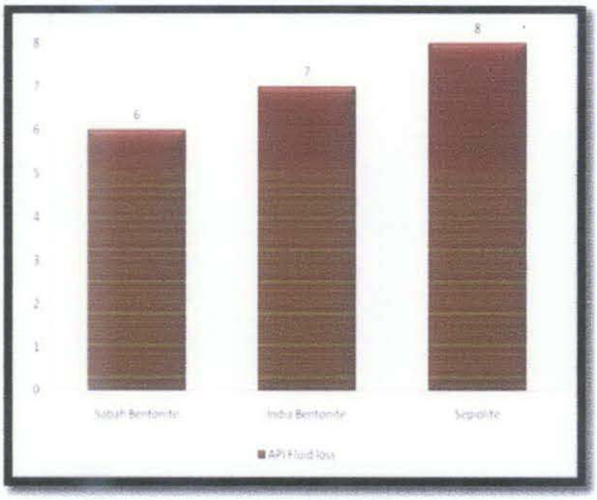


Figure14: Performance of API fluid loss, 10 lb/gal WBM, KCL concentration of 5 lb

A comparison between the graph showing performance of API fluid loss, 10 lb/gal WBM, KCL concentration of 10 lb and 5 lb, reveals differences. Sepiolite generates higher fluid loss compare to Sabah and India Bentonite. Several errors could explain the differences. Sepiolite mud has distinguished behavior at ambient condition. Mixing time and mixing speed effect filtration properties due to grain size. If grain size is reduced, more surface area is exposed and led to increase in solid concentration. Generally the fluid loss arises with increased solids content. The thickness of the cake is influenced by the particle size and the particle size distribution.

Apart from that, the thickness of the filter cake is assumed to be proportional to the filter loss for any given mud. According to the above graphs, high fluid loss can be resulted due to grain size distribution in the mud. These factors are negligible for Bentonite mud system because mixing time and speed do not give significant variation in result.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 CONCLUSIONS

1. This project has managed to determine rheological properties of Sepiolite as a drilling fluid.

2. Comparison study of rheological properties has been made with standard product of Bentonite and proven to be superior in term of rheological properties followed by Sabah and India Bentonite.

3. Sepiolite mud gives best rheological value if the liquid phase contains less salt. However, acceptable viscosities are still attained even if liquid phase is fully saturated with KCl at atmospheric conditions.

5.2 RECOMMENDATIONS

1. There are more rooms for further investigations for example study regarding additives must be used to control filtration properties.

2. Sepiolite rheological properties can be experimented at 120 °F using heater cup and dynamic filtration equipment can be used to gain viscosity at accurate borehole condition.

3. Since Sepiolite mud offers best viscosities and the fluid loss can be treated using additives, Sepiolite mud is a good candidate as a drilling fluid particularly in geothermal well and high temperature high salinity well.

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APPENDICES

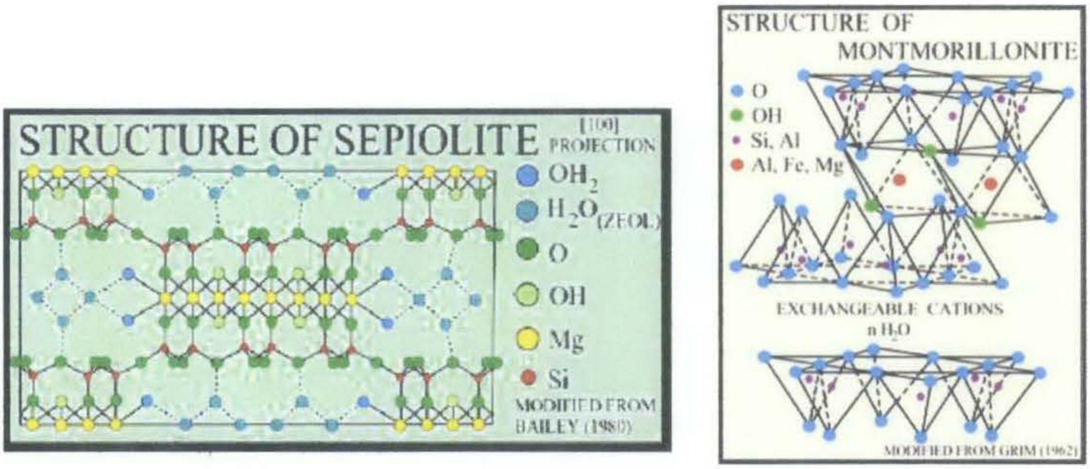

Figure 1 & 2: Basic Structure of Sepiolite Clay and Montmorillonite Clay [1], [5]

Test slurries: 20 lb Sepiolite in Saturated NaCl water as outline in API Specification RP 13A for oil well drilling-fluid materials.

