

# **Study on Ferruginous Clay as Additive in Drilling Fluid**

by

**Muhammad Faiz Bin Abdullah**

Dissertation submitted in partial fulfillment of

the requirements for the

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**(Petroleum Engineering)**

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**Universiti Teknologi PETRONAS  
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# CERTIFICATION OF APPROVAL

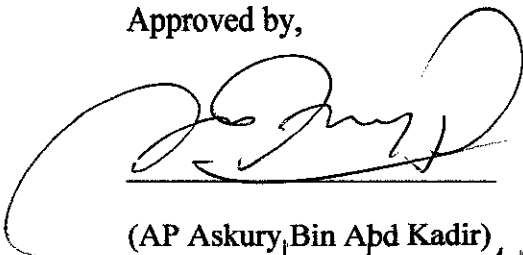
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A project dissertation submitted to the  
Petroleum Engineering Programme  
Universiti Teknologi PETRONAS  
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Bachelor of Engineering (Hons)  
(Petroleum Engineering)

Approved by,



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UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

August 2011

## **CERTIFICATION OF ORIGINALITY**

This is to certify that I, Muhammad Faiz Bin Abdullah (I/C No: 900722-11-5621), am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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## **ABSTRACT**

Drilling fluid is one of the vital factors in drilling a successful well. Any drilling fluid must have common properties that facilitate safe and satisfactory completion of the well, where the main component is clay. Clay is a widely distributed, abundant mineral resource of major industrial importance for an enormous variety of uses. The main objective of the whole project is to determine the feasibility of ferruginous clay as additive in drilling fluid. The scope of study includes investigating the ferruginous clay sample taken from Gunung Rapat area near Ipoh, Perak and evaluates its performance and behavior in drilling fluids. This project is conducted on experimental basis. Physical properties and mineral composition of the sample are determined before being tested in drilling fluid to evaluate its performance.

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## ABBREVIATIONS AND NOMENCLATURES

API	American Petroleum Institute
LI	Liquidity index
LL	Liquid limit
NAF	Non-aqueous fluid
OCMA	Oil Companies Mineral Association
PI	Plasticity index
PL	Plastic limit
PV	Plastic viscosity
SEM	Scanning electron microscope
SL	Shrinkage limit
$W_L$	Water content at liquid limit
$W_P$	Water content at plastic limit
$W_S$	Water content at shrinkage limit
WBM	Water-based mud
XRD	X-ray diffraction
YP	Yield point
$\mu$	Viscosity, cp
$\gamma$	Shear stress, dynes/cm <sup>2</sup>
$\tau$	Shear rate, sec <sup>-1</sup>

# CHAPTER 1: INTRODUCTION

## 1.1 Project Background

Drilling fluid is one of the vital factors in drilling a successful well. Any drilling fluid must have common properties which facilitate safe and satisfactory completion of the well such as bottom hole cleaning, removal of cuttings to the surface, controlling formation pressure, and maintain wellbore stability. These functions are controlled by the rheological and filtration properties of mud.

The main component of water base mud is clay (mostly bentonite). The special properties of bentonite are ability to form thixotropic gels with water, an ability to absorb large quantities of water with an accompanying increase in volume of as much as 12–15 times its dry bulk, and a high cation exchange capacity. The gelling and swelling qualities of clays impart colloidal properties to drilling mud that make them different from viscous liquids such as honey or lubricating oil (Joel & Nwokoye, 2010).

Sodium montmorillonite and calcium montmorillonite are the two major forms of bentonite. Some of the best deposits of sodium montmorillonite are found in Wyoming. Currently, bentonite deposits in Wyoming make up 70 percent of the world's known supply. Wyoming bentonite is transported to countries in every area around the world, including Malaysia. A good bentonite for a drilling fluid requires montmorillonite with sodium and calcium as the minor-cations associated with its exchange sites (Al-Homadhi, 2007). A drilling grade bentonite must readily disperse in water to produce a thixotropic or shear thinning fluid which possesses gel strength and a low fluid loss to the formation (Al-Homadhi, 2007).

However, up till date in Malaysia, most of the bentonite in use in oil field operations had been imported. In order to reduce the overall cost of bentonite used in local oil and gas industry, the feasibility study of clay in Ipoh area had been initiated. Before this clay can be used, its mineralogy must be determined as bentonite first; then their physical and chemical properties and their performance as compared with the standard bentonite must be determined.

## 1.2 Problem Statement

Clay is a widely distributed, abundant mineral resource of major industrial importance for an enormous variety of uses. In both value and amount of annual production, it is one of the leading minerals worldwide. Thus, it is important to conduct a study on clay deposits in Malaysia for potential development.

Currently in drilling fluid technology, bentonite is widely used as viscosifier to give rheological properties to the mud. By extension, the term bentonite is applied commercially to any plastic, colloidal, and swelling clay regardless of its geological origin. Such clays are ordinarily composed largely of minerals of the montmorillonite group. It is desired that the sample of ferruginous clay taken from field near Ipoh, Perak has a potential values to be developed as commercial bentonite.

Thus, the purpose of this project therefore was to undertake a comparative performance evaluation of Malaysian clay with imported bentonite so that the local bentonite could meet API specification and could be utilized for oil well operations in Malaysia. The successfully treated bentonite will be used as a material in drilling mud, which is cheaper as compared to the imported Wyoming bentonite and standard grade bentonite.

### 1.3 Objectives and Scope of Study

For this project, a ferruginous clay sample is taken from Gunung Rapat area near Ipoh, Perak to be evaluated as potential drilling fluid additive in petroleum industry. The objectives of the project are based on the evaluation and process of the testing methods, which are:

1. To determine the mineral content of a clay sample from Ipoh, Perak.
2. To evaluate the physical properties of the clay.
3. To evaluate the performance of the clay sample in water-based mud system.

The scope of study mainly investigates the ferruginous clay sample taken from Gunung Rapat area near Ipoh, Perak and evaluates its performance and behavior in drilling fluids. This experimental works can be divided into two major parts – mineral content/composition determination and engineering properties evaluation. Then, further research may be conducted to compare the performance of the clay sample as drilling fluid additive. A limited amount of tests will be prioritized; in order to fit within the time frame, hence proper research must be done beforehand. For example, engineering properties evaluation will focuses more on Atterberg limits test and limited amount of mud formulation will be prepared for further evaluation in drilling fluids. Result collected from experiments will be analyzed and discussed.



*Figure 1: Ferruginous clay sample.*

## CHAPTER 2: LITERATURE REVIEW

### 2.1 Clay

Clay is a widely distributed, abundant mineral resource of major industrial importance for an enormous variety of uses (Ampian, 1985). In both value and amount of annual production, it is one of the leading minerals worldwide. The term 'clay' refers to a naturally occurring material composed primarily of fine-grained minerals, which is generally plastic at appropriate water contents and will harden with dried or fired (Guggenheim & Martin, 1995). Clay deposits are mostly composed of clay minerals, a subtype of phyllosilicate minerals. It may contain organic materials which do not impart plasticity. Clay that bears a significant amount of iron ore (hematite,  $\text{Fe}_2\text{O}_3$  and magnetite,  $\text{Fe}_3\text{O}_4$ ) in the clay deposits is termed ferruginous clay.

Clays are distinguished from other fine-grained soils by differences in size and mineralogy. Silts, which are fine-grained soils that do not include clay minerals, tend to have larger particle sizes than clays. Geologists and soil scientists usually consider the separation to occur at a particle size of  $2\ \mu\text{m}$  (clays being finer than silts) (Guggenheim & Martin, 1995). Silts and clays also can be distinguished based on the plasticity properties of the soil, as measured by the soils' Atterberg limits.

#### 2.1.1 Bentonite

Bentonite is a rock formed of highly colloidal and plastic clays composed mainly of montmorillonite, a clay mineral of the smectite group, and is produced by in situ devitrification of volcanic ash (Parker, 1988). The transformation of ash to bentonite apparently takes place only in water (certainly seawater, probably alkaline lakes, and possibly other fresh water) during or after deposition (Patterson & Murray, 1983). In addition to montmorillonite, bentonite may also contain feldspar, biotite, kaolinite, illite, cristobalite, pyroxene, zircon, and crystalline quartz (Parkes, 1982). There are different types of bentonites, and their names depend on the dominant elements, such as potassium (K), sodium (Na), calcium (Ca), and aluminum (Al). For industrial purposes, two main classes of bentonite exist: sodium and calcium bentonite.

