

ASSESSMENT OF BENEFICIATION PROCESS OF A NIGERIAN BARITE  
SOURCE FOR UTILIZATION AS OIL DRILLING MUD

A Thesis Presented to the Department of  
Materials Science and Engineering

African University of Science and Technology



In Partial Fulfillment of the Requirements for the Degree of  
Master of Science in Materials Science and Engineering (Minerals Processing)

By

Afolayan David Oluwasegun

Abuja, Nigeria

December, 2017.

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## **CERTIFICATION**

This is to certify that the thesis titled “ASSESSMENT OF BENEFICIATION PROCESS OF A NIGERIAN BARITE SOURCE FOR UTILIZATION AS OIL DRILLING MUD” submitted to the school of postgraduate studies, African University of Science and Technology (AUST), Abuja, Nigeria for the award of the Master’s degree is a record of original research carried out by Afolayan David Oluwasegun in the Department of Materials Science and Engineering

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## ABSTRACT

The texture and size distribution of various species of gangue minerals affect the beneficiation of barite and lower the specific gravity of barite as-mined. About 9% of the barite used in the oil industry in Nigeria is imported, even though it is found in abundance in eight states of the country. This study has examined barite from deposits in Torkula, Benue State of Nigeria for application as oil drilling mud. Physico-chemical and rheological tests were conducted, specific gravity measurements were taken along with moisture content, hardness, gel strength, viscosity and yield point. Mineral samples collected were found to have SG ranging between 3.98 and 4.42, a pH of 8.015, moisture content of about 0.161% and a Mohr's hardness of between 3.5 and 5.0. Slight beneficiation processes are required to completely refine the barite mineral from all the gangue minerals, and to meet the API, DPR standards for oil drilling.

**KEYWORDS:** Gangue minerals, barite, specific gravity, minerals processing strategies, oil drilling mud

To the glory and praise of the Almighty God, the giver of all wisdom, to my family and the overall benefits of mankind, I sincerely dedicated this work.

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## PREFACE

Beneficiation process encompasses safe value addition strategies employed to upgrade and enrich the inherent qualities of a mineral in its crude form. Each of this strategy increases the economic worth, market value and the processability of the ore either as primary materials or an intermediate product. Within the industrial relevance, each stage of value addition opens up new levels of comparative advantage for minerals and the creation of job opportunities.

On the Torkula barite field, the beneficiation process begins from the mining of the vein up to the final stage. Other operational stages include size reduction and control, enrichment (washing and screening), upgrading, materials handling, wear in operation, operational value and environment. However, these depend on the grade of the crude barite, the nature or form of the associated gangue minerals and most importantly, the liberation sizes.

In Nigeria and other countries in the sub-Sahara Africa, barite deposits are widespread and are available in commercial quantity. However, the entire barite market has been taken over by foreign investors who have succeeded in mining and exporting the mineral in the crude form, either with little or without the addition of value. As a result of this, it has become so unrealistic for many of the indigenous oil and gas industries to depend on the locally processed barite.

It is therefore noteworthy to examine and assess the efficiency of the indigenous beneficiation process adopted by local investors of barite mineral for oil drilling mud and other industrial requirements. This enables us to design a workable frame work for any industrial processes, both at the ordinary and specialized level of application.

Conclusively, several works have been done, commercialized and documented on verifiable techniques at the laboratory scale. However, there is a critical need to conduct comprehensive tests and analysis at the molecular level in order to justify the state and properties of barite minerals on the site. This is achievable through the evaluation of the

beneficiated product on the basis of the American Petroleum Institute (API), Department of Petroleum Resources (DPR), quality and environmental system standards.

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## LIST OF ABBREVIATION AND SYMBOLS

- API: American Petroleum Institute
- API RP: American Petroleum Institute Recommended practice
- API Spec.: API Specification
- @: At
- ASM: Artisanal and small-scale mining
- ASTM: American Standard Testing Materials
- AV: Apparent Viscosity
- AWBM: Alternative Water-Based Drilling Mud
- BaO: Barium Oxide
- BaCO<sub>3</sub>, BaCl<sub>2</sub>, Ba (OH): Barium Carbonate, Barium Chloride, Barium hydroxide
- BHCT: Bottom hole circulating temperature
- BaSO<sub>4</sub>: Barium Sulphate
- CEC: Cation Exchange Capacity
- Dial read: Dial reading
- DPR: Department of Petroleum Resources
- EDTA: Ethylene diamine tetra acetic acid
- EPA: Environmental Protection Agency
- EP: Extreme pressure
- ERD: Extended reach drilling
- ES: Electrical Stability
- Exp.Val.: Experimental value
- Exp. Pulv.: Pulverized Samples
- F: Fahrenheit
- FDI: Foreign Direct Investment
- FTIR: Fourier Transform Infrared Spectroscopy

