

**The Suitability of Barite from East Coast of Peninsular
Malaysia as Weighting Agent in Drilling Fluid**

by

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

JANUARY 2010

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
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Universiti Teknologi PETRONAS
in partial fulfilment of the requirement for the
BACHELOR OF ENGINEERING (Hons)
(CHEMICAL ENGINEERING)

Approved by,

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May 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.

HO CHING SHEARN

ABSTRACT

A drilling fluid, or mud, is any fluid that is used in a rotary drilling operation in which that fluid is circulated or pumped from the surface, down the drill string, through the bit, and back to the surface via the annulus.

Drilling fluid is crucial for a successful drilling program. From functioning to transport the rock cuttings to surface, its applications have become more diverse nowadays as the drilling operations are becoming more and more complex. One physical property of a drilling fluid critical to carry out many of the desired functions is its density.

The density of drilling fluid is adjusted to balance or somewhat to overbalance pore pressures encountered in the course of drilling. Imparting positive pressure into the formation is of paramount importance in order to prevent inflow of formation fluid. Moreover, the pressure of mud column is important to stabilize the uncased section, particularly in the unconsolidated formation.

Among the additives that are designed to give certain properties to drilling fluid, a weighting agent is used to increase the density of drilling fluids while minimizing the increase in the resistance to flow of the drilling fluid. Several minerals have been identified to be potentially used as weighting agents including barite, hematite, dolomite and illmenite. However, a combination of factors has made barite to be the most preferred choice compared to others. A drilling grade barite normally includes at least 85% by weight particles less than 75 microns and greater than 6 microns in equivalent spherical diameter^[6]. (Nattier & Shumate, 2003)

Naturally occurring barite contains 50-70 percent BaSO₄. The remaining impurities are quartz and various silicates that may include dispersible clays. In order to be used as drilling-grade barite, the barite ore must meet the physical and chemical requirements as dictated in API 13-A: Barite physical and chemical requirements.^[3]

ACKNOWLEDGEMENT

I would like to extend my gratitude to the coordinators of the Final Year Project from the Chemical Engineering Department, namely Mr Mohd Tazli, Assoc Prof Dr Thanabalan Murugesan, Dr Khalik b Mohd Sabil, and Dr. Mohanad M.A.A El-Harbawi for their dedication and efforts in guiding me into better understanding about this research-based course.

I would also like to thank my supervisor, Ap Dr Chow Weng Sum for his guidance, advices, assistance and support throughout the project. He had suggested me to take on this project and has since been very kind to help in various ways.

Besides that, I am also grateful to the lab technician, Cik Imma who had allowed me to carry out my laboratory testing in the geo-technical laboratory. She had been very sincere and helpful.

My appreciation is also extended to the family members and fellow friends who had given moral support to motivate me to pursue to greater heights in our project.

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CHAPTER 1.

INTRODUCTION

1.1. Background of Study

Nowadays, drilling fluid has very diverse applications in rotary drilling operations, among which including to cool and lubricate the drill bits, to transport the rock cutting from downhole to surface, to counter the downhole pressure and the likes.

A critical property differentiating the effectiveness of various wellbore fluids in achieving these functions is density, or mass per unit volume. The wellbore fluid must have sufficient density in order to carry the cuttings to the surface. Density also contributes to the stability of the borehole by increasing the pressure exerted by the wellbore fluid onto the surface of the formation downhole. The column of fluid in the borehole exerts hydrostatic pressure (also known as pressure head) proportional to the depth of the hole and the density of the fluid. Therefore, one can stabilize the borehole and prevent the undesirable inflow of reservoir fluids by carefully monitoring the density of the wellbore fluid to ensure that an adequate amount of hydrostatic pressure is maintained.

In this respect, the governing parameter of drilling fluid is its density, which is controlled by the addition of weighting agent. Several minerals have been used by the industry to raise the density of drilling fluid, but barite is the most preferred among all.

1.2. Problem Statement

The use of barite (Barium Sulfate, BaSO_4) as weighting agent in drilling fluid is thus far the most widespread compared to any other minerals such as hematite (Fe_2O_3), illmentite ($\text{FeO}\cdot\text{TiO}_2$), siderite (FeCO_3), dolomite (CaCO_3 MgCO_3), calcite (CaCO_3), galena (PbS), and etc. The reason is because that barite is highly dense and chemically inert in addition to its availability in abundance, relatively non-abrasiveness and moderate cost.

However, naturally occurring barite is always contaminated by some common accessory minerals: (1) silicates, such as quartz and chert; (2) carbonate compounds, such as siderite and dolomite; (3) metallic oxide and sulfide compounds. Although these minerals are normally insoluble, they can, under certain conditions, react with other components in some types of drilling fluids and cause adverse changes in the drilling fluid properties as well as producing undesirable performance in some drilling fluids. The density of barite may be reduced due to presence of impurities. Moreover, the impurities can influence the particle size distribution to which barite ores can be ground. The American Petroleum Institute (API) has issued international standards to which ground barite must comply for use as drilling grade barite under API Specification 13A, Section 2.

Barite is a dense mineral comprising barium sulfate (BaSO_4) that commonly associated with a range of accessory minerals, such as quartz, chert, dolomite, calcite, siderite and metal sulfides. Being used as a weighting agent for all types of drilling fluids, barites are mined in many areas worldwide and shipped as ore to grinding plants in strategic locations. Pure barium sulfate has a specific gravity of 4.50 g/cm^3 , but drilling-grade barite is expected to have a specific gravity of at least 4.20 g/cm^3 in accordance to API specifications. Contaminants in barite, such as cement, siderite, pyrrhotite, gypsum, anhydrite and more remarkably soluble calcium, can cause problems in certain mud systems and should be evaluated in any quality assurance program for drilling-mud additives.

1.3. Objectives and Scopes of Study

The objective of this final year project is to evaluate and to ascertain the suitability of ground barites from East Coast of Peninsular Malaysia as weighting agent in drilling fluid.

On top of that, it is also aimed to compare the properties of the Malaysian barites with those of China and Cambodia barites as these two countries are supplying drilling grade barite to a mineral company in Terengganu in light of barite shortage in Malaysia.

The scope of study mainly focuses on the laboratory tests which look into the properties of the barite samples and subsequently compare the results with the specifications with regard to the barite physical and chemical requirements as dictated by API 13A, Section 2.

CHAPTER 2. LITERATURE REVIEW

2.1. Principal Functions of Drilling Fluid

Success in drilling a hole through the earth's crust depends significantly on the performance of the fluid being circulated down the rotating drill pipe, through the bit, and up the annular space between the pipe and the formation or steel casing, to the surface in a continuous loop. Historically, the first purpose of drilling fluid was to serve as a vehicle for removal of cuttings from borehole, but now it serves diverse applications.

Drilling fluids are necessary for the followings:

- Transport cuttings from the wellbore to surface.
- Deliver hydraulic energy to keep the area under the bit clean and therefore the bit will not regrind the old cuttings which can cause reduced rate of penetration (ROP).
- Control the subsurface pressure (pore pressure) to prevent kick which is as a result of involuntary influx of pressurized formation fluids – oil, gas or water into the well. Under extreme circumstances, a kick may cause a catastrophic blowout;
- Suspend the cuttings when circulation stops. The solids start to settle leading to problems like bridging off of the wellbore, stuck pipe, hole fill, loss of hydrostatic pressure. A measure of mud's ability to suspend cuttings in the hole when circulation stops is called gel strength.
- Control torque (reduce friction) between the drilling string and the sides of the hole;
- Deposit a thin and low-permeability filter cake which seals the pores and other openings in formation penetrated by the bits to prevent lost circulation or leakoff which can cause formation damage in form of reduced formation permeability;
- Cool and lubricate the drill string and bit

- Assist in formation evaluation by facilitating the collection and interpretation of information available from drill cuttings, cores and electrical logs

The following side effects should be minimized when using drilling fluid to accomplish the above functions: (1) formation damage, especially those that may be productive; (2) casing and drill string corrosion; (3) penetration rate reduction; (3) circulation, surge, and swabbing problems (suction); (4) lost circulation; (5) drill string stuck; (6) well bore erosion; (7) mud pump wear; (8) cement and environmental contamination.

2.2. Pore Pressure

Pore pressure is the pressure exerted by the fluids in the pores of the formation. It is a function of the depth of the porous medium, the density of the formation fluids, and the density of the overlying rock, whether the rock is self-supporting or is supported by the fluid, tectonic activity and surface terrain.

