# STUDY ON BATU GAJAH CLAY AS DRILLING FLUID MATERIALS

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**Study On Batu Gajah Clay as Drilling Fluid Materials**

By

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Dissertation submitted in partial fulfillment of
the requirements of the
Bachelor of Engineering (Hons)
(Civil Engineering)

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# CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
Civil Engineering Programme
Universiti Teknologi PETRONAS
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Approved:

Assoc. Prof. Dr. Nasiman B. Sapari
Project Supervisor

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

July 2010

# 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.

______________________________

(Yasmin Bt. Khamis)

# ABSTRACT

CLAY is a common material used for water based drilling fluid. The montmorillonite found in clay is beneficial for the drilling fluid technology. This project aims to evaluate the suitability of the clay as the drilling fluid material. This project is to analyze the physical, chemical, and mineralogy and also the rheological properties of the clay sample from Batu Gajah. Based on the physical analysis, the clay was found to be silt with high plasticity value. The clay then analyzed for its mineral composition using X Ray Diffractometer, and the sample was proven to be mainly composed of quartz. X Ray Fluorescence (XRF) analysis shows that the ratio of $Al_2O_3/SiO_2$ is 1/3 which however proven that they clay sample contains high montmorillonite. The clay sample was shown to have low capacity of water adsorption as it obtained very low value of Cation Exchanged Capacity (CEC) compared to the reference bentonite. The clay has weak rheological properties such as the viscosity, yield point and gel strength. Due to the weak rheological properties, the clay sample experienced huge losses of filtration that caused the mud cake thickness to be higher than the reference bentonite. Overall, the raw clay sample is not suitable to be formulated to drilling fluid. Therefore, there are recommendations for the clay sample to improve its weak properties. Organic polymer can be used to control the rheology and filtrate loss for water based drilling fluid.

# ACKNOWLEDGEMENT

This project has taken about a year of planning and working processes in order to fulfill the requirements of Final Year Project subject. In a year duration, many people had being involved either directly or indirectly whose contribute in assorted ways to this project in order to make it a success. Thus, it is a pleasure for me to convey my heartfelt gratitude to them all in this humble acknowledgement.

In the first place, I would like to record my gratitude to Assoc. Prof. Dr. Nasiman Bin Sapari. He was not only served as my supervisor but also encouraged and challenged me throughout his constructive comments, critics and valuable suggestions. Besides, his full supports and advices had actually inspired me and develop my skills as a researcher and engineer to be.

It is also a pleasure to pay tribute to the lecturers from Petroleum and Geoscience Department, Dr. Sonny Irawan and Dr. Azurein Jaafar for their endless support and guidance throughout this project. I also wish to express my sincere and warm thanks to the technicians both in Civil and Petroleum Engineering Laboratory for their cooperation in making my project a great success.

Not to forget, my parents and family members who have given a never-ending support in the quest to gain valuable knowledge and experience in this university throughout the completion of this project. They deserve a special mention for their inseparable supports and prayers.

Certainly on the list of peoples to be thank of are all friends from all around and also to those people that not be mentioned here. Your names may slip of my mind at this moment, but your actions and helps stay in my mind forever.

And last but not least, grateful gratitude to the almighty God that has made all things possible.

# TABLE OF CONTENTS

## LIST OF FIGURES

# LIST OF TABLES

# CHAPTER 1

# PROJECT BACKGROUND

## 1.1 BACKGROUND OF STUDY

The title for the author's Final Year Project is "Local Materials for Drilling Fluid Formulation". Local materials can be specifically defined as Bentonite. Bentonite is an absorbent aluminium phyllosilicate, generally impure clay consisting mostly of montmorillonite. Montmorillonite is said to be used in oil and gas industry as a component of drilling fluid which is capable of making the mud slurry viscous and helps in keeping the drill bit cool and removes the drilled solid. Drilling fluid that contains high montmorillonite is believed to make a good and effective fluid compared to Synthetic Based Mud which is widely used now.

It is crucial to keep developing the drilling fluid system as there are many drilling challenges relating the fundamental objective of maintaining a workable wellbore. Most operational problem is interrelated for example loss of circulation will lead to a drop in hydrostatic pressure in the wellbore. Incident like this is common in drilling practice; therefore a good drilling fluid system will benefit the drilling system. Besides enable to increase productivity, it is also will enable to prevent conditions that lead to wellbore instability.

There are few spotted areas in Perak believed to have clay that contains high montmorillonite. The areas are around Gerik and also Batu Gajah. The author has taken the sample from the Batu Gajah area and is running some test on the samples to investigate and identify the properties of the sample and also its suitability as drilling fluid.

## 1.2 PROBLEM STATEMENT

Producing drilling mud or also known as drilling fluid can be very costly for the oil companies. Drilling mud can cost roughly up to 10 percent of the total cost of oilwell drilling and completion. This problem can be overcome by utilizing the natural resources to produce a formula for drilling fluid. This project will focus in producing water based mud or WBM which usually consists of water and clays.

## 1.3 OBJECTIVES OF STUDY

The objectives of the project are as below:

i) To identify the physical characteristics and properties of the clay sample
ii) To identify the mineralogy properties of the clay sample
iii) To identify the rheology properties of the clay sample
iv) To repeat all the identifications on the reference bentonite
v) To study and propose modifications for the clay sample if it does not meet the standards of drilling fluid based on the reference bentonite properties and API Specifications 13A (Specifications of Drilling Fluid Materials).

## 1.4 RELEVANCY OF THE PROJECT

The tasks are basically first to check the physical and chemical properties of the samples taken continued by formulating the fluid using the samples believed to be montmorillonite clay. If this study can prove that the areas do have the potential montmorillonite clay or bentonites to be made the drilling fluid, then it will be able to make such a significant contribution to the oil and gas industry as well as the country.

## 1.5 FEASIBILITY OF THE PROJECT

As explained in project background, the author has almost ten months to complete this study. The first two months will be allocated for in depth study and research on the topics related. During this time, the author will gather as much journals as possible regarding the topic. Furthermore, the author will seek opinions from the lecturers related to this study as well as people from the industry like the engineers and technicians.

# CHAPTER 2

# LITERATURE REVIEW

Throughout these two months of study, the author has found several paperwork related to the research topic. Below is the literature review done on some of the paperwork of related topic.

## 2.1 HISTORY OF DRILLING MUDS

According to Lewis J.O and Mc Murray (1916) " Early years of oil and gas development, drilling mud was defined as the mixture of water with any clayey materials which will remain suspended in water for a considerable time and is free from sand, lime cuttings or similar materials. The weight of the mud or fluid should lie between the specific gravity values 1.05 to 1.15(8.75 to 9.58 ppg). A good drilling fluid was considered to be thick enough to cloc the pores of sand and rock and not pass into them. A thick fluid was considered as having the advantages of plugging the sand, preventing caving of formations and holding back high gas pressures."

## 2.2 DRILLING FLUID FROM LOCAL BENTONITE

Lewis J.O and Mc Murray (1916) also points out that Bentonite is an absorbent aluminium phyllosilicate, generally impure clay consisting mostly of montmorillonite. There are a few types of bentonites and their names depend on the dominant elements, such as K, Na, Ca, and Al. As noted in several places in the geologic literature, there are some nomenclatorial problems with the classification of bentonite clays. Bentonite usually forms from weathering of volcanic ash, most often in the presence of water.

Sonny Irawan and Ariffin Samsuri (2009) have done research on the development of Sabah bentonite as drilling mud material has been initiated. In the drilling industry, bentonite is generally classified as sodium (Na) or calcium (Ca) types, depending on dominant exchangeable ion.

In this study, two groups of local bentonite samples were collected from Andrassy area in Tawau district and from Mansuli area in Lahad Datu district. The field sampling from Mansuli area were taken from mainly from the area underlain by the Ayer Formation, which is collectively from the Segama Group and is interpreted to be Miocene in age. The Andrassy area is underlain mainly by the high level of alluvium and volcanic rock, and occur in bed underlying in Pleistocene to Holocene in age. Utilization of bentonite after their extraction and adds value to Sabah economy by minimizing imported bentonite used by oil and gas industry.

Following the is flow chart of the upgrading the Sabah bentonite to be made the drilling fluid;

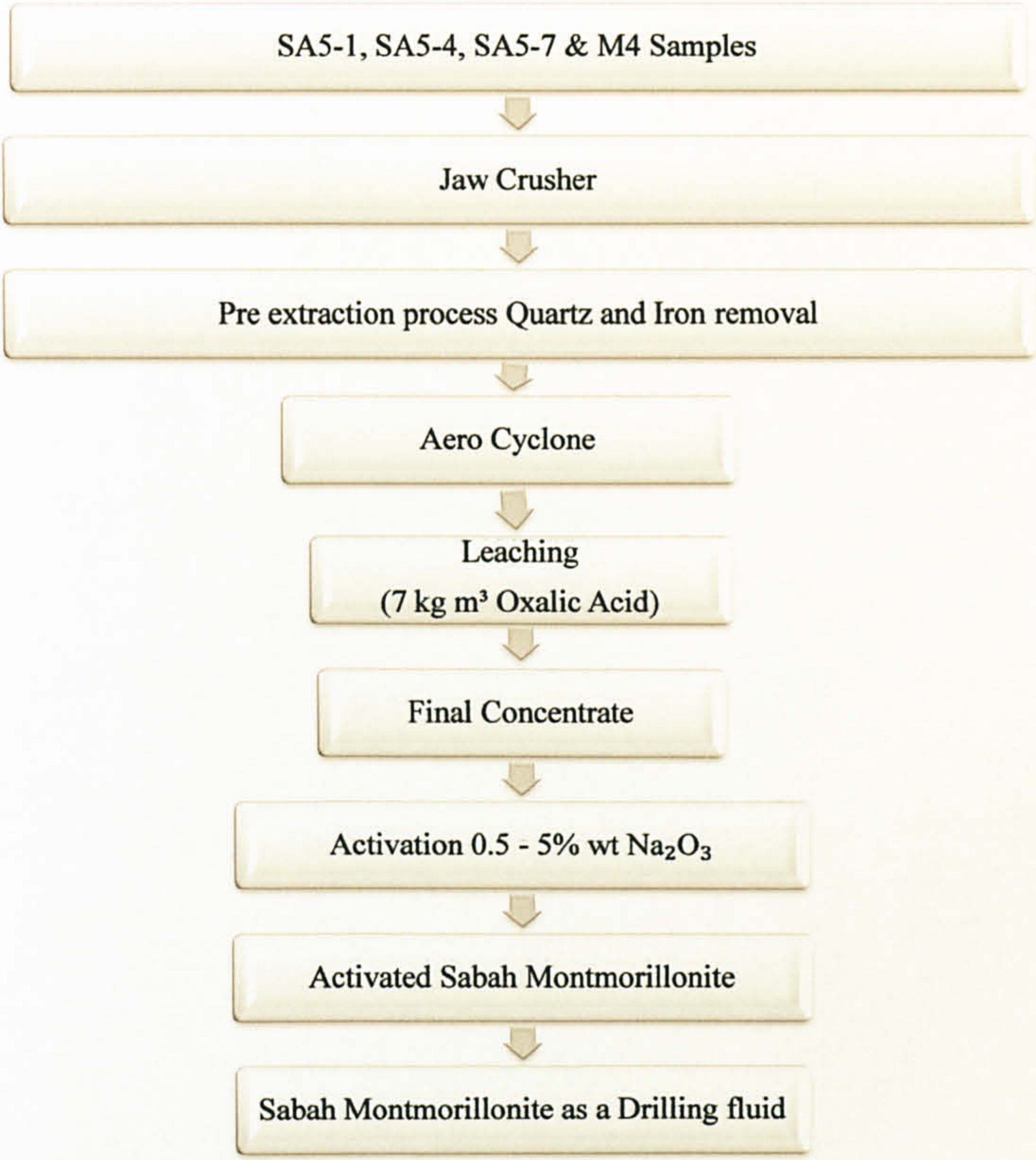


Figure 2.1:A flow sheet for the upgrading Sabah bentonite and its use in drilling fluid