GDP: Gross Domestic Product

GoM: Gulf of Mexico

GSM: Gel Strength at 10 minutes

GSS: Gel Strength at 10 seconds

HPHT: High-Pressure High-Temperature

IADC: International Association of Drilling Contractors

ICDD: International Centre for Diffraction Data

IITA: International Institute for Tropical Agriculture

IR: Infrared

ISO: International Standards Organization

JCPDS: Joint Committee on Powder Diffraction Standards

KBr: Potassium bromide

kg/m<sup>3</sup>: Kilogram per cube meter

KH<sub>2</sub>PO<sub>4</sub>: Potassium dihydrogen Phosphate

lb/lbbl: Pound per pound litres

LGAs: Local Government Area

LLEM: Liquid–liquid extraction method

LOI: Loss on Ignition

LPOs: Legal Process Outsourcings

LTM: Low Toxicity Oil-Based Drilling Mud

M. B. A.: Methylene Blue Absorption

M. B. C.: Methylene Blue Capacity

MCO: Mining Cadastre Office

mg/L: Milligram per Litres

mg/kg: Milligram per Kilogram

MMSD: Ministry of Mines and Steels Development

MT: Metric Tonnes

MW: Mud Weight

NGSA: Nigeria Geological Survey Agency

NMDC: National Metallurgical Development Centre

NPDC: Nigeria Petroleum Development Company

NPDC Ref: Nigeria Petroleum Development Company

N-S: North-South

NW-SE: North West- South East

N-W: North-West

OBM: Oil-Based Mud

OCMA: Oil Companies Materials Association

$\Omega m$ : Ohms meter

Pb-Zn: Lead-Zinc

pH: Power of Hydroxide

PIN: Polyguard Investment Nigeria

POCEMA: Pollution Control and Environmental Management

Ppg: Part Per Galloon

PV: Plastic Viscosity

Ref. Samp: Reference Sample

ROM: Run-of-mine

ROP: Rate of penetration

RPM: Revolution Per Minute

SAP: Structural Adjustment Programme

SBM: Synthetic-Based Drilling Mud

SEM: Scanning electron microscope

SG: Specific Gravity

SrSO<sub>4</sub>: Celestine

SMEW: Standard Methods for the Examination of Water and Wastewater

Sqrt: Square Root

TPA: Tonnes per annum

UCA: Autoclave system or the ultrasonic Cement Analyzer

UHMWPE: Ultra high Molecular Weight Polyethylene

UKOOA: United Kingdom Offshore Oils Association

UV: Ultra-violent

XRD: X-ray diffractrometer

XRF: X-ray fluorescence

YP: Yield point

## **1.0 Introduction**

### **1.1 Background to the Study**

Barite or Barytes, an unusually heavy non-metallic, chemically stable (inert) and insoluble mineral, with Mohr's hardness of 3-3.5, density of 4.48g/cm<sup>3</sup> and a specific gravity between 3.5 and 5.3. It contains between 76-87% BaSO<sub>4</sub>, gangue minerals of between 13% and 24% silica, iron oxide, quartz, Celestine (SrSO<sub>4</sub>), mercury (Hg) and cadmium (Cd), fluorite, chalcopryrite and sulphide minerals such as galena. Up to 80% of barite worldwide is used as a weighting agent for drilling fluids. Nigeria barite contains very low mercury (Hg) and cadmium (Cd) value, making it very suitable for all types of drilling fluid systems (oil or water based) (NGSA, 2010; MMSD, 2010). It is chemically inert to all drilling fluids additives used in offshore drilling technology. High quality micronized barium sulphate (BaSO<sub>4</sub>), within the American Petroleum Institute (API) 13A specifications (3-74 micron range) is used in oil and gas exploration to increase mud density up to 2.52 SG (21lb./gal; 2.5g/cm<sup>3</sup>), suppress high formation pressures and prevent blowouts, to stabilize the borehole or used in kiln fluid, and prepare solids-laden plugs for well control application. As a well is drilled, the bit passes through various formations, each with different characteristics. The deeper the hole, the more barite is needed as a percentage of the total mud mix. The weight of this mixture counteracts the force of the oil and gas when it is released from the ground, which allows the oil and gas rig operators to prevent the explosive release of the oil and gas from the ground (Miller, 2009; NGSA, 2010; MMSD, 2010; API, 2004, API, 2010).

Nigeria currently holds the fourth largest deposit of barite in the world, with an estimated reserve of over 21 Million metric tons. Ironically, Nigeria is not anywhere in the list of the 16th globally recognized largest producers of barite in the world. According to literatures, China the largest producers, produces 3.6 Million MT per annum; India, 1 Million MT per year and USA, 675 Thousand MT per year. Within Africa continent, Morocco is the 5th largest in the world followed by Algeria the 14th with production capacities of 142 Thousand MT and 47 Thousand MT per annum respectively. Nigeria is among the others, which does not account up to 5% of the world production (Ebechidi, 2014; NGSA, 2010). Principal factors responsible for this menace are the uncontrolled importation of barite, no or little access to mining site, absence of mechanized mining companies, uncontrolled artisanal and

small-scale mining (ASM) activities, and lack of LPOs from oil and gas companies to local producers. As a result, local investors are not motivated to consider this as a viable option, whereas, local miners cannot meet up with the demand. The two main areas of concern are processing and the beneficiation (value addition) of the mined barite. Similarly, up to 80% of the mining activities in Nigeria are predominantly artisanal, with diggers, hoes and shovels, in Nasarawa, Benue and Cross Rivers States where high level of barite mining occurs. However, the Nigeria government and some of the indigenous oil industries have shown positive interest in the mining and processing Nigeria barite for oil drilling activities, but such plans are yet to be realized (MMSD, 2010, MMSD, 2013).

Crude or primary barite (run-of-mine) requires some upgrading to minimum purity, density and specific gravity. It is ground to a small, uniform size before it is used as a weighting agent in petroleum well drilling mud specification for barite, filler or extender, or an addition to industrial products. Weighting material with a specific gravity between 4.2 and 4.5 is used to increase the apparent density of a liquid drilling fluid system. Barite ( $\text{BaSO}_4$ ) is the most common weighting agent used today in all drilling fluids. It is mined from layers of sedimentary rock which is formed when barite is precipitated onto the bottom of the ocean, and ground to an API specification (API, 2004, API, 2010; Ariffin, 2000). Although, barite is a by-product of mining lead, zinc, silver, or other metal ores, smaller mines utilize barite from veins, which is formed by precipitation in hot subterranean waters. As a result of the high specific gravity of barite, nearly three quarters of the domestic output of the mineral is used in oil industry as drilling fluids. Due to the increasing drilling activities, the demand for high quality barite as drilling fluid weighting agent has been increasingly difficult to meet. L In addition, the supply of barite is geographically limited with high transportation cost and substandard products. The consumption of drilling mud fluctuates from year to year, which is largely traceable to the price and amount of exploration drilling for oil and gas (Tanko et al., 2015). However, the demand for this very important mineral is quite high. In Nigeria, estimated demand is up to 255,000.00MT per annum. The local market price of this mineral is about N50, 000.00 per ton, but it is imported at N170, 000.00 per ton. To encourage local involvement and industrial participation which is nearly zero, and improve the quality and quantity of output, it has then become imperative to focus attention on Research and

Development, and revitalize the neglected exploration and exploitation of barite deposits abound in Nigeria (Ebechidi, 2014; Adewale and Salihu, 2014).

Barite, as a weighting agent constitutes 58% and 69% of weight in water and oil based drilling fluid respectively. Drilling fluid properties and circulation rates determine the incessant pressure losses in the drill string and the available pressure at the bit for optimized drilling performance. Its rate of penetration (ROP) is also affected by the density of the mud and nature of the suspended solids. Although, the drilling grade barite is appealing over other choices in weighting material due to its unique characteristics. The oilfield application of barite is regulated for chemical purity due to the presence of some heavy metals in various amounts. On the other hand, the texture and size distribution of various species of gangue minerals affect the beneficiation of barite and lower the specific gravity of the barite as-mined. Similarly, the specific gravity (SG) of mine materials varies between 3.9 and 4.4. However, some samples have SG values of less than 3.6, such as the silica rich varieties and other impurities in barite are not suitable for use unless beneficiated (IADC Drilling Manual, 2014; NGS, 2010; MMSD, 2010; MMSD, 2013). The attainment of these standards has become clearly impossible due the high cost of mechanized mining and processing equipment, impacting negatively on investment interests. Up to 97% of the barite used in the oil industry in Nigeria is imported, even though it is found in eight states which are within the Benue Trough, one of the Cretaceous sedimentary basins in Nigeria. Within these geographic areas, drilling grade barites are environmentally acceptable and stable, from the standpoint of disposal as part of the used drilling fluid (Ukooa, 1999a; Ibe et al., 2016; Tanko et al., 2015; MMSD, 2010). So, there is the need for a critical reappraisal on the local barite deposits in Nigeria to address its availability and suitability as a weighting agent in drilling mud system. To do this, some physico-chemical properties, materials behaviour and the environmental impact of the barite mineral deposits from Torkula, Guma Barite Field must be examined, analyzed and correlated with the existing American Petroleum Institute (API) Standards.

## **1.2 Problem Statement**

The American Petroleum Institute (API) introduced a new barite grade (SG 4.1) in August, 2010, in addition to the long-standing 4.2 specification. This was due to the failure of most

developing countries in meeting up with the acceptable quality standards. The intention was not motivated with the notion to replace the 4.2 grade, but to provide the end-user with choice as to which material to use. This change was driven in part by a shortage of SG 4.2 barite, due to the absence of large scale barite value addition facilities in the country. Drilling-grade barite is specified, authorized and introduced by the API and must meet certain SG, chemical and sizing requirements. The API specifications for the drilling grade barite are quite detailed. Beyond specific gravity and density, both physical and chemical requirements such as the water soluble earth metals, the particle size and shape must be consistent with the global standards. In Nigeria, barite deposits from Alosi, Azara, Wuse, mines have specific gravity lower than the API standards, while others are within the specifications (Oden, 2012). On the other hand, run-of-mine from Ibi, Alifokpa, Gabu, Osina, Kumar, Konshisha, Didango and Turkula mines tend to have high specific gravities (above 4.2) and as such are useful for blending to upgrade the lower grade materials (Oden, 2012). Altogether there are more mines producing above API grade materials than those producing below API grade barite (Oden, 2012; API, 2004, API, 2010).