There are two types of geological conditions affecting pore pressure:

- *Normally pressure formation*, which have a self-supporting structure of solid particles and thus the pore pressure depends only on the weight of the overlying pore fluid;
- *Abnormally pressure or geopressured formations*, which are not fully compacted into a self-supporting structure and thus the pore fluid must bear the weight of some or all of the overlying sediments as well as the weight of the overlying fluids. The hydrostatic pressure gradient of formation fluids varies from 0.43 psi/ft to over 0.52psi/ft, depending on the salinity of the water.

2.3. Formation Pressure Control

Control of influx of fluid from pressurized subsurface formations is an important aspect of any drilling operation. If uncontrolled, fluid influx can lead to a blowout and fire, frequently with catastrophic results in terms of loss of life, damage to properties, and pollution of the seaway.

Hydrostatic pressure is the force a stagnant fluid exerts on the sides and bottom of a container. The column of drilling fluid in the hole exerts a hydrostatic pressure which increases with the true vertical depth of the hole and the density of the fluid. This can be expressed with the following formulas:

$$\text{Hydrostatic Pressure (psi)} = \text{Depth (ft)} \times \text{Fluid Density (ppg)} \times 0.052$$

$$\text{Hydrostatic Pressure (kPa)} = \text{Depth (m)} \times \text{Fluid Density (s.g)} \times 9.7983$$

$$\text{Hydrostatic Pressure (kg / cm}^2\text{)} = \text{Depth (m)} \times \text{Fluid Density (s.g)} / 10$$

This pressure is used to control the flow of formation fluid into the wellbore and to prevent lost circulation, hole instability, and stuck pipe. If the pressure exerted by the mud column in the wellbore is insufficient, the formation fluids (liquid or gas) will flow toward the wellbore. In the extreme case, failure to maintain adequate fluid density can lead to a catastrophic, uncontrolled inflow of gas or oil known as “blow-out” A blow-out is an uncontrolled escape of drilling fluid, gas, oil, or water from a well, caused by the formation pressure being greater than the hydrostatic head of the fluid in the hole.

If the formation is mechanically stressed, as a result of exerting pressure greater than the hydrostatic pressure, the walls of the wellbore will tend to collapse and result in borehole instability. On the other hand, if the hydrostatic pressure is greater than the pore pressure, lost circulation and/or stuck pipe may occur. Also, if the hydrostatic pressure exceeds the fracture pressure of the formation, an induced fracture can result in similar problems of stuck pipe and lost circulation.

2.4. Density of Drilling Fluid

Being required to perform a wide range of functions, drilling fluid inevitably has complex formulation which is specific to different geologic situations. There are four dominant properties of drilling fluids that should be controlled to address the necessary functions in the various geological situations: (1) density; (2) viscosity (rheological properties); (3) fluid loss (filtration); (4) inhibition.

Of the aforementioned physical properties, density of drilling fluid is particularly critical in carrying out many of the desired functions. The density and depth of a column of drilling fluid determine the pressure exerted by the drilling fluid on the surrounding formation. In practice, the density of a drilling fluid is carefully controlled to exert sufficient downhole pressure to stabilize the uncased section of borehole and prevent the inflow of oil and gas from the formations that are spud.

Hereinafter, the focus will be on the density property of drilling fluid which plays important roles to: (1) to control the pore pressure to prevent inflow of formation fluid; (2) to lay down a thin, low-permeability filter cake on the wall of the hole; (3) to maintain hole stability, especially in the case of plastic formations like rock salt and unconsolidated formation.

By convention, density is called the mud weight, which is measured in pounds per gallon (lbm/gal, or ppg), grams per cubic centimeter (specific gravity [sg]), or, occasionally, pounds per cubic foot (lbm/ft^3), pounds per square inch per thousand feet ($\text{lbm}/\text{in.}^2/1000 \text{ ft}$), or kilograms per cubic meter (kg/m^3).

When drilling, the downhole pressure varies for different depth and different geologic conditions. Correcting the density of the drilling fluid plays a key role in balancing formation pressures. The rule of thumb is to maintain the mud weight high enough to control formation fluids, but not so high as to induce fracture which causes formation damage in terms of reduced permeability. Hence, the mud weight must be below the fracture point of the weakest formation, but above the highest pore pressure observed.

WS Training (2003) points out that for determining appropriate mud weight, the equivalent circulating density (ECD) must be taken into account to avoid fracturing the formation ^[4]. ECD is defined as the sum of hydrostatic pressure resulting from the column of mud (and cuttings) in the annulus, plus the pressure drop in the annulus during circulating. ECD is calculated by adding the mud weight to the pressure loss in the annulus and dividing by the true vertical depth multiplied by 0.052. Mathematically, ECD is given as the following.

$$ECD = Mud\ Weight\ (ppg) + \left[\frac{Annular\ Pressure\ Loss\ (psi)}{0.052 \times True\ Vertical\ Depth\ (feet)} \right]$$

Pressure loss in the annulus is due to the frictional forces on the mud as it moves up the annulus and is dependent on the velocity, density, flow properties, and geometry of the hole. There should be at least 0.5 lbm/gal (0.0599 g/cm³) equivalent mud weight between the equivalent circulating density and fracture pressure. Between the pore pressure and ECD, a 0.2- to 0.5-ppg equivalent differential pressure should be kept as a safety margin. However, the differential pressure should be kept as low as possible to improve the rate of penetration whereby the lower the differential pressure, the faster the penetration. In fact, there are some situations where a negative differential is used to drill, such as in hard formations, some reservoirs, or lost circulation zones. This technique of drilling is called underbalanced drilling (UDB).

Excessive drilling fluid density has several setbacks including: (1) if the mud weight is too high, there may be an increased chance of exceeding the fracture gradient, which can cause lost circulation; (2) stuck pipe may occur due to differential pressure sticking, which means the force that holds pipe against the wall of the hole, due to the differential between formation pressure and hydrostatic pressure; (3) decreased rate of penetration (ROP); and (4) unnecessarily increase the mud costs.

2.5. Characteristics of Barite

Barite (Barium Sulfate, BaSO_4) composes of 58.84% Barium, 13.74% Sulfur, and 27.42% Oxygen. It is the primary ore of barium. The Mohs hardness of barite is 2.5 – 3.5.

Barite is a relatively inert mineral with a high density. It is virtually insoluble in water, and does not react with other components of the mud. Calcium sulfate, sometimes present as gypsum or anhydrite associated with barite, is objectionable as a contaminant of lightly-treated, fresh water mud. Sulfide minerals, such as pyrite and sphalerite, if present, may undergo oxidation with the formation of soluble salts that adversely affect the mud performance.

Barite most often occurs in hydrothermal veins and as veins in limestone. Commercial deposits of barite occur as vein or cavity-filling deposits, residue deposits, and bedded deposits.

2.6. Barite as Weighting Agent in Drilling Fluid

In many drilling situations, it is necessary to increase the density of the fluid by adding so-called “weighting materials”. Any substance that is denser than water and that does not adversely affect other properties of the mud can be used as a weighting agent.

Weighting materials are various inert, high-density particulate solid materials with a normal particle size smaller than 75 microns and preferably smaller than 50 microns. Finely ground barite, hematite and ilmenite are some examples of known weighting materials ^[6]. (Nattier & Shummate, 2003)

According to Darley and Gray (1988) ^[1]

Several factors in addition to chemical inertness and specific gravity affect the use of a substance as a weighting material. First, the substance should be available in large

quantities. It should be easily ground to the preferred particle-size distribution, and relatively nonabrasive. It should also be moderate in cost, and not injurious or objectionable to the drilling crew or the surroundings. Consideration of these factors, along with chemical inertness and specific gravity, has made barite the only mineral now used in significant quantities in the United States to raise the density of mud. (p.545)

Barite is the only material which is used in substantial quantities to increase the density of mud. Consequently, the commercial product has been tested extensively for many years in an effort to define suitable specifications. (p.134)

Pure barium sulfate has a specific gravity of 4.50 g/cm^3 , but drilling-grade barite is expected to have a specific gravity of at least 4.20 g/cm^3 to meet API specifications. Because of mineral impurities, commercial barite may vary in colour from off-white to grey to red or brown. Common accessory minerals are silicates such as quartz and chert, carbonate compounds such as siderite and dolomite, and metallic oxide and sulfide compounds. Although these minerals are normally insoluble, they can, under certain conditions, react with other components in some types of drilling fluids and cause adverse changes in the drilling fluid properties. These contaminants can cause problems in certain mud systems and should be evaluated in any quality assurance program for drilling-mud additives.

