ANALYSIS ON CHARACTERISTIC OF SABAH BENTONITE AT HIGH TEMPERATURE UNDER DYNAMIC CONDITION

by

Zainur Azwin binti Mokhtar

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

JULY 2008

Universiti Teknologi PETRONAS
Bandar Seri Iskandar
31750 Tronoh
Perak Darul Ridzuan.

CERTIFICATION OF APPROVAL

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

Zainur Azwin binti Mokhtar

ABSTRACT

This project is carried out to study the properties of Bentonite, particularly Sabah Bentonite. A series of experiments, by mixing the two Bentonite are conducted and comparing them to API specification, under dynamic condition, as to stimulate the real drilling process to achieve a more accurate result. The rheology properties of the drilling fluids, such as density, viscosity and yield point are obtained by conducting experiments using few apparatus, such as mud balance and direct indicating viscometer. The Fann 90 Dynamic Filtration Test Apparatus is used to get the outcome of the study by comparing the parameters of dynamic filtration rate and static filtration rate. The experiments are conducted first in Low Temperature (LT) condition and the target composition, 55% Sabah Bentonite and 45% Indian Bentonite is selected for the High Temperature (HT) condition. Sodium Hydroxide (NaOH) is then added from 0.5 ppb to 4.0 ppb for further rheological and filtration loss evaluation. The comparison of rheological properties of the three drilling fluids: Indian, Sabah and Mixture. For Sabah and Mixture, as the temperature increases, the clay swelling decreases, thus the PV, YP and GS of the mud decreases. For Indian, as the temperature increases, it caused the bentonite to become more dispersed, increasing the number of individual platelets in the suspension, thus PV, YP and GS increases. For mixture of 45% Indian and 55% Sabah bentonite, the dynamic filtration is higher than static condition at lower temperature but lower in high temperature as they are no real correlation between static and dynamic condition. Optimum of 0.5 ppb of NaOH added improved the rheological properties but not the filtration properties. The economic analysis showed that treated Andrassy sample is much cheaper than the commercial bentonite (60.71 % cheaper). This directly increases the gross domestic product (GDP) of Malaysia and can beneficiated the country. Further recommendations are suggested for next research.

ACKNOWLEDGEMENTS

First and foremost, I would like to extend the gratitude to Allah the Almighty with His blessings I have managed to undergo and finish the project in time.

I also would like to offer my appreciation to Universiti Teknologi PETRONAS (UTP) for offering and giving me an opportunity to take the Final Year Project (FYP) course. Also, I would like to acknowledge the FYP committee of the Mechanical Engineering Department for the approval for me to work on this project.

Highest appreciation and gratitude for my Project Supervisor, Dr. SONNY IRAWAN for his supervision, evaluation, guidance and support given to me from the beginning of the project until the end. His advises and help contributed a lot in completing the project.

Furthermore, I am glad to acknowledge all the Petroleum Engineering and Mechanical Engineering technicians, for their effortless help for me to complete the experiments. Also, to other Mechanical and Petroleum engineering lecturers for their advises and guidance.

Last but not least, I would like to thank everyone who was involved directly or indirectly for their contributions and moral support. I would like to apologize for any inconvenient and mistakes.

THANK YOU.

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CHAPTER 1

INTRODUCTION

1.1 Project Background

Drilling fluids, or mud, is any fluid that is used in a drilling operation, specifically to minimize the concentration of cuttings around the drill bit and throughout the well bore. The study of the behavior of these fluids can help to improve the rheological properties of the fluids, thus enhancing the production. Bentonite is an absorbent aluminium phyllosilicate generally impure clay consisting mostly of montmorillonite and widely used as a mud additive for viscosity and filtration control.

Indian Bentonite is a common and widely used drilling fluid, which meet the API Standard. This Bentonite is capable of absorbing 7 to 10 times its own weight in water, and swelling up to 18 times its dry volume. Sabah Bentonite is a local mud material, which is being developed to meet the API Standard requirements.

The experiments conducted in this project focuses on the mixture of Sabah Bentonite and Indian Bentonite at different percentages for high temperature and high pressure at dynamic condition to determine the dynamic filtration rate and compare it with static filtration rate, obtained from the previous project done.

1.2 Problem Statement

Drilling mud, a type of drilling fluid, also known as spud mud, is a fluid used in operations to drill boreholes into the earth. Often used while drilling oil and natural gas wells and on exploration drilling rigs but can also be used for much simpler holes, such as water wells. Bentonite is a common additive to spud muds to control viscosity.

To enhance the production, experiments are conducted for a more economical drilling fluid. This particular project focuses on experimentation of mixture of Sabah and Indian Bentonite at dynamic condition to study their rheological properties, thus study can be carried out for improvements.

1.3 Significance of Study

The study of characteristic of Sabah Bentonite at high temperature and pressure at dynamic condition can stimulate the real drilling process, thus produce a more accurate results than in static condition. From the previous project done, the static filtration is obtained from two phases; namely the low temperature (LT) and high temperature (HT). The composition of 55% of Sabah Bentonite and 45% of Indian Bentonite was chosen to stimulate the dynamic drilling condition by rolling the mud for 16 hours. The results for real dynamic filtration rate determined the outcome of the project, mainly comparing the results of the mixture of the selected composition and also with Sabah and Indian Bentonite itself. Improvement is then suggested so the second part can be carried out, depending on the effectiveness of the apparatus and the feasibility of the project. Thus, this project is carried out to study drilling fluids for a more economical drilling process.

1.4 Objectives

There are a few objectives of this project. The objectives are created to reach its main purpose of conducting the project, as stated in the followings:

1. To measure the rheological properties of the drilling fluids in dynamic condition
2. To observe the effect of Low Temperature Low Pressure (LTLP) and High Temperature High Pressure (HTHP) to the properties of the bentonites
3. To compare the properties for dynamic condition and static condition of the mixture of bentonites
4. To determine the concentration of Sodium Hydroxide (NaOH) to be added to improve the composition and result of the drilling fluids

1.5 Scope of Study

The project focuses on several aspects, namely;

1. The experiments conducted for this project will be conducted on Low Temperature (LT) first, and selected sample of composition will be tested in High Temperature (HT) condition
2. The following parameters discussed are the scope of study for this project are the mud density, plastic viscosity and yield point, and the parameters from the Dynamic Filtration Test which comparing the dynamic filtration rate and the static filtration rate.
3. The second part of the project will focus on the improvements, such as adding additives to the bentonite to achieve better results, depending on the effectiveness of the equipment and the feasibility of the project.

CHAPTER 2

LITERATURE REVIEW AND THEORY

2.1 Bentonite

Bentonite is clay with montmorillonite as its main mineral. It has many applications in various industries, such as in petroleum industry as drilling mud material and oilwell cement additives. Bentonite has an expanding lattice, where all layers surface are available for hydration and cation exchange. Bentonite structure is classified as dioctahedral, having two thirds of the octahedral sites occupied by trivalent cations. The use of bentonite depends on their properties; hence it is desirable to consider uses and properties together. The properties of bentonite are contingent upon the fact that they are composed of smectite clay minerals (Wikipedia, 2007, p.1).

Bentonite, when added to freshwater mud, makes the mud gel, causes the mud to stiffen (solidify like gelatin) when circulation stops. This stiffening effect holds the cuttings in place within the mud instead of allowing them to fall to the bottom of the hole. When the mud starts moving again, the mud reliquefies and flows normally (V.Dyke, 1998, p.13).

Clay water interaction is an all-inclusive term to describe various progressive interactions between clay minerals and water. In the dry state, clay packets exist in face-to-face stacks like a deck of playing cards, but clay packets begin to change when exposed to water.

There are five descriptive terms describe the progressive interactions that can occur in a clay-water system, such as a water mud. The first one explains on hydration occurs as clay packets absorb water and swell. Then, dispersion (or disaggregation) causes clay platelets to break apart and disperse into the water due to loss of attractive forces as water forces the platelets farther apart. Thirdly, flocculation begins when mechanical shearing stops and platelets previously dispersed come together due to the attractive force of surface charges on the platelets. Fourth, deflocculation, the opposite effect, occurs by addition of chemical deflocculant to flocculated mud; the positive edge charges are covered and attraction forces are greatly reduced. Fifth, aggregation, a result of ionic or thermal conditions, alters the hydrational layer around clay platelets, removes the deflocculant from positive edge charges and allows platelets to assume a face-to-face structure.

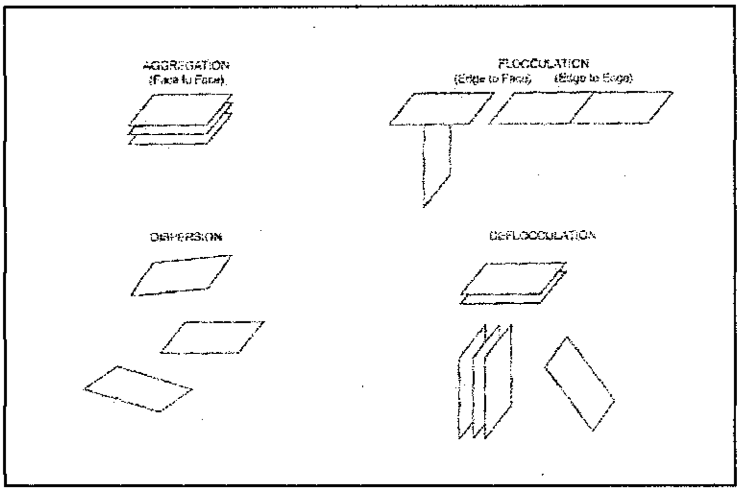

Figure 2.1: Clay-Water System Interaction [9]

2.2 Filter Cake

Filter cake, also called mud cake or wall cake, is a plasterlike coating of mud on the walls of the hole. In a porous, permeable formation, hydrostatic pressure squeezes the liquid part of a drilling mud into the formation. If this filtration process stayed constant throughout drilling, the crew would have to add liquid to the mud continuously to make up for the liquid lost to the formation. However, the solid material in the mud is left behind as a filter cake. A good filter cake slows the loss of liquid, called fluid loss, from the mud to a very low rate (V.Dyke, 1998, p.16).