However, there is a need for a proper analysis and documentation of research results on different categories of barite deposits in Nigeria. In addition to these, drilling companies have started to focus on heavy metal content in particular mercury (Hg <1ppm) and cadmium (Cd <3ppm) as specified by the US Environmental Protection Agency (EPA) for the Gulf of Mexico (GoM). Others such as silver (Ag), arsenic (As), chromium (Cr), copper (Cu), lead (Pb), selenium (Se) and zinc (Zn), etc. is expected to be within the API specifications. The procedures for measurement of the drilling fluid properties is expected to be within the API Recommended Practices 13B-1 and 13B-2 for water-based drilling fluids. These procedures are revised and extended periodically as improvements are made and new tests are developed. Consequently, the market structure has undergone a major change compared to the previous period. This critically concerns the more stringent quality requirements for the different utilizations: oil service companies, in addition to OCMA (Oil Companies Materials Association) standards. This disparity has resulted into low market demands and geometric fall in market prices (Scogings and Hughes, 2014; IADC Drilling Manual, 2014, Ebechidi, 2014; MMSD, 2013).

The detailed identification and localization of barite has become intensely difficult due to the associated unconformities and major faults. Its structural features, mineralization and the accumulation of the barite deposits within the Benue trough may occur in lenses and impervious covers. Due to this uniqueness, there is a need for a thorough quality assurance tests on the Nigeria barite deposits, prior to its mining, during processing and post processing operations (Asokm, 2015; Adamu et al., 2014). To do this, the beneficiation route of any ore deposit must be designed to suit its overall peculiarities in order to address several disparities as a result of the geological characteristics. Presently, barite deposits in Turkula, Guma Field has no reliable data that describes its mineralogical characteristics and its beneficiation route. Substantial deposits of barite have been identified but not quantified, in Turkula, Guma Field. These have not yet been documented, probably due to remote location and access to the mining site. Their field characteristics, grade, dimensions, and abundance are not known well enough. The rareness of these relevant data on the mineral deposits continues to constitute a major barrier towards their economic exploitation and utilization. With an imagined increase in the industrial application of barite particularly for drilling purposes, there is a need to carry out some studies on the beneficiation and recovery of barite. This will serve as a means to diversify the national economy, increase the use and exploitation of available raw materials (Obi et al., 2014; Ene et al., 2012; Scogings, 2014).

The geometry and type of barite deposits have affected mining economics and processing complexity. Specific dips of barite veins tend to favours manual mining while some are problematic to artisanal miners. The quality of Nigeria barite differs with depth. The quality of material from the top part of a vein (0-5m depth) is always lower than that from the deeper parts of the same vein (Oden, 2012). This means that much of the publicized low quality of Nigerian barite is largely traceable to the minerals processing strategies employed in the field in question and the level of mining. Also, more than a few of the veins that produced low quality barite in the first decade of production, when mining was at the top 0-5m depth, produces better quality materials at depths of 7-10m. The era of the low quality stigma associated with Nigerian barite from the trough has affected its marketability. This is due to the pattern and forms of mining. The major method of mining barite in Nigeria involves the use of explosives. This is done without the stripping of the overburden, which could allow for blending of the less dense top, with the denser bottom material, to form an API grade

batch (Oden, 2012; Adamu et al., 2014). All these conditions are necessary prerequisite to ensure that the quality of the processed industrial grade barite reflects the promising potentials in-situ. Moreover, the sudden shift from coal and other minerals towards barite mining in Nigeria is largely due to the Federal Government's policy on the use of the locally available raw materials. This has resulted to fewer legal but numerous illegal mining of barite. In addition to this, mining and processing of barite has produced vast quantities of mine rocks and mine tailings. Mining activities within the Benue trough have contributed immensely to the disequilibrium of mineral elements and terrestrial ecosystem due to the excavation of large amount of sands called overburden (Tchokossa et al., 2012; Scogings, 2014).

In recent time, there has not been any meaningful partnership with an Organized Private Sector to meet some of the challenges in the mining sector. This is also necessary to promote the exploration, exploitation and production of barite required to meet the need of the international oil companies in Nigeria. In spite of the huge and promising potentials that have been identified in Guma barite Field and other fields in the Benue trough, large-scale monetization of barite mining in the trough still remains unrealistic. This is due to the illegal batching of Nigeria barite by oil service companies, calling for the need to import high grade barite, necessary to meet the demands for the operation (Oden, 2012; Brobst, 1994). China, India, Morocco, USA, Canada, Algeria and many other countries that are endowed with barite in huge commercial quantities have the locally-based investment and market with promising demand for the mineral deposits prior to the exploration. Unlike Nigeria, there were only foreign investors without any promise and plan to develop the local economy (Brobst, 1994).

In addition to all these, the first barite field where mining started in Nigeria is among those with the lowest quality. As a result of the numerous setbacks, it has become very difficult to reconcile the qualities of the locally processed barite with other locally processed barite offered for sale in the global market. While all these are problems, seeking for a way to address them is the basic focus of this thesis work. Hence, understudying the indigenous mining and mineral processing strategies employed at a typical mining site is the option that was considered. This is critical to identify the peculiar and specific limitations to attract Local

and Foreign Direct Investment in the production of barite. There can be an exception when barites are processed within the acceptable API specifications and to produce the pure white colour in line with international best practice (Etim, 2015; Searls, 1996; Nigeria Bariod Investment, 1996b).

### **1.3 Unresolved Issues**

The API standards and requirement for a drilling grade barite is beyond the purity, density and specific gravity. There are other physico-chemical properties. These are size distribution, the amount of alkaline metals and residue such as calcium, extractable carbonate, moisture content, floatation chemicals, hematite, etc. Relevant researches in this area have only focused on meeting the specification for SG and density without much emphases on the purity and other performance indices for particulate mechanics. The mineralization and the actual content of the gangue mineral in barite ROM are needed to be characterized prior to the processing and beneficiation of barite. Each gangue mineral has a selective method of extraction and the level to which the API standards cover. These minerals are difficult to recover from the barite deposits. However, grinding and size reduction of the barite run-of-mine (ROM) is critical to the recovery of barite. The finer the particles, the better the specific gravity and density.

The geological formation of barite results into different grades, and classified based on the volume of the gangue minerals intermixed during the solidification of the molten magma. During mining and processing of the minerals, the ROM of these grades was not taken through a screening process. However, these ought to be screened into various grades on the basis of their purity in accordance to the company specification for barite. In addition to these, many of the indigenous mining companies, Artisanal and Small scale miners (ASM) in Nigeria have not adopted these practices, which have greatly affected the quality of the mined minerals. Moreover, the quality of barite differs with respect to the depth of the vein. Barite mineral at different depths is mined and processed together without screening. This adulterates the potentials of the mineral that possesses better qualities, within and above the API specification and standards. The screening of products according to grades and qualities prior to the milling of barite is critical to other value-addition techniques and processing of

the mineral. With this, the cost of crushing and beneficiation of barite should reduce, hence this study.