The sample later went through some other few experiments to have its physical and chemical properties determined. The physical properties include the pH, cation exchange capacity (CEC) and specific surface area (SSA) while the physical properties are the liquid limit (LL), plastic limit (PL) and plasticity index (PI), moisture absorption (MA), moisture content (MC) and ignition loss (IL).

To continue this research, the sample was then taken to be tested its rheological properties like the apparent viscosity (A.V), the plastic viscosity (P.V) and also the yield point (Y.P).

After experiments and tests are completed, all the data on the properties are gathered to make comparisons. Qualitative and quantitative analysis are done to see whether if there are improvements on the mineral compositions after organic acid beneficiation. There are actually significant changes on the montmorillonite contents on the sample after beneficiated by the oxalic acid. It had successfully removed iron content as impurities in the sample.

For the conclusion of this study, Sabah bentonite can be beneficiated based on organic acid treatment by applying organic acid concentration of 7.1 $kgm^3$, pH less than 2, 80°C temperature and 2 hours stirring time. The beneficiated bentonite performance as drilling mud material can be improved by 3% wt Tannathin addition.

In the same context of study, Emad S. Al Homadhi concluded that it is very crucial for his country Arab Saudi to find local materials to be made the drilling fluid as present consumption of bentonite clay in the drilling operations in Saudi Arabia alone can reach over 100 thousand tons a year and all of it is imported from USA.

The properties of the local mud can be improved economically by adding some cheep materials to the prepared mud to enhance its viscosity and filtration loss (such as CMC, Drispac polymer, and bentonite extenders). Also shearing speed can be used as an enhancement method to improve the dispersion rate of the clay suspension, and hence increase viscosity and decrease filtration loss. But the used shearing speed should be limited to a practical and economical speed. The bentonite extender can be either a salt or a polymer; it enhances viscosity buildup by slightly flocculating the bentonite suspension. Sodium carbonate is an example of a salt that can be added as an extender. Soaking raw bentonite with a solution of sodium salt resulted, through ion exchange, in a higher sodium bentonite content. More effective extenders than inorganic salts are the high molecular weight polymers.

In order to produce a product which can compete with the imported bentonite and to meet the API standards, he suggested enhancing the performance of the local Bentonite by adding salt extender (Soda Ash) at a concentration of 5 % and by adding cheap polymer such as Drispac at a very low concentration of 0.5 % of the added Bentonite

## 2.3 CLAY IDENTIFICATION TESTS

### 2.3.1 X Ray Diffraction (XRD)

XRD is used to determine the mineralogical composition of the raw material components as well as qualitative and quantitative phase analysis of multiphase mixtures. No quantitative estimation phases in these adsorbents have been made but their characterization of XRD patterns indicates the presence of quartz, kaolinite, hematite, illite, and tridymite as the major phases (Pretti Sagar Nayak, BK Singh,2007a).

### 2.3.2 X Ray Fluorescence (XRF)

XRF is a technique performed to know the chemical compositions of the minerals that are present in the clay. Below is the example of the chemical analysis (Pretti Sagar Nayak. BK Singh, 2007b)

*Table 2.1: Example of chemical analysis of clay*

| Chemical Composition | Weight Percentage (%) |
|---|---|
| SiO2 | 48.12 |
| $Al_2O_3$ | 34.54 |
| $Fe_2O_3$ | 2.48 |
| Cao | 0.83 |
| .Mgo | 0.50 |

### 2.3.3 Moisture Content Test

Water content or moisture content is the quantity of water contained in a material, such as soil (called soil moisture), rock, ceramics, or wood on a volumetric or gravimetric basis. The property is used in a wide range of scientific and technical areas, and is expressed as a ratio, which can range from 0 (completely dry) to the value of the materials' porosity at saturation.

### 2.3. Cation Exchanged Capacity (CEC)

The capacity of the soil to hold on these cations are called the cation exchanged capacity (CEC). These cations are held by negatively charged clay and organic matter particles in the soil through electrostatic forces (negative soil particles attracted to positive charged particles). Thus, the CEC of a soil represents the total amount of exchangeable cations that soil can absorb (Ketterings, Reid, Rao, 2007).
The primary factor determining CEC is the clay and organic matter content of the soil. Higher quantities of clay and organic matter result in higher CEC. Different types of clay have different CECs. Amounts of negative and positive charges are both expressed in miliequivalent. One miliequivalent of negative charge on a clay particls is neutralized by one miliequivalent of cation. A miliequivalent takes into account both the weight and the charge of cation (Camberato, 2001)

- Transmit hydraulic energy to tools and bit
- Minimize reservoir damage
- Permit adequate formation evaluation
- Control corrosion
- Facilitate cementing and completion
- Minimize impact on the environment
- Inhibit gas hydrate formation

### 2.4.3 Properties of Drilling Fluids

Properties of drilling fluids are usually been categorized in rheology. Rheology is the study of the deformation and flow of matter (Growcock, Harvey, 2005). The effectiveness of the drilling fluids are related to these properties; (Dyke, Kate, 2000).

- Viscosity

Viscosity is resistance to flow. Drilling fluids with high viscosity resist flow more than a drilling fluid with low viscosity. A viscous drilling fluid can transport more and heavier cuttings, so a fluid often contains a material to increase its viscosity. Bentonite a commonly used colloidal clay is made up of montmorillonite clay mineral that disperses into small particle in water and hydrate and makes the fluids very viscous.

Based on O.A Falode et al (2008a), viscosity of a mud is a function of three components which are the plastic viscosity, yield point and gel strength.

Plastic viscosity is the resistance to flow in mud caused by the friction between suspended particles and the viscosity of the base fluid. Yield point is a measure of the forces between the particles in mud. The resistance to initial flow or the stress required to start fluid movement then is the yield point, which is measured under flowing conditions. Gel strength is a measure of the same interparticle forces of the mud as determinded by the yield point but the difference is gel strength is measured with the mud at rest.

- Density

Under normal drilling condition, preventing these fluids from flowing into the wellbore is crucial. Underbalanced drilling allows fast penetration rates because hydrostatic pressure is low. The density and weight determines the hydrostatic pressure of the fluids.

- pH of water

The pH is the the response of how acidic or alkaline is the water. Bentonite will perform the best with a pH range 7 to 9. This property is able to affect mud mixing, viscosity, water loss and core recovery.

- Solid content

Solids are added to the fluid or accumulate in the fluid. Solids may be added to increase viscosity or to increase weight. Examples of the solids are barite and hematite.

# CHAPTER 3

# METHODOLOGY

## 3.1 PROJECT METHODOLOGY

The project method will be according to Table 3.1

*Table 3.1: Methodology of project*

| | | |
|---|---|---|
| **Literature** | Literature Studies | Literature studies are conducted on the properties of potential drilling fluid and others related to the subject. This will provide an overview as well as the flow of the project. |
| **Experiements/Analysis** | Physical Properties | Identify the physical properties of the sample retrieved. The experiments will be moisture content, liquid limit, plastic limit, plasticity index, density, specific gravity |
| | Mineralogy & Chemical Properties | Identify the mineralogy properties of the clay sample. XRD or X Ray Diffraction will be done on the clay sample to identify the mineralogy composition of the clay sample. X Ray Fluorescence will also be used for chemical analysis of the clay sample. |
| | Rheology Properties & Wall Filtration | Identify the rhelogy properties of the clay sample. The rheology properties are viscosity, apparent viscosity, and plastic viscosity and yield point. Wall filtration test will also be conducted using Standard API Filter Press. |
| | Comparison & Analysis using API 13A | All the experiments will be repeated using reference bentonite to compare the the properties with the clay sample, API 13A will also be used in analyzing the suitability of the clay sample to be formulated to drilling fluid. |
| **Modifications** | Propose modifications to improve the properties | Chemicals will be used to optimize and increase drilling efficiency properties of the clay,for example Xanthan Gum to improve the viscosity of the clay. |

For moisture contents, the mass of wet clay before and after oven dried will be weighed. The experiment is done using three samples. The temperature of oven dried is around 105°C to 110°C. The moisture contents than calculated with the formula below.

$$W = \frac{(m^2 - m^3)}{(m^3 - m_1)} x\ 100\ \% \qquad (1)$$

Where

W is the moisture content in percentage (%)

$m_1$ is the mass of container (g)

$m_2$ is the mass of container and wet soil (g)

$m_3$ is the mass of container and dry soil (g)

For plastic limit, three sets of same sample weights if 20g which passes the 425 μm test sieve is dried partially on plate until it becomes plastic enough to be shaped into the ball. The sample then moulded in fingers to equalize the distribution of moisture contents and then the sample is formed into a thread about 6mm diameter, The diameter then reduced to the diameter of the thread to about 3mm. The samples then moulded again until it shears both longitudinally and transversely. In order to reform a thread, the sample is rolled and the first crumbling point is the plastic limit. The moiture content is calculated using over drying method and the average moisture content is the plastic limit.

Liquid Limit is determined by doing a cone penetrator experiment. The relationship between moisture content and cone penetration are plotted. The liquid limit is indicated by the moisture contents corresponding to a cone penetration of 20mm.