Fluid loss that is too high is a problem for several reasons. One is that as long as the fluid filters into the formation, the filter cake keeps getting thicker. It may eventually become thick enough to reduce the diameter of the hole, causing tight spots where the drill string can get stuck. Second, muds with a high fluid loss may sometimes cause sloughing and caving of shale formations. Some shales are sensitive to water. That is, shale absorbs the water, swells up, and sloughs into the hole (V.Dyke, 1998, p.16). The liquid will finally entering the producing zones (the zones containing oil) may reduce the rate of oil flow when the well is ready to produce. This phenomenon happens because, like shales, some producing zones are sensitive to water. The water enters tiny openings of the zone and causes the surrounding rock to swell, thus blocking permeability. This blocked permeability is formation damage. A good filter cake is thin, slick and virtually impermeable. The crew may add finely ground clays or other substances such as polymers to drilling mud to improve its ability to form a filter cake, or wall-building ability (Soliman, 1994, p.6).

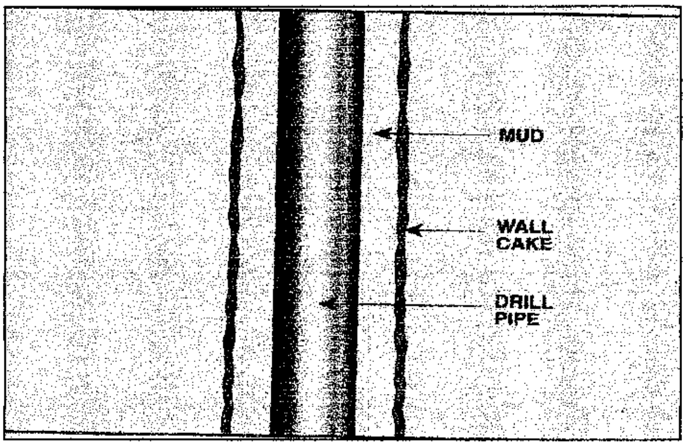

Figure 2.2: Formation of impermeable wall cake (V.Dyke, 1998, p.16)

2.3 Flows through Filter Cake

Drag force is exerted on the solid particles and cumulative as liquid moves towards the filter medium as suspended solids are deposited during cake filtration. On moving through the cake from towards the filter medium the drag pressure, P_S increases and hydraulic pressure, P_L decreases.

However, all filter cakes do undergo some compaction as P_S increases. These compressible filter cakes exhibit non-linear permeability and porosity profiles with maximum porosity at the cake surface ($P_S = 0$) and minimum at the filter medium surface ($P_S = dP$). The treatment of filtration is based on these three concepts; flow through filter cakes is laminar, all of the factors contributing to the resistance of the filter cake and fluid flow can be grouped into a single term called specific resistance and hydraulic pressure is converted by fluid drag to mechanical pressure on solids (K.G Arthur, 1988, p. 1).

2.4 Static and Dynamic Condition

There are two (2) types of filtration involved in drilling an oilwell:

1. Static filtration which takes place when mud is not being circulated and filter cake grows undisturbed.
2. Dynamic filtration, when mud is being circulated and growth of filter cake is limited by erosive action of mud stream.

Dynamic filtration rates are much higher than static rates, and most of the filtrate invading subsurface formations does so under dynamic conditions. The filtration properties of drilling fluids are usually evaluated and controlled by the API filter loss test, which is a static test, and is therefore not a reliable guide to downhole filtration unless the differences between static and dynamic filtration are appreciated, and the test results interpreted accordingly [1]. In this project, a special instrument, Fann Model 90 Dynamic Filtration System is used specifically for simulating dynamic condition thus comparisons can be made. Dynamic filtration rate can be evaluated over any interval during the filtration process. This is accomplished by calculating the rate of change in the filter volume versus time (ml/min). It is desirable to have a low filtration rate. The rate should be less than 0.2 ml/min for most oil well drilling fluid systems.

2.5 Effect of Temperature and Pressure

Fluid loss must withstand the temperatures in the wells, generally from about 100 to about 500° F. Bore hole temperatures can vary from ambient up to about 500° F. and pressures from atmospheric up to about 20,000 psi. Temperature and pressure conditions such as these can have an adverse effect on bore hole fluids causing them to destabilize if they contain additives.

These pressures and temperatures have a very strong effect in forcing the drilling fluid not only to the surface, but also against the side of the bore hole causing either filtrate loss or a break through of the drilling fluid, as well as the oil or gas under pressure into the permeable strata considerably below the opening of the well at the surface [11].

2.6 Additive

The use of rheology modification agents, frequently thixotropic agents, for aqueous fluids has been common practice in a large number of industries. These fluids include, for example, oil field drilling fluids, metal-working fluids, mining fluids, fire control fluids, agricultural organic formulations, water-based paints and coating fluids, stripping fluids, and the like. For each of these, and other, applications, the rheology modification agents serve very specific purposes tailored to the function for which the fluid is being employed. Because a variety of properties are frequently needed for a given fluid, the rheology modification agent is frequently used in conjunction with other types of agents or additives, in order to produce a final fluid suitable to a given application. However, it is generally desirable to reduce the number of such agents or additives as much as possible, in order to facilitate the ease of production and use and therefore to also minimize cost. [18]

Certain inorganic materials, including mineral clays such as bentonite and attapulgite, are commonly used for rheology modification of aqueous fluids. For example, bentonite, which is one of the most popular of the clay materials for use in drilling muds, is thixotropic in combination with water and is also relatively stable to temperature extremes. Furthermore, it imparts resistance to oxidation and durability when exposed to high shear conditions. These clays may be used with other types of agents or densifiers, such as iron oxide or barium sulfate, which enhance the ability of the fluid to resist pressures such as are encountered in subterranean excavations.

Aqueous fluids containing only clays for rheology modification suffer problems, however, including compromise in the presence of polyvalent cations, such as calcium and magnesium. These cations are frequently present in drilling formations and can cause the drilling mud to become so thick at higher temperatures that thinners may be required. Some clay systems also suffer from undesirable reactivity and temperature degradation, and may not be adequately consistent in composition from batch to batch. [18]

For some applications it is particularly desirable to use a rheology modification agent which exhibits the ability to "shear-thin", i.e., to show a rapid or near-instantaneous and predictable viscosity reduction upon application of shear forces, followed by a rapid or near-instantaneous return to original viscosity when the shear forces are removed. Clays alone may effectively thicken a composition, but generally do not offer this shear-thinning capability. While there are newer rheology modification agents that do offer shear-thinning capability, they tend to be much more expensive than clay and may be more difficult to employ under typical field conditions. For this project, Sodium Hydroxide (NaOH) is added to bentonite to improve its rheological properties, especially in enhancing composition of Sodium in Sabah Bentonite.

CHAPTER 3

METHODOLOGY / PROJECT WORK

3.1 Methodology

Wikipedia states that methodology includes the following concepts as they relate to a particular discipline or field of inquiry:

1. A collection of theories, concept or ideas;
2. Comparative study of different approaches; and
3. Critique of the individual methods

The following is the methodology used to carry out this project, to be conducted within a certain time range, as shown in the flowchart and Gantt chart below.

For the first semester, or first part of the project will be discussing on the literature review, aim to understand the drilling operations, drilling fluids, especially on Sabah Bentonite and Indian Bentonite and also to understand the mechanism of the Dynamic Filtration apparatus. A few initial experiments is also conducted in the first part to evaluate and study the parameter of the dynamic filtration test, so improvements can be suggested on the later chapter of this research.

The references for the first part of this project are from books, journals, lab manuals and standards.