The assay and recovery of barite, and other major gangue minerals are quite low. Most of these minerals have been lost or washed away within the waste water. This has resulted into the dispersion of the minerals, increase in the soil toxicity and other environmental degradation. Hence, the recovered concentrate does not consolidate the actual volume of the run-of-mine. Surface mining has dominated the exploitation and exploration of most minerals in Nigeria. Due to this dilemma, research results and data from several experimental works on barite do not correlate and reflect the geological evidences. Research and development on Nigeria barite has remained ineffective as a result of the weak local involvement and industrial participation. The Government-researcher-industry-community nexus needs to be formulated and fully established. The field work and experiences are required to build up a synergy among the University, miners and the host community. This is highly necessary to upgrade indigenous mining and processing of barite, and to address the environmental danger to humans and vegetation.

#### **1.4 Aim and Objectives**

The aim of this research is to examine the qualities, potentials and the suitability of Nigeria barite deposits in Torkula, Guma barite Field, as candidate for oil drilling mud application. In essence, the goal is to resolve the issues enumerated above as much as possible by means of scientific methods, within the American Petroleum Institute (API) Specifications and Standards. These specific objectives are to:

- (i). Characterization of on-site processed barite from Torkula mine field.
- (ii). Determine the physico-chemical properties of barite beyond SG and density of on-site processed barite.
- (iii). Evaluate the rheological properties of Torkula Barite within the API Specifications and Standards.
- (iv). Evaluate the current processing equipment and machineries, and on-site beneficiation of Torkula barite.

## **1.5 Justification**

Torkula barite, Guma field in Benue State, has high concentration of barite veins. Due to its size, the field holds a lot of promise, even though the known veins are currently fewer than in some fields for example, Azara field. However, there is barite quality contrast between the top part and the lower portions of the vein. Higher quality barite tends to come from the lower portions. While much of the low quality stigma associated with Nigerian barite from the trough might be relieved when mining gets down to good depths. It could still be proven that the locally produced barite in Nigeria to a large extent is yet to meet or agree with the standards specified and spelt out by the America Petroleum Institute (API).

Over 90% of mining activities in the Nigeria are carried out by Artisanal and Small-scale Miners (ASM). Up to 75% of this is carried out illegally. Although, Artisanal and Small-scale Mining (ASM) is an important part of the Nigerian mining sector which has the potential to raise internally generated revenue and enhance foreign exchange earnings, create employment, reduce poverty and encourage Foreign Direct Investment (FDI). These potentials have remained unaccomplished as a result of unregulated practices, weak polices and inadequate training, non-existence of large scale mining and low technology input. However, the present status of the Nigeria mining sector presents an immense opportunity for economic growth and development. It currently accounts for 0.02% of exports, 0.3% of national employment and contributes 0.5%, an estimated \$2.0 billion to the nation's GDP which is still low in comparison to global trends for the sector (MMSD, 2010).

The barite market structure has under gone a major change compared to the previous period. This primarily concerns the more stringent quality requirements for the different utilizations, such as the oil service companies, Oil Companies Materials and Association standards. Nigeria barite has not been able to gain access into the global market due to the presence of floatation reagent and pollutant. In addition to all these, the rheological, chemical and physical properties of Nigeria barite varies at different locations. Nigeria Barite Vein is comparatively different with respect to locations. Hence, there is a need to ascertain the quality and other industrial applications of barite necessary for an efficient and economic use of in both civil and industrial applications

## 1.6 Scope of Work

The physico-chemical and rheological properties of barite can be measured or determined through several techniques depending on the occurrence of the barite vein and the characteristics of the associated gangue minerals present in the barite ROM. To do these, the phase, elemental and morphological analysis of barite from Torkula, Guma field was carried out, using X-ray diffractometer, (XRD), X-ray fluorescence (XRF) and Scanning electron microscope (SEM) respectively. In addition, rheological properties such as the mud weight, pH value, gel strength of the weighting agent, plastic and apparent viscosity, compressibility strength and compatibility tests, fluid loss and free fluid tests, specific gravity, yield point and thickening time were examined. These physico-chemical, rheological and structural properties were compared with the Combined API/Company Specifications for Barite, the API Recommended Practices 13B-1 and 13B-2 for water-based drilling fluids. Prior to the laboratory tests and analysis, a field study was done for four (4) weeks at the polyguard Investment Nigeria limited, Hungwa, Torkula, Guma Barite field. There, the mining and processing activities such as size reduction and control (screening), enrichment (washing and separation), upgrading by sedimentation and material handling (loading, unloading and conveying) were studied for future enhancement.

To this end, as a way of summary, conversely:

- ❖ Chapter 2 significantly reviews previous work on Nigeria barite with envances on research focusing on the Benue through. This is focused on the introduction to the field characteristics and quality issues of Barite Vein in the Torkula, Guma field of the Benue Trough, the exploration and exploitation of barite minerals around the region. It also understudies the characteristics of barite mining, the operation and effect of the ASM in Nigeria. Elaborate consideration was given to the basics of indigenous mining and processing, in comparison to the API Specifications. This forms the research framework for an extensive outlook.
- ❖ Chapter 3 summarily discussed the proceedings for sample preparation, materials requirement and methodologies employed in the characterization of Barite from Turkula Field, measurement of relevant physico-chemical and rheological properties.

- ❖ Chapter 4 presented, discussed and analyzed the results of the entire work.
- ❖ On a significant note in Chapter 5, the research work was concluded, valuable recommendations were made, and solutions were proffered to the identified problems. Similarly, brief details of future significance on beneficiation methods were highlighted, with sufficient data for possible further analyses on the subject matter.

## 2.0 Literature Review

### 2.1 General Introduction

Barite or Baryte is the foremost mineral of barium with chemical formula  $\text{BaSO}_4$  in the proportion of 66%  $\text{BaO}$  and 34%  $\text{SO}_3$ . It is a naturally-occurring barium sulphate ( $\text{BaSO}_4$ ) used most predominantly for industrial purposes. It owes this industrial value to its heaviness; with a specific gravity of 4.5, and its unusually heavy non-metallic mineral. The mineral may occur in veins, stratiform beds and lenses in addition to residual deposits. The largest deposits currently mined are stratiform beds in China, India and the US. In Nigeria, the three major geological types of barite ore deposits occur in the form of vein, cavity filling and residual or bedded deposits. Barite seldom exists in nature as a pure end member but rather as solid solutions. This may appear to be continuous between baryte and celestine ( $\text{SrSO}_4$ ) (Hanor, 2000; Zhu, 2004; Monnin and Cividini, 2006) but then discontinuous with anhydrite ( $\text{CaSO}_4$ ). The substitution of Ba in barite ( $\text{BaSO}_4$ ) with other cations (such as  $\text{K}^+$ ,  $\text{Mg}^{2+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Sr}^{2+}$ ,  $\text{Ra}^{2+}$ ,  $\text{Pb}^{2+}$ ,  $\text{La}^{3+}$ ,  $\text{Ce}^{3+}$ ,  $\text{Lu}^{3+}$ ,  $\text{Eu}^{2+}$ ) hinge on its degree of similarity in ionic charge, ionic radius and electronegativity (Hanor, 2000). Barite is typically associated with one or more of the following minerals: celestine ( $\text{SrSO}_4$ ), galena ( $\text{PbS}$ ), sphalerite ( $\text{ZnS}$ ), pyrite ( $\text{FeS}_2$ ), quartz ( $\text{SiO}_2$ ), calcite ( $\text{CaCO}_3$ ), dolomite ( $\text{Ca,Mg}(\text{CO}_3)$ ), marcasite ( $\text{FeS}_2$ ), chalcopyrite ( $\text{CuFeS}_2$ ), fluorite ( $\text{CaF}_2$ ), siderite ( $\text{FeCO}_3$ ) and witherite ( $\text{BaCO}_3$ ). China is the foremost producer of barite in the world followed by India with the Mangampet deposit in Cuddapah district of Andhra Pradesh being the single largest deposit in the world (Nwoko and Onyemaobi, 1997; Searls, 1996; Oladapo and Adeoye-oladapo, 2011; Ngukposu, 2015). The distribution of major barite deposits in some parts of Nigeria is presented in Figure 2.1.