Cation Exchange Capacity is measured using Methylene Blue Test (MBT). Approximately 1 gram of montmorillonite sample will be tested in 50ml of distilled with about 0.5 ml of 5N sulphuric acid added. The montmorillonite solution will then be boiled gently for 10 minutes. The CEC value will be measured by conductomercial titration after cation exchange, meq/100 grams. Based on studies done by Sonny Irawan and Ariffin Samsuri on "Beneficiation of an Andrassy and Mansuli Bentonite as a Drilling Mud Material", a sodium based bentonite should have CEC value same as that of montmorillonite (80 -100 meq/100 grams).

The pH value is determined by the pH meter and chemical composition is identified by the X Ray Fluorescence (XRF) analysis.

To determine the density, specific gravity, viscosity and also the rheology properties, the clay sample has to be diluted with distilled water. Below is a simple diagram to show the flow of these experiments. These experiments are done in Petroleum and Geosciences Department.

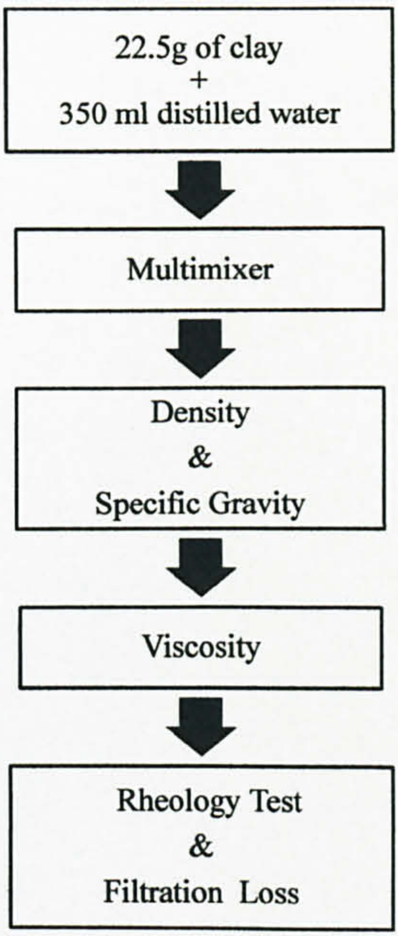


Figure 3.1: Flow of experiments in Petroleum & Geoscience Department

22.5 g of clay sample is diluted with 350ml distilled water and then stirred in multimixer. This is to allow the solution to be dispersed uniformly. Density and specific gravity is determined by using Baroid Mud Balance whereas viscosity with Marsh Funnel. Rheology Test is done using viscometer and last but not least wall filtration test is completed using Standard API Filter Press. The equipments will be explained further in the next section.

To determine the viscosity of the clay sample, the Marsh Funnel is used. Viscosity is the time required for clay to fill the receiving vessel to the 1- quart (946 ml) level.

For rheology tests, viscometer will be used to determine all three properties, the apparent viscosity, plastic viscosity and also the yield point. The reading of the viscometer at 600 rpm is taken as the apparent viscosity (A.V). Plastic viscosity will be determined by the difference between the readings of 600 rpm and 300 rpm. On the other hand, yield point will be calculated from the difference between the reading of viscometer at 300 rpm and plastic viscosity.

## 3.2 TOOLS/EQUIPMENTS

For physical tests, basically the equipments used can be found in Geotechnical Laboratory in Civil Engineering Department. Those equipments like the compaction machine, the cone penetrator, high temperature ovens and sieves.

Figure 3.2: Clay sample

Figure 3.3: Compaction Machine

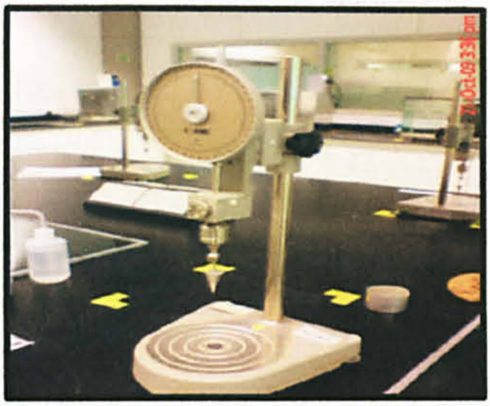

Figure 3.4: Cone Penetrator Method

For mineralogy analysis, the equipments that will be used are XRD equipment, model D8 and its function is to characterize the crystallographic structure, crystallite size (grain size) and preferred orientation in polycrystallite or powdered solid samples. XRD will determine the mineral composition of the clay sample. Examples of minerals are quartz, kaolinite, illite and others.

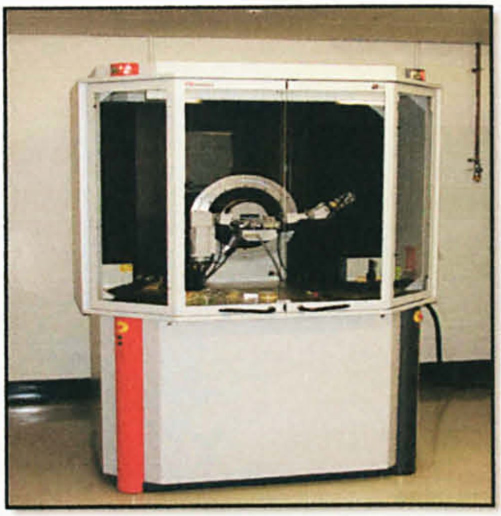

Figure 3.5: XRD Equipment

For chemical analysis, X Ray Fluorescence (XRF) is used to determine the main chemical elements of the clay sample such as Magnesium, Calcium, Kalium and others. To determine the alkalinity of the clay sample, pH value will be identified using the pH meter Model Oakton. Below are the figures of the equipment to perform the chemical analysis.

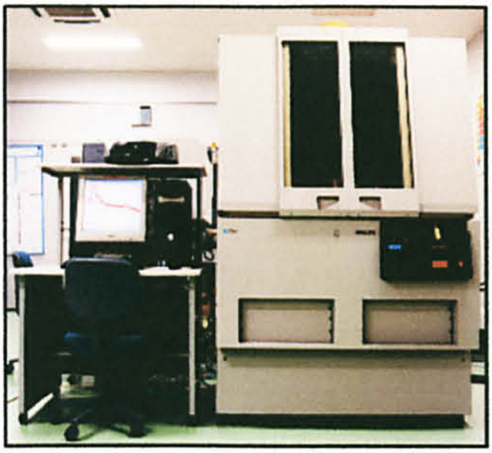

Figure 3.6: XRF Spectrometer

Figure 3.7: pH Meter

The Baroid Mud Balance is used to determine density of the drilling fluid. The instrument consists of a constant volume cup with a lever arm and rider calibrated to read directly the density of the fluid in ppg (water = 8.33), pcf (water 62.4), specific gravity (water = 1.0) and pressure gradient in psi/1000 ft, (water = 433).

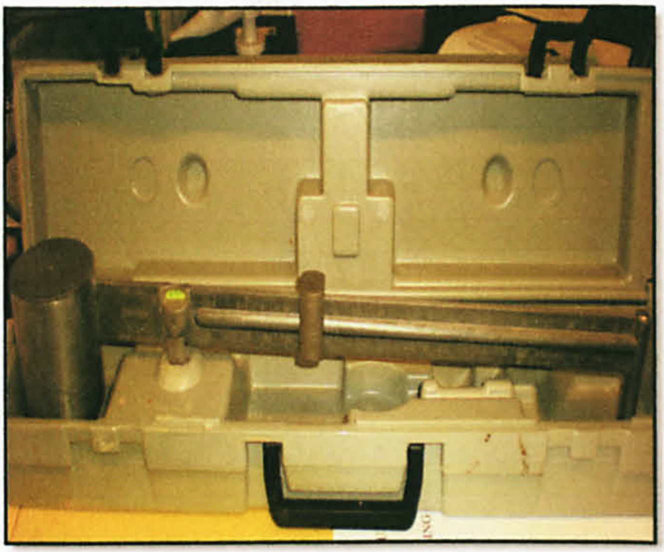

Figure 3.8: Typical Mud Balance

The Marsh Funnel is a device that is common to every drilling rig. The viscosity is reported in seconds allowed to flow out of the funnel. API specifications call for 1500ml and one quart (946ml) out. For API water at 70F + 0.5F = 26+0.5 sec. The Marsh Funnel measures the apparent viscosity.

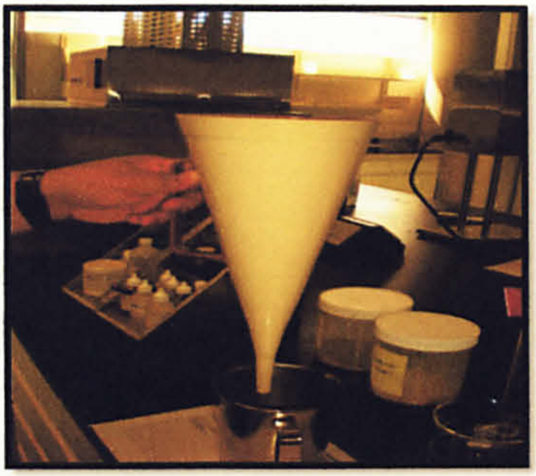

Figure 3.9: Marsh Funnel and One Litre Cup

The FANN (Model 35A) Viscometer is a coaxial cylindrical rotational viscometer, used to determine single or multipoint viscosities. It has fixed speeds of 3 (GEL), 6, 100, 200, 300, and 600 RPM that are switch selectable with the RPM knob.

Figure 3.10: Viscometer

Standard API Filter Press is used to measure filtration rate at elevated temperatures and pressure. Filter press used for filtration tests consist of four independent filter cells mounted on a common frame. Each cell has its own valve such that any or all the cells could be operational at the same time. Toggle valve on the top of each cell could be operated independently for the supply of air for each individual cell. Special high pressure and high temperature filtration tests are run in the laboratory simulating formation temperature and formation back pressure.

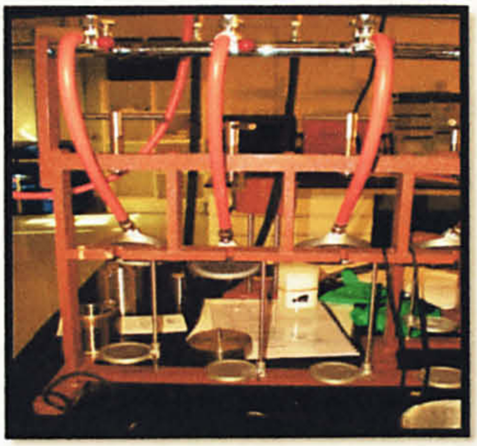

Figure 3.11: Standard API Filter Press

# CHAPTER 4

# RESULTS AND DISCUSSIONS

## 4.1 MOISTURE CONTENT

Below are the simplified calculations on the moisture contents. The experiment was done on three sets of the same example and the moisture contents are determined by the average percentage.