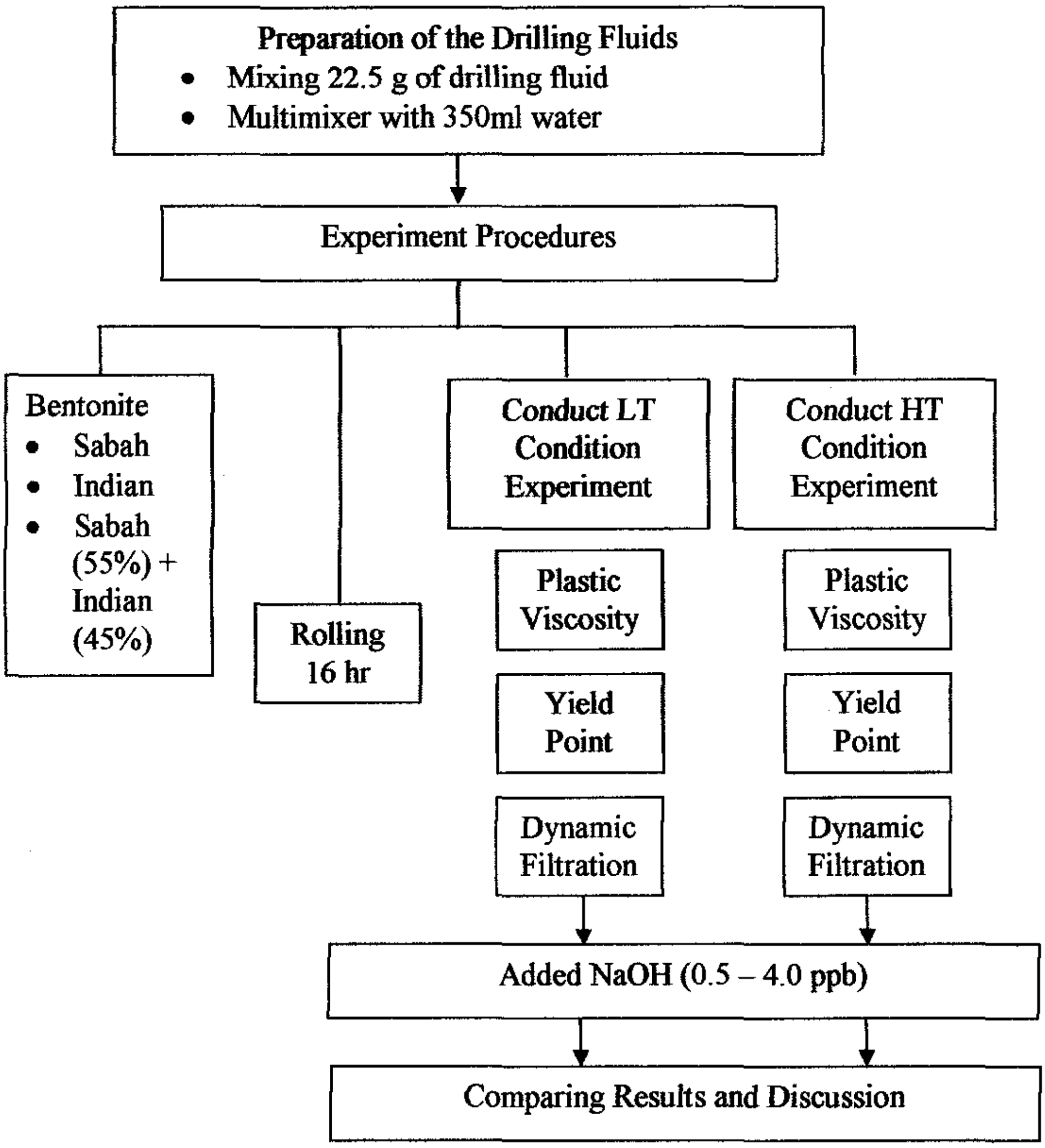

Figure 3.1: Methodology Flowchart

3.2 Fluids Preparation

The experiments are conducted in the lab for optimum results, especially for this research, where pressure and temperature play a vital role. The purpose of this experiment is to study the effect of pressure and temperature to different percentages of Sabah and Indian Bentonite, therefore the standard drilling mud samples are prepared according to API 13. The sample contains water as base fluid plus bentonite as viscosifier.

1. Add 22.5 grams (+ 0.01 gram) of bentonite powder in 350 ml (+ 5 ml) of distilled water
2. Stir for five (5) minutes using Fann Multimixer
3. The container is examined and any bentonite adhering to the container walls is dislodged by scraping the side using spatula.
4. The stirring continued until the bentonite has mixed well, usually takes up till 25 minutes.
5. Machine is stopped and observed if the mixture has no (or less) bentonite powder and it is ready to be used.

3.3 Density

A mud balance is used to measure density. It is a beam balance which works like a scale in a doctor's office (See Figure 3.3). It has a cup on one end to hold a mud sample and an arm with a sliding weight on it. The arm rests on a fulcrum. The balance weighs a precise volume of mud and automatically divides the weight by the volume to give a direct reading on the mud's weight. To use the mud balance, fill the cup, move the sliding weight until the arm balances, and read the density from a scaled marked on the instrument.

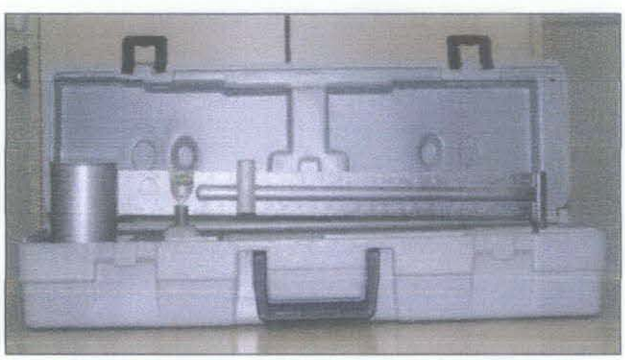

Figure 3.3: Fann Mud Balance

3.4 Viscosity and Gel Strength

Viscosity and gel strength is measured using a direct-indicating viscometer. It has a weight (bob) inside the rotor. A spring restraints the movement of the bob, and a dial on top of the viscometer indicates how far the spring moves. The mud's viscosity influences the spring's movement so that thinner muds let the spring move more than thicker muds. The instrument measures a mud's plastic viscosity and the yield point. Plastic viscosity is a fluid's resistance to flow because of friction. The yield point is a fluid's resistance to flow that resulted from the attraction between clay particles.

Figure 3.4: Fann Direct Indicating Viscometer

3.4.1 Measuring viscosity

1. Fill the cup with mud and place it under the rotor. Adjust the rotor so that the mud in the cup meets a scribe line on the rotor.
2. Set the rotor to turn at 600 revolutions per minute (rpm). Note the dial and wait for the reading to reach a steady state value.
3. Record this value.
4. Stop the rotor, press the button down, and turn the rotor at 300 rpm. Allow enough time at this speed for the reading to become steady.
5. Record the reading at 300 rpm. The calculation for plastic viscosity and yield point are as follows:

Plastic viscosity

Reading at 600 rpm – Reading at 300 rpm .... (3.1)

Yield point

Reading at 300 rpm – Plastic viscosity .... (3.2)

3.4.2 Gel Strength

To measure gel strength requires two tests on the same mud sample at the same temperature. The first, the 10-second test, shows how quickly the mud gels. The second, the 10-minute test, shows the extent of gelling – how thick it gets after it has the chance to gel.

For 10-second test:

1. Lower the viscometer's rotor and bob into a cup of mud and stir it for 10 seconds.
2. Then, let the mud sit still for 10 second.
3. Next, stir the mud very slowly, at 300 rpm.
4. Take a reading from the dial on top of the viscometer, which gives the 10-second gel strength in pounds per 100 square feet (kilograms per 10 square metres).

For 10-minute test:

1. Stir the muds for 10 seconds as before, but let the mud sit still for 10 minutes.
2. Then, slowly stir the mud at 300 rpm to obtain the 10-minute gel strength reading.

3.5 Dynamic Filtration

Filtration tests measure the relative amount of water in the mud that escapes into a permeable formation and the thickness of the filter cake deposited on the walls of the hole. The test can be made at a low or high temperature and pressure.

For this project, Fann Model 90 Dynamic Filtration System is a computerized instrument for conducting filter cake, formation, fluid loss and permeability analysis. The Model 90 simulates the down hole conditions of pressure, temperature and shear rate. Test parameters input into Model 90 will optimize the test simulation. The Filter Core medium is a thick walled cylinder with formation-like characteristics to simulate the depositing and build up of cake on the formation. The porosity and permeability simulates the fluid loss to the formation. The fluid loss rate can be tracked as well as the fluid volume measured.

Results of the tests are shown on the screen while the test is in progress and may be read on the screen after the test is complete. A summary of the test results is also available on the screen.

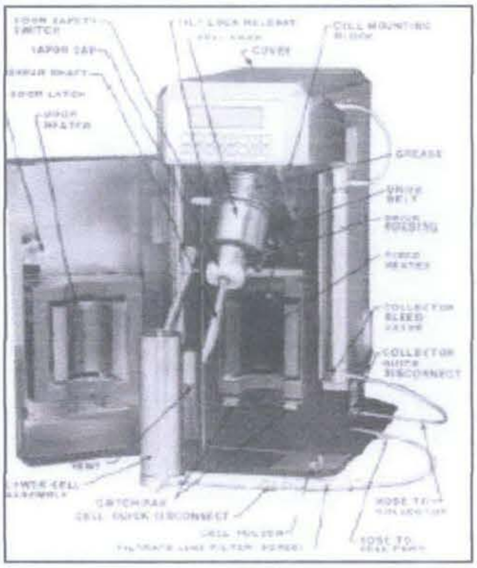

Figure 3.5: Fann 90 Dynamic Filtration Apparatus

3.5.1 Test Sequence

A test sequence consists of a sequence of steps made up of the desired (target) conditions and the time intended to attain or maintain them. The system is capable of slow ramping of the controlled variables. For example, this allows a steady increase of temperature. The followings are the test sequence used for this project:

1. Step 1 – Establishes the static conditions
   Static pressure is applied and sample is heated to the desired temperature.
   Step time is set to "0" to obtain test conditions in the minimum time. Since step conditions have not stabilized, filtrate is not collected during this step. The differential pressure is set to "0" and filtrate flow is set OFF.

2. Step 2 – Sets the differential pressure

   These steps starts after the conditions of step 1 have been met. Zero time is programmed for this step, and target static pressure, shear rate and filtrate flows are the same as Step 1.

3. Step 3 – Allows the differential pressure

   This is a step that gives time for all the conditions established in step 1 and 2 to stabilize before starting filtration. A "2" minute step time is set, and the other parameters are the same as step 1 and 2.

4. Step 4 – Collects dynamic filtration data

   This is the step where all the conditions of the test are held constant while filtrate is collected. The conditions of this step are the same as step 3, except the filtrate flow is "ON".

5. Step 5 – Cools down and vents the pressure

   During this step the cell is cooled to a temperature where it can be handled safely and the pressure is vented. The step time and target static pressure are set to "0". The pressure is maintained at 6.79 psi / °F above 122 °F. The instrument reduces the pressure as the cell temperature drops and cell will be completely vented by the time the target temperature is reached. This is usually temperature of 115 °F (46 °C). The target shear rate is 100 / s to aid in cooling and filtrate flow is "OFF".