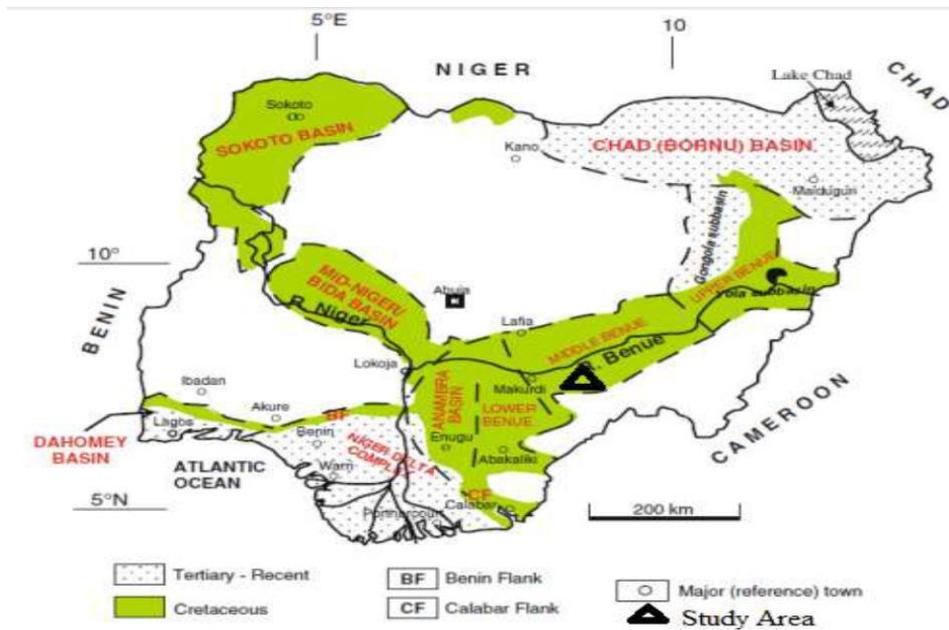


Figure 2.1: Map of Nigeria showing the Barite mineralized Zones around the Benue Trough (Source: Ehirim et al., 2016)

Torkula barite of Guma field in Benue State holds a lot of promise, even though its known veins are currently fewer than in Azara field. This field is made up of Torkula, Kaseyo, Zanzan, Iye, University of Agriculture campus, at Makurdi, etc. Guma is a local government area in Benue State of Nigeria with the total land mass of about 28,882km<sup>2</sup>. It is surrounded with sedimentary rocks and the soil is dominantly associated with mineral deposits like lead, salt, barite, feldspar, and brown earth from volcanic eruption. Guma lies between latitude N7 32 and N8 51 and longitude E9 35 and E9 22 with an average high rainfall of about 1198 – 1798mm annually. Relative humidity is between 43 – 86%, temperature ranges from 27°C and 37°C (Sombo et al., 2016). The highest concentration of barite veins in this field is within and around Torkula because of its size. However, there is barite quality contrast between the top part and the lower portions of the vein. Higher quality barite tends to come from the lower portions, which increases with depth. The quality of the Nigerian barytes is moderate to high, with fewer cases below the minimum global specification. This disparity is due to major impurities such as quartz, iron oxide (goethite), fluorite, and carbonates of iron, calcium and magnesium. These impurities incline to reduce the specific gravity of the unprocessed barytes, increase the cost of the processing and the wear rate of the mills rapidly.

The goethite and silica impurities can be removed by magnetic and gravity separation. Once processed the specific gravity of the Nigerian barytes increases and meets 4.20 specified values by American Petroleum Institute (API) (Oden, 2012; Fatoye et al., 2014; API, 2004, API, 2010).

Weighting agents are the most abundant component of drilling mud (Neff, 2005). They are used to prevent blowouts by sustaining the borehole pressure (Sadiq et. al., 2003). This is obtained by mixing barite in water with other materials to form a mud, and then pumped into the drill hole. The weight of the mixtures counteracts the force of the oil and gas when it is released, which allows the oil and gas rig operators to prevent the explosive release of the oil and gas from the ground. The levels of barite within a drilling mud increase from use at the seabed surface to the bottom of a deep well, increasing from roughly  $6.3\text{kg/m}^3$  to  $2000\text{kg/m}^3$  respectively (Neff, 2005). Primary barite used as weighting agent, includes crude barite and products of simple beneficiation methods, such as washing, jigging, heavy media separation, tabling, flotation, and magnetic separation. This mostly requires grinding to a small, uniform size prior to its application as a weighting agent in petroleum well drilling mud (API or OCMA specification barite). Barite used for drilling can be blue, black, brown, or gray depending on the associated gangue minerals. However, it is also expected that such barite is finely ground, dense, soft, and chemically inert. In addition to these, the American Petroleum Institute (API) specifications recommended SG of 4.2 or more, an allowable percentage of iron oxide, free-soluble salts, and that 90% to 95% of the material must pass through a 325-mesh screen. Similarly in offshore drilling, the U.S. Environmental Protection Agency limits the content of mercury and cadmium to 1 milligram and 3 milligrams per kilogram of barite respectively (Sadiq et al., 2003; Strachan, 2010; Searls, 1996).

## **2.2. Field Characteristics and Quality Issues of Barite Vein in the Torkula, Guma field of the Benue Trough**

Several works have been done on barite deposits and mineralization around the Benue Trough but the huge deposits and high quality minerals in Torkula Village areas of Guma LGA still remains as one of the endowed secret of the Benue trough. Presently, the area has three barite mineralized zones, one galena mineralized zone and many other mineralized zones yet to be discovered. Out of these three locations, only one has been leased to an

indigenous investor. This site was leased to Polyguard Investment (Nig.) Ltd in conjunction with the Benue State Investment Subsidiary in 2007. Artisanal mining activities commenced in 2011, and have dug three (3) pits. The quality of barite and other associated minerals explored from these pits has attracted the attention of the Minister of Solid Minerals, Ministry of Mines and Steel Development (MMSD) and the Benue State Ministry of Mines and Steel Development. The estimated resource calculation done by the Nigerian Geological Survey Agency and National Metallurgical Development Centre, Jos, revealed a reserve estimate of over 300, 000 tons/m<sup>3</sup> and about 240, 000 tons/m<sup>3</sup> for the second and third pits respectively. The total depth of the mine pit is 40m, having a total mine opening face of 63m and an average depth of 19m as reserves for the ore. However, the level of mining has reached a depth of 35m, with much of the minerals unexplored. No further research work has been done on the site other than the mining technical report. Similarly, the geochemical analysis of the minerals deposits on the site has revealed the presence of barite and galena, with a specific gravity of 4.21 and 7.11 for barite and galena respectively, at a depth of 6m. Below this depth, the analysis revealed a SG of 4.5 and 7.4 for barite and galena respectively (PIN, 2017; Sambo et al., 2016; Afolayan, 2017).