Set 1

$$\frac{38.0 - 34.4}{34.4 - 18.7} \times 100\% = 23\%$$

Set 2

$$\frac{30.8 - 28.3}{28.3 - 18.6} \times 100\% = 26\%$$

Set 3

$$\frac{31.3 - 29.2}{29.2 - 20.9} \times 100\% = 25.3\%$$

**MOISTURE CONTENTS**

$$\frac{23 + 26 + 25.3}{3} = 24.80\%$$

## 4.2 PLASTIC LIMIT

The formula to calculate the plastic limit is a just the same with the over dryingmethod. The calculation of the plastic limit is shown below ;

Set 1

$$\frac{26.58 - 24.87}{24.87 - 20.18} \times 100\% = 36.46\%$$

Set 2

$$\frac{26.82 - 24.85}{24.85 - 19.37} \times 100\% = 35.95\%$$

Set 3

$$\frac{26.37 - 25.00}{25.00 - 21.32} \times 100\% = 37.23\%$$

Set 4

$$\frac{23.53 - 22.42}{22.42 - 19.41} \times 100\% = 36.88\%$$

**PLASTIC LIMIT**

$$\frac{\mathbf{36.46 + 35.95 + 37.23 + 36.88}}{\mathbf{4}} = \mathbf{36.63\%}$$

## 4.3 LIQUID LIMIT

Liquid limit is determined by the moisture content corresponds to a cone penetration of 20 mm. below is the graph of penetration of cone vs moisture content.

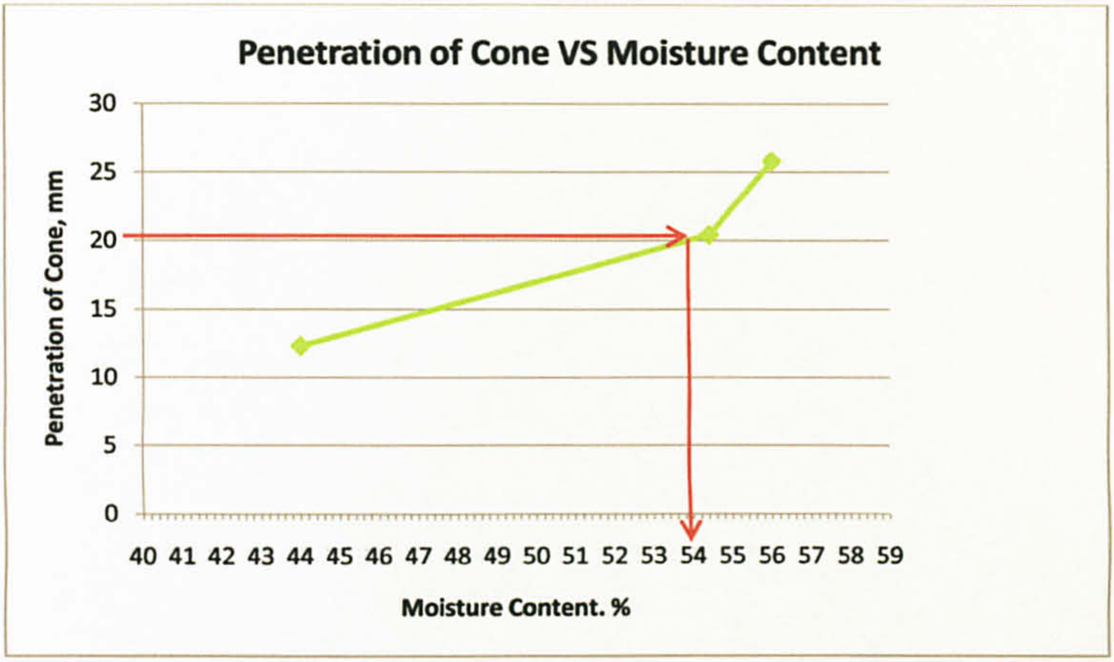


Figure 4.1 : Penetration of Cone VS Moisture Content

From the above graph, the moisture content corresponds to a cone penetration of 20mm is 54%. Thus, the liquid limit is 54%.

## 4.4 PLASTICITY INDEX

$$Ip = LL - PL \qquad (2)$$

$$Ip = 54\% - 36.63\%$$

$$= 17.37\%$$

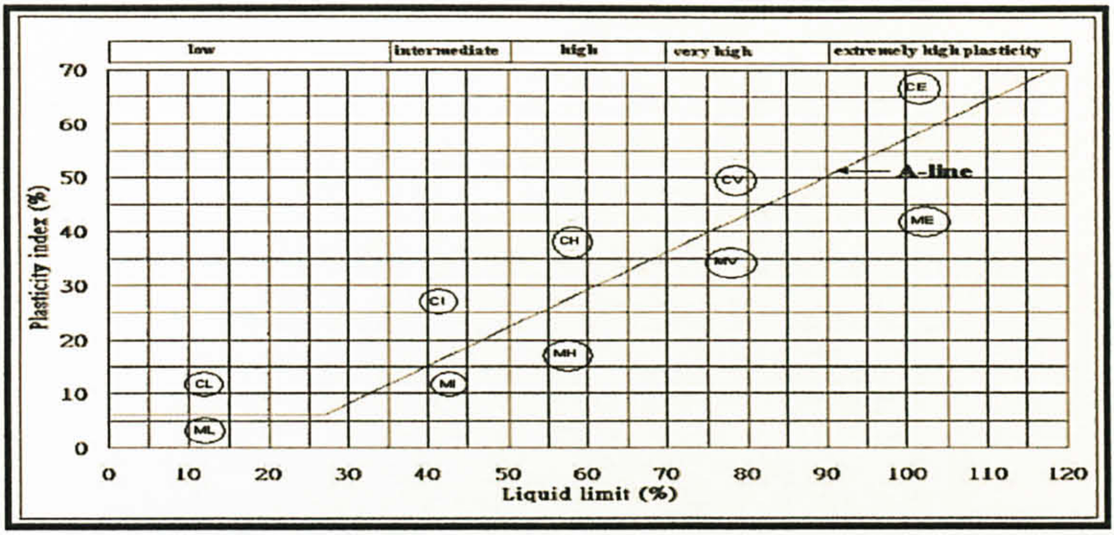


Figure 4.2 : Plasticity Chart For the Classification of Soils

*Table 4.1 : The Atterberg Limit for Batu Gajah clay and reference bentonite*

| Sample | Liquid Limit | Plastic Limit | Plasticity Index |
|---|---|---|---|
| Reference bentonite | 700 | 65 | 635 |
| Batu Gajah clay | 54 | 36.63 | 17.37 |

Based on the Plasticity Chart, the sample can be identified as silt with high plasticity value. The plasticity index for Batu Gajah clay was found to be lower than the reference bentonite. The plasticity index indicates the compressibility of the clay. The greater the plasticity index value, the more compressible is the clay. This property also related to the swelling characteristics of the clay. The higher is the plasticity index, the clay tend to swell better.

## 4.5 X RAY DIFFRACTION (XRD)

The purpose of the X Ray Diffraction on the clay sample is to analyze the mineralogical composition of the clay sample. Below are the figures of the X Ray Difractogram of the clay sample and also the Wyoming and Pindiga bentonite which are also used as the materials for drilling fluid. This is to compare and observe the similarities between the materials as for potential drilling fluid.

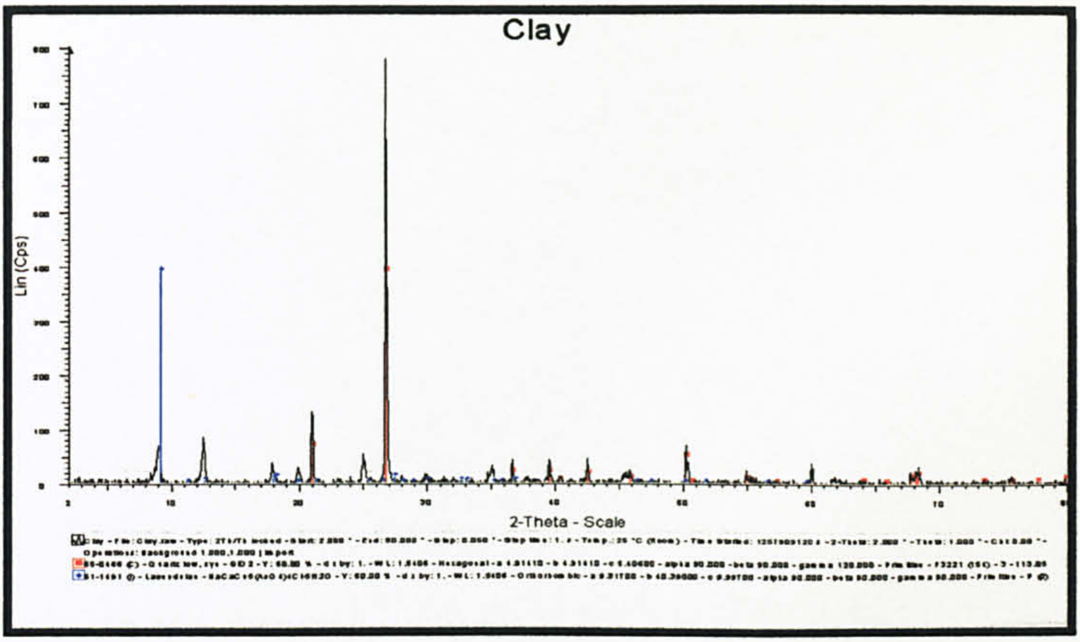


Figure 4.3 : X Ray Difractogram of the clay sample

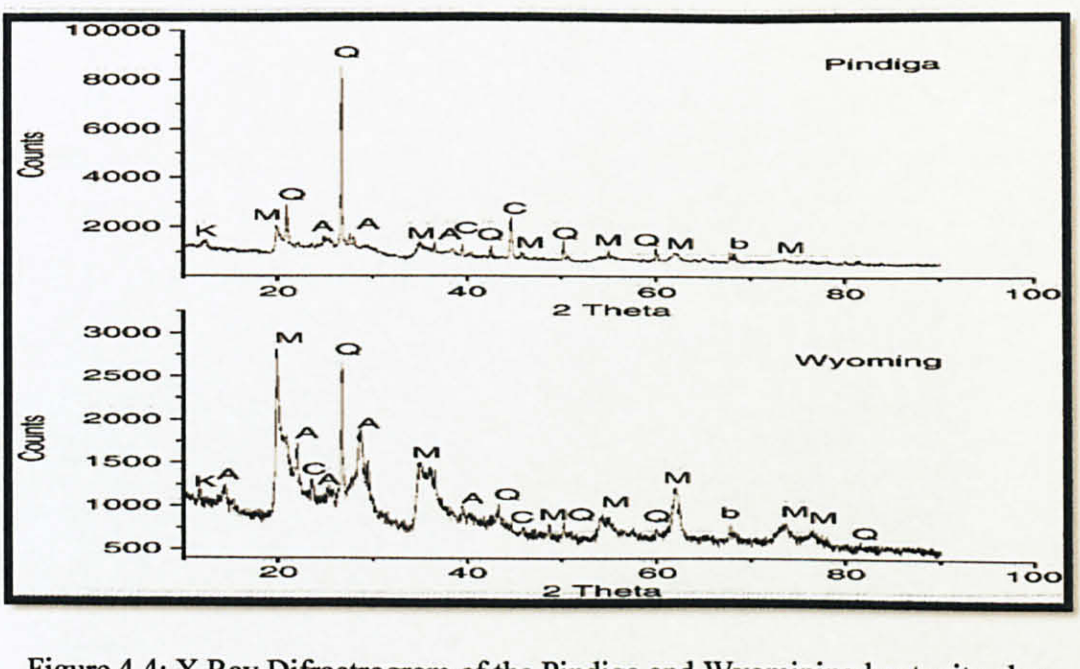


Figure 4.4: X Ray Difractrogram of the Pindiga and Wyomining bentonite clay
(O.A. Falode et al.,2008)

Based on the XRD analysis on the samples above, it was observed that both clay sample and Pindiga and Wyomining bentonite clay are mainly composed of quartz minerals. Therefore, the clay sample can be said to be suitable as drilling fluid materials. This is also mentioned in API Specifications 13A (Drilling Fluid Materials) that drilling material like clay should contain minerals like quartz, mica, feldspar, and calcite.