By extension, the term bentonite is applied commercially to any plastic, colloidal, and swelling clay regardless of its geological origin (Adamis & Williams, 2005). Such clays are ordinarily composed largely of minerals of the smectite group. Smectite is a group of clay minerals that includes montmorillonite, saponite, sauconite, beidellite, nontronite, etc., as shown in Figure 2 (Rieder, et al., 1998; Bailey, 1980) below.

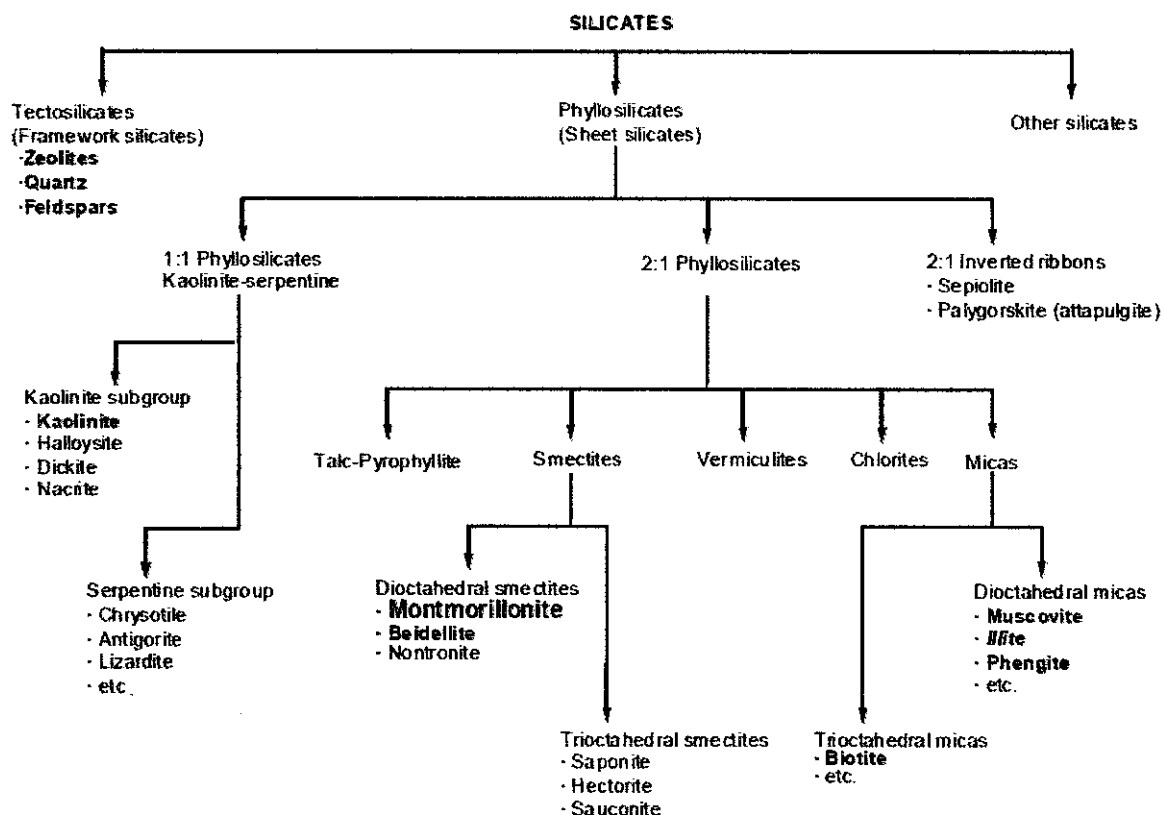


Figure 2: Classification of silicate minerals.

Freshly exposed bentonite is white to pale green or blue and, with exposure, darkens in time to yellow, red, or brown (Parker, 1988). The special properties of bentonite are an ability to form thixotropic gels with water, an ability to absorb large quantities of water with an accompanying increase in volume of as much as 12 – 15 times its dry bulk, and a high cation exchange capacity (Adamis & Williams, 2005).



### *2.1.2 Analytical Method*

There is no single or simple procedure for the positive identification of smectite group or other aluminosilicates or for their quantification in clay samples. The application of several methods may be necessary for even approximate identification and rough quantification (Adamis & Williams, 2005). These methods include X-ray diffraction (XRD), scanning electron microscopy, energy-dispersive X-ray (EDX) analysis, differential thermal analysis, and infrared spectroscopy.

X-ray powder diffraction analysis is the basic technique for clay mineral analysis (Moore & Reynolds, 1989). X-ray diffraction patterns are obtained and compared with standards for identification of minerals. Comparisons are complicated, however, by variations in diffraction patterns arising from differences in amounts of absorbed water, by the presence of imperfections in the crystal lattice structure of the minerals, and by mixed-layer structures formed by interstratification of minerals within a single particle (Grim, 1968). Approximate quantification of mineral abundance in samples containing several minerals is possible, although subject to a variety of complications and errors (Starkey, Blackmon, & Hauff, 1984).

Transmission electron microscopy is valuable for identifying aluminosilicates with a distinctive morphology (Starkey, Blackmon, & Hauff, 1984). Energy-dispersive X-ray analysis may permit the rapid identification of individual clay mineral particles (Lee, 1993). Differential thermal analysis (DTA) is based on temperature differences between the sample and a thermally inert material during heating or cooling and is most useful for mineral identification in samples composed mainly or entirely of a single clay mineral (Smykatz-Kloss, 1974).

### *2.1.3 Plasticity Properties*

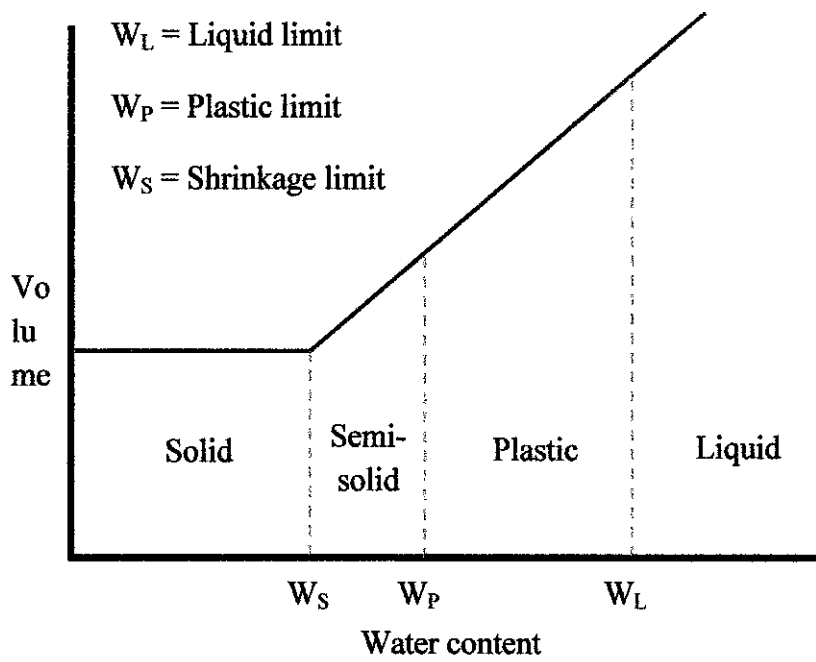
The main physical engineering properties of the clay sample that will be determined are the Atterberg limits. The Atterberg limits are a basic measure of the nature of a fine-grained soil. Depending on the water content of the soil, it may appear in four states – solid, semi-solid, plastic, and liquid. In each state the consistency and behavior of a soil is different and thus so are its engineering properties. Thus, the boundary between each state can be defined based on a change in the soil's behavior. These boundaries are referred to as the Atterberg limits which consist of:

1. Shrinkage limit (SL) – Water content where further loss of moisture will not result in any more volume reduction.
2. Plastic limit (PL) – Water content where soil transitions between brittle and plastic behavior.
3. Liquid limit (LL) – Water content at which a soil changes from plastic to liquid behavior.

The Atterberg limits are not only used to identify the soil's classification, but it allows for the use of empirical correlations for some other engineering properties, include:

1. Plasticity index (PI) – A measure of the plasticity of a soil. The plasticity index is the size of the range of water contents where the soil exhibits plastic properties. By calculations, it is the difference between the liquid limit and the plastic limit ( $PI = LL - PL$ ). Soils with a high PI tend to be clay; those with a lower PI tend to be silt.
2. Liquidity index (LI) – It is used for scaling the natural water content of a soil sample to the limits.