The presence of certain accessory materials in a barite can produce undesirable performance in some drilling fluids. These accessory materials include naturally occurring minerals such as gypsum (calcium sulfate dihydrate, $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$), siderite (iron carbonate, FeCO_3), dolomite (calcium magnesium carbonate, $\text{CaMg}(\text{CO}_3)_2$) and pyrrhotite (iron sulfide). Gypsum is somewhat soluble in water and releases ions of calcium, one of the alkaline earth metals. Other minerals such as siderite, dolomite, and pyrrhotite, though not soluble in water to a significant degree, can release their anions in the presence of hydroxyl ions. Carbonate ions are released from siderite and dolomite in the presence of hydroxyl ions, and sulfide ions are released from pyrrhotite at high temperature in combination with high pH.

Barites are mined in many areas worldwide and shipped as ore to grinding plants in strategic locations, where API specifies grinding to a particle size of 3 to 74 microns.

According to API Specification 13A, Section 2 (Specifications for Drilling-Fluid Materials) published in February 2004, for use as drilling-grade barite, the naturally occurring barite should conform to four physical and chemical requirements as given in Table 2.1. ^[3]

Table 2.1: Barite physical and chemical requirements

Requirement	Standard
Density	4.20 g/cm ³ , minimum
Water-soluble alkaline earth metals, as calcium	250 mg/kg, maximum
Residue greater than 75 µm	maximum mass fraction 3.0 %
Particles less than 6 µm in equivalent spherical diameter	maximum mass fraction 30 %

*Courtesy API Specification 13A / ISO 13500:1998

Table 2.2: Materials used to increase the density of drilling fluids

Material	Principal component	Specific Gravity	Hardness, Moh's Scale
Galena	Pbs	7.4 - 7.7	2.5 - 2.7
Hematite	Fe ₂ O ₃	4.9 - 5.3	5.5 - 6.5
Magnetite	Fe ₃ O ₄	5.0 - 5.2	5.5 - 6.5
Iron Oxide	Fe ₂ O ₃	4.7	
Illmenite	FeO.TiO ₂	4.5 - 5.1	5.0 - 6.0
Barite	BaSO ₄	4.2 - 4.5	2.5 - 3.5
Siderite	FeCO ₃	3.7 - 3.9	3.5 - 4.0
Celestite	SrSO ₄	3.7 - 3.9	3.0 - 3.5
Dolomite	CaCO ₃ MgCO ₃	2.8 - 2.9	3.5 - 4
Calcite	CaCO ₃	2.6 - 2.8	3

Note: Devised by German mineralogist Friedrich Mohs, the Mohs Scale grades minerals on a comparative scale from 1 (very soft) to 10 (very hard) ^[9]. The scale states that a mineral of a given hardness rating will scratch other minerals of the same rating, as well as any minerals of a lower hardness rating.

2.7. Effect of Size of Weighting Agent Particles on Drilling Fluid

Particle size and density determine the mass of the weighting agents, which in turn correlates to the degree of sag. Sag is sediment arises from the settling out of particles (weighting agents and cuttings) from the wellbore fluid. It can lead to a multitude of well-related problems such as lost circulation, loss of well control, stuck pipe, and poor cement jobs. It is influenced by a variety of factors related to operational practices or drilling fluid conditions such as: low-shear condition, drillstring rotation, time, well design, drilling fluid formulation and properties, and the mass of weighting agent.

Massam, Browne, & Kapilla (2005) states that although it follows that lighter and finer particles will sag less in theory, conventional view is that reducing weighting agent particle size causes an undesirable increase in the fluid's viscosity, particularly its plastic viscosity ^[5]. Plastic viscosity is generally perceived as a measure of the internal resistance to fluid flow that may be attributable to the amount, type or size of the solids present in a given fluid. It has been theorized that this increase in plastic viscosity attributable to the reduction in particle size, and thereby increasing the total particle surface area due to a corresponding increase in the volume of fluids, such as water or drilling fluid, absorbed to the particle surfaces. Thus, particle sizes below 10 μ m have been disfavored.

Because of the mass of the weighting agent, various additives are often incorporated to produce a rheology sufficient to allow the wellbore fluid to suspend the material without settlement or "sag" under either dynamic or static conditions. Such additives may include a gelling agent, such as bentonite for water-based fluid or organically modified bentonite for oil-based fluid. A balance exists between adding a sufficient amount of gelling agent to increase the suspension of the fluid without also increasing the viscosity resulting in reduced pumpability. One may also add a soluble viscosifier such as xanthan gum to slow the rate of sedimentation of the weighting agent.

2.8. Density and Flow Property of Drilling Fluid

Nattier & Shumate (2003) discussed in their paper that as a result of large quantities of weight materials which are often added to drilling fluids, the impact of a weight material on the flow properties (such as viscosity) of a drilling fluid is a major concern ^[6]. It is necessary that the required amount of weight agent can be added with a minimum adverse increase in the resistance to flow of the fluid. There is also an upper limit to the amount of weight material that can be added to a drilling fluid before it becomes too thick to be of any practical use.

Various approaches have been devised to provide the desired density with a minimum thickening of the drilling fluid. One obvious approach has been to utilize weight materials of increasingly higher specific gravity to reduce the quantity of weight materials needed to achieve a given drilling fluid density. Another method is disclosed in U.S Pat. No. 5,007,480 for using a manganomanganic oxide (Mn_3O_4) particulate material having a particle size of at least 98% below 10 microns in combination with conventional weight materials such as barite to obtain a drilling mud with a higher density than what is possible using barite alone. Moreover, chemical dispersants are also used to thin the drilling fluid.

2.9. Effect of Water-soluble Alkaline Earth Metals (Ca^{2+}) Content on Drilling Fluid

Asides from flow rate, a fairly high viscosity is necessary to clean the hole. When the circulation is stopped, the rock cuttings fall back down the hole. Some problems may arise from this phenomenon including bridging off of the wellbore, stuck pipe, hole fill and loss of hydrostatic pressure.

Ideally, the viscosity should be high enough to assist in cuttings removal by carrying the cuttings to the surface and to support weighting materials such as barite. Appropriate gel strength should suspend weighting agents, such as barite, but avoid significantly increasing wellbore pressure when breaking circulation. Gel strength is a measure of a fluid's ability to hold particles in suspension.

Bentonite, an additive commonly used for viscosity and fluid loss control in some water-based muds, is sensitive to water contamination. The pH, hardness, and salt content of the make up water determine the yield and performance of the bentonite clay. Hard water, due to the presence of dissolved calcium salts, can result in unsatisfactory performance of bentonite clay mud. Calcium salts impair the suspending and sealing properties of bentonite clay. Soda ash is used to treat hard water usually 1 to 2 pounds of soda ash will be sufficient treat for both high pH and hardness.

On other note, the efficiency of bentonite is also affected by the pH range, whereby low pH and extremely high pH water will adversely affect the bentonite clay performance. The pH of the make up water should be within the alkaline range. On the pH scale, the acidity range is from less than 1 to just below 7. The alkalinity range is just above 7 to 14. A pH of 7 is considered neutral. The pH desired for mixing bentonite clay is between 8.5 and 10 on the pH scale. A pH of 9 is considered perfect. If the pH of the make up water is low, it will affect the hydration of the bentonite clay. The make up water should be treated with soda ash, Sodium Carbonate (NaCO_3), to raise pH. If the pH of the make up water exceeds 11 pH, flocculation of the bentonite clays can occur.

Furthermore, according to Amoco Production Company (1994), the calcium ion is a major contaminant of water-based drilling fluids ^[12]. Calcium contamination drastically changes the nature of freshwater in clay-based systems. The calcium ion tends to replace sodium ions on the clay surface through a base exchange. The bound layer of water on the clay platelets is reduced, resulting in diminished hydration or swelling characteristics. The effects of calcium contamination on deflocculated mud are increased fluid loss, yield point and gel strengths.

CHAPTER 3. METHODOLOGY

Four experiments are to be carried out. The results from each of the experiments are to be compared with respective requirements as given in Table 1.

The four experiments are:

- Particle density
- Water-soluble alkaline earth metals as calcium
- Residue of diameter greater than 75 μm
- Particles less than 6 μm in equivalent spherical diameter.

Note: The reagents and apparatus, procedures as well as calculation necessary for carrying out each of the experiments are given in Appendices.

Five samples of barite ores from five different locations had been obtained for carrying out the quality assurance testing.