3.5.2 Experiment Parameters

There are six (6) parameters, for the target conditions for every step that should be set before starting the experiment:

1. Step time
2. Static pressure (psi)
3. Differential pressure (psi)

4. Temperature (°F)
5. Shear rate (1/s)
6. Filtrate flow (On / Off)

For Low Temperature Low Pressure (LTLP) condition and High Temperature High Pressure (HTHP), the followings are the parameters set constant for the target conditions of the experiments:

Table 3.5.2.1 : Constant Parameters for LTLP Condition

Parameters	**Target Conditions**
Static Pressure (psi)	200
Differential Pressure (psi)	100
Temperature (°F)	86 (30°C)
Shear rate (1/s)	100

Table 3.5.2.2: Constant Parameters for HTHP Condition

Parameters	**Target Conditions**
Static Pressure (psi)	500
Differential Pressure (psi)	400
Temperature (°F)	167 (75°C) 248 (120°C) 302 (150°C)
Shear rate (1/s)	100

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Rheology Test

Until this part of the project, the rheology test was conducted to determine density, viscosity and yield point of Sabah and Indian Bentonite, also the mixture of 45% of Indian bentonite and 55% of Sabah bentonite at room temperature. The following is the results obtained for the experiment:

Table 4.1.1: Comparison of Rheological Properties between Sabah and Indian Bentonite at Low Temperature

Properties	Types of Drilling Fluid			
	API Standard	**Sabah**	**Indian**	**55% Sabah, 45% Indian**
Density (lb/gal)	n/a	1.017	8.60	8.50
Viscometer Reading at 600 rpm / min	Minimum 30	3	30	7
Plastic Viscosity (cp)	n/a	2	9	4
Yield Point (lb / 100 ft^2)	n/a	1	12	1
YP/PV	Maximum 3	0.50	1.33	0.25

Table 4.1.2: Comparison of Rheological Properties at High Temperature

Properties	45% Indian, 55% Sabah			Indian			Sabah		
	75°C	120°C	150°C	75°C	120°C	150°C	75°C	120°C	150°C
Viscometer Reading at 600 rpm / min	17	13	11	77	79	85	3	2	2
Plastic Viscosity (PV)	6	5	5	25	26	29	2	1	0.5
Yield Point (YP) (lb / 100 ft^2)	5	3	1	27	27	27	1	1	1
YP/PV	0.83	0.6	0.2	1.08	1.03	0.93	0.5	0.5	2
Gel Strength (10 s)	12	9	8	56	57	69	2	2	1
Gel Strength (10 m)	13	10	9	54	60	65	2	2	2
Density	8.5	8.6	8.5	8.3	8.75	8.7	8.4	8.4	8.3

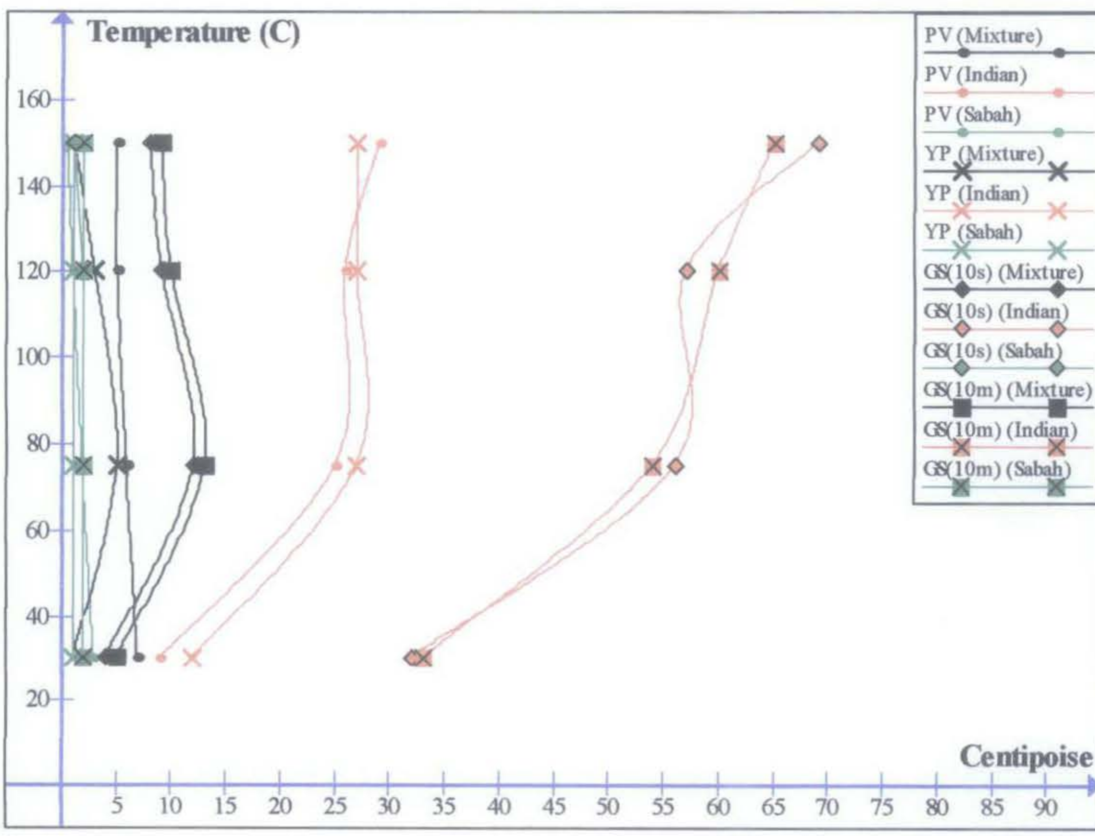

Figure 4.1.1: Comparison of Rheology Properties

From the result above, the density stays almost the same for the three bentonites, with Indian bentonite having the highest density. Indian bentonite has higher density than Sabah bentonite because it has higher content of montmorillonite, which is a very soft phyllosilicate mineral that typically forms in microscopic crystals, forming clay.

Montmorillonite function is making the mud slurry viscous which helps in keeping the drill bit cool and removing drilled solids.

For the viscosity test, using Indian Bentonite, the following results, Sabah Bentonite has not meet the API standard for viscometer reading at 600 rpm / min and has much lower plastic viscosity and yield point than Indian Bentonite. The low plastic viscosity might result the Sabah Bentonite fails to control the magnitude of shear stress of fluids and caused fluid failure during operation. The low yield point may cause Sabah Bentonite to fail in developing and retaining its structure during operation. The mixture 55% Sabah Bentonite and 45% of Indian Bentonite showed an increment of viscometer reading and plastic viscosity, but still way lower than the API Standard.

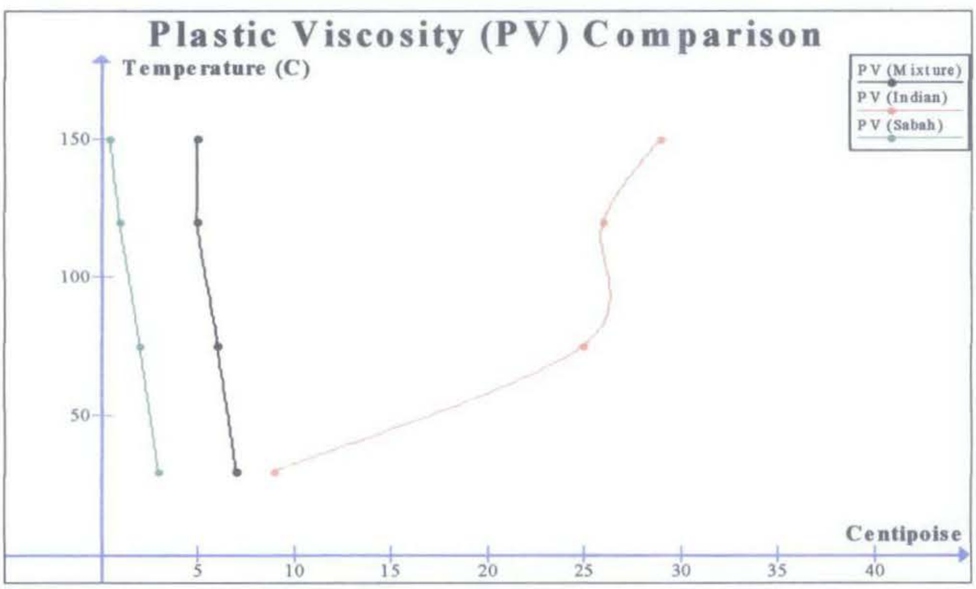

Figure 4.1.2: Plastic Viscosity (PV) Comparison

The viscometer reading shows the decrease of viscometer reading as the temperature goes up for Sabah and Mixture bentonite. This is because as the temperature increases, the clay swelling decreases, thus the viscosity of the mud is decreases. However, it was the opposite condition for Indian bentonite. The plastic viscosity increases with temperature due to shear thickening effect. It caused the bentonite to become more dispersed, increasing the number of individual platelets in the suspension. The plastic viscosity, PV, is a measure of the internal resistance to fluid flow attributable to the amount, type, and size of solids present in a given fluid.

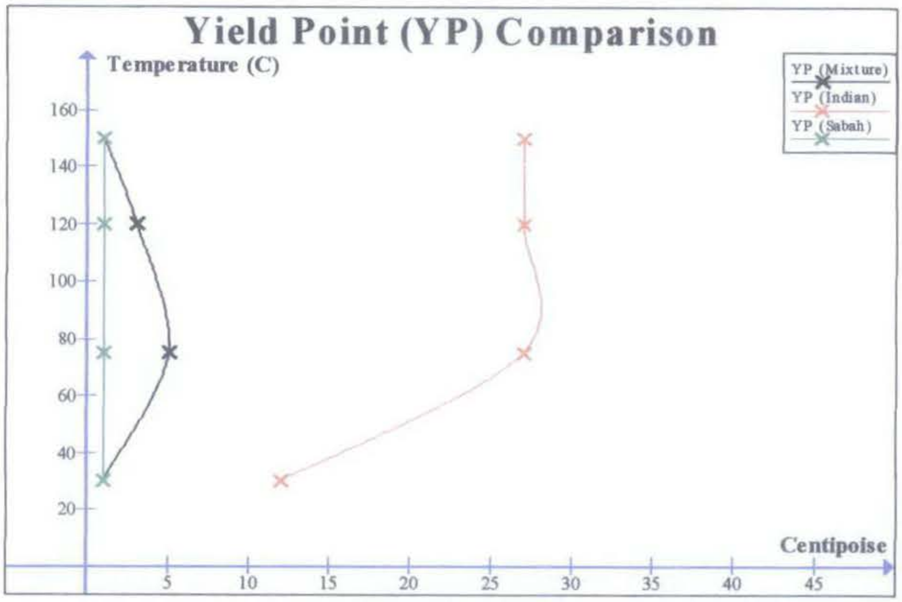

Figure 4.1.3: Yield Point (YP) Comparison

The yield point (YP) is decreasing with an increase of temperature for Sabah and Mixture Bentonite while increasing for Indian Bentonite. YP is the resistance to initial flow, or represents the stress required to start fluid movement. This resistance is believed to be due to electrical charges located on or near the surfaces of the particles. Values of yield point and thixotropy, respectively, are measurements of the same fluid properties under dynamic and static states. Thus, the reading from the rolled bentonite indicated the same result for dynamic condition.