### **2.2.1. Types of Occurrence, Host Rocks and Distribution of barite Mineralization**

The mineralization of Torkula barite is a vein-type, displaying distinct features suggestive of open-space deposition and cavity fillings such as large, interlocked, ephedra crystal growths, cocks-comb structure and mineral inclusions. Host rock to the mineralization is the ubiquitous sandstone and shale within the prospect. The trend of these mineralized veins was observed along the NW-SE axis, with steep dips to the southwest. These are by far the most consistent rock types that host barites in the trough. In addition to these, basement gneisses have been observed to host barite and fine grained granite. The persistence of barite veins into the basement complex beneath the Cretaceous cover is an indication of both the depth and width of the Cretaceous deformation, resulting into residual or bedded deposits. Further review on Benue Trough mineralization confirms the evidence of wall rock alteration, mineral zoning, structural relations and stratigraphic controls. The area has undergone a pronounced tectonic activity resulting in shearing and fracturing. However, the sheared zone and fractures are potential channels for mineralization fluids. There are interceptions of both

pure and intercalated barite at various depths between 16m and 23m, having over 250, 000 tons/m<sup>3</sup> ore deposits at 70% ore concentrate (PIN, 2017; Afolayan, 2017).

### **2.2.2. Barite Field and Attitude of Barite Vein**

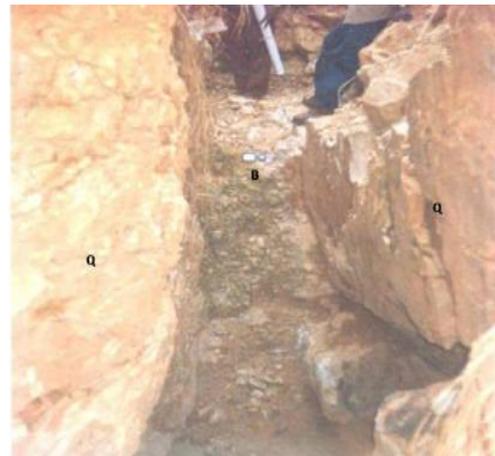
Torkula barite of the Guma barite field is made up of Hungwa village in the North, Western and Southern part of Torkula area. This field has the highest concentration of barite veins at a point amidst other fields around and within the Benue Trough. Currently, the known vein and reserves is fewer than in Azara field. However, it holds a lot of promise and the quality of minerals are within and a little above the API Specifications. The barite occurrences in Hungwa of the Torkula barite field reveals summary of the entire existing position of barites mining activities in the area within the trough. Consistently, every “ROM” mined and processed from the site tend to have high specific gravities of 4.2 and above, and as such are useful for blending to upgrade the lower grade materials obtained from Nasarawa and other mines such as Alosi, Azara, Wuse, Kuduku, Pupule, Apawa, Tombu and Bunde lessel. This development guarantees that Nigeria does not actually need to import high grade barite from foreign countries. In the same vein, there are more mines producing above the API grade materials than those producing below API grade barite. The observed zonation in the magnetic data and the potassic alteration in the ternary image are indication of hydrothermal alteration associated with mineralization. These zones has high potassium level with a low thorium/potassium ratio anomalies, indicating potassium enrichment associated with sericitic, K-feldspar, sandstones, shale, calcite, hematite and pyrite alterations. The regional and localized aeromagnetic anomalies with associated lineaments are the target for Barites and Lead-Zinc veins (Oden, 2012; Afolayan, 2017).

Barite veins in the Torkula barite field incline to be persistent, even if discontinuously so. The vein at the first pit is stratified and can be traced discontinuously for up to about 6km, while a section of the second has been traced geographically for up to 3km. On the other hand, the vein has indicated a length of 1km, as observed from a large scale map of the mining site produced by the Nigerian Geological Survey Agency (NGSA, 2010). The most prominent mineral vein trend is that in the NW-SE quadrants, orthogonal or nearly so, to the axis of the Benue Trough (N7° 12'54"/E9°0'22'). The dips of barite veins may be high (Figures 2.2 and 2.3) of the order of 80° to 90°, a condition that favours manual and

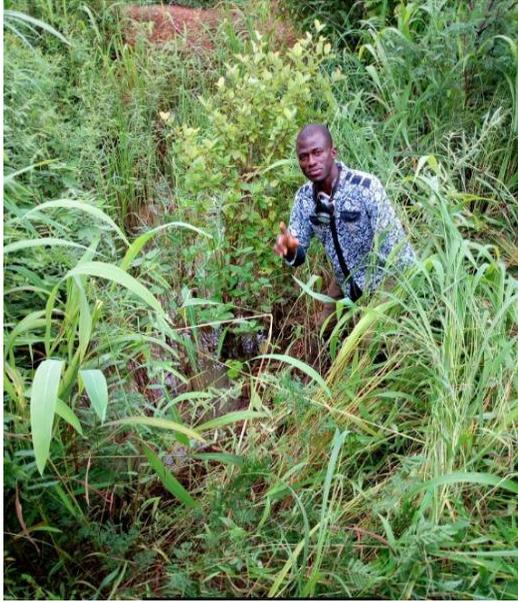
mechanized mining. Low angle veins are problematic to artisanal miners since they have to continue to strip the overburden to keep their operations on the surface. They also seem to result in more extensive damage to the environment when mined, than vertical veins (Oden, 2012). The very brittle barite veins and host rocks in Torkula areas are occasionally fractured, whereas secondary minerals like quartz, feldspar, hematite, calcite and pyrite fill the fractures to act as binding cement. This is partly responsible for the high quality of barite from the pits. The most dominant vein structure in profile is the residual or bedded and vertical block structural type. Barite in a vein tends to fold and solidifies into a block irrespective of the size of the block. The most frequently occurring vein width is between 1m and 30m. The wide veins (4-25m) are by far more frequently occurring than wide veins (1-2m). This has fluid pressure and  $BaSO_4$  availability implications for the mineralizing fluids during the Cretaceous period. Barite mineralization in the barite field is associated with quite a range of minerals to varying degrees, such as half-half associations between barite and galena, sphalerite, hematite, calcite, lots of quartz and feldspar. The most pure and unassociated barite that is transparent and clear dominate the site (Sambo et al., 2016; Afolayan, 2017; PIN, 2017).



(a)



(b)



(b)



(d)

Figure 2.2: barite Mines at Hungwa, Torkula barite field, Jobe mine, Seriki Farm, Azara, showing high angle (90o) dip of the vein and Alosi main vein, where the barite B is sandwiched between microcrystalline quartz Q (Source: Afolayan, 2017, Oden, 2012)



Figure 2.3: Mine barite Pit 2 at Hungwa, Torkula barite field (Source: Afolayan, 2017)



Figure 2.4: Mine barite Pit 3 at Hungwa, Torkula barite field during the enrichment and upgrading process (Source: Afolayan, 2017)

### **2.3 Exploration and Exploitation of Barite Minerals**

Exploitation of mineral resources has anticipated leading significance in several developing countries including Nigeria. Most importantly, Nigeria is endowed with abundant mineral resources, which have contributed immensely to the national wealth with associated socio-economic benefits. Mineral resources amidst other reserves have to pass through the stages of exploration, mining and processing before they are harnessed (Adekoya, 2003; Ajakaiye, 1985). However, there are different types of environmental damage and hazards that inevitably accompany the three stages of mineral development. Relatively more recently in the oil and gas industries, barite, limestone, marble and rock aggregates have been playing an increasing role in the national socio-economic development and growth. They generate appreciable internal revenue and foreign exchange earnings. Moreover, this have surpassed other economic minerals by generating over 90% of the export earnings, which is more than 50% of the national revenue. To a large extent, the scale of operations involved in exploration, mining and processing of a mineral determines the intensity and extent of environmental degradation (Aigbedion and Iyayi, 2007).