The barite ores obtained were firstly grinded into powder form. The processes for crushing the mineral ores are discussed in a book by Geological Survey Malaysia (1981) ^[13]. Rocks are first broken into tiny pieces with a hammer so that they can be accommodated by the jaw crusher. The samples must be put through the jaw crusher three times before they are finally crushed to minus 80 mesh in the plate crusher. Between samples, the jaw crusher and the plates must be cleaned by blowing air into them and wiping to prevent contamination. Finally, the minus 80 mesh material is crushed to minus 150 to minus 200 mesh in an agate mortar crusher.

The locations from where the samples are collected include:

- Ulu Sok, Kelantan
- Tasik Chini, Pahang
- Sungai Tregan, Terengganu
- Guangdong, China
- Cambodia

CHAPTER 4. RESULTS AND DISCUSSION

4.1. Results of Particle Density Experiment

Table 4.1: Raw and calculated data of particle density experiment for all the barite samples

	Source of barite ores				
	Terengganu	Pahang	China	Cambodia	Kelantan
Mass of jar+gas jar+plate, m1 (g)	538.00	537.34	538.24	535.96	532.36
Mass of jar+gas jar+plate+barite, m2 (g)	941.32	933.93	937.76	935.55	932.51
Mass of jar+gas jar+plate+barite+water, m3 (g)	1869.81	1852.29	1873.04	1860.82	1864.03
Mass of jar+gas jar+plate+water, m4 (g)	1558.02	1555.62	1566.54	1556.27	1556.16
Mass of barite = m2-m1 (g)	403.32	396.59	399.52	399.59	400.15
Mass of water in full jar = m4-m1 (g)	1020.02	1018.28	1028.30	1020.31	1023.80
Mass of water used = m3-m2 (g)	928.49	918.36	935.28	925.27	931.52
Volume of barite (m4-m1)-(m3-m2) (ML)	91.53	99.92	93.02	95.04	92.28
Density (Mg/m ³)	4.406	3.969	4.295	4.204	4.336
Density (g/cm ³)	4.406	3.969	4.295	4.204	4.336

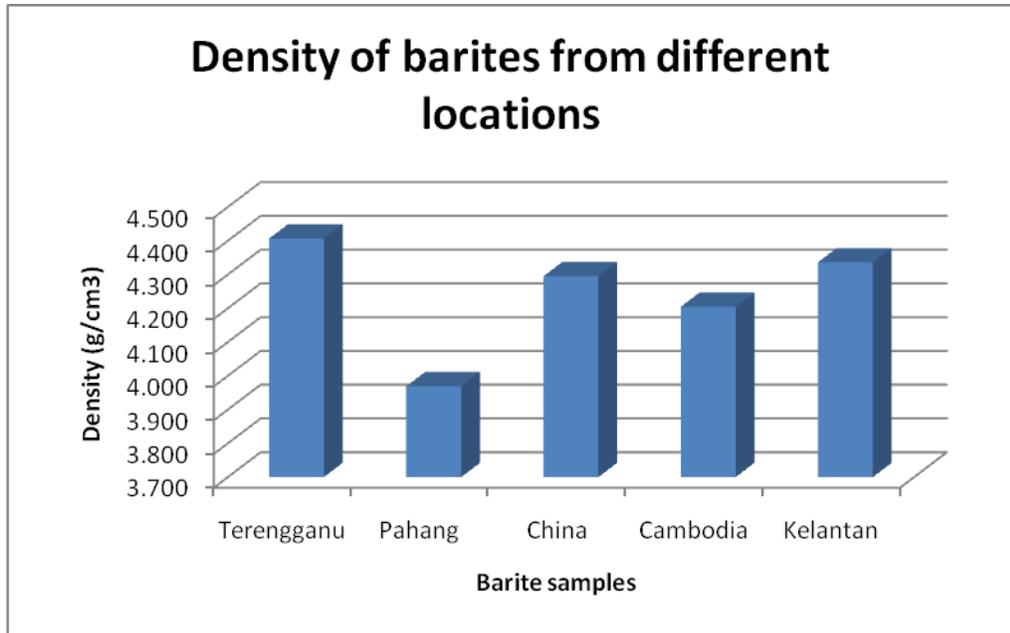


Figure 4-1: Density of barites from different locations

Discussion

From Figure 4-1, it can be deduced that the density of Terengganu barite is the highest (4.406 g/cm³), followed by that of Kelantan barite (4.336 g/cm³), that of China barite (4.295 g/cm³), that of Cambodia barite (4.204 g/cm³) and that of Pahang barite (3.969 g/cm³) is the lowest among the samples. Higher the density of a barite, more favorable it is provided that the other factors are not taken into account. Thus, Terengganu barite can be said as the best of all the barite samples.

According the density requirement given in API Specification 13A, the minimum density of that a barite sample should have in order for it to be used as drilling grade barite is 4.20 g/cm³. As all the barite samples meet the requirement except for Pahang barite, they are considered to be potentially suitable drilling grade barite.

4.2. Result of Particle Less Than 6 μm in Equivalent Spherical Diameter (% finer, S6) Experiment

Note: Calculation for particle less than 6 μm in equivalent spherical diameter is given in Appendix E.

Table 4.2: Raw and calculated data of (% finer, S6) experiment for Terengganu barite

Time , t (min)	Temperature , T (C)	Hydrometer reading, H	Water Viscosity , η (mPa.s)	Effective depth , L (cm)	Particle diameter , d_e (μm)	% Finer , S	% Finer , S6
10	24.4	1.005	0.9018	15	11.03	4.65	
20	24.4	1.005	0.9018	15	7.80	4.65	
30	24.4	1.004	0.9018	15.2	6.41	3.04	
40	24.4	1.003	0.9018	15.5	5.61	1.43	2.21

Table 4.3: Raw and calculated data of (% finer, S6) experiment for Pahang barite

Time , t (min)	Temperature , T (C)	Hydrometer reading, H	Water Viscosity , η (mPa.s)	Effective depth , L (cm)	Particle diameter , d_e (μm)	% Finer , S	% Finer , S6
10	24.4	1.001	0.9018	16.0	12.20	-1.86	
20	24.4	1.000	0.9018	16.3	8.71	-3.54	
30	24.4	1.000	0.9018	16.3	7.11	-3.54	
40	24.4	1.000	0.9018	16.3	6.16	-3.54	

Table 4.4: Raw and calculated data of (% finer, S6) experiment for China barite

Time , t (min)	Temperature , T (C)	Hydrometer reading, H	Water Viscosity , η (mPa.s)	Effective depth , L (cm)	Particle diameter , d_e (μm)	% Finer , S	% Finer , S6
10	24.4	1.005	0.9018	15.0	11.21	4.70	
20	24.4	1.004	0.9018	15.2	7.98	3.07	
30	24.4	1.003	0.9018	15.5	6.58	1.44	
40	24.4	1.003	0.9018	15.5	5.70	1.44	1.44

Table 4.5: Raw data and calculated data of (% finer, S6) experiment for Cambodia barite

Time , t (min)	Temperature , T (C)	Hydrometer reading, H	Water Viscosity , η (mPa.s)	Effective depth , L (cm)	Particle diameter , d_e (μm)	% Finer , S	% Finer , S6
10	24.4	1.014	0.9018	12.6	10.42	19.49	
20	24.4	1.011	0.9018	13.4	7.60	14.57	
30	24.4	1.010	0.9018	13.7	6.27	12.93	
40	24.4	1.008	0.9018	14.2	5.53	9.65	11.71

Table 4.6: Raw and calculated data of (% finer, S6) experiment for Kelantan barite

Time , t (min)	Temperature , T (C)	Hydrometer reading, H	Water Viscosity , η (mPa.s)	Effective depth , L (cm)	Particle diameter , d_e (μm)	% Finer , S	% Finer , S6
10	24.4	1.007	0.9018	14.4	10.92	7.94	
20	24.4	1.006	0.9018	14.7	7.80	6.31	
30	24.4	1.005	0.9018	15.0	6.43	4.69	
40	24.4	1.004	0.9018	15.2	5.61	3.06	3.83