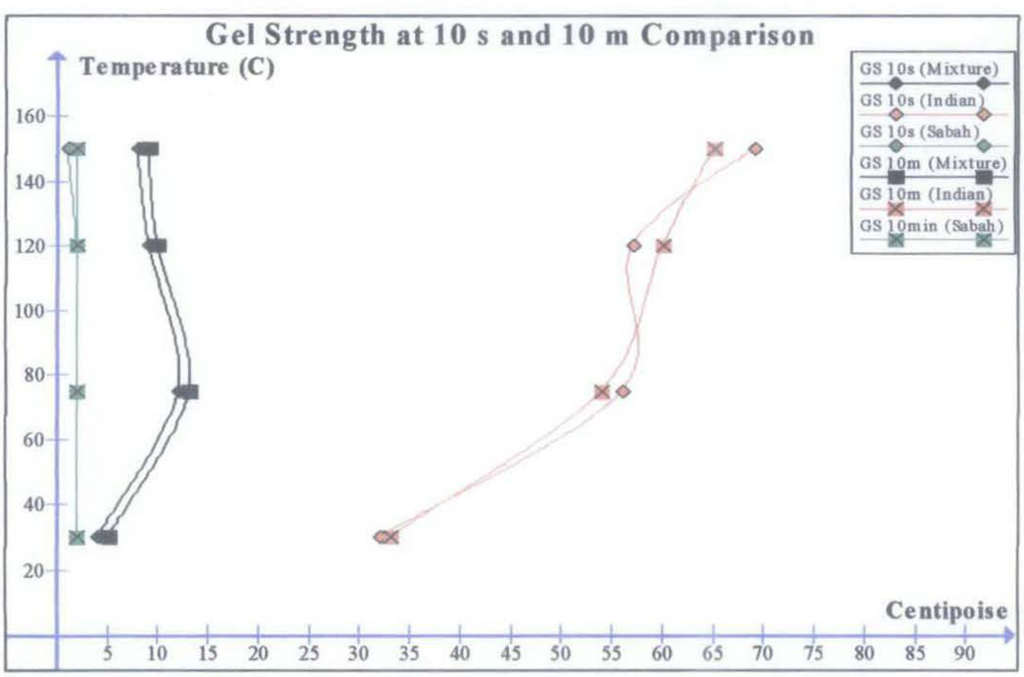

Figure 4.1.4: Gel Strength (GS) Comparison

Gel strength showed decrease through temperature except for Indian bentonite which has increased as temperature increased. The exposure to high temperatures for long times caused the bentonite to become more dispersed, increasing the number of individual platelets in the suspension and increasing viscosities at low shear rates.

4.2 Dynamic Filtration Test

The Fann 90 Dynamic Filtration apparatus is relatively new equipment in Petroleum Department, even in Petronas. Thus, a trial experiment is conducted first using plain water to test the functionality of the equipment. After the first trial and results can be obtained, the first experiment using Indian bentonite is conducted. The LTLP condition is used in this experiment, using the parameters shown in the methodology part. The following is the result for dynamic filtration in the first 30 minutes of the experiment.

Table 4.2: Comparison of Filtrate Volume between 55% Sabah Bentonite 45% Indian Bentonite at Static (S) and Dynamic (D) Condition

Condition		S 100% Indian	D 100% Indian	S	D	S	D	S	D	S	D
Temperature		30°C	30°C	30°C	30°C	75°C	75°C	120°C	120°C	150°C	150°C
Static Pressure (psi)		200	200	200	200	500	500	500	500	500	500
Differential Pressure (psi)		100	100	100	100	400	400	400	400	400	400
Filtrate Volume	7.5 min	2.60	9.84	7.30	12.39	9.00	16.56	22.50	17.09	22.40	18.88
	30 min	4.00	14.29	9.80	25.74	10.40	24.38	38.10	35.24	37.90	36.19

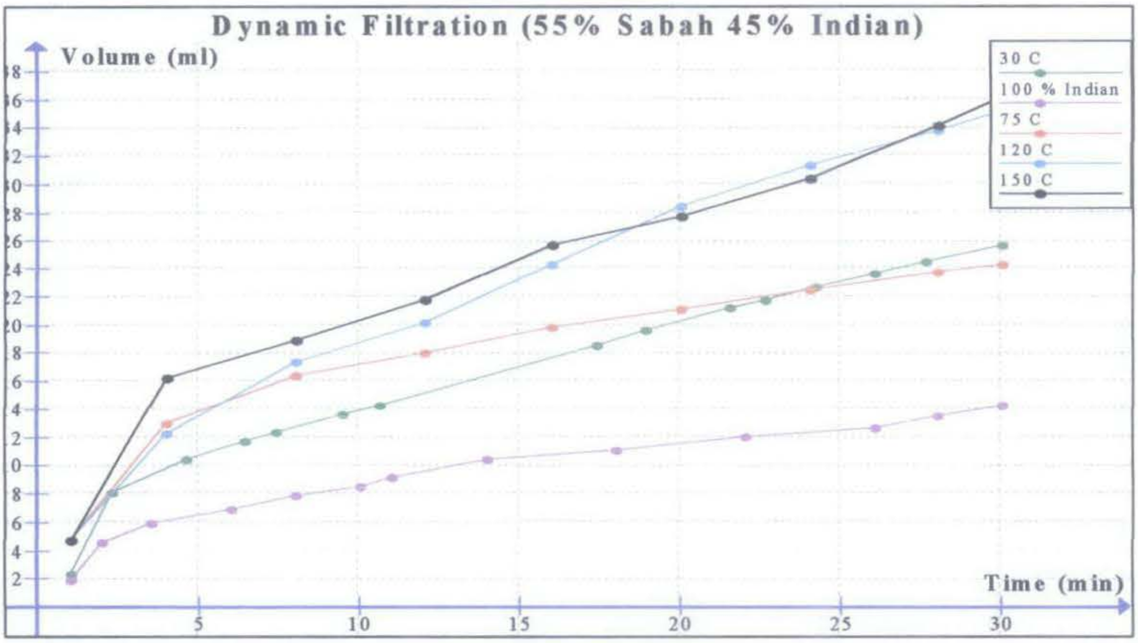

Figure 4.2.1: Graph of Filtrate Volume (ml) versus Time (min) under Dynamic Condition

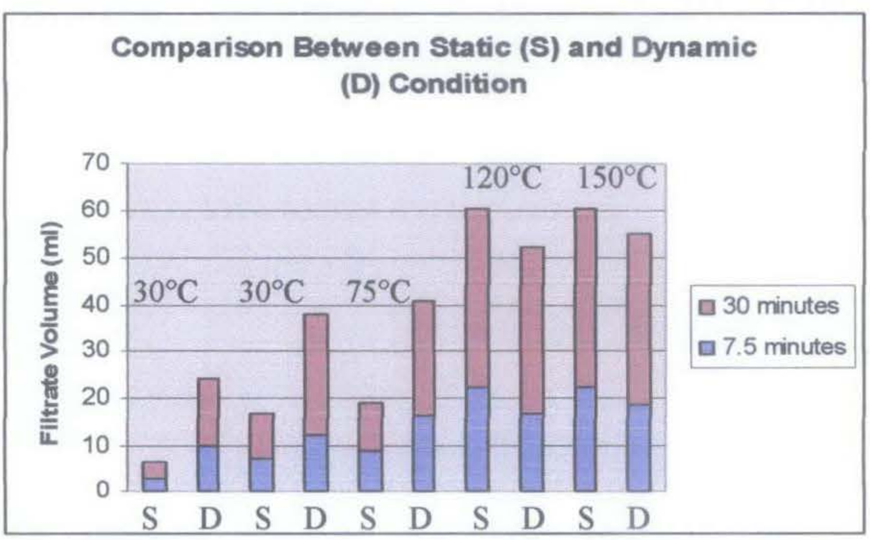

Figure 4.2.2: Comparison between Static (S) and Dynamic Filtration (D)

Based on the result, the filtrate rate are observed to decrease with time first, and then regulate constantly. Filtration occurs radially from the inside of the filter core to the outside. At the same time, the filter cake is formed on the inside of the filter to simulate filter cake formation on the wall of a borehole. A polished stainless steel shear bob runs through the central axis of the filter core. The shear bob is rotated to produce a concentric cylinder type shear across the filtration surface.