Further details on the relationship between the soil volume and water content of the soil can be explained by figure below:



*Figure 3: Atterberg limits and soil volume relationships.*

## 2.2 Drilling Fluid

By definition, drilling fluid is a fluid used to drill boreholes into the earth. Liquid drilling fluid is often called drilling mud. Figure 4 below shows the three main types of drilling fluids, which are water-based mud (WBM) which can be either dispersed and non-dispersed, non-aqueous fluid (NAF), usually called synthetic-based mud (SBM) or oil-based mud (OBM), and gaseous drilling fluid.

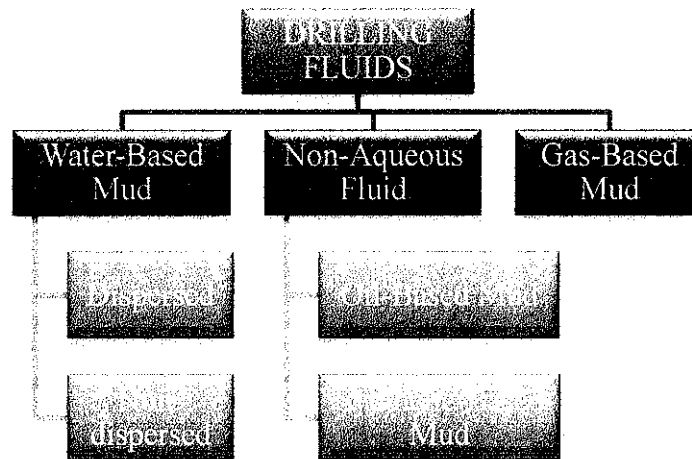


Figure 4: Classification of drilling fluids.

The difference in one drilling fluid to other drilling fluid is the chemical compositions in the formulation, as shown in Figure 5 below.

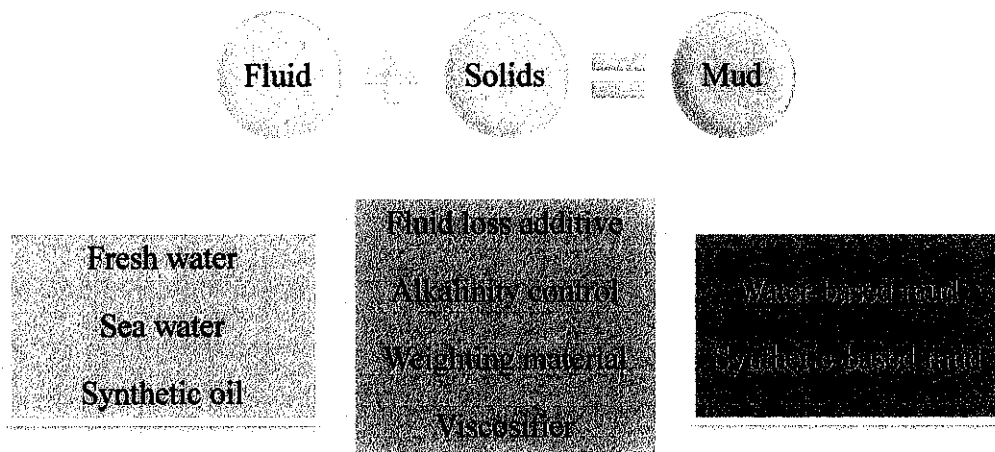


Figure 5: General composition of a drilling fluid system.

### 2.2.1 Functions of Drilling Fluid

The objective of a drilling operation is to drill, evaluate and complete a well that will produce oil and/or gas efficiently. Drilling fluids perform numerous essential functions that help make this possible. Drilling fluids are designed and formulated to perform three prime functions as tabulated in Table 1 below:

*Table 1: Primary functions of drilling fluids.*

PRIMARY FUNCTIONS	
Control formation pressure	A drilling fluid controls the subsurface pressure by its hydrostatic pressure. Hydrostatic pressure is the force exerted by a fluid column and depends on the mud density and true vertical depth (TVD).
Transport cuttings	As drilled cuttings are generated by the bit, they must be removed from the wellbore. To do so, drilling fluid is circulated down the drill string and through the bit, transporting the cuttings up the annulus to the surface. Cuttings removal is a function of cuttings size, shape and density combined with rate of penetration (ROP), drill string rotation, plus the viscosity, density and annular velocity of the drilling fluid.
Maintain stable wellbore	Borehole instability is a natural result of the unequal mechanical stresses and physico-chemical interactions and pressures created when surfaces are exposed in the process of drilling a well. The drilling fluid must overcome both the tendency for the hole to collapse from mechanical failure and/or from chemical interaction of the formation with the drilling fluid.

Secondary functions of a drilling fluid include:

1. Support weight of tubulars.
2. Cool and lubricate the bit and drill string.
3. Transmit hydraulic horsepower to bit.
4. Provide medium for wireline logging.
5. Assist in the gathering of subsurface geological data and formation evaluation.

### *2.2.2 Clay in Drilling Fluid*

Clays used in drilling fluid are:

1. Bentonite – Used for viscosity and fluid loss control for some WBM.
2. Organophilic bentonite – Used for viscosity and fluid loss control in NAF.
3. Attapulgite – Used for viscosity in salt and for viscosity in high temperature WBM.
4. Hectorite – Viscosifier in high temperature WBM.

Bentonite in the commercial market can be grouped into several grades, which are:

1. Wyoming bentonite – Pure sodium montmorillonite. This is the best grade of bentonite. Wyoming bentonite possesses unique characteristics rarely found anywhere else. It can swell up to 16 times its original size, and absorb up to 10 times its own weight in water. This is mostly due to the presence of sodium instead of calcium, more commonly found in bentonite.
2. API bentonite – Is montmorillonite that meets API standards on viscosity and filtration control. It may be (and usually is) treated with polymers/extenders (sodium polyacrylate) to attain the API grade.
3. OCMA bentonite – Calcium montmorillonite, pertaining to drilling-grade bentonite clay with API/ISO specifications. API specifications for this clay are similar to those of OCMA.

### 2.2.3 Clay Chemistry for Drilling Fluid

Clays play a major role in drilling fluid technology. Every stage of drilling a hole brings in contact with the clays. Thus, understanding of clay chemistry is essential in selection of drilling fluid system & borehole stability.

Chemically clays are aluminosilicates. Clay minerals are a part of a general group within the phyllosilicates (layered silicates). Most clays are chemically and structurally analogous to each other but contain varying amounts of water and allow varying levels of substitution in their cations. There are two basic building units from which all the different clay minerals are constructed:

1. Tetrahedral layer – In each tetrahedral unit, a silicon atom is located in the center of the tetrahedron equidistant from the four oxygen atoms.
2. Octahedral layer – In each octahedral unit, an aluminum (or magnesium) atom is located in the center of the octahedron equidistant from the six oxygen atoms.

There are more than 400 reported clay mineral names due to different combination of the basic building blocks and 26 different clay mineral groups. Each clay mineral type exhibits different characteristics and is deposited in a different environment. Montmorillonite (bentonite) clays are expandable, thus absorb water; while kaolinite, illite, and chlorite are not expandable, and thus do not absorb water.

Clay charges are important as they determine properties such as ion exchange, swelling behavior, and viscosity of muds. Charges can arise from two ways: broken edges on clay particles (induced charges) and by substitution of ions in the clay structure (permanent charges). In tetrahedral layer, some  $\text{Si}^{4+}$  can be replaced by  $\text{Al}^{3+}$  or  $\text{Fe}^{3+}$ . In octahedral layer, some  $\text{Al}^{3+}$  can be replaced by  $\text{Mg}^{2+}$  or  $\text{Fe}^{2+}$ . These substitutions produce sheets with net negative charge satisfied by adsorption of cations. Isomorphous substitution is the main reason why clays have ion exchange properties and is the reason why montmorillonite swells in water. Cation exchange capacity of clay can be measured by methylene blue test or chemical analysis of displaced cations.