Table 4.7: Summary of % finer, S6 of barite from different locations

Location	% Finer, S6
Terengganu	2.21
China	1.44
Cambodia	11.71
Kelantan	3.83

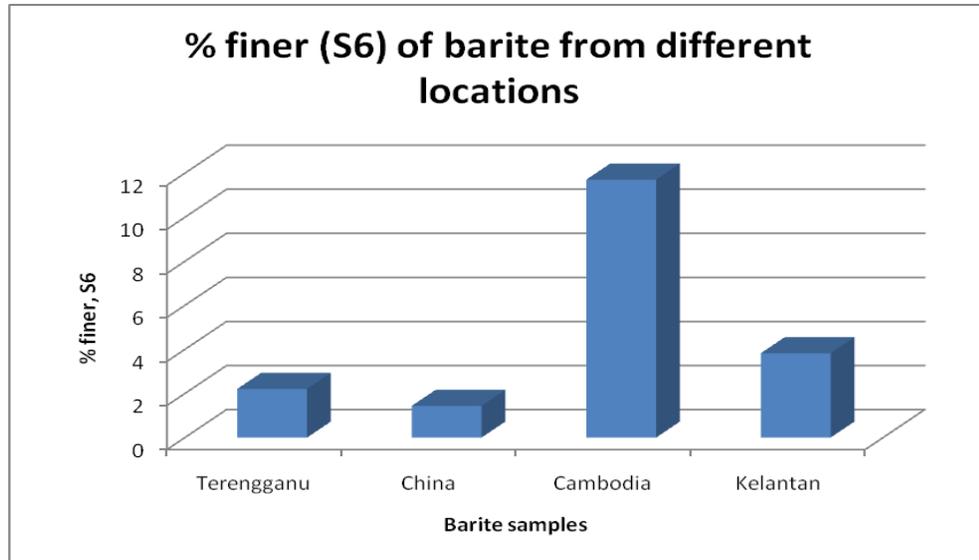


Figure 4-2: % finer, S6 of barite from different locations

Discussion

It is theorized that lighter and finer particles will sag less. On the other hand, reducing particle size of weighting agent is conventionally thought to be able to increase the plastic viscosity of fluid undesirably. In other words, overly fine weighting agent particles can thicken the drilling fluid.

The requirement given by API Specification 13A on particles less than 6 μ m in equivalent spherical diameter (% finer, S6) says that a drilling grade barite should contain at most 30% by mass of (% finer, S6). All the barite samples meet this requirement except for Pahang barite.

As shown in Figure 4-2, the content of particle less than 6 μ m in equivalent spherical diameter (% finer, S6) in Cambodia barite is the highest (11.71%), followed by that in Kelantan barite (3.83%), Terengganu (2.21%) and lastly, that of China barite (1.44%) is the lowest. It is to note that Pahang barite mainly consists of particle courser than 6 μ m in equivalent spherical diameter and thus its results are not in the range to allow for the application of the formulation to calculate (% finer, S6). China barite can be said to be most desired one as it sags the least and more importantly, it will cause the least undesirable increase in plastic viscosity of drilling fluid and thus leading to reduced pumpability.

4.3. Results of Residue of Diameter Greater than 75 μ m Experiment

Table 4.8: Raw and calculated data of residue of diameter greater than 75 μ m Experiment

	Barite samples					
	Terengganu	Pahang	China	Cambodia	Kelantan	Base
m1, mass of sample (g)	50.00	50.00	50.00	50.45	50.00	50.00
m2, mass of residue+bowl (g)	180.13	181.94	184.17	181.90	180.01	91.90
m3, mass of bowl (g)	178.69	179.00	182.26	180.68	178.33	91.00
m4, mass of residue (g)	1.44	2.94	1.91	1.22	1.68	0.90
Uncorrected Rm	2.87	5.88	3.81	2.41	3.36	1.80
Corrected Rm	2.17	5.18	3.11	1.71	2.66	1.10

Note: Calculation for residue of diameter greater than 75 μ m (Rm) is given in Appendix G

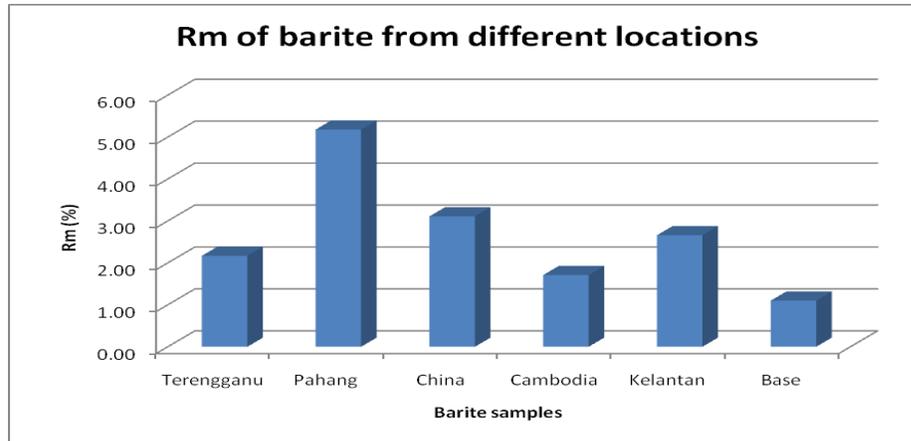


Figure 4-3: Residue of diameter greater than 75 μ m for barite from different locations

Discussion

The problem of sag arises due to the presence of courser particle size. The API Specification 13A states that for use as drilling grade barite, the residue greater than 75 μ m of any barite sample should not exceed 3.0% by mass.

Figure 4-3 illustrates that all the barite sample meet the API requirement except for Pahang barite. An additional sample, termed as “Base” is a commercial drilling grade barite. Putting the barite samples in ranking, Cambodia barite has the least residue greater than 75 μ m and thus is the most preferred. This is then followed by Terengganu barite, Kelantan barite, China barite and lastly Pahang barite.

4.4. Results of Water-soluble Alkaline Earth Metal, as Calcium Experiment

Table 4.9: Elemental analysis of barites from different locations using XRF

Terengganu		Kelantan		China	
Element	Mass fraction (%)	Element	Mass fraction (%)	Element	Mass fraction (%)
O	27.000	O	26.000	O	27.000
Al	0.451	Al	0.000	K	0.029
Si	0.796	Si	0.700	Si	1.650
P	0.423	P	0.387	P	0.405
S	11.950	S	11.880	S	11.970
Ca	0.015	Ca	0.019	Ca	0.017
Ti	0.190	Ti	0.170	Ti	0.170
Fe	0.040	Fe	0.010	Fe	0.112
Cu	0.014	Cu	0.018	Cu	0.021
Sr	0.198	Sr	1.340	Sr	1.311
Ba	58.940	Ba	58.930	Ba	56.740
P2O5	1.205	SiO2	1.197	P2O5	1.243
K2O	0.000	K2O	0.000	K2O	0.035

Cambodia		Pahang		Base	
Element	Mass fraction (%)	Element	Mass fraction (%)	Element	Mass fraction (%)
O	29.000	O	41.800	O	26.000
Al	1.350	Al	1.450	Al	0.140
Si	4.330	Si	12.270	Si	0.000
P	0.391	P	0.437	P	0.390
S	10.660	S	9.300	S	11.860
Ca	0.023	Ca	0.035	Ca	0.017
Ti	0.191	Ti	0.013	Ti	0.190
Fe	0.658	Fe	0.319	Fe	0.000
Cu	0.013	Zr	0.009	Cu	0.017
Sr	0.336	Cl	1.320	Sr	0.963
Ba	52.250	Ba	33.060	Ba	60.450
TiO2	0.319			Al2O3	1.224
K	0.171			Cl	0.000
Mn	0.086				
Zn	0.005				
Ar	1.154				

Table 4.10: Concentration of Ca in barite from different locations

Location	% of Ca	mg Ca in 5g sample	mg Ca / g sample
Terengganu	0.015	0.0008	152.00
Kelantan	0.019	0.0010	192.00
China	0.017	0.0009	170.00
Cambodia	0.023	0.0012	232.00
Pahang	0.035	0.0017	347.00
Base	0.017	0.0008	166.00

Note: Calculation for converting mass fraction (wt%) Ca into concentration (mg Ca/kg barite) is given in Appendix F