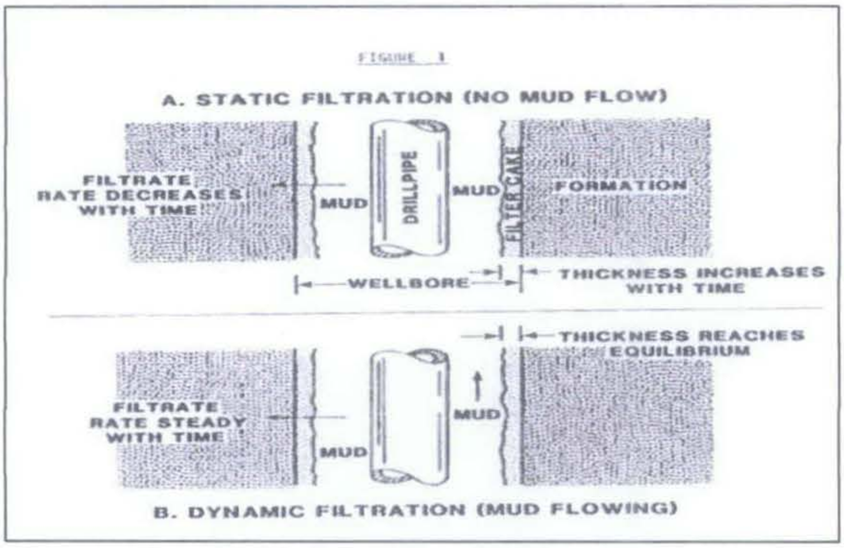

Figure 4.2.3: Mechanism of Static and Dynamic Filtration

For static condition, the filtration rate decreases with time, thus the filtrate volume collected increases. Filter cake forms when the pressure in the well bore exceeds the formation pressure. For dynamic filtration, the filter cake thickness increases until the rate of deposition equals the rate of erosion by the flowing mud. During this cake-building time the filtration rate into the formation decreases. The filtration rate stabilizes when the filter cake thickness reach equilibrium [11]. Hence, the result showed the filtrate volume increases as the filtration rate decreases until it reaches equilibrium.

However, when comparing dynamic filtrate volume and static filtrate volume, there is no clear correlation between them. Based on findings by Vaussard et al:

1. Dynamic rates was reduced by a period of static filtration, but increased if annular flow rate was increased.
2. Invert emulsion cakes were easily erodable, resulting in higher dynamic rates than would have been expected from their API filter losses. Spurt losses were high when solid contents were low.
3. Dynamic rates were independent of rock properties except for coarse sintered media.

There were studies to determine the connection between static and dynamic filtration but none are confirmed. The apparatus efficiency and accuracy also plays part in determining the static and dynamic filtration rates.

4.3 Sodium Hydroxide (NaOH) Additive

Table 4.3.1: XRF Results for Indian and Sabah Bentonite

No	Elements	Indian (%)	Sabah (%)
1	Titanium (TiO_2)	1.03	0.83
2	Sodium (Na_2O)	2.70	0.56
3	Potassium (K_2O)	0.17	3.03
4	Magnesium (MgO)	3.14	2.30
5	Calcium (CaO)	2.51	0.26
6	Ferric Oxide (Fe_2O_3)	10.4	5.63
7	Alumina (Al_2O_3)	21.6	21.3
8	Silica (SiO_2)	57.6	65.6
9	Loss of ignition	0.85	0.49

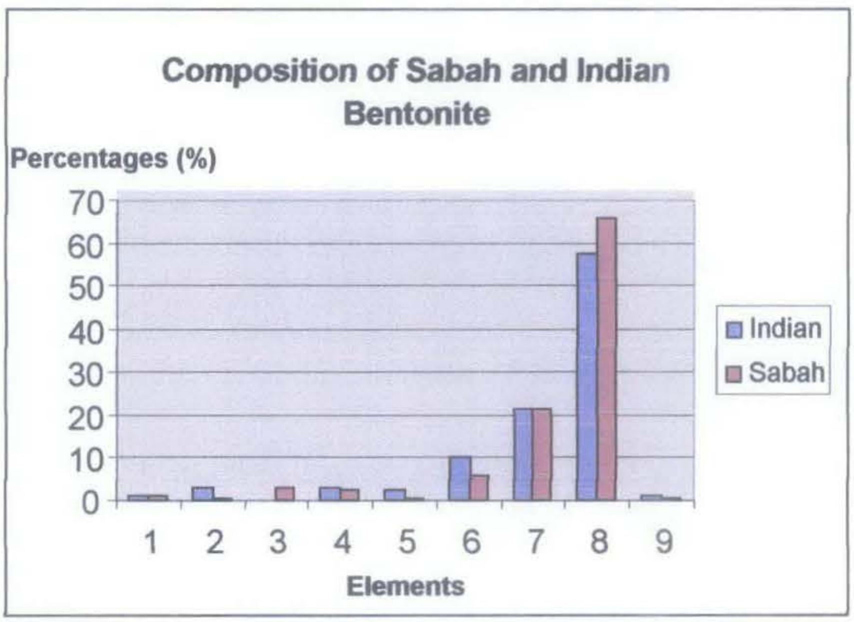

Figure 4.3.1: XRF Result for Composition of Indian and Sabah Bentonite

Darley and Gray (1988) stated that the interaction between sodium (monovelent) with montmorillonite will determine the swelling of the particles. High sodium content results in high swelling and thus high density and viscosity of the fluid. As comparison, the Sodium content of Sabah bentonite is 0.58% as compared to Indian bentonite sodium content, which is 2.70%. The XRF was conducted for the mixture of 55% Sabah Bentonite and 45% Indian Bentonite in powder form but it had failed due to the different composition added together without any chemical reactions. Sodium Hydroxide (NaOH) or known as caustic soda is added as an additive to increase the amount of sodium in Sabah Bentonite. The results are as followings:

Table 4.3.2: Rheology Properties

Properties / Weight	Indian	Mix	0.5	1.0	1.5	2.0	2.5	3.0	3.5	4.0
Viscometer Reading at 600 rpm / min	30	7	32	32	38	39	32	39	35	35
Plastic Viscosity (cp)	9	4	4	8	7	6	7	7	6	6
Yield Point (lb / 100 ft^2)	12	1	24	16	24	27	18	25	23	23
YP/PV	1.33	0.25	6.00	2.00	3.42	4.50	2.57	3.57	3.83	3.83
Gel Strength (10 s)	27	3	29	43	30	32	25	30	28	28
Gel Strength (10 m)	28	2	29	42	28	29	22	28	25	25
Density (lb / gal)	8.60	8.50	8.70	8.70	8.70	8.70	8.70	8.70	8.65	8.50

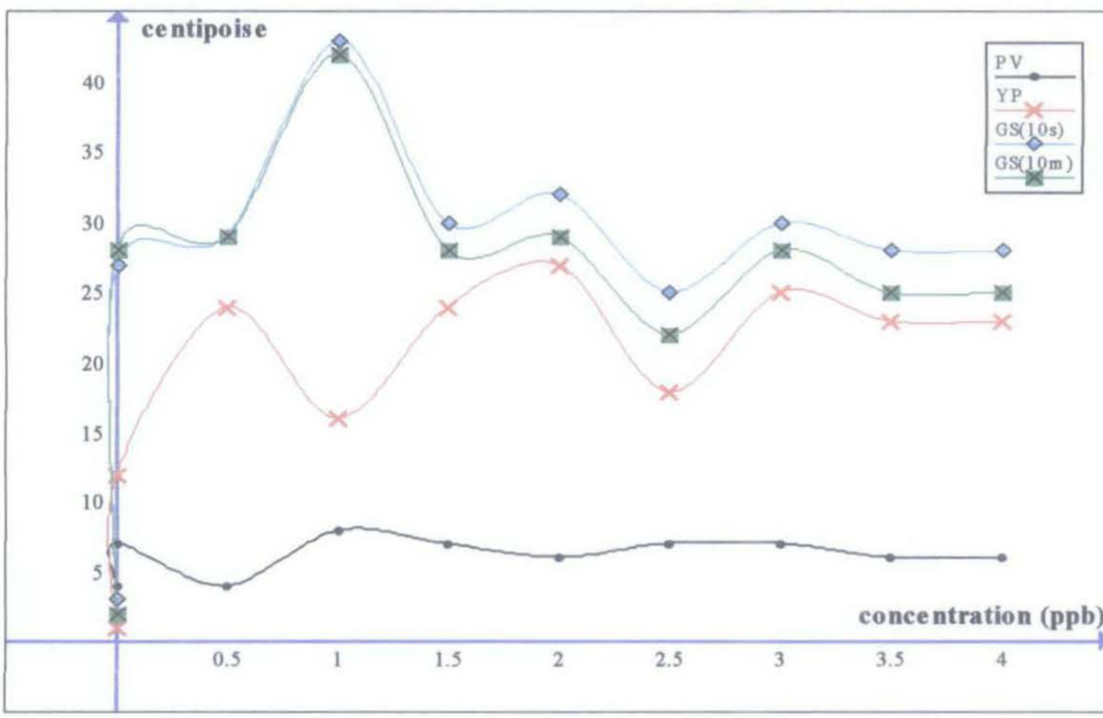

Figure 4.3.2: Rheology Properties of Sodium Hydroxide Additive

Table 4.3.3: Filtration Volume

Condition / Weight		Indian	Mix	0.5	1.0	1.5	2.0	2.5	3.0	3.5	4.0
Temperature		30°C	30°C	30°C	30°C	30°C	30°C	30°C	30°C	30°C	30°C
Filtrate Volume (ml)	7.5 min	2.60	7.30	12.20	22.00	24.00	29.40	31.40	32.80	33.50	34.20
	30 min	4.00	9.80	27.00	46.00	55.20	63.80	66.50	68.40	69.30	69.60