Sample No	Description
1	Sepiolite + 20 Mesh Screen
2	Sepiolite - 20 Mesh Screen
3	Sepiolite - 20 + 40 Mesh Screen
4	Sepiolite - 40 + 60 Mesh Screen
5	Sepiolite - 60 + 100 Mesh Screen
6	Sepiolite - 100 Mesh Screen

Table 1: Effect of Grind Size on the Rheological Properties of Sepiolite Slurries [5]

Sample	1	2	3	4	5	6
Apparent Visc	31.5	19.5	17.5	15	15	13.5
600 Reading	63	39	35	30	30	27
300 Reading	56	33	28	26	27	24
PV	7	6	7	4	3	3
YP	49	27	21	22	24	21

Table 2: Rheological properties at ambient temperature

Mud/Initial Properties	1	2	3	1	2	3
Rheological properties at	77 F					
Mud weight, ppg	10					
600 rpm reading	54	58	45	70	75	60
300 rpm reading	34	37	29	45	48	38
200 rpm reading	19	22	14	24	30	20
100 rpm reading	11	12	8	13	20	10
6 rpm reading	5	6	4	8	10	7
3 rpm reading	3	4	2	5	6	4
PV, cP	20	21	16	25	27	22
YP, lb/100ft2	14	16	13	20	21	16
Gel strength 10 s, lb/100ft2	4	5	3	8	9	5
Gel strength 10 m, lb/100ft2	3	4	2	5	6	4
API FL, cc/30 min,ml	5	6	7	6	7	8
PH	9.2	9.4	9.1	9.4	9.4	9.4

(green) = 5 lb of KCL

(light orange) = 10 lb of KCL

Table 3: After hot rolled 16 hours of rheological properties at 150ºF

Mud / 16 Hours AHR Properties, 150ºF	1	2	3	1	2	3
Rheological properties at	77 F					
Mud weight, ppg	10					
600 rpm reading	49	54	40	60	72	57
300 rpm reading	30	34	25	38	46	36
200 rpm reading	17	20	16	22	28	18
100 rpm reading	9	10	8	12	18	8
6 rpm reading	4	5	3	7	8	4
3 rpm reading	3	3	1	6	5	3
PV, cP	19	20	15	22	26	21
YP, lb/100ft2	11	14	10	16	20	15
Gel strength 10 s, lb/100ft2	5	6	3	6	7	4
Gel strength 10 m, lb/100ft2	3	5	2	4	5	3

(green) = 5 lb of KCL

(orange) = 10 lb of KCL

Table 4: After hot rolled 16 hours of rheological properties at 250ºF

Mud / 16 Hours AHR Properties, 250ºF	1	2	3	1	2	3
Rheological properties at	77 F					
Mud weight, ppg	10					
600 rpm reading	26	30	20	34	48	28
300 rpm reading	16	19	12	22	30	18
200 rpm reading	10	12	8	12	14	10
100 rpm reading	6	8	4	8	10	7
6 rpm reading	3	4	2	5	7	3
3 rpm reading	2	3	1	3	4	2
PV, cP	10	11	8	12	18	10
YP, lb/100ft2	6	8	4	10	12	8
Gel strength 10 s, lb/100ft2	2	2	2	5	6	3
Gel strength 10 m, lb/100ft2	1	1	1	4	3	2

(green) = 5 lb of KCL

(peach) = 10 lb of KCL

The following table represents the activities, start date and end date.

Table 5: Project activities

Activities	From Date	To Date
Measurement of rheological properties	19/10/2010	31/01/2011
Comparison study of Sepiolite with Bentonite	1/2/2011	28/02/2011
Documentation of result	1/3/2010	14/3/2010

Gantt chart below consists of timeline and activities as well as timeline and the project milestone:

Table 6: Key milestone

Time	10-Jul					10-Jan				
Milestone	7	8	9	10	11	1	2	3	4	5
Completion of physical and chemical properties of Sepiolite										
Completion of rheological test										
Project completion										