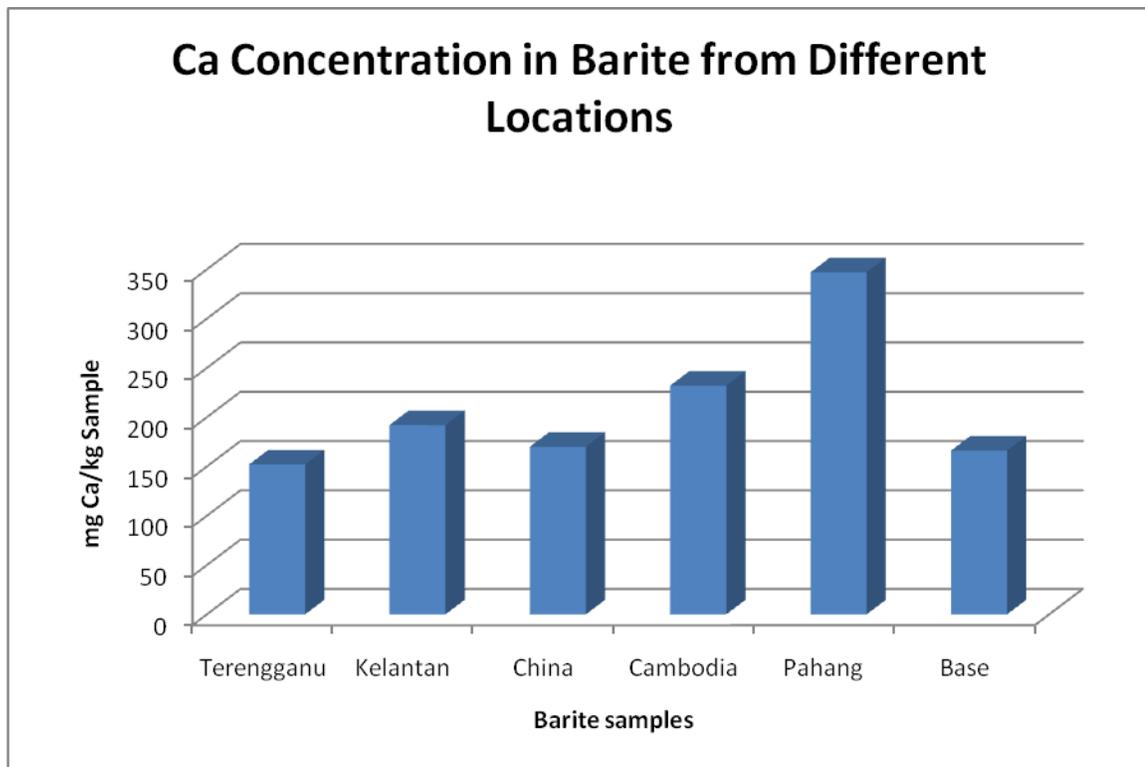


Figure 4-4: Concentration of Ca in barite from different locations

Discussion

The concentration of soluble calcium salt in barite can lead to undesirable effect on the performance of bentonite which is an important additive in water-based muds as viscosity and fluid loss control. Some calcium salts dissolve in water and thus increase the hardness of water which then weakens the ability of bentonite in suspending the cuttings and sealing the pore throats to prevent fluid loss. In brief, the harder the water, the more difficult it is to get bentonite to yield.

From Figure 4-4, it is apparent that all the barite samples except Pahang barite conform to the requirement on water-soluble alkaline earth metals, as calcium given by API Specification 13A, which is 250mg/kg at most. An additional sample, termed as “Base” is a commercial drilling grade barite. From the figure, the concentration of calcium in Terengganu barite is the lowest, followed by that of China barite, Kelantan barite, Cambodia barite, and lastly that of Pahang barite.

4.5. Overall Discussion

On a scale from 1 (the most superior) to 5 (the least superior), Table 4.11 gives a summary of which barite is more superior than the others in terms of different criteria of interest.

Table 4.11: Summary of superiority of each barite sample

	Barite samples				
	Terengganu	Kelantan	China	Cambodia	Pahang
Density	1	2	3	4	5
Particle less than 6 μm in equivalent spherical diameter	2	3	1	4	5
Residue greater than 75 μm	2	3	4	1	5
Water-soluble alkaline earth metals (Ca)	1	3	2	4	5
Total	6	11	10	13	20

Terengganu barite has the least total score and therefore is considered as the best barite among the samples. It is followed by China barite, Kelantan barite, Cambodia barite and lastly the Pahang barite which is not even suitable to be used as weighting agent in drilling fluid.

CONCLUSION

Five barite samples (three from localities in the east coast of peninsular Malaysia and two from China and Cambodia) had been obtained and subsequently been tested on their compliance with the physical and chemical requirements given in API Specification 13A for use as drilling grade barite.

The results have revealed that all the barite samples are suitable for use as drilling grade barite except for Pahang barite, which complies with none of the requirements.

A weighting agent is used to increase the density of drilling fluids while minimizing the increase in the resistance to flow of the drilling fluid. Amongst all the barite samples, Terengganu barite is of the highest quality. The quality of Kelantan barite is comparable to that of standard commercial barites like China and Cambodia barites. Pahang barite is of inferior quality.

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APPENDICES

Appendix A: Experiment of Particle Density of Barite by Using the Large Pyknometer Method

Reagent and apparatus

A set of pyknometer, a glass rod, an electronic balance, a thermometer and drying oven



Figure A-1: Pyknometers being filled with barite solutions during the experiment.

Procedure

1. A sample of barite of about 1.5kg is taken. The sample is sieved. The course particles retained on a 20mm test sieve is broken down to less than that size,
2. The sample is divided into 2 specimens, each weighting 400g by riffing.
3. These specimens are put into the oven for drying at 105°C – 110°C and then the specimens are stored in an airtight container until required.
4. Clean and dry the pyknometer and the whole assembly is weighed to the nearest 0.5g (m1).
5. The screw top is removed and the first specimen is transferred from its sealed container directly into the jar.
6. The jar and its content and the screw top assembly is weighed to the nearest 0.5g (m2).
7. Water at temperature of the average room temperature is added to about half fill of the jar. The mixture is stirred thoroughly with a glass rod to remove air trapped in the particles
8. The screw cap assembly is fit and it is tightened so that the reference marks coincide. The pyknometer is filled with water.
9. The pyknometer is agitated by shaking. Air is allowed to escape and froth is allowed to disperse. The pyknometer is left standing for at least 24 hours at room temperature.
10. The pyknometer is top up with water so that the water surface is flush with the hole in the conical cap. Make notes that air bubbles or froth are not trapped under the cap.
11. The pyknometer is dry on the outside and the whole is weighed to the nearest 0.5g (m3).
12. The pyknometer is emptied, washed thoroughly and filled completely with water at room temperature. Make sure that the reference marks on the screw top coincide, that no air bubble are entrapped, and that the water surface is flush with the hole in the conical cap.
13. The pyknometer is dried on the outside and weighed to the nearest 0.5g (m4).
14. Step 4-12 is repeated by using the second specimen of the same barite so that two values of particle density can be obtained.

Calculation

$$\text{Density, Mg / m}^3 = \frac{m_2 - m_1}{(m_4 - m_1) - (m_3 - m_2)}$$

Where:-

m1 is the mass of pyknometer + cap assembly (g)

m2 is mass of pyknometer + cap + soil (g)

m3 is mass of pyknometer + cap + soil + water (g)

m4 is mass of pyknometer + cap + water (g)

Appendix B: Experiment of Water-Soluble Alkaline Earth Metals as Calcium

X-ray fluorescence (XRF) spectroscopy is used for determination of calcium concentration. XRF is basically the emission of characteristic "secondary" (or fluorescent) X-rays from a material that has been excited by bombarding with high-energy X-rays or gamma rays. Originally, the technique is mainly used to analyze geological samples. Fuelled by the advancements in computer and technology, it has since been widely used for elemental analysis and chemical analysis, particularly in the investigation of metals, glass, ceramics and building materials, and for research in geochemistry, forensic science and archaeology. It has the advantages being easy sample preparation, nondestructive rapid multi-element analysis, and the ability to screen unknowns in a wide array of sample matrices such as liquids, solids, slurries, powders, pastes, thin films, air filters, and many others.

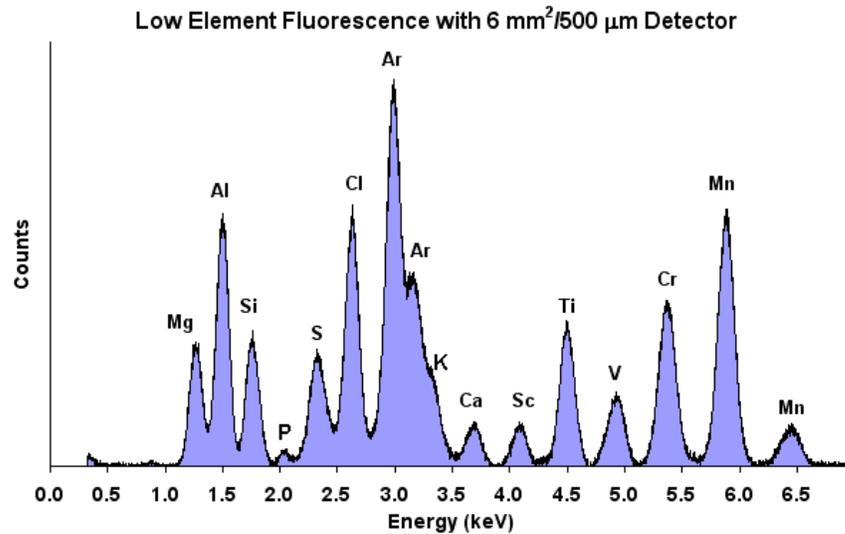


Figure A-1: Low element fluorescence with 6mm²/500µm detector

In addition to calcium concentration, the concentration of other elements that present in the barite samples had also been determined. Having more information about the content of barite samples may sometime be handy especially when drilling a more sensitive environment that requires a more complex formulation of drilling fluid.