#### 2.2.4 Rheology

Rheology is the science of the deformation and flow of matter. When applied to drilling fluids, rheology deals with the relationship between shear rate and shear stress. Shear rate is the change in fluid velocity divided by the gap or width of the channel through which the fluid moving in laminar flow whereas shear stress is the force per unit area required to move a fluid at a given shear rate. Viscosity is the resistance of fluid to flow or deform. In mathematical definition it is a fluid shear stress divided by corresponding shear rate.

$$\mu = \tau/\gamma$$

Plastic viscosity (PV) is resistance to flow due to mechanical friction. This friction is caused by solids concentration, size and shape of solids, and viscosity of the fluid phase. Yield point (YP) is the initial resistance to flow cause by electrochemical forces between the particles. YP is important to evaluate the ability of mud to lift cuttings out of the annulus. Using Fann 35 viscometer, these properties can be determined by:

$$\text{Plastic viscosity,} \quad PV = R_{600} - R_{300}$$

$$\text{Yield point,} \quad YP = R_{300} - PV$$

where  $R_{600}$  is the viscometer dial reading at 600 rpm;

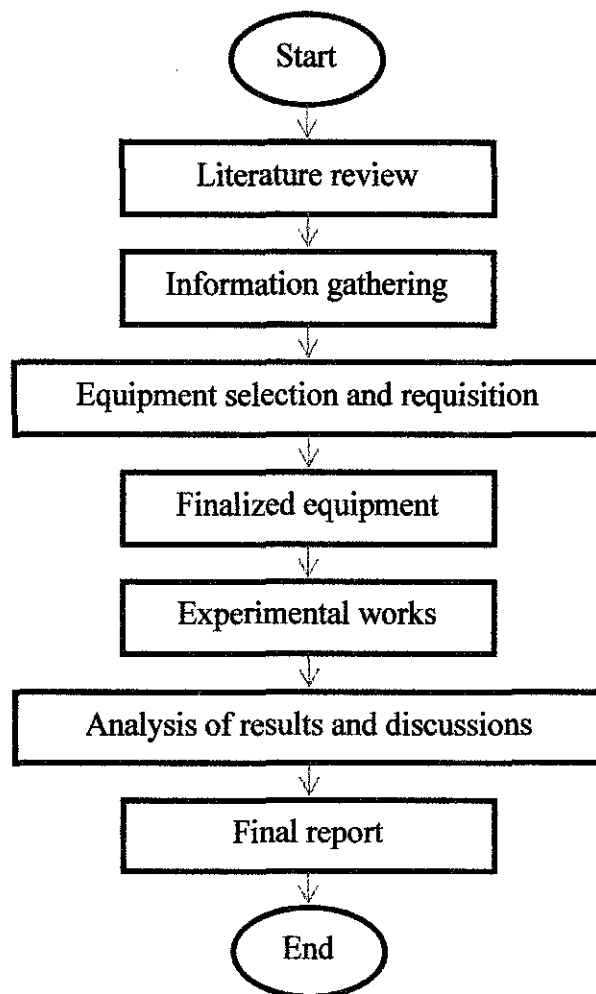
$R_{300}$  is the viscometer dial reading at 300 rpm.



## CHAPTER 3: METHODOLOGY

### 3.1 Research Methodology

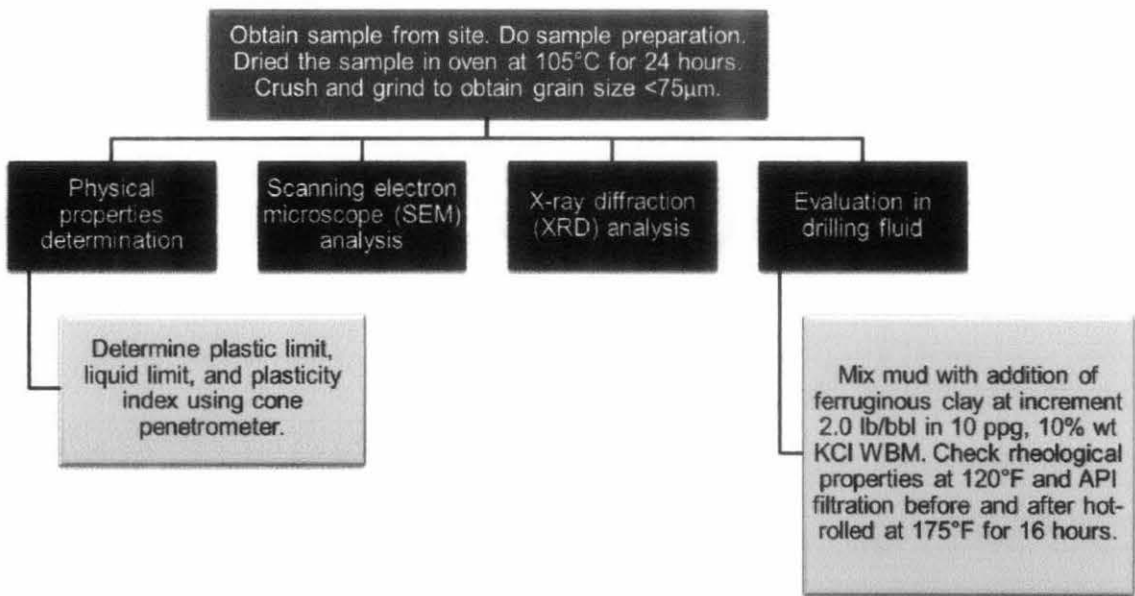
In completing the project, student plays an important role as an investigator/researcher; doing all the literature study and look for his/her own approach to work on the topic. Thus, assistance and supervision from the assigned supervisor is essential to ensure the student is on the right path and follow the schedule. This could be done through a good communication medium such as weekly meeting, progress report and consultations. Progress report shall be submitted according to the schedule so that any corrective measure can be taken and indirectly both student and supervisor will have good and up-to-date information.



*Figure 6: Research methodology.*

### 3.2 Project Activities

This project is conducted on experimental basis. The sample will be taken from the site and in situ properties and data will be collected, which consists of color and texture of the sample, nature of clay formation and clay deposits. Further experimental works that will be done include, sample preparation, clay identification, physical properties determination, chemical properties determination, and evaluation of performance as drilling fluid additive. The general view of project activities are as shown in figure below.



*Figure 7: Project activities.*

#### 3.2.1 Sample Preparation

Bulk samples of raw ferruginous clay were obtained from Gunung Rapat area near Ipoh, Perak. Clay sample from the field will be dried in the oven for 24 hours at 105°C to remove the moisture content of the clay. The sample then will be crushed using mortar grinder till it become powder. Selection of grain size is very important to obtain maximum cation exchange. API Specification 13-A states that the particle size to be used should less than 74µm, which can be achieved by sieving.

### *3.2.2 Clay Identification*

The X-ray diffraction (XRD) and the scanning electron microscope (SEM) methods are considered as the most reliable methods to identify clay minerals. They are easy and fast identification methods. The result of the X-ray method is an oscillating curve with several high peaks. The peaks are the identity of the tested clay. Each clay type can give a certain distinctive one or more peaks that are used to distinguish it from other clay types. The SEM result will show an image of the clay particle at certain magnification scale. For XRD, the mineralogical constituents within the clay sample can be directly characterized.

### *3.2.3 Physical Properties Determination*

These properties including Atterberg limits, moisture content (MC), and moisture absorption (MA). The physical study values are used to obtain information on the nature and quality of the mineral by using Atterberg limits test, such as plastic limit (PL), liquid limit (LL), and plasticity index (PI). The standard testing methods for liquid and plastic limit are according to BS 1337: Part 2: 4.3/4.4.

The plastic limit was simply the moisture content at which a ball of clay when rolled to a diameter of 3 mm. Liquid limit is determined using cone penetrometer method. On the other hand, plasticity index is the difference between liquid limit and plastic limit.

### *3.2.4 Performance as Drilling Fluid Material*

The clay sample undergoes a series of testing based on API (American Petroleum Institute) specification 13-A, and OCMA (Oil Companies Materials Association) specification. For easier purposes, the clay sample will be tested in a basic water-based mud system. First, the clay will be evaluated using testing procedures for bentonite to see its performance as drilling grade bentonite. Comparison will be made between the clay sample and the standard bentonite.

The basis of bentonite testing is to see the suspension properties of the sample. 22.5g of clay is added to 350 cm<sup>3</sup> (1 lab barrel) of deionized water while stirring for 20 minutes. The suspension is then aged up to 16 hours in a sealed container at room temperature before all the properties are evaluated.

Plastic viscosity,  $PV = R_{600} - R_{300}$

Yield point,  $YP = R_{300} - PV$

where  $R_{600}$  is the viscometer dial reading at 600 rpm

$R_{300}$  is the viscometer dial reading at 300 rpm

Filtrate volume,  $V$  is given by  $V = 2V_C$  where  $V_C$  is the filtrate volume collected between 7.5 min to 30 min.