## 4.6 CATION EXCHANGE CAPACITY (CEC)

The capacity of clay to absorb cations from solution may be measured by the uptake of methylene blue, a cationic dye. This test is to determine the amount of clay in the drilling fluid. CEC is dependent on the type and quality of mineral present (Baroid Petroleum Services, 1984a). Below is the table of CEC value for both the clay sample and also the reference bentonite.

*Table 4.2: Cation Exchange Capacity (CEC) value for clay sample and also the reference bentonite*

| Sample | CEC (meg/100g) |
|---|---|
| Clay sample | 45.5 |
| Reference bentonite | 70 |

Based on the CEC values above, it is shown that the CEC value of the clay sample is very low compared to the reference bentonite. This shows that the clay has low capability of water adsorption. This low value of CEC usually leads to the swelling characteristic of clay. Sodium type usually has higher value of swelling strain as compared to the calcium type (Hideo Komine, 2003). This is because sodium has low cation replaceability capacity (McBride, 1994).

CEC value can be improved by undergoing a physical treatment using a high intensity wet magnetic (Balakrishnan S.,2009). This method is to separate the impurities and that causes the low CEC value.

## 4.7 RAY FLUORESCENCE (XRF)

X Ray Fluorescence or XRF is a chemical analysis that purposely to identify the chemical composition of any materials which specifically for this research is clay. This analysis is considered very crucial as each chemical component that consists in the clay sample will contribute to different properties for example Magnesium Oxide or MgO which commonly known as the gel strength enhancer. Below are the tables of the chemical analysis done both for clay sample and reference bentonite.

*Table 4.3 Analysis of chemical elements of clay sample using XRF (%)*

| **O** | **Mg** | **Al** | **Si** | **P** | **K** | **Ca** |
|---|---|---|---|---|---|---|
| 49 | 0.35 | 13.13 | 30.83 | 0.37 | 4.48 | 0.10 |
| **Ti** | **Cr** | **Fe** | **Rb** | **Zr** | **$K_2O$** | **$V_2O_5$** |
| 0.62 | 0.01 | 0.77 | 0.02 | 0.01 | 1.25 | 0 |

*Table 4.4: Analysis of chemical compound of clay sample using XRF (%)*

| **MgO** | **$Al_2O_3$** | **$SiO_2$** | **$P_2O_5$** | **$SO_3$** | **$K_2O$** | **CaO** |
|---|---|---|---|---|---|---|
| 0.754 | 21.02 | 62.01 | 2.00 | 1.08 | 4.382 | 0.703 |
| **$TiO_2$** | **MnO** | **$Fe_2O_3$** | **ZnO** | **$Rb_2O$** | **SrO** | **$ZrO_2$** |
| 0.885 | 0.0917 | 6.984 | 0.0163 | 0.0252 | 0.0065 | 0.0432 |

*Table 4.5: Analysis of chemical compound of reference bentonite using XRF (%)*

| **MgO** | **$Al_2O_3$** | **$SiO_2$** | **$P_2O_5$** | **$SO_3$** | **$K_2O$** | **CaO** |
|---|---|---|---|---|---|---|
| 1.04 | 18.77 | 65.48 | 0.06 | 0 | 0.78 | 1.18 |
| **$TiO_2$** | **MnO** | **$Fe_2O_3$** | **ZnO** | **$Rb_2O$** | **SrO** | **$ZrO_2$** |
| 0.13 | 0.0 | 3.79 | 0.0 | 0.0 | 0.0 | 0.0 |

According to Chilingarian and Vorabtr (1983), bentonite clay used in drilling fluid is montmorillonite Chemical analysis results from tables above showed that the $Al_2O_3/SiO_2$ ratio in clay sample and also the reference bentonite were approximately 1/3 which is already expected for Montmorillonite, is the main component of bentonite. This is also proven that the clay sample is suitable for drilling fluid material based on its chemical and mineral composition. Below is the simple formula that proves the ratio of $Al_2O_3/SiO_2$ is 1/3.

$$Ratio\ for\ Al_2O_3/SiO_2 = \frac{Percentage\ of\ Al_2O_3\ (\%)}{Percentage\ of\ SiO_2\ (\%)} \qquad (3)$$

Rheological properties are affected by the clay percentages or low non clay mineral. The clay samples and the reference bentonite contained high montmorillonite, therefore it should have high rheological properties and also has good swelling capacity. Besides that, high montmorillonite usually indicates the capability of the clay to form viscous suspension at low concentrations of clay.

## 4.8 DENSITY & SPECIFIC GRAVITY

The density of the drilling fluid must be controlled to provide adequate hydrostatic head to prevent influx of formation fluids, but not so high as to cause loss of circulation or adversely affect the drilling rate and damaging the formation (Drilling Engineering Laboratary Manual, 2010).

The experiments for density and specific gravity were done on three sets of the same clay sample and also the reference bentonite. The average value will then be calculated and comparisons between both will be discussed.

*Table 4.6: Density and specific gravity for clay sample*

| Sample No | Density (ppg) | Specific Gravity |
|---|---|---|
| 1 | 8.5 | 1.05 |
| 2 | 8.8 | 1.06 |
| 3 | 8.8 | 1.02 |
| Average | 8.7 | 1.04 |

*Table 4.7: Density and specific gravity for reference bentonite*

| Sample No | Density (ppg) | Specific Gravity |
|---|---|---|
| 1 | 8.5 | 1.06 |
| 2 | 8.6 | 1.07 |
| 3 | 8.7 | 1.04 |
| Average | 8.6 | 1.06 |

Based on the results above, it can be concluded that the density and the specific gravity for both clay sample and reference are found to be similar. This showed that the weight of the clay sample is suitable and adequate to be made as drilling fluid. According to an Engineering Manual 1110- 1-1804 (2001), the desired density of drilling fluid for most drilling operations is usually less than 1080kg/m$^3$ or 9 (lb/gal or ppg).

## 4.9 MARSH FUNNEL VISCOSITY

The desired viscosity for a particular drilling operation is influenced by several factors, including mud density, hole size, pumping rate, drilling rate, pressure system and requirements and hold problem. The indicated viscosity as obtained by any instrument is valid only for that rate of shear and will differ to some degree when measured at a different rate of shear.

The values for Marsh Funnel Viscosity for both clay sample and reference bentonite are shown in the table below;

*Table 4.8.: Marsh Funnel Viscosity for clay sample and reference bentonite*

| | Marsh Funnel Viscosity (sec) | |
|---|---|---|
| Sample No. | Clay Sample | Reference Bentonite |
| 1 | 14.47 | 18.53 |
| 2 | 14.75 | 18.28 |
| 3 | 14.50 | 18.18 |
| Average | 14.57 | 18.33 |

Based on the table above, it is found that the clay sample has lower viscosity compared to the reference bentonite. This can be improved by using various thickeners to control the viscosity of the clay for example Xantham Gum, Guar Gum, Glycol and others. However, this property is not suitable for the analysis of the whole performance of the circulating system. Marsh Funnel viscosity is very important to the hole cleaning (Baroid Petroleum Services, 1984b)

### 4.10 RHEOLOGICAL PROPERTIES

Rheology refers to the deformation and flow behaviour of all forms of matter. This property is important in the design of circulating system required to accomplish certain desired objectives in drilling operation. Rheological properties include apparent viscosity, plastic viscosity, yield point and also gel strength. The values determined for these properties will be presented in the table shown below;

$$\text{Apparent Viscosity} = \frac{[600\text{r/min}]}{2}$$

$$\text{Plastic Viscosity} = [600\ \text{r/min}] - [300\text{r/min}]$$

$$\text{Yield Point} = [300\text{r/min}] - [\text{plastic viscosity}] \qquad (4)$$

*Table 4.9: Rheological properties for clay sample*

| Sample No | Viscosity(cp)31 | | | | | Gel Strength lb/100ft² | |
|---|---|---|---|---|---|---|---|
| | 600Φ | 300Φ | μp | μa | Yp lb/100 ft² | Initial at 10 sec | Final 10 Min (Gel) |
| 1 | 3.0 | 1.5 | 1.5 | 1.5 | 0 | 3.0 | 4.0 |
| 2 | 3.0 | 1.5 | 1.5 | 1.5 | 0 | 3.0 | 4.0 |
| 3 | 3.0 | 1.5 | 1.5 | 1.5 | 0 | 3.0 | 4.0 |
| Avg | 3.0 | 1.5 | 1.5 | 1.5 | 0 | 3.0 | 4.0 |

*Table 4.10: Rheological properties for reference bentonite*

| Sample No | Viscosity(cp) | | | | | Gel Strength lb/100ft² | |
|---|---|---|---|---|---|---|---|
| | 600Φ | 300Φ | μp | μa | Yp lb/100 ft² | Initial at 10 sec | Final 10 Min (Gel) |
| 1 | 14.0 | 9.0 | 5.0 | 7.0 | 4.0 | 3.0 | 15.0 |
| 2 | 14.0 | 9.0 | 5.0 | 7.0 | 4.0 | 3.0 | 15.0 |
| 3 | 14.0 | 9.0 | 5.0 | 7.0 | 4.0 | 3.0 | 15.0 |
| Avg | 14.0 | 9.0 | 5.0 | 7.0 | 4.0 | 3.0 | 15.0 |

Based on the rheological properties above, comparisons have been made between the properties of the clay sample and also the reference bentonite. It was found that ;

a) The reading for 600Φ and 300Φ for clay sample were 3.0 centipoises and 1.5 centipoise and reference bentonite were 14.0 centipoise and 9.0 centipoise.

b) The plastic viscosity of the clay sample is 1.5 centipoise whereas for reference bentonite was 5.0 centipoise

c) The apparent viscosity of the clay sample is only 1.5 centipoise whereas for reference bentonite was 7.0 centipoise.

d) The yield point for clay sample was 0 centipoise whereas for reference bentonite was 4.0 centipoise

The clay sample has very weak rheological characteristics. Due to this, the clay sample has to be added organic polymer which is commonly used to control the rheology and filtrate loss for water based drilling fluid. According to Vikas Mahto and VP. Sharma (2004a), the tamarind gum is a low cost viscosity modifier which is widely used in drilling fluid formulation. It is preferable as compared to the other visocisty modifier like guar gum as it is seven times cheaper (Khoja and Halbe, 2001)

Since tamarind gum is a carbohydrate polymer (Parija et al., 2001; Whistler and Bemiller, 1973) and it consists of hydroxyl groups which increases the apparent viscosity, plastic viscosity, yield point, gel strength. This may be due to hydrogen bonding of hydroxyl group of tamarind gum with clay.