Appendix C: Experiment of Residue of Diameter Greater than 75µm

Reagent and apparatus

- **Sodium hexametaphosphate** (CAS No.10124-56-8).
- **Oven**, regulated to $105\text{ }^{\circ}\text{C} \pm 3^{\circ}\text{C}$.
- **Desiccator** with calcium sulfate (CAS No. 7778-18-9) desiccant or equivalent.
- **Balance**, with an accuracy of 0,01 g.
- **Mixer** (e.g. Multimixer® Model 9B with 9B29X impellers or equivalent), having each spindle fitted with a single sine-wave impeller, approximately 25 mm in diameter, mounted flash side up.
- **Container**, of approximate dimensions 180 mm deep, 97 mm i.d. top, 70 mm i.d. bottom (e.g. Hamilton Beach® mixer cup No. M110-D6) , or equivalent).
- **75µm sieve**, conforming to the requirements of ASTM E11, of approximate dimensions 76 mm diameter and 69 mm from top of frame to wire cloth.
NOTE: Supplier's verification that sieve conforms to ASTM E11 is satisfactory evidence of compliance.
- **Spray nozzle** with 1/4 TT body (Spraying Systems Co., No. TG 6.5 tip with 1/4 TT body7), or equivalent) attached to water line with 90° elbow.
- **Water pressure regulator**, capable of regulation to $69\text{ kPa} \pm 7\text{ kPa}$.
- **Evaporating dish** or functional equivalent.
- **Wash bottle**.



Figure A-2: Sieving the commercial barite

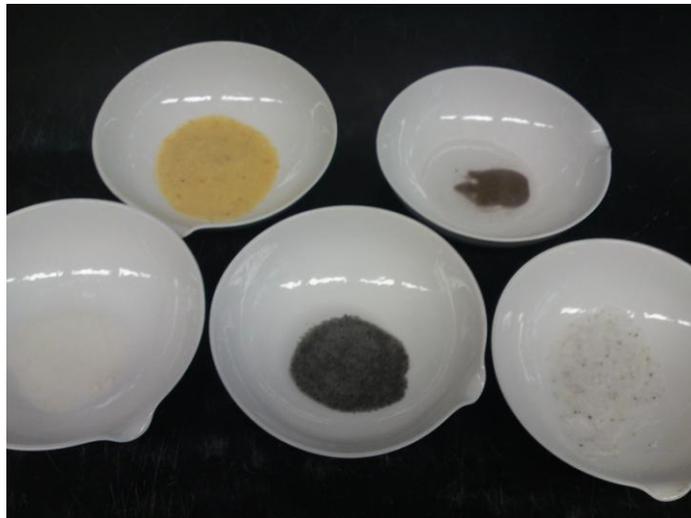


Figure A-3: The wet residue of all the barite solutions in evaporating dishes.

Procedure

1. If required, equilibrate approximately 60 g of dried barite in a desiccator.
2. Weigh $50 \text{ g} \pm 0.01 \text{ g}$ of dried barite. Add the weighed sample to approximately 350 cm³ of water containing about 0.2 g of sodium hexametaphosphate. Stir on the mixer for $5 \text{ min} \pm 1 \text{ min}$.
3. Transfer the sample to the 75- μm sieve. Use wash bottle to remove all material from the container to the sieve. Wash the material on the sieve with water controlled to $69 \text{ kPa} \pm 7 \text{ kPa}$ from a spray nozzle for $2 \text{ min} \pm 15 \text{ s}$. While washing, hold the tip of the spray nozzle approximately in the plane of the top of sieve and move the spray of water repeatedly over the sample.
4. Wash the residue from the sieve into a tared evaporating dish and decant excess clear water.
5. Dry the residue in the oven to a constant mass. Record residue mass and total drying time.

Hamilton Beach® mixer cup No. M110-D is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

Spraying Systems Company, No. TG 6.5 tip with 1/4 TT body is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

Calculation

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

Where C is the correction factor as given in Table A-1.

Table A-1: Correction values (C) for 75 μ m sieves

Working sieve size Average opening ^a , μm	Correction size ^b :	
	Barite/Haematite	Bentonite
70	-0,7	-0,3
71	-0,6	-0,2
72	-0,4	0
73	-0,3	0
74	-0,1	0
75	0	0
76	+ 0,1	0
77	+ 0,3	0
78	+ 0,4	0
79	+ 0,6	+ 0,2
80	+ 0,7	+ 0,3

Appendix D: Experiment of Particle Less Than 6 μ m In the Equivalent Spherical Diameter For Sedimentation Method.

Reagent and Apparatus

- **Dispersant solution**, comprising 40 g \pm 0.1 g sodium hexametaphosphate and 3.60 g \pm 0.1 g anhydrous sodium carbonate (CAS No. 497-19-8) per 1 000 cm³ of solution. The sodium carbonate is used to adjust the pH of the solution to approximately 9.0.
- **Oven**, regulated to 105 °C \pm 3 °C.
- **Desiccator**, with calcium sulfate (CAS No. 7778-18-9) desiccant, or equivalent.
- **Balance**, with an accuracy of 0.01 g.
- **Mixer** (e.g. Multimixer Model 9B with 9B29X impellers or equivalent), having each spindle fitted with a single sine-wave impeller approximately 25 mm in diameter mounted flash side up.
- **Container**, of approximate dimensions 180 mm deep, 97 mm i.d. top, 70 mm i.d. bottom (e.g. Hamilton Beach mixer cup No. M110-D, or equivalent).
- **Glass sedimentation cylinder**, approximately 457 mm in height and 63 mm in diameter, marked for a volume of 1 000 cm³ (ASTM D-422-63).
- **Rubber stopper**, Number 13.
- **Water bath or constant-temperature room**, capable of maintaining a convenient constant temperature at 24 °C \pm 5 °C.
- **Thermometer**, including the range 16 °C \pm 0.5 °C to 32 °C \pm 0.5 °C.
- **Hydrometer**, ASTM 151H, graduated to read specific gravity of suspension.
- **Timer**, mechanical or electrical, with an accuracy of 0.1 min over the test period.



Figure A-4: The barite solutions contained by sedimentation cylinder being put in 24°C water bath.

Procedure for sedimentation method

1. Weigh $80 \text{ g} \pm 0.1 \text{ g}$ of dry barite and place in container.
2. Add $125 \text{ cm}^3 \pm 2 \text{ cm}^3$ ($127 \text{ g} \pm 2 \text{ g}$) of dispersant solution (from 7.11.6). Dilute to approximately 400 cm^3 with deionized water. Rinse all adhering particles from spatula into suspension.
3. Stir $5 \text{ min} \pm 0.5 \text{ min}$ on mixer.
4. Transfer the suspension to the sedimentation cylinder. Rinse container with deionized water to assure that all sample particles are transferred to the sedimentation cylinder.
5. Add deionized water to the 1000 cm^3 mark. Mix the contents thoroughly by constantly changing the cylinder from the upright to the inverted position and back for $60 \text{ s} \pm 5 \text{ s}$ while holding a No. 13 rubber stopper in the top of the cylinder.

This is a critical step. Suspension shall be homogeneous at start of sedimentation. This is difficult to obtain because of the high density of barite.