Then, the clay sample will be evaluated in 1.2SG, 10% wt KCl water-based mud system to see the effects of the clay addition into the mud on certain properties like rheology and filtration control. A base mud will be prepared to compare the performance of clay when added into the mud system. The amount of clay addition is set at an increment of 2.0 lb/bbl for each formulation. The formulations for the base mud are as follow:

*Table 2: Mud formulation.*

Products	Concentration (lb/bbl)	Functions
Distilled water	313.74	Base fluid
Soda ash	0.20	Water hardness reducer
Potassium chloride	36.48	Water activity and density
Hydrated bentonite	5.00	Viscosifier
Poly-anionic cellulose	2.00	Filtration control
Non-fermenting starch	6.00	Filtration control
API barite	56.65	Weighting agent
Caustic soda	0.10	pH control
Ferruginous clay		

3.3 Gantt Chart

Table 3: Gantt chart for FYP 1.

Activities	Final Year Project I (FYP-1)													
	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Gather information regarding clay, drilling fluid and testing procedures			■	■	■	■	■							
In situ data collection								■	■	■				
Sample preparation and mineralogy determination using SEM and XRD											■	■	■	■
Physical properties evaluation using Atterberg limits test														
Data collection and interpretation														
Comparison against the standard material as drilling fluid additive														

Table 4: Gantt chart for FYP 2.

Activities	Final Year Project II (FYP-2)													
	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Gather information regarding clay, drilling fluid and testing procedures														
In situ data collection														
Sample preparation and mineralogy determination using SEM and XRD	■													
Physical properties evaluation using Atterberg limits test		■	■	■	■									
Data collection and interpretation						■	■							
Comparison against the standard material as drilling fluid additive								■	■	■	■	■		

### 3.4 Key Milestones

Table 5: Key milestones for project activities.

Milestone	Final Year Project II (FYP-2)													
	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Completion of physical properties tests														
Completion of mineralogy analysis														
Completion of sample evaluation as drilling fluid material														

### 3.5 Tools Required

Table 6: Tools required for each activity.

Activity	Equipment
Sample preparation	1. Mortar grinder 2. Oven 3. 75 $\mu$ m, 425 $\mu$ m sieve
Clay identification	4. X-ray diffraction (XRD) 5. Scanning electron microscope (SEM)
Physical properties determination	6. Cone penetrometer
Drilling fluid testing	7. Mixer 8. Mud balance 9. Fann 35 viscometer with heating cup 10. API filter press 11. Aging cells 12. Roller oven

## CHAPTER 4: RESULTS AND DISCUSSION

### 4.1 Determination of Plasticity Index

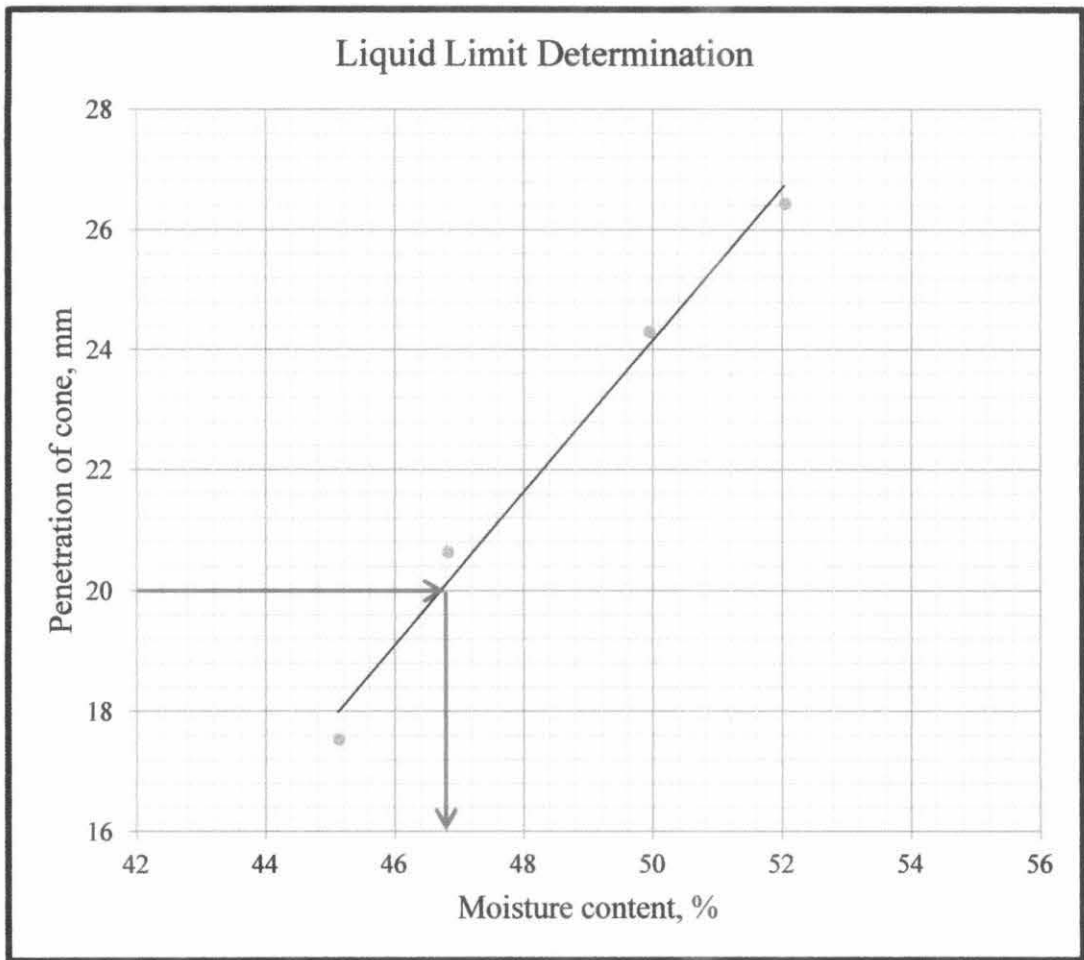
Plastic limit, liquid limit (cone penetrometer method), and plasticity index of ferruginous clay are determined according to BS 1337: Part 2. The plastic limit is the empirically established moisture content at which a soil becomes too dry to be plastic. The liquid limit is the empirically established moisture content at which a soil passes from the liquid state to the plastic state. It is used together with the plastic limit to determine the plasticity index which when plotted against the liquid limit on the plasticity chart provides a means of classifying cohesive soils. The results are as follow:

*Table 7: Plastic limit results.*

<b>Plastic Limit</b>	<b>Test no.</b>	<b>1</b>	<b>2</b>	<b>3</b>	<b>4</b>	<b>Average</b>
Container no.		1	2	3	4	
Mass of wet soil + container	g	36.34	24.79	23.58	23.79	
Mass of dry soil + container	g	34.40	23.20	22.40	22.40	
Mass of container	g	29.47	18.87	18.96	19.67	
Mass of moisture	g	1.94	1.59	1.18	1.39	
Mass of dry soil	g	4.93	4.33	3.44	3.73	
Moisture content	%	39.35	36.72	34.30	37.27	<b>36.91</b>

*Table 8: Liquid limit results (cone penetrometer method).*

<b>Liquid Limit</b>	<b>Test no.</b>	<b>1</b>	<b>2</b>	<b>3</b>	<b>4</b>
Average penetration	mm	17.53	20.63	24.30	26.43
Container no.		1	2	3	4
Mass of wet soil + container	g	48.91	43.32	42.94	51.36
Mass of dry soil + container	g	39.50	35.50	34.80	40.30
Mass of container	g	18.65	18.80	18.50	19.05
Mass of moisture	g	9.41	7.82	8.14	11.06
Mass of dry soil	g	20.85	16.70	16.30	21.25
Moisture content	%	45.13	46.87	49.94	52.05