Besides the tamarind gum, polyanionic cellulose (PAC) acts as viscosity modifier in the fresh water and salt water systems (Bruton et al., 2000). This is proven in the research of Vikas Mahto and VP. Sharma (2004b) which explained that the viscosity (apparent, plastic), yield point increases with the usage of PAC. This will be discussed thoroughly in the last chapter of the report.

### 4.11 FILTRATION LOSS

The loss of liquid or fluid loss from a mud due to filtration is controlled by the filter cake formed of the solid constituents in the drilling fluid. The experiment consists of measuring the volume of liquid forced through the mud cake into the formation drilled in a 30 minute period under given pressure and temperature.

*Table 4.11: Filtration loss for clay sample*

| Time(min) | Volume($cm^{3)}$ | Time(min) | Volume($cm^{3)}$ |
|---|---|---|---|
| 2 | 75 | 16 | 260 |
| 4 | 125 | 18 | 285 |
| 6 | 175 | 20 | 297 |
| 8 | 200 | 22 | 319 |
| 10 | 218 | 24 | 332 |
| 12 | 230 | 26 | 344 |
| 14 | 246 | | |

*Table 4.12: Filtration loss for reference bentonite*

| Time(min) | Volume($cm^{3)}$ | Time(min) | Volume($cm^{3)}$ |
|---|---|---|---|
| 2 | 4.0 | 16 | 12.0 |
| 4 | 5.5 | 18 | 12.0 |
| 6 | 6.5 | 20 | 14.0 |
| 8 | 8.0 | 22 | 14.3 |
| 10 | 9.0 | 24 | 15.0 |
| 12 | 10.0 | 26 | 15.5 |
| 14 | 11.0 | 28 | 16.0 |
| | | 30 | 16.5 |

Based on the filtration loss of both clay sample and reference bentonite above, it was observed that the clay has a very weak filtration and wall building properties. This is because they clay allows huge losses of fluid for just 26 minutes period. This is very different compared to the filtration loss of the reference bentonite which only experienced 16.5 $cm^3$ of fluid losses. Therefore, it is very crucial to correct the fluid loss control by forming a thin and tough filter cake on the walls of the hole of permeable formation. This is to prevent excessive loss of filtrate. There are chemical additives that can be added to improve this character which is by adding thinner for example lignite-based "thinners". The lignite-based "thinners" are like

a) CARBONOX®
b) CC-16™
c) K-LIG™

Mud Cake Thickness

Mud cake is the compacted material remaining after the filtration loss tests. The higher the filtration loss the thicker is the mud cake. In a real drilling activity, mud cake is formed on the walls of borehole. It is identified using vernier calliper and measured in either 32nd of an inch or cm. Below is the value shown for mud cake thickness for both clay sample and reference bentonite.

*Table 4.13: Mud Cake Thickness for clay sample and reference bentonite*

| | Mud Cake Thickness (mm) | |
|---|---|---|
| Sample No. | Clay Sample | Reference Bentonite |
| 1 | 3.59 | 2.06 |
| 2 | 3.12 | 2.53 |
| 3 | 3.30 | 2.91 |
| Average | 3.34 | 2.42 |

Based on the value obtained for mud cake thickness above, it is clearly seen that the thickness of the clay sample is much higher than the reference bentonite. This is related to the huge filtration loss experienced by clay sample which has been explained in the section above. The build up of mud cake is due to the loss of fluid to the formation. These problems will lead to a tight hole, increased torque, increased circulating pressures and can cause stuck pipe (O.A. Falode et. al, 2008b)

# CHAPTER 5

# CONCLUSIONS & RECOMMENDATIONS

Based on the research done, it can be concluded that the raw clay sample is not suitable to be made as drilling fluid due to its weakness on some properties. The weaknesses are;

a) It has low Cation Exchanged Capacity (CEC)

b) It has low viscosity; this is proven in the Marsh Funnel experiment.

c) It has very poor rheological properties like apparent viscosity, plastic viscosity, yield point and also gel strength.

d) It allows huge filtration loss, resulting a thick filter/mud cake

Based on the research, there are two ways of improving the weak characteristics of waster based drilling fluid. Those are;

- Biopolymers

Biopolymers used are polysaccharides. For example, Xanthan gum which has high properties viscosifiers with low concentration. BIOVIS is a non ionoc biopolymer produced through fermentation of carbohydrates. BIOVIS solutions exhibit exceptional shear thinning rheology and suspending abilities. The advantages of BIOVIS are higher thermal stability, pH stability and tolerance of divalent and trivalent cations. It is also will turn into gel in a static condition. BIOVIS gel has a good carrying capacity (Biovis, 2003).

Xanthan gum is a type of biopolymer that will enhance the properties of drilling fluid. It is used as a rheology control agent in aqueous systems and as a stabilizer for emulsions and suspensions.

The polyanionic cellulose (PAC) is acts as a fluid loss inhibitor for fresh water and salt water muds and also as viscosity modifier in the system. PAC is available in two types (high or low viscosity grade), both allows the same amount of fluid loss but different values of viscosities. It will not experience bacterial degradation as well.
PAC will increase the adsorption and flocculation due to hydrogen bonding between solid surfaces and the hydroxyl groups on the polymer. This will also result a tremendous improvement on the viscosity (apparent, plastic), yield point, yield point/plastic viscosity ratio and gel strength (Gungor, 2000).

As explained in the chapter 4, tamarind gum is also used in developing good and high potential drilling fluid. This material was found to be very beneficial for drilling fluid as it proven to be able in improving the properties of drilling fluid for examples, yield point, viscosity and also yield point/plastic viscosity. This may be due to the hydrogen bonding of hydroxyl group of tamarind gum with clay.

Adding the biopolymers to the drilling fluid will also reduce the formation damage during the drilling operations. . Formation damage is caused by the invasion of excessive fluid or fine solids that may contain of chemicals. It will be decreased if the filtration of drilling fluid is decreasing as well. Biopolymers are capable of reducing the fluid filtration due its substance that enables the adsorption of polymers on silica surfaces and on the edge of clay lattice (Bennion et al., 2000).

It is in fact will produce an optimum result if two types of biopolymers or organic polymers are combined with specific quantity. This was proven in the same research by V. Mahto and V. P Sharma (2004). They made comparisons of permeability of distilled water and also formulated drilling fluid. The fluid is formulated by mixing bentonite, PAC and also tamarind gum in specific amount. The formation damage is decreased probably because of less adsorption of polymer on the core due to less polymer concentration. Low pH also contributes to less formation damage as at low pH the dissolution of silica and subsequently releasing of fines inside the formation is less.

- Using additives

Additives used are to improve the properties of drilling fluids. There are many additives involved in developing the high potential drilling fluid. Examples of additives used are they clay High Mod Prima (HMP), the calcium carbonate and the potassium chloride. HMP usually used to simulate drilled solids. Calcium carbonate is used to increase the fluid density. This is important to increase the viscosity of the drilling fluid as well as to prevent from fluid loss.

Potassium ion is used in drilling fluids to aid in stabilization of shales and to control swelling clays. Inhibition is obtained with KCl in two ways. The Chloride ion ($Cl^{-}$) prevents water from entering the clay matrix by what is called "mass action". This is good to control the filtration loss as well as to prevent the formation damage due to excessive fluid loss. The potassium ion fits well in the pore space and shrinks the space where water can be drawn into the clay resulting the swelling to be decreased (GEO Drilling Fluids, Inc., 2001).

- Beneficiation process

Beneficiation process is a process to remove the impurities in the clay sample. For example, beneficiation by using oxalic acid had successfully removed its smectite content by removing the the gangue minerals like iron and free silica (Sonny Irawan and Ariffin Samsuri). Besides that, there are other few improvements resulting from this particular beneficiation process. Those are;

a) The moisture content of the beneficiated sample increased due to the free silica content
b) The moisture adsorption of the beneficiated sample is increased due to iron reduced and also the free silica content
c) The plasticity index is increased resulting from the improvement of the water adsorption capabilities
d) The CEC value of the beneficiated sample is improved indicating that the calcium ions had been replaced by sodium ions which will improve the hydration and also the swelling capability.

Based on the study d one by M.S Hassan et al. (1998), there are various improvements on the drilling properties of the bentonite clay after undergoing the beneficiation process. This beneficiation process differs from the previous one as it used hydrochloric acid instead of oxalic acid. The details of the method for this experiment are explained in a flowsheet attached in the Appendix C. This particular beneficiation process managed to improve the properties of the drilling fluid in certain aspects.

a) According to the chemical analysis of the beneficiated sample, the free silica and calcite contents have decreased significantly.
b) The swelling indices is increased after the beneficiation process.
c) The filtration loss is decreased due to the increase of smectite content
d) The plastic viscosity is increased.

Another example of the beneficiation process is obtained from the study by O.A Falode et al.(2008). This process uses sodium carbonate ($Na_2CO_3$), starch, and potash. The beneficiated clay from this process has resulted the rheological, gel and filtration characteristics that satisfy API specification.

From the experiment, the filtration loss showed a very dramatic decreased when the clay concentration and the amount of Na2CO3 present in the mud increased. Potash was found to give the most affect on the rheological properties. It also managed to increase the plastic viscosity of the drilling fluid by 750%.