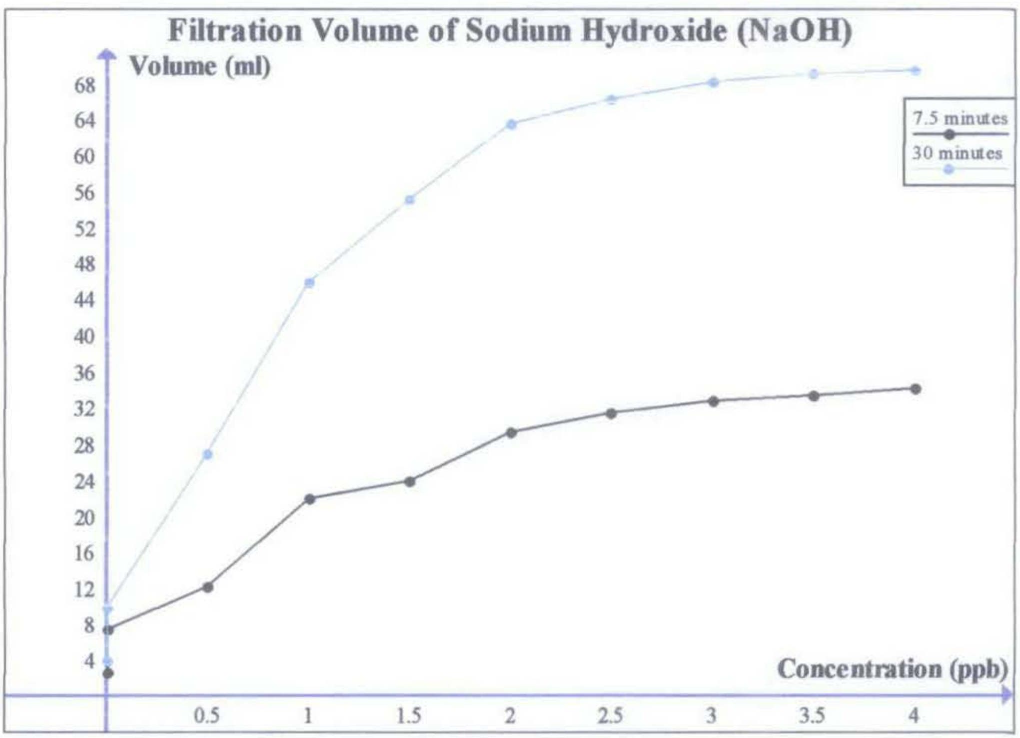

Figure 4.3.3: Filtration Volume of Sodium Hydroxide Additive

Based on the results obtained, Sodium Hydroxide (NaOH) helps in improving its rheological properties but failed to improve its filtration properties. Addition of sodium hydroxide and other monovelent cation to an aqueous suspension of dispersed hydrated clay increases the ionic atmospheric charge from the sodium and chloride ions. The hydroxide ions increase the negative charge of the solution and the sodium ions the positive charge. The increased number of sodium ions means that some approach the planar surface of the clay closer than the cation previously associated with it, thereby tending to decrease the sheet negative charge and the repulsive charge between sheets.

The repulsive charge between the ionic atmosphere and reduced planar charge will remain high, however, because of the increased atmospheric negative charge. The reduced repulsive charge between sheets, plus the attractive edge valencies, plus the high repulsive charge of the ionic atmosphere negative charges, forces the clay sheets to flocculate or cluster in lumps. These lumps are more difficult to move than the individual clay sheets, as manifest by an increase in viscosity and gel strength. In addition, once the sheets are not available in individual form to seal openings, the fluid loss increases. If the result of flocculation is a regrouping of the sheets in stacked parallel layers (aggregated), then viscosity and gel strength will decrease and fluid loss will increase. Since such a regrouping is a matter of statistical chance it is seldom that the randomly oriented sheets will completely restack themselves. In all probability some restacking occurs, with the remaining plates interlocked at random angles. [3]

The increase of sodium hydroxide (NaOH) concentration also increases the mud's pH. Under high pH conditions the mud viscosity is unduly high because of the effect of the hydroxyl radical. A condition where NaOH is present and conducive to the development of sodium clays from the shales drilled with their great hydration and dispersion effects which add viscosity to the mud. This maybe highly undesirable and supposed to be kept at minimum pH 8.0 to 9.0.

4.4 Economic Analysis

In Malaysia, the bentonite occurs in Sabah is confirmed as calcium variety (Radzuan Junin *et al.*, 1992). So, this study had provide an alternative way to beneficiated and activated these bentonite to provide better product, the sodium based bentonite. Based on previous study (Leyong, 2003 and Sarimah Man *et.al.*, 2000) for Malaysian bentonite, the bentonite deposit in Mansuli area is about 1.5 million tons while in Andrassy area is about 3.6 million tons with average thickness varies from 1 meter to 2.5 meters. So, from this study, only one of the Andrassy bentonite (SA5-4) can be upgraded to meet the API specifications with the addition of polymer dispersant. Therefore, only one third of the Andrassy bentonite deposits can be used as a material in drilling mud and the amount is around 1.2 million tons (Sarimah Man *et al.*, 2000).

In order to drill average oilwell geometry, an amount of 270 tons of bentonite is needed to prepare the required drilling mud. The cost of bentonite per well of beneficiated Andrassy samples compared to the commercial bentonite is shown in Table 4.4. From Table 4.4, it is indicated that the cost per well when used a beneficiated Andrassy sample is much lower than the commercial bentonite (60.71 % cheaper). Based on forecasting of 50 development and 10 exploration wells to be drilled per year, the requirement for bentonite as a material is about 16 000 tons per year. If the treated Andrassy sample can be produced and used widely as a material in drilling mud, a total cost of around RM 1.5 million could be saved per year. In addition, because of its promising economic potentiality, the beneficiated bentonite can be a major contributor to the Malaysia economic. If around 50 000 tons of beneficiated bentonite can be exported as a drilling mud material to other countries, an amount of RM 3 million can be earned. This directly increases the gross domestic product (GDP) of the country. Although the untreated Andrassy didn't meet API requirements, the treatment undergoing will improve the properties of the bentonite and the economic analysis will be done in later stage of research for cost comparison.

Table 4.4: Comparison of Cost between Commercial Bentonite and Beneficiated Andrassy Sample

Cost	Commercial Bentonite	Beneficiated Andrassy sample
Price of oxalic acid required to extract per ton of bentonite (US$)	-	9*
Price of Tannathin/CMC required to activate		
1 ton bentonite (US$)		6*
Processing cost per ton of bentonite (US$)	-	10*
Total Price per ton (US$)	42*	25
Total Cost (RM)	148.68	88.50
Total Cost per well (US$)	11 340*	4 455*
Total Cost per well (RM)	40 144	15 771

* Source: KMC Sdn Bhd.; US$ 1 = RM 3.54

CHAPTER 5

CONCLUSION

The results obtained after the initial experiments are shown and discussed to get a better understanding of the research, such as:

1. The comparison of rheological properties of the three drilling fluids: Indian, Sabah and Mixture. For Sabah and Mixture, as the temperature increases, the clay swelling decreases, thus the PV, YP and GS of the mud decreases. For Indian, as the temperature increases, it caused the bentonite to become more dispersed, increasing the number of individual platelets in the suspension, thus PV, YP and GS increases.
2. For mixture of 45% Indian and 55% Sabah bentonite, the dynamic filtration is higher than static condition at lower temperature but lower in high temperature as they are no real correlation between static and dynamic condition.
3. Concentration of Sodium Hydroxide (NaOH) from 2 ppb till 4 ppb is added And optimum of 0.5 ppb of NaOH added improved the rheological properties but not the filtration properties.
4. The economic analysis showed that treated Andrassy sample is much cheaper than the commercial bentonite (60.71 % cheaper). This directly increases the gross domestic product (GDP) of Malaysia and can beneficiated the country.

CHAPTER 6

RECOMMENDATION

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

1. The Sabah Bentonite is treated with certain composition, mainly to improve the properties to meet API standard requirements, with suitable additives.
2. Adding suitable chemical additives to improve the properties of Sabah Bentonite at high temperature under dynamic condition, specifically in improving its filtration properties, such as starch or lignite.
3. Experimentation of Sabah Bentonite to be conducted at high pressure and under dynamic condition thus the rheological and filtration properties can be observed.

REFERENCE

[1] H.C.H Darley, G.R Gray, "*Composition and Properties of Drilling and Completion Fluids, Fifth Edition*", Houston, Texas, Gulf Professional Publishing.

[2] Dyke K.V. 1998, *Drilling Fluids, Mud Pumps, and Conditioning Equipment, Unit 1, Lesson 7, First Edition*, Houston, Texas, Petroleum Extension Service.

[3] W.F Rogers, 1963, *Composition and Properties of Oil Well Drilling Fluids*, Houston, Texas, Gulf Publishing Company.

[4]. H. Rabia, 1985, *Oilwell Drilling Engineering Principles and Practice*, United Kingdom, Graham and Trotman.

[5] ASME Shale Shaker Committee, 2005, *Drilling Fluids Processing Handbook*, United Kingdom, Elsevier

[6] K.G. Arthur, E.P.D.S Ltd, and J.M. Peden, "*The Evaluation of Drilling Fluid Filter Cake Properties and Their Influence on Fluid Loss*", SPE 17617, SPE International Meeting on Petroleum Engineering, Tianjin, China, November 1-4, 1988.

[7] B. Vicki, C. Julie, "*Silicone Resin for Drilling Fluid Loss Control*", Dow Corning Corporation (2200 West Salzburg Road, Midland, MI 48686-0994, US), August, 29, 2007.

[8] T.A Jones, L. Quintero, "*Innovation and Flexibility for Drill-in Fluids Design*", AADE-04-DF-HO-08, AADE Drilling Fluid Conference, Houston, Texas, April 6 – 7, 2004.

[9] American Association of Drilling Engineer (AADE), 1999. *Chapter 9 Drilling Fluids*. Ref No. DE/9, Version 1.1 Date of Issue 10/3/99.

[10] Crawford, Donald , *"High pressure, high temperature (HPHT) fluid loss control aid for drilling fluids"* , Belle Chasse, LA, May 28, 2002.

[11] M.Scott Quigley, Henry A. Seal, *"Dynamic Filtration Unit and Process of Use"*, Mobil Oil Corporation, New York, November, 18, 1986.

[13] American Petroleum Institute, ANSI/ API/ ISO13500, *"Specification for Drilling Fluids Material, API Specifications 13A, Sixteenth Edition"*, February 2004.