6. Set the cylinder into the water bath (or counter-top of constant-temperature room) and simultaneously start the timer. Hang the thermometer in the suspension.
7. Take hydrometer readings at intervals of 10 min ± 0.1 min, 20 min ± 0.1 min, 30 min ± 0.1 min, and 40 min ± 0.1 min (or until the first point below the 6 mm value is reached). To take a hydrometer reading, carefully and slowly lower the hydrometer to approximately the 1,020 reading before releasing. After hydrometer stabilizes, read the top of the meniscus at the prescribed time. Carefully and slowly remove hydrometer, rinse with deionized water, and dry after each reading. The hydrometer shall be removed immediately after each reading to eliminate particle build-up on the shoulders which causes erroneous results. All hydrometer readings shall be done with a minimum of fluid disturbance to preserve suspension-settling equilibrium.
8. Record time, min (t), temperature, °C or °F (T), and hydrometer reading (H) on data sheet.
NOTE: Temperature may be measured in either °C or °F as long as all measurements and calculations are consistent in units, including hydrometer calibration.
9. For each time interval, determine water viscosity (*h*) and effective hydrometer depth (L) from Table 3 and Table 4. Record on data sheet.

Calculation

1. From hydrometer calibration (5.7) enter hydrometer correction slope (Mc) and hydrometer correction intercept (Bc) into data sheet.
2. Calculate sample constant (Ks) as follows (or determine from Table 5) and enter into data sheet:

$$Ks = \frac{100 \times \rho}{m(\rho - 1)}$$

Where:

Ks is the sample constant

ρ is the sample density, in grams per cubic centimeter

m is the sample mass, in grams

3. Calculate and enter onto data sheet the equivalent spherical diameter (d_e) for each time interval as follows:

$$d_e = 17.5 \sqrt{\frac{\eta L}{(\rho - 1)t}}$$

Where:

d_e is the equivalent spherical diameter, in micrometres

η is the viscosity of water, in millipascal seconds

ρ is the density of sample, in grams per cubic centimeter

t is the time, in minutes

L is the effective depth, in centimetres

4. Calculate and enter onto data sheet the cumulative percent finer for the equivalent particle diameter (d_e) immediately greater than 6 μm (SH) and the equivalent particle diameter (d_e) immediately less than 6 mm (SL) as follows:

$$S = K_s[(M_c \times T) - B_c + (H - 1)1000]$$

Where:

S is the cumulative percent finer than size

K_s is the sample constant

M_c is the hydrometer correction slope

T is the suspension temperature, $^{\circ}\text{C}$

B_c is the hydrometer correction intercept

H is the hydrometer reading

5. Calculate and enter onto data sheet cumulative percent less than 6 mm (S_6) as follows:

$$S_6 = \left[\left(\frac{SH - SL}{dH - dL} \right) (6 - dL) \right] + SL$$

Where:

S₆ is the cumulative percent less than 6µm

SH is the cumulative percent for point immediately greater than 6µm

SL is the cumulative percent for point immediately less than 6µm

dH is the equivalent particle diameter immediately greater than 6µm

dL is the equivalent particle diameter immediately less than 6µm

Table 3 – Viscosity of water at various temperatures

Temperature °C (°F)	Viscosity mPa·s	Temperature °C (°F)	Viscosity mPa·s
15,6 (60)	1,1211	22,2 (72)	0,9498
16,1 (61)	1,1050	22,8 (73)	0,9374
16,7 (62)	1,0893	23,3 (74)	0,9253
17,2 (63)	1,0739	23,9 (75)	0,9134
17,8 (64)	1,0589	24,4 (76)	0,9018
18,3 (65)	1,0442	25,0 (77)	0,8904
18,9 (66)	1,0298	25,6 (78)	0,8792
19,4 (67)	1,0158	26,1 (79)	0,8683
20,0 (68)	1,0020	26,7 (80)	0,8576
20,6 (69)	0,9885	27,2 (81)	0,8470
21,1 (70)	0,9753	27,8 (82)	0,8367
21,7 (71)	0,9624	28,3 (83)	0,8266

Table 4 – Values of effective depth based on readings on hydrometer ASTM 151H used in specific sedimentation cylinder

Uncorrected hydrometer reading	Effective depth L , cm	Uncorrected hydrometer reading	Effective depth L , cm
1,000	16,3	1,020	11,0
1,001	16,0	1,021	10,7
1,002	15,8	1,022	10,5
1,003	15,5	1,023	10,2
1,004	15,2	1,024	10,0
1,005	15,0	1,025	9,7
1,006	14,7	1,026	9,4
1,007	14,4	1,027	9,2
1,008	14,2	1,028	8,9
1,009	13,9	1,029	8,6
1,010	13,7	1,030	8,4
1,011	13,4	1,031	8,1
1,012	13,1	1,032	7,8
1,013	12,9	1,033	7,6
1,014	12,6	1,034	7,3
1,015	12,3	1,035	7,0
1,016	12,1	1,036	6,8
1,017	11,8	1,037	6,5
1,018	11,5	1,038	6,2
1,019	11,3		

Table 5 – Sample constant (Ks) for 80.0 g sample mass

Sample density g/cm ³	Sample constant, K _s
4,20	1,641
4,21	1,640
4,22	1,639
4,23	1,637
4,24	1,636
4,25	1,635
4,26	1,634
4,27	1,633
4,28	1,631
4,29	1,630
4,30	1,629
4,31	1,628
4,32	1,627
4,33	1,625
4,34	1,624
4,35	1,623
4,36	1,622
4,37	1,621
4,38	1,620
4,39	1,619
4,40	1,618

Appendix E: Calculation for particle less than 6 µm in equivalent spherical diameter

The followings demonstrate the steps involved to calculate the particle less than 6 µm in equivalent spherical diameter of Terengganu barite.

Calibration of hydrometer ASTM 151H

The hydrometer correction slope, $M_c = 0.26$ (from API manual)

The hydrometer correction interception, $B_c = 8.469$ (from API manual)

Sample mass = 80g

Sample density = 4.406 (from Table 4.1)

Sample constant, K_s

$$K_s = \frac{100 \times \rho}{m(\rho - 1)}$$

$$K_s = \frac{100 \times 4.406}{80(4.406 - 1)} = 1.612$$

Calculation of d_e for 20 min reading

$$d_e = 17.5 \sqrt{\frac{\eta L}{(\rho - 1)t}}$$

$$d_e = 17.5 \sqrt{\frac{0.908 \times 15}{(4.406 - 1)20}} = 7.80$$

Cumulative percent finer than size, S

$$S = K_s [(Mc \times T) - Bc + (H - 1)1000]$$

$$S = 1.612 [(0.26 \times 24.4) - 8.64 + (1.005 - 1)1000] = 4.65\%$$

Calculation for percent of diameter less than 6 μm

$$S_6 = \left[\left(\frac{SH - SL}{dH - dL} \right) (6 - dL) \right] + SL$$

$$S_6 = \left[\left(\frac{3.04 - 1.43}{6.41 - 5.61} \right) (6 - 5.61) \right] + 1.43 = 2.21\%$$

Appendix F: Calculation for converting mass fraction (wt%) Ca into concentration (mg Ca/kg barite)

The following calculation is with respect to converting the mass fraction Ca (wt%) of Terengganu barite into concentration (mg Ca/ kg barite).

From Table 4.8, the mass fraction Ca in percentage of Terengganu barite is 0.015 %.

Assume a basic of 100g of barite sample, thus mass of Ca present is:

$$\text{Mass Ca (g) in 100g of barite} = \frac{100\text{g}}{100\%} \times 0.015\% = 0.015\text{g}$$

$$\frac{0.015\text{g Ca}}{100\text{g Barite}} \times \frac{1000\text{mg Ca}}{1\text{g Ca}} \times \frac{1000\text{g Barite}}{1\text{Kg Barite}} = 150\text{ g Ca / Kg Barite}$$

Appendix G: Calculation for residue of diameter greater than 75µm (Rm)

The following calculation is based on the results of Terengganu barite.

Calculation for the mass of the residue, (g)

$$\text{mass of residue} = \text{mass of (residue + bowl)} - \text{mass of bowl}$$

$$\text{mass of residue} = 180.13\text{g} - 178.69\text{g}$$

$$\text{mass of residue} = 1.44\text{g}$$

Calculation for the uncorrected residue, mass fraction (%) of diameter greater than 75µm, Uncorrected Rm

$$\text{Uncorrected } Rm = 100 \left(\frac{\text{mass of residue, g}}{\text{mass of sample, g}} \right)$$

$$\text{Uncorrected } Rm = 100 \left(\frac{1.44\text{g}}{50.00\text{g}} \right)$$

$$\text{Uncorrected } Rm = 2.87\%$$

From Table A-1, the correction values, C is determined to be -0.7 since the average opening of my working sieve size is 70µm.

Calculation for the corrected residue, mass fraction (%) of diameter greater than 75µm, Rm

$$Rm = \text{uncorrected } Rm + C$$

$$Rm = 2.87\% - 0.7\%$$

$$Rm = 2.17\%$$