As the wealth from hydrocarbons and crude oil reserves continue to diminish with the growing uncertainty in global demand and supply, all attentions are now being fixated on solid minerals as alternative sources of revenue and employment for the teeming population. Similarly, most solid mineral occurrences in Nigeria are located in the Benue trough (Tate, 1959; Offodile, 1976). Farrington (1952), Tate (1959) and Offodile (1976) in Ehirim et al., (2016) have reported the occurrence of barite as an epigenetic deposits arising from products of hydrothermal activity at shallow depths and low temperature. However, they may occur either as massive vein bound deposits or gangue minerals associated with lead-zinc mineralization in fractures, fissures, and open space fillings in sedimentary and igneous rocks. These veins are exceptionally varied in character and form, well developed in hard sandstone units and less likely in shales.

The exploration for near-surface mineral deposits has turn out to be more difficult due to the complexities in geology. However, several geophysical techniques have been adopted in the search for geobodies associated with mineralization, as required to identify areas of ore mineralization (Bishop and Emerson, 1992; Maxwell, 2002). Each of these techniques depends upon detecting variations in one or more of the physical properties of rocks such as Electrical resistivity, Density, Magnetic susceptibility and Seismic velocities, which varies within wide limits. Barite has a high resistivity compared to its host rock. This makes it easily detectable by the electrical method because of the resistivity contrast with the host rock. Electrical resistivity methods have variously been used in the exploration of barite veins by several researchers. Results of these studies revealed that high resistivity signatures were diagnostic of barite vein structures and depend on the  $\text{BaSO}_4$ , to which barite mineralization could be associated (Ehirim et al., 2016).

### **2.3.1 Barite Deposits and Quality in Nigeria: An Extensive Outlook**

In Nigeria, the barite at the center of barite vein is considered to be of pure grade while barite at the contact with the wall rock is considered moderate. The high grade varieties can be used in oil and gas industries while the low grade can be used in paint and glass industries. This classification into two grades is done in terms of quality and specification for use in many industries. These include grade 1 containing the smoky and whitish varieties with a minimum of 92%  $\text{BaSO}_4$  and grade 2 containing the pinkish varieties with 77%  $\text{BaSO}_4$ . However, the

barite produced in Nigeria to a large extent, is yet to meet the standards specified globally, based on NPDC's experience. Consequently, such barite is normally delivered in bags of 1.5MT as bulk materials in trucks or vessels, depending on the terrain of operation (Ayim and Enoch, 2009; Ene et al., 2012).

A typical barite veins in Nigeria appear to have its origins in the basement complex beneath the Cretaceous sedimentary cover. This makes the mineralization more epigenetic, the vein orientations are strongly and structurally controlled on the basis of its formation and in a parallel direction to the prevailing maximum compressive stress. Similarly, such orientations enable grain growth and vein expansion to occur parallel to the low energy orientation. However, the presence of the two sets of barite vein trends in the Benue Trough is, as yet, one of the greatest incontrovertible evidences of two phases of post sedimentary deformations in the trough. The higher occurrence of narrow veins over wide ones specifies that hydrothermal fluids were not rich in barium sulphate, and were generally of low pressure or low volume in the trough. Consequently, more than 95% of barite occurrences observed in the trough are discordant mineral veins and is less frequent. For large-scale monetization of barite mining in Nigeria, several promising fields have been identified. Beyond these fields, there are more other veins producing medium to high quality barite than those of low quality material as clearly shown in Table 1. In addition to these, practicing proper and adequate batching of barite by indigenous oil service companies is critical to address the need for high grade barite. (Oden, 2012; Ene et al., 2012).

Table 2.1: Geotechnical and geochemical Properties of Barite Ores from Gabu-Alifokpa Barite Fields (Source: Ene and Okogbue, 2016).

Sample Location	Vein Depth (m)	Specific Gravity	Porosity (%)	Water Absorption Capacity (%)	Moisture Content (%)	Uniaxial Compressive Strength (N/mm <sup>2</sup> )
Gabu						
A	0-32	3.1 <sup>a</sup>	0.3	8	0.3	43
B	33-36	3.2	0.2	2	0.8	38
C	37-45	3.6	0.2	7	0.5	23
D	46-48	4.2	0.1	6	0.2	20
Alifokpa						
A	0-29	2.9	0.5	12	0.4	40
B	30-34	3.3	0.3	10	0.6	34
C	35-43	3.9	0.2	9	0.4	14
D	44-47	4.5	0.1	2	0.2	11
Osina						
A	0-24	3.1	0.2	5	0.24	39
B	25-35	3.7	0.4	11	0.34	31
C	36-37	3.7	0.4	11	0.45	18
D	38-39	4.3	0.3	4	0.21	12

### 2.3.2 Barite in Taraba State, Nigeria

Taraba State in Nigeria constitutes major part of the Middle Benue Trough and hosts barite in five of its LGAs, namely: Ibi, Lao, Yoro, Sardauna and Karim Lamido. This barites resource is compered in the igneous-metamorphic rocks of the Pre-Cambrian, also in sandstones, shale, mudstone, siltstone and limestone of the Benue Trough Sedimentary Formations (Fatoye et al., 2014). However, its mineralization and deposition are in the form of fissure filling with hydrothermal solutions formed by the closing in of the Benue Trough. At these mineralized zones and locations, the resource is hosted in porphyritic granites and fine grained sandstones. Similarly, vein length persists and extends over 3.5 to 5km range, whereas its width ranges from 3.5 to 5 meters. To process high grade barite for industrial applications, impurities such as Quartz and sulphide minerals like galena and sphalerite are properly separated in order to enrich the barite mineral (MMSD, 2010; Ebechidi, 2014). The inferred barite resource base in Taraba State, Nigeria is approximately 8,960,000 metric tons, measured to 20m depth or more. Most significantly, the quality of deposit is appropriate for the API Specifications; with most of the resource having SG close to 4.2. Considering the size of individual veins present, investment in heavy equipment to mine at greater depth is considered valuable and productive (MMSD, 2010).

### 2.3.3 Barite in Nasarawa State, Nigeria

In Keana and other areas in Nasarawa State, barites occur in the form of veins having a width varying between a few centimetres to 3.5 metres, and in length between just under 1.0 to over 4.0km. Geotechnical and geochemical analyses conducted on samples from those areas revealed that most samples contain between 76% and 87% BaSO<sub>4</sub>, and that specific gravity of mine materials varies between 3.9 and 4.4. Similarly, this grade range is within the 4.2 minimum specification of the American Petroleum Institute (API) (MMSD, 2010). However, some tested samples with 3.6 SG or less were as a result of their richness in silica, and other gangue minerals which include quartz, celestite (SrSO<sub>4</sub>) and iron oxide (Fe<sub>2</sub>O<sub>3</sub>). To improve on its suitability and proficiency in diverse applications, adequate enrichment and upgrading techniques are properly implemented. The assessed amount of barite resource in Nasarawa is put at 3,243,376 metric tons, considered at an average SG of 4.0 and a projected vein depth of 20m (MMSD, 2010). In addition to all these, some sections reportedly contain fluorite as a gangue mineral in barite which is traceable to sandstones of the Keana Formation of Cenomanian age (Ebechidi, 2014, Fatoye et al., 2014).