[14] N.M Borah, R.K Mallick, *"Mud Filtrate Invasion Profile – Its Influence on Wireline Logs and Hydrocarbon Producibility : A Case Study"*, SPE 39510, SPE India Oil and Gas Conference and Exhibition, New Delhi, India, February 17-19, 1998.

[15] M.Shakir, *"Analysis on Characteristics of Sabah Bentonite at High Temperature Environment"*, Universiti Teknologi Petronas, July 2007.

[16] Wikipedia. December 2007 < http://en.wikipedia.org/wiki/Methodology>.

[17] Wikipedia. November 2007 < http://en.wikipedia.org/wiki/Bentonite >.

[18] Hoy, Edgar Franklin, *"Additives for Preparing Rheology-modified Aqueous Fluids"*, Kingwood, TX, US, June 2005.

[19] Lecture Materials, *"Advanced Engineering Polymers, Ceramics and Composites,"* Universiti Teknologi Petronas (UTP), July 2008.

[20] Schlumberger Oilfield Glossary, < http://www.glossary.oilfield.slb.com >.

APPENDICES

APPENDIX 1 (Gantt Chart for Semester 1)

No.	Detail/ Week	1	2	3	4	5	6	7		8	9	10	11	12	13	14
1	Selection of Topic															
2	Preliminary Research Work • Preparation of Drilling Fluids • Further research of theories															
3	Submission of Preliminary Report															
					○											
4	Continue Research Work • Preparation of the Experiments									○						
5	Submission of Progress Report															
6	Initial Experiments • To observe the parameters such as plastic viscosity, yield point and dynamic filtration and comparing with static filtration															
															○	
7	Submission of Interim Report Final Draft															○
8	Oral Presentation															

○ Suggested milestone   Process

APPENDIX 2 (Gantt Chart for Semester 2)

No.	Detail/ Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14	SW	EW
1	Project Work Continue																
	-Dynamic Filtration																
	- Rheology Properties																
2	Submission of Progress Report 1				15/8												
3	Project Work Continue																
	-Additives of NaOH																
4	Submission of Progress Report 2																
5	Project work continue																
	-Additives of NaOH																
6	Submission of Dissertation Final Draft																
7	Oral Presentation																
8	Submission of Project Dissertation (Hardbound)																

SW Study Week   **EW** Exam Week

9 Bentonite

9.1 Description

9.1.1 Drilling grade bentonite is a naturally occurring clay containing the clay mineral smectite. It may also contain accessory minerals such as quartz, mica, feldspar and calcite.

9.1.2 Drilling grade bentonite shall be deemed to meet the requirements of this International Standard if a composite sample representing no more than one day's production conforms to the physical requirements of Table 12, represents the product produced, and is controlled by the manufacturer.

Table 12 — Bentonite physical requirements

Requirement	Standard
Suspension properties	
Viscometer dial reading at 600 r/min	minimum 30
Yield point/plastic viscosity ratio	maximum 3
Filtrate volume	maximum 15,0 cm^3
Residue of diameter greater than 75 µm	maximum mass fraction 4,0 %

9.2 Suspension properties — Apparatus

9.2.1 Thermometer, including the range 0 °C ± 0,5 °C to 105 °C ± 0,5 °C .

9.2.2 Balance, with an accuracy of 0,01 g.

9.2.3 Mixer (e.g. Multimixer® Model 9B with 9B29X impellers or equivalent), having spindle fitted with a single sine-wave impeller approximately 25 mm in diameter mounted flash side up.

9.2.4 Container, of approximate dimensions 180 mm deep, 97 mm i.d. top, 70 mm i.d. bottom (e.g. Hamilton® Beach mixer cup No. M110-D, or equivalent).

9.2.5 Spatula.

9.2.6 Motor-driven direct-indicating viscometer, as referenced in API RP 13B-1, subclause 4.3.

9.2.7 Filter press, as referenced in API RP 13B-1, subclause 5.2.1.

9.2.8 Graduated cylinders (TC), of capacities 500 cm3 ± 5 cm3 and 10 cm3 ± 0,1 cm3

9.2.9 Deionized or distilled water.
9.2.10 Container, with lid, of capacity about 500 cm3.

9.2.11 Timers, two, mechanical or electrical, with an accuracy of 0,1 min over the test interval.

9.3 Suspension properties — Procedure for rheology

9.3.1 Prepare a suspension of the bentonite. Add 22,5 g ± 0,01 g of clay (as-received) to 350 cm3 ± 5 cm3 of deionized water while stirring on the mixer.

9.3.2 After stirring 5 min ± 0,5 min, remove the container from mixer and scrape its sides with the spatula todislodge any bentonite adhering to container walls. Be sure all bentonite clinging to the spatula is incorporated into the suspension.

9.3.3 Replace the container on the mixer and continue to stir. The container may need to be removed from the mixer and the sides scraped to dislodge any clay clinging to container walls after another 5 min and after 10 min.Total stirring time shall equal 20 min ± 1 min.

9.3.4 Age the bentonite suspension up to 16 h in a sealed or covered container at room temperature. Record storage temperature and storage duration.

9.3.5 After ageing bentonite suspension, shake well and then pour the suspension into the mixer container. Stir the suspension on the mixer for 5 min ± 0.5 min.

9.3.6 Pour the suspension into the viscometer cup provided with the direct indicating viscometer. The dial readings at 600 r/min and 300 r/min rotor speed settings of the viscometer shall be recorded when a constant value for each r/min is reached. Readings shall be taken at a suspension test temperature of 25 °C ± 1 °C.

9.4 Suspension properties — Procedure for rheology calculations

Plastic viscosity = [600 r/min reading] 2 [300 r/min reading] (15)

Yield point = [300 r/min reading] 2 [plastic viscosity] (16)

Yield point/plastic viscosity ratio = [yield point (equation 16)] / [plastic viscosity (equation 15)] (17)

Record calculated values for plastic viscosity, yield point, and yield point/plastic viscosity ratio.

9.5 Suspension properties — Procedure for filtrate volume

9.5.1 Recombine all of the suspension as prepared and tested in 9.3 and stir in container for 1 min ± 0,5 min on the mixer. Adjust suspension temperature to 25 °C ± 1 °C.

9.5.2 Pour the suspension into the filter press cell. Before adding the suspension, be sure each part of the filter cell is dry and that all gaskets are not distorted or worn. Pour the mud to within about 13 mm of the top of the cell. Complete assembly of the filter press cell. Place filter cell in frame and close relief valve. Place a container under the drain tube.

9.5.3 Set one timer for 7,5 min ± 0,1 min and the second timer for 30 min ± 0,1 min. Start both timers and adjust pressure on cell to 690 kPa ± 35 kPa. Both of these steps shall be completed in less than 15 s. Pressure shall be supplied by compressed air, nitrogen or helium.

9.5.4 At 7,5 min ± 0,1 min on the first timer, remove the container and any adhering liquid on the drain tube and discard. Place the dry 10 cm3 graduated cylinder under the drain tube and continue collecting filtrate until the end of the second timer set at 30 min. Remove the graduated cylinder and record the volume of filtrate collected.

9.6 Suspension properties — Calculation of filtrate volume

Calculate the filtrate volume of the clay suspension as:

Filtrate volume, $cm^3 = 2 \times Vc$ (18)

where Vc is the filtrate volume collected between 7,5 min and 30 min.

Record calculated filtrate volume.

9.7 Residue of diameter greater than 75 mm — Reagent and apparatus

9.7.1 Sodium hexametaphosphate (CAS No. 10124-56-8).

9.7.2 Oven, regulated to 105 °C ± 3 °C.

9.7.3 Balance, with an accuracy of 0,01 g.

9.7.4 Mixer (e.g. Multimixer Model 9B with 9B29X impellers or equivalent), having each spindle fitted with a single sine-wave impeller, approximately 25 mm in diameter, mounted flash side up.

9.7.5 Container of approximate dimensions 180 mm deep, 97 mm i.d. top, 70 mm i.d. bottom (e.g. Hamilton Beach mixer cup No. M110-D, or equivalent).

9.7.6 Spatula.

9.7.7 75-mm sieve, conforming to the requirements of ASTM E11, of approximate dimensions 76 mm diameter, and 69 mm from top of frame to wire cloth.

NOTE Supplier's verification that sieve conforms to ASTM E11 is satisfactory evidence of compliance.

9.7.8 Spray nozzle, 1/4 TT body (Spraying Systems Co., No. TG 6.5 tip with 1/4 TT body or equivalent) attached to water line with 90° elbow.

9.7.9 Water-pressure regulator, capable of regulation to 69 kPa ± 7 kPa.

9.7.10 Evaporating dish.

9.7.11 Wash bottle.

9.8 Residue of diameter greater than 75 mm — Procedure

9.8.1 Weigh 10 g ± 0,01 g of bentonite.

9.8.2 Add the weighed bentonite sample to approximately 350 cm3 of water containing about 0,2 g of sodium hexametaphosphate while stirring on the mixer.

9.8.3 Stir suspension on the mixer for 30 min ± 1 min.

9.8.4 Transfer the sample to the sieve. Use the wash bottle to remove all material from the container to the sieve. Wash the material on the sieve with water controlled to 69 kPa ± 7 kPa from the spray nozzle for 2 min ± 15 s. While washing, hold the tip of the spray nozzle approximately in the plane of the top of sieve and move the spray of water repeatedly over the sample.

9.8.5 Wash the residue from the sieve into a tared evaporating dish and decant excess clear water.

9.8.6 Dry the residue in the oven to a constant mass. Record residue mass and total drying time.

9.9 Residue of diameter greater than 75 mm — Calculation

$$\text{Residue, mass fraction (\%) greater than 75 µm diameter} = 100\left(\frac{\text{mass of residue, g}}{\text{mass of sample, g}}\right) + C$$

where C is the correction factor calculated from 5.5.

Record calculated value.