Starch beneficiated drilling fluid contributed most on the filtration properties. As a result, the combination between Potash and starch will make the best beneficiating materials that will significantly increase the potential of the clay to become drilling fluids. It can be studied and enhanced further by investigating the effects of the additives at different thermodynamic conditions. The properties of the clay before and after the beneficiation process were compared and the graphs are shown in the Appendix E and Appendix F.

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# APPENDICES

# APPENDIX A

| Products | Commercial name | Company | Chemical composition | Use |
|---|---|---|---|---|
| Clay | H.M.P | | $Al_4 Si_4O_{10} (OH)_8$ | Vary the load factor |
| Biopolymere 1 scleroglucan | Biovis | Degussa (ex skw polymers) | | Viscosifier, easily biodegradable |
| Biopolymere 2 Xanthan gum | Idvis | Schlumbreger | | Viscosifier, easily biodegradable |
| Weight material | Id40 | | $CaCo_3$ | Lost circulation material, acid soluble |
| Starch | Idflo | Schlumberger | $C_6 H_{10} o_5$ | Viscosifier |
| Potassium chloride | | Panreac | KCL | Potassium salt for inhibition and density |
| Biocide/bactericide | Paraformaldéhyde | Gifrer Barbezat | $O\text{-}ch_2\text{-}$<br>$O\text{-}ch_2\text{-}$<br>$O\text{-}ch_2$ | Protect the starches and polymers against the parasite |
| Potassium hydroxide | | Panreac | KOH | Control pH to 8.5-9.0 |

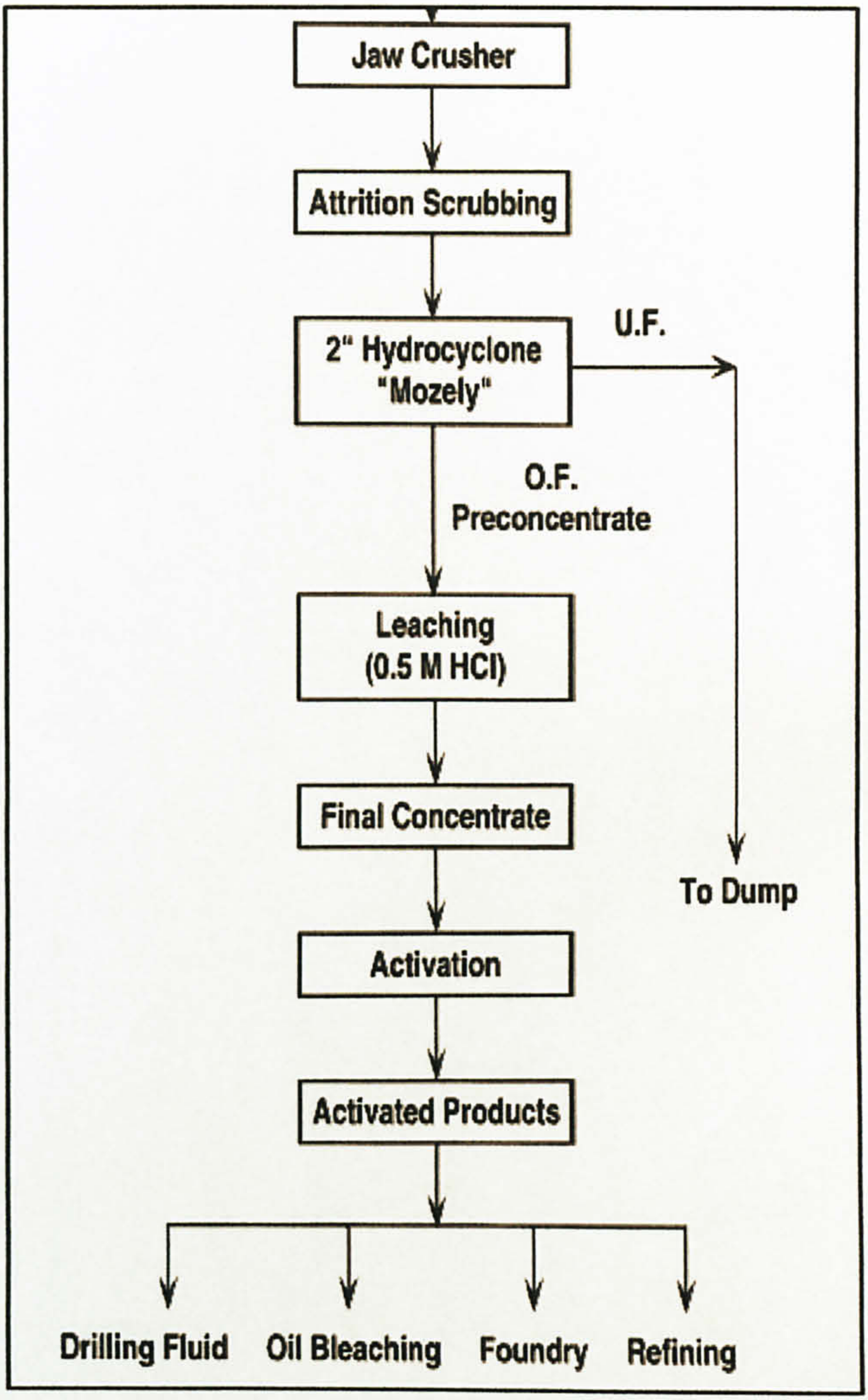
Biopolymers products and their functions (S. Baba Hamed et al., 2009)

## APPENDIX B

| Property | Influences | Desirable Limit | Control |
|---|---|---|---|
| Density (Weight) | Drilling rate<br>Hole stability | Less than about 1,080 kg/m$^3$ (9.0 lb/gal) (mud balance) | Dilute with water or remove solids to decrease<br>Add barium to increase |
| Viscosity | Cuttings transport<br>Cuttings settlement<br>Circulation pressures | 34-40 sec/dm$^3$ (32-38 sec/qt) (Marsh funnel and measuring cup) | Add water, phosphates, or lignites to thin<br>Add bentonite or polymers to thicken |
| Filtration | Wall cake thickness | Very thin (less than 0.2 cm {1/16 in.}) | Control density and viscosity of mud<br>Polymers |
| Sand content | Mud density<br>Abrasion to equipment<br>Drilling rate | Less than 2 percent by volume | Add water to lower viscosity<br>Good mud pit design<br>Use desander |
| pH (Acidity or alkalinity) | Mud properties<br>Filtration control<br>Hole stability<br>Corrosion of equipment | 8.5 to 9.5 (Neutral is 7.0) | Increase with sodium carbonate<br>Decrease with sodium bicarbonate |
| Calcium content[1] (Hard water) | Mud properties<br>Filtration control | Less than 100 parts per million (ppm) calcium | Pretreat mixing water with sodium bicarbonate |

Properties of Drilling Fluid (N.L. Baroid / N.L Industries, Inc.)

# APPENDIX C




Flowsheet for the beneficiation of Abu Zeneiman bentonite ores

(M.S. Hassan et al,1998)