*Figure 8: Relationship between moisture content and cone penetration.*

For liquid limit determination using cone penetrometer method, the liquid limit of soil is defined as the moisture content corresponding to a cone penetration of 20mm. Since one cannot get the exact cone penetration of 20mm, a plot of several values of cone penetration against its respective moisture content with a best straight line fitting the plotted points is used to determine the moisture content at 20mm penetration of cone. From the plotted graph above, the liquid limit is **46.8**. Plasticity index may be calculated as:

$$PI = LL - PL = 46.8 - 36.9 = 9.9$$



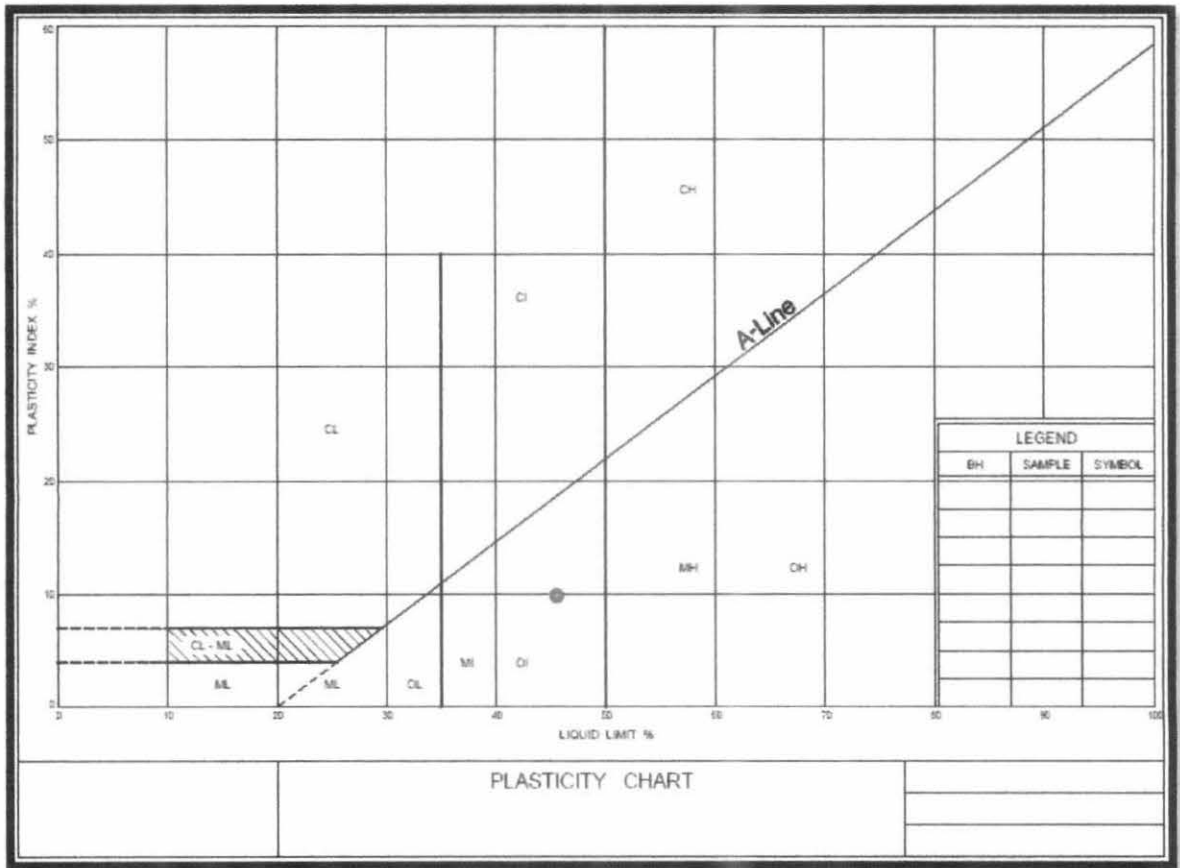


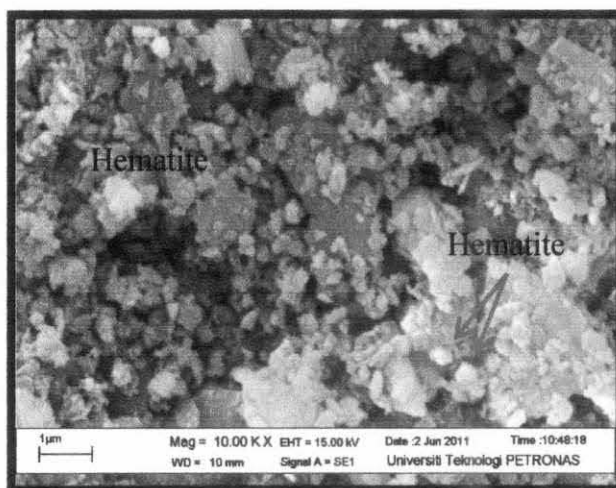
Figure 9: Plasticity chart.

A plot of plasticity index against the liquid limit on the plasticity chart provides a means of classifying fine soils. A clay sample will lie above the A-line whereas a plot below the A-line will indicate silt. From the graph above, the red dot is the plot of the sample, which indicates that the sample has intermediate plasticity. Since the plot lies below the A-line, the sample can be classified as silt, most probably due to high content of non-swelling minerals such as hematite.

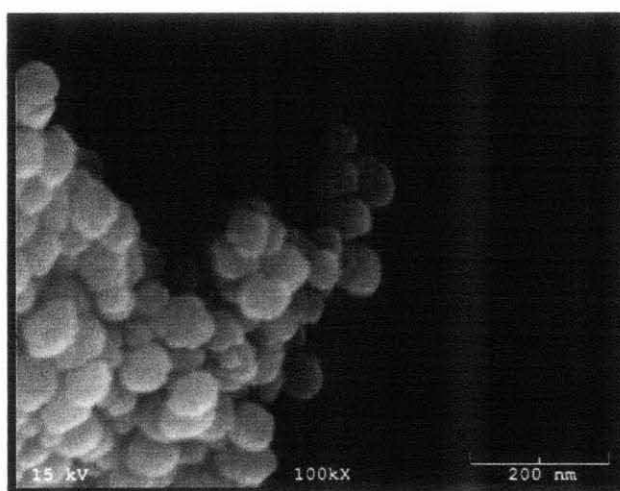
## 4.2 Clay Identification

### 4.2.1 Scanning Electron Microscopy

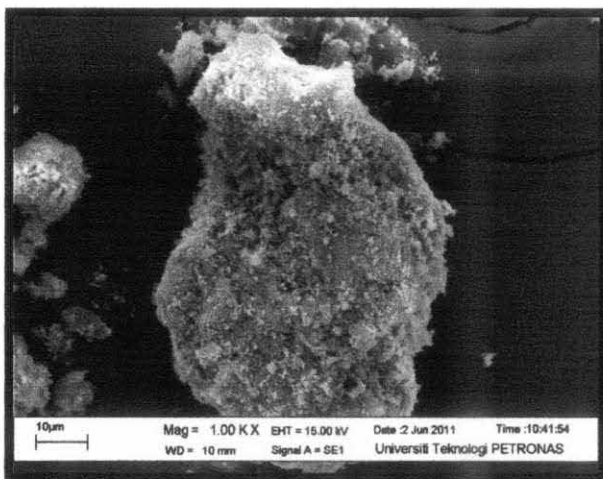
From the images taken from the microscope, the sample seems to have hematite as minor mineral constituents by comparing to the image of aggregated hematite nanoparticles as shown in figures below, which proves that the clay is ferruginous. No other description can be determined by the images due to insufficient information.



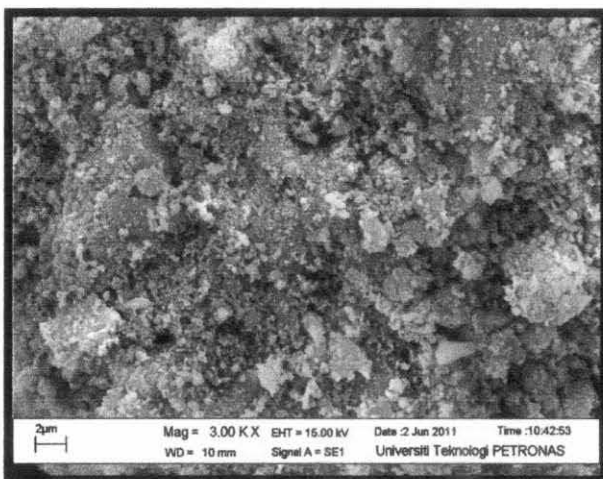
*Figure 10: Ferruginous clay (magnification 10000X).*



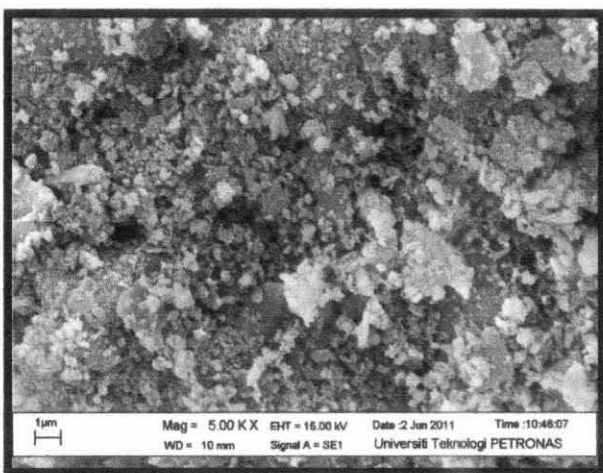
*Figure 11: Aggregated hematite nanoparticles (magnification 100000X).*