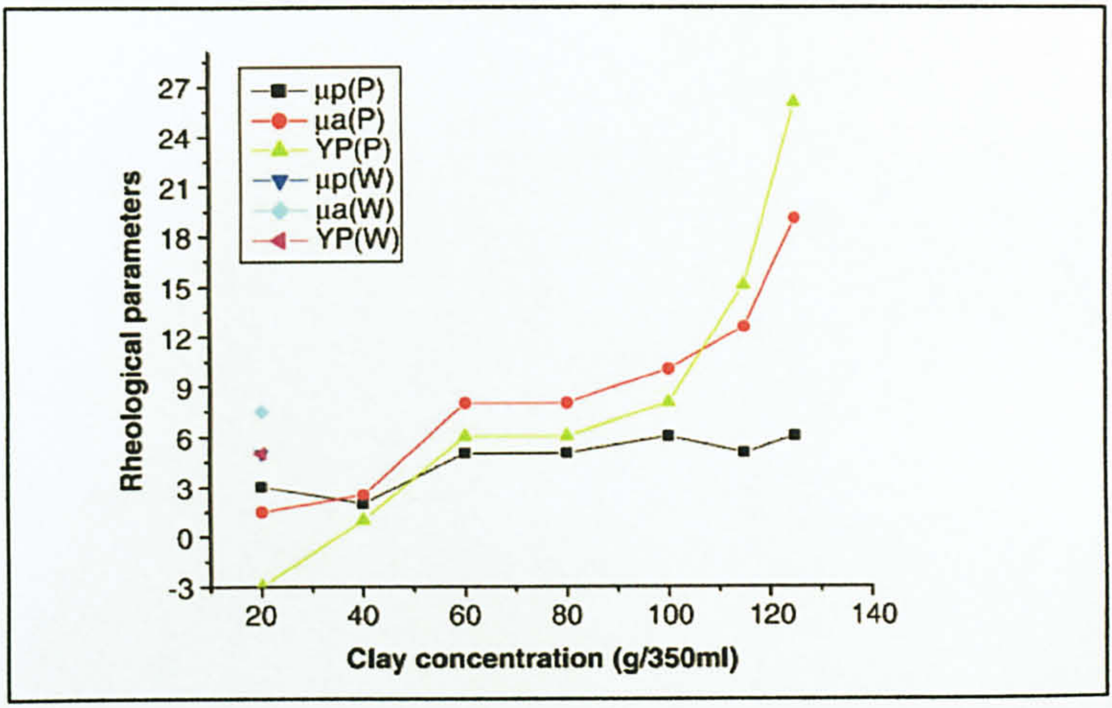
# APPENDIX D

Chemical analysis of the investigated samples

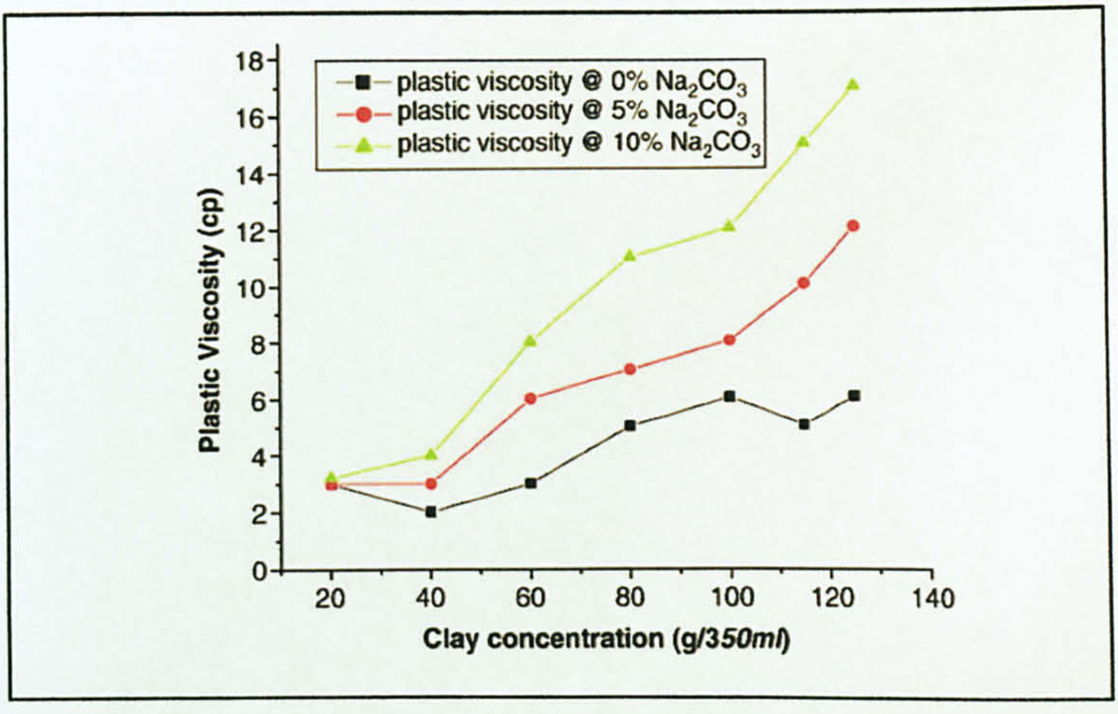
| Samples | $SiO_2$ (%) | $TiO_2$ (%) | $Al_2O_3$ (%) | $Fe_2O_3$ (%) | MgO (%) | CaO (%) | $Na_2O$ (%) | $K_2O$ (%) | LOI(%) |
|---|---|---|---|---|---|---|---|---|---|
| MJ-48 | 40.67 | 0.03 | 7.64 | 0.79 | 0.80 | 19.69 | 6.15 | 0.79 | 20.0 |
| MJ-50 | 32.20 | 0.46 | 6.60 | 3.00 | 0.80 | 22.84 | 2.33 | 0.80 | 28.2 |
| MJ-51 | 31.00 | 0.36 | 6.50 | 3.20 | 1.20 | 21.28 | 2.23 | 0.94 | 30.8 |
| MJ-52 | 34.17 | 0.41 | 5.60 | 2.80 | 1.60 | 22.96 | 1.92 | 0.94 | 27.1 |
| MJ-53 | 39.18 | 0.25 | 6.10 | 0.63 | 0.83 | 22.72 | 5.39 | 0.73 | 22.0 |
| MJ-56 | 31.86 | 0.16 | 7.24 | 0.75 | 0.79 | 36.94 | 4.27 | 0.75 | 26.0 |
| Pre-concentrate | 42.40 | 0.83 | 11.62 | 7.06 | 1.89 | 10.5 | 1.59 | 0.98 | 24.0 |
| Final concentrate | 51.01 | 1.15 | 15.40 | 8.21 | 2.44 | 1.77 | 0.35 | 0.77 | 18.0 |
| Clay fraction | 50.40 | 1.25 | 15.62 | 7.81 | 2.12 | 1.55 | 0.36 | 0.65 | 18.8 |

Chemical analysis of the clay sample after beneficiation process with hydrochloric acid and activation with $Na_2CO_3$ (M.S. Hassan et al,1998)

# APPENDIX E

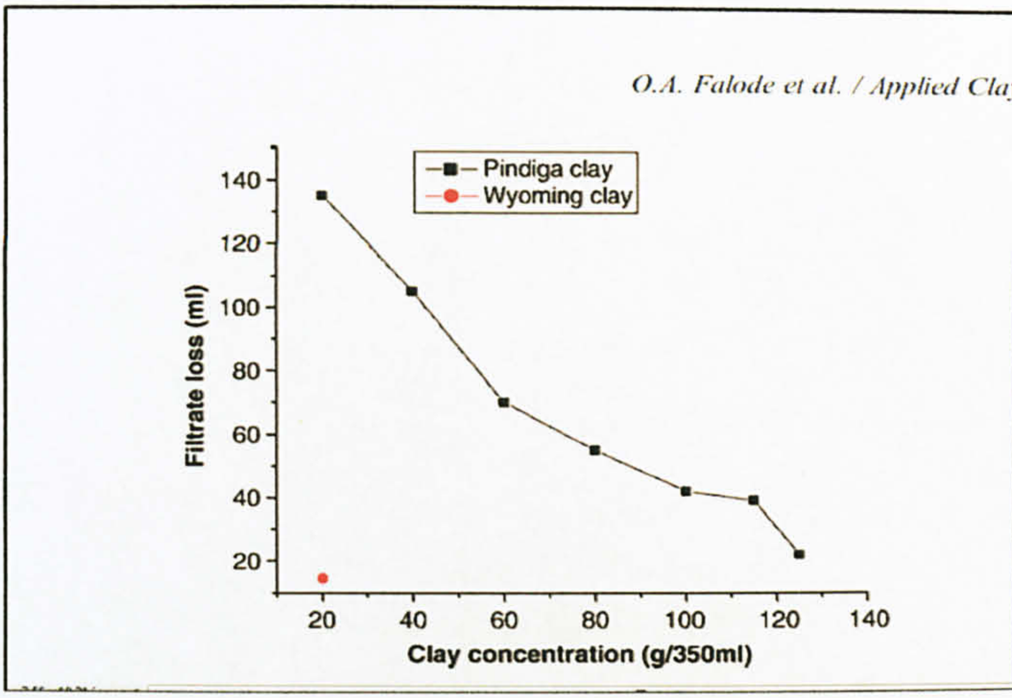



Variation of rheological parameters with clay (Pindiga – P and Wyoming– W) concentration (O.A. Falode et al., 2008)



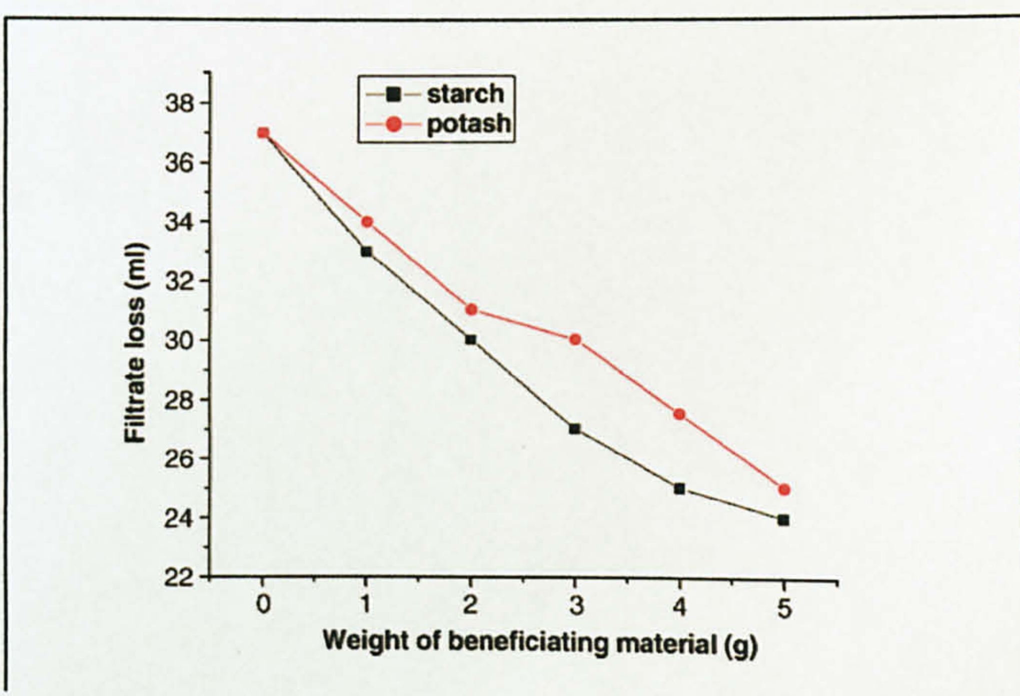

Rheological properties of the Pindiga Clay mud sample beneficiated with $Na_2CO_3$ (O.A. Falode et al., 2008)

# APPENDIX F



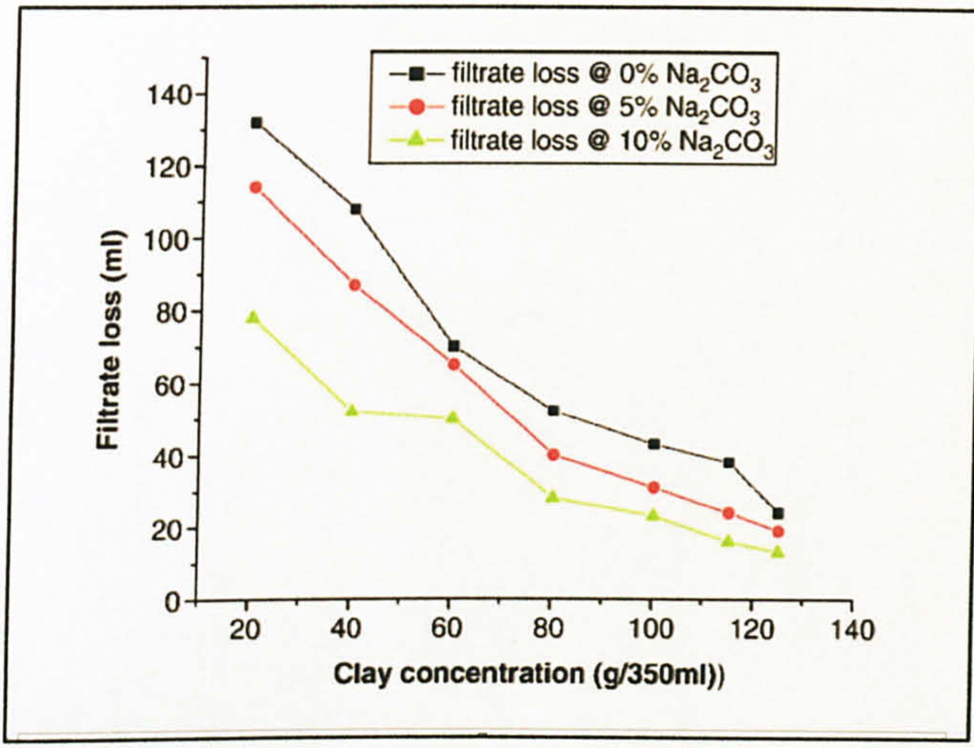

Variation of filtration loss with clay concentration

(O.A. Falode et al., 2008)




Filtration properties of the starch and potash beneficiated with Pindiga clay mud

(O.A. Falode et al., 2008)




Filtration properties of the $Na_2CO_3$ beneficiated with Pindiga clay mud

(O.A. Falode et al., 2008)