*Figure 12: Ferruginous clay (magnification 1000X).*



*Figure 13: Ferruginous clay (magnification 3000X).*



*Figure 14: Ferruginous clay (magnification 5000X).*

## 4.2.2 X-Ray Diffraction (XRD) Analysis

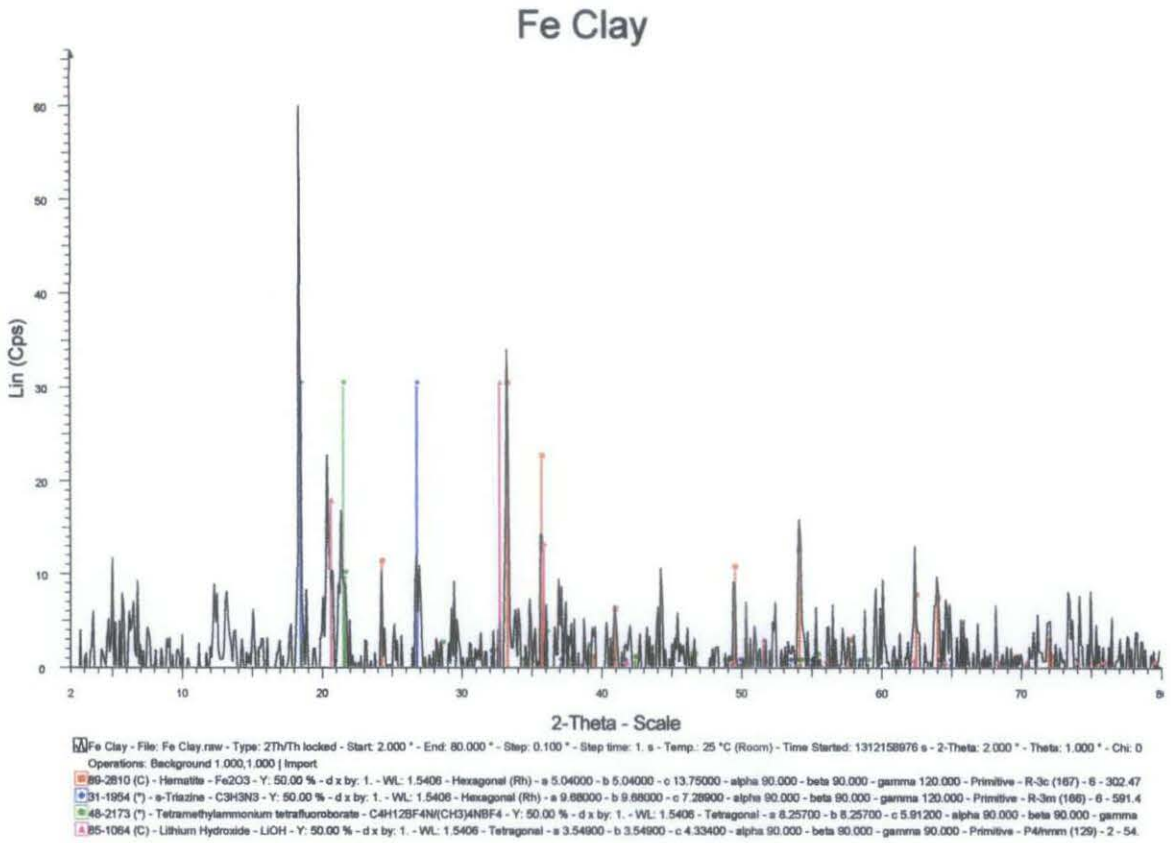


Figure 15: X-ray diffraction (XRD) analysis.

From the graph, it can be concluded that the sample were made of four major minerals which are hematite, C<sub>3</sub>H<sub>3</sub>N<sub>3</sub>, (CH<sub>3</sub>)<sub>4</sub>NBF<sub>4</sub>, and lithium hydroxide.

### 4.3 Performance as Drilling Fluid Material

#### 4.3.1 Evaluation as Bentonite

Preliminary evaluation on ferruginous clay as bentonite in drilling fluid is conducted. Drilling grade bentonite shall be deemed to meet the requirements of API and OCMA standard.

*Table 9: Suspension properties of ferruginous clay as compared to standard bentonite.*

Test Parameter	API 13-A Specification	OCMA Specification	Standard Bentonite	Ferruginous Clay
Viscometer dial reading at 600 rpm	minimum 30	minimum 30	32	5
Yield point/plastic viscosity ratio	maximum 3	maximum 6	1.75	0.5
Filtrate volume, cm <sup>3</sup>	maximum 15	maximum 16	14	>30

The results shown in Table 9 indicate that the suspension properties of the clay sample almost failed to meet the requirements that had been set by API and OCMA for drilling grade bentonite. From physical indication, there are almost no gelling effects for the drilling mud prepared using untreated clay. Also, fluid loss control capability of the ferruginous clay is very poor and failed to meet the standard requirement.



### 4.3.2 Evaluation in Drilling Fluid

Table 10: Raw data of mud evaluation.

Product		Base	A	B	C	D	E
Fresh water		313.74	313.5	313.3	313.0	312.8	312.5
Soda ash		0.20	0.2	0.2	0.2	0.2	0.2
KCl (10%wt, 96% purity)		36.48	36.5	36.4	36.4	36.4	36.3
HYDRO-STAR NF		6.00	6.0	6.0	6.0	6.0	6.0
HYDRO-PAC R		2.00	2.0	2.0	2.0	2.0	2.0
Bentonite		5.00	5.0	5.0	5.0	5.0	5.0
Ferruginous clay		0.00	2.0	4.0	6.0	8.0	10.0
Caustic soda		0.10	0.1	0.1	0.1	0.1	0.1
API barite		56.65	54.9	53.2	51.5	49.7	48.0
<b>Initial results</b>	<b>Spec</b>	<b>10 ppg</b>	<b>10 ppg</b>	<b>10 ppg</b>	<b>10 ppg</b>	<b>10 ppg</b>	<b>10 ppg</b>
Rheology at		120°F	120°F	120°F	120°F	120°F	120°F
600 rpm		71	74	71	74	76	79
300 rpm		47	50	49	51	53	56
200 rpm		37	40	38	40	41	42
100 rpm		24	25	24	26	27	29
6 rpm		11	11	12	11	10	11
3 rpm		9	10	11	9	9	10
PV, cP		24	24	22	23	23	23
YP, lb/100ft <sup>2</sup>	10 - 20	23	26	27	28	30	33
10s gel strength, lb/100ft <sup>2</sup>		9	10	11	10	9	10
10min gel strength, lb/100ft <sup>2</sup>		12	13	13	13	12	13
API filtrate, cc		3.5	3.3	3.4	3.5	3.4	3.5
Mud cake thickness, mm							
pH	9.5-10						
<b>AHR results</b>		175°F	175°F	175°F	175°F	175°F	175°F
Rheology at		120°F	120°F	120°F	120°F	120°F	120°F
600 rpm		61	61	60	63	62	64
300 rpm		40	41	40	42	41	42
200 rpm		30	31	31	32	31	31
100 rpm		21	21	21	21	21	20
6 rpm		8	8	7	8	8	8
3 rpm		6	6	6	7	7	7
PV, cP		21	20	20	21	21	22
YP, lb/100ft <sup>2</sup>	10 - 20	19	21	20	21	20	20
10s gel strength, lb/100ft <sup>2</sup>		6	6	6	7	7	7
10min gel strength, lb/100ft <sup>2</sup>		7	8	7	8	8	8
API filtrate, cc		4.8	4.8	4.9	5.0	5.0	4.8
Mud cake thickness, mm							
pH	9.5-10						

Further evaluation is done to see the effects of the clay to the performance of the mud. The clay sample will be added in 1.2SG, 10% wt KCl water-based mud system to see the effects of the clay addition into the mud on certain properties like rheology and filtration control. A base mud will be prepared to compare the performance of clay when added into the mud system.

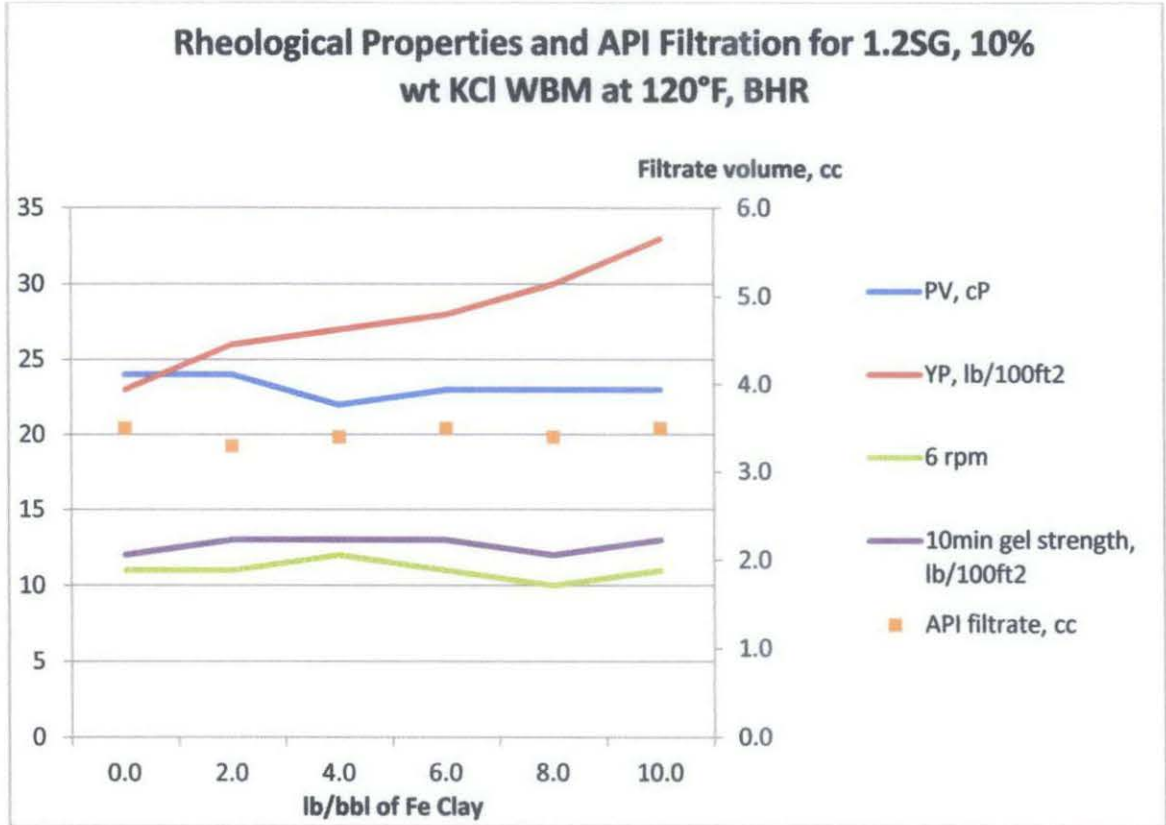
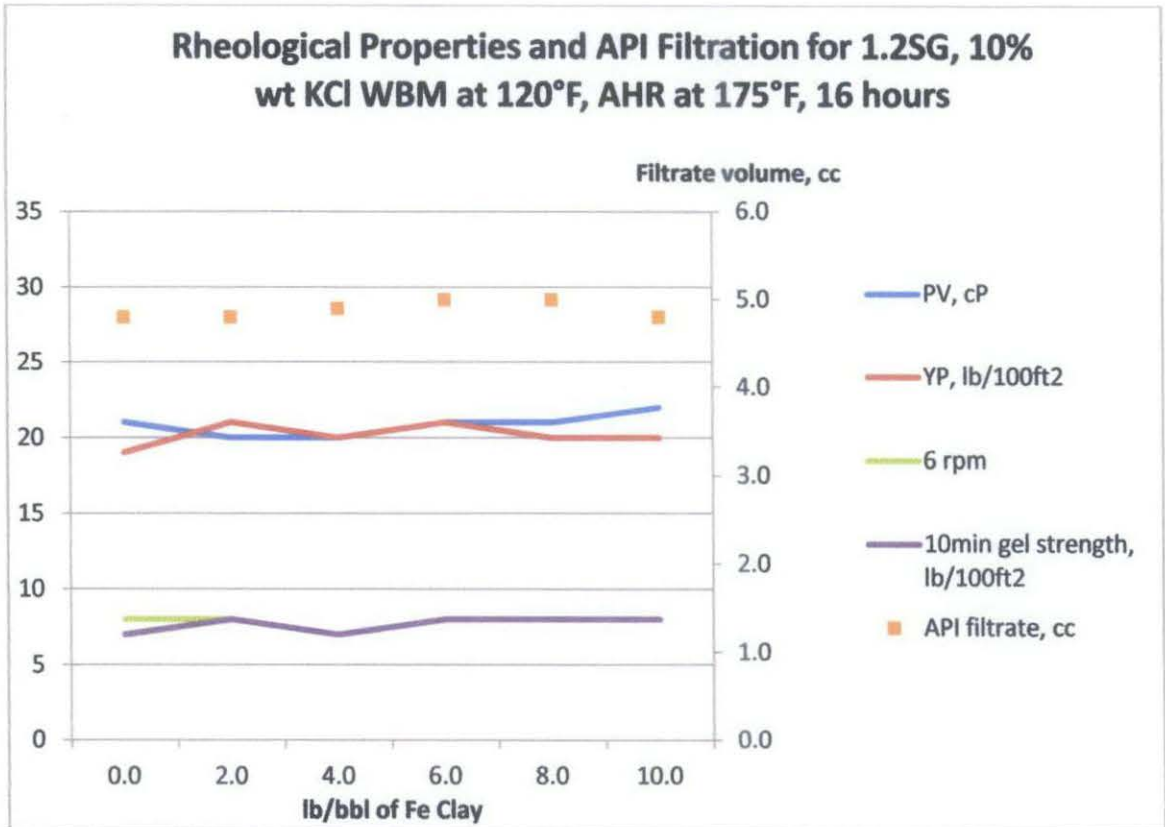


Figure 16: Rheological properties and API filtration for 1.2SG, 10% wt KCl WBM at 120°F, before hot-rolled.

From Figure 16, as the amount of ferruginous clay used increases by 2.0 lb/bbl, the YP value increases from 23 to 33. Meanwhile, the value for PV seems to be constant. Thus, the addition of ferruginous clay increases the YP value without increasing the PV value at initial mixing before hot-rolled. Other rheological properties (6 rpm and 10' gel strength) as well as the filtrate volume do not give any evident changes as the amount of ferruginous clay increases.



*Figure 17: Rheological properties and API filtration for 1.2SG, 10% wt KCl WBM at 120°F, after hot-rolled at 175°F, 16 hours.*

From Figure 17, it can be seen that all rheological properties (PV, YP, 6 rpm, and 10' gel strength) do not show any significant changes as the amount of ferruginous clay used increases by 2.0 lb/bbl. The same thing happens to API filtration, where the volume of filtrate does not change much as the amount of ferruginous clay increased. By comparing to Figure 16, it may be noticed that the rheological properties decrease after hot-rolled at 175°F for 16 hours, while the filtrate volume increases after hot-rolled.



## CHAPTER 5: CONCLUSION AND RECOMMENDATION

### 5.1 Conclusion

1. The clay sample can be classified into silt of intermediate plasticity (35-50). So, the clay has a moderate plasticity.
2. From both SEM images and XRD analysis, hematite is one of the major constituents of the clay, proving that the clay is ferruginous.
3. For clay performance in drilling fluid, yield point (YP) value before hot-rolled increases with increasing amount of ferruginous clay used. Other rheological properties and API filtration did not show any trend. All properties after hot-rolled at 175°F for 16 hours did not show any changes with increasing amount of ferruginous clay. From here, it can be concluded that small amount of ferruginous clay give a significant increase in yield point (YP) value at initial mixing without increasing the plastic viscosity (PV) value even though it has a moderate swelling capacity. Thus, it can be used as alternative clay to change certain rheological properties before hot-rolled and successfully tested as potential drilling fluid additive.

### 5.2 Recommendation

The suggestion for future work to expand and continue this study is by conducting more evaluations on clay deposits in other part of Malaysia. It is cost effective to utilize the local clay rocks, though the cost for processing the local modified samples may appear to be higher. The comparative advantage of local content realization and enhancement of home-based technology should outweigh cost consideration. The cost of importing drilling fluid additives could be uneconomical if one could exploit the local resources within Malaysia itself. Overall, this study is relevant to meet the major objective which is to use local clay for drilling purposes, which subsequently reduces the operating cost.

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