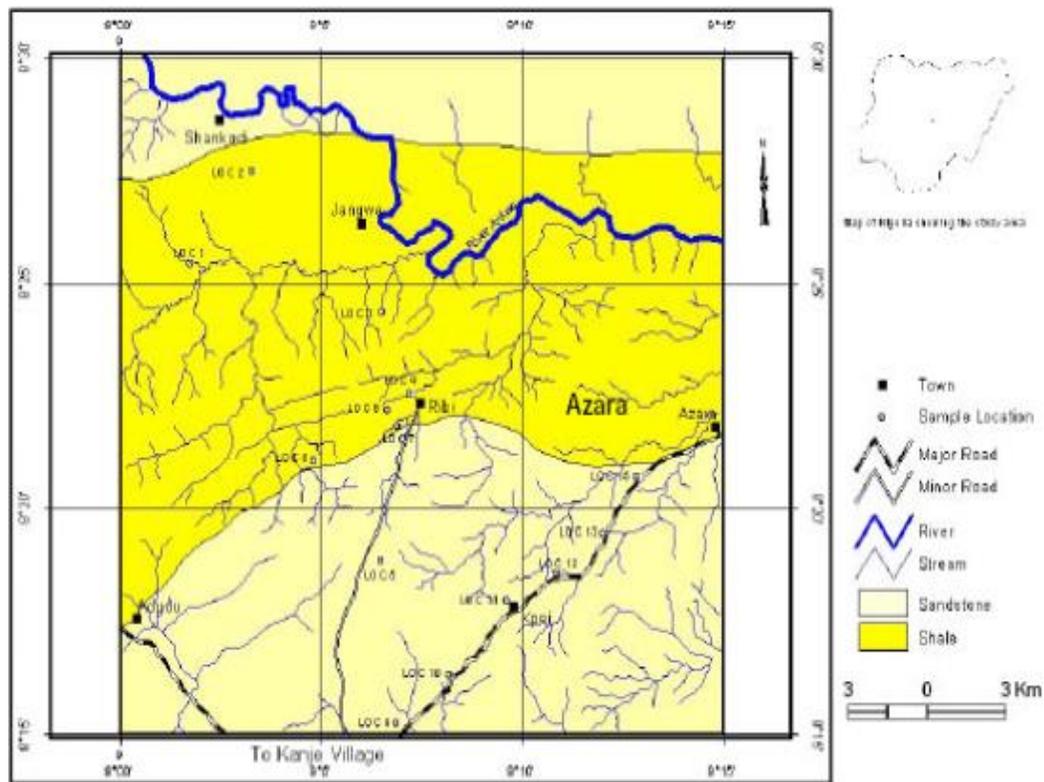


Figure 2.5: Geological map of part of Akiri sheet 232 N. W., Keana, Nasarawa State

### 2.3.4 Barite in Plateau State, Nigeria

The barites deposit in Azara occurs as hydrothermal veins within the Cretaceous Keana sandstone of the Middle Benue Trough. Faya with the best quality barites in the state where a prominent well developed vein, was estimated to contain about 500,000 metric tons of resource, having the specific gravity varying between 4.0 and 4.2. This best known deposit of barites in Nigeria has about twenty identified hydrothermal veins. These mineralized veins were found to contain Calcite, Fluorite and Celestine ( $\text{SrSO}_4$ ) as gangue minerals, striking generally in the NE-SW, NW-SE and E-W directions (Chaanda et al., 2010). However, the presence of these impurities has lowered the SG. In order to make a balance of these properties, available data have revealed that such deposits contain very low mercury (Hg) and cadmium (Cd) value making it very suitable for drilling offshore (MMSD, 2010; Fatoye et al., 2014).

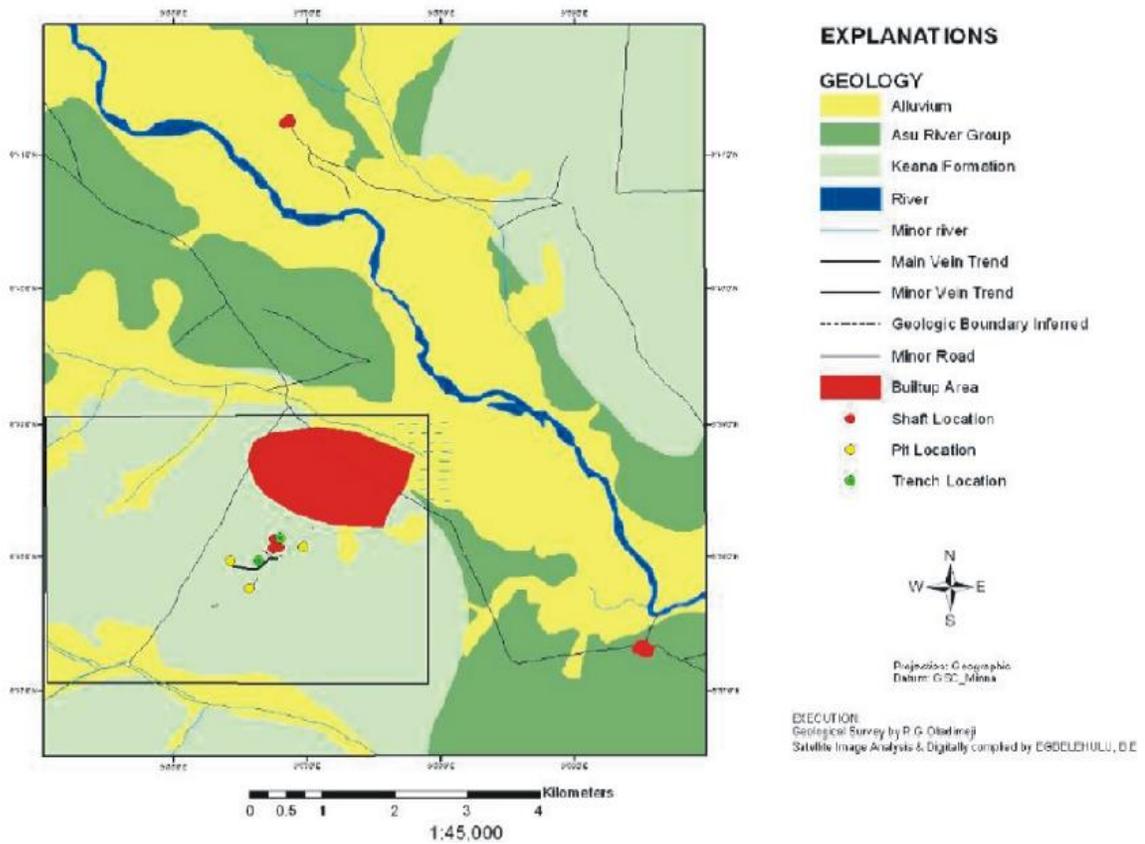


Figure 2.6: Barite occurrence in Plateau State, Nigeria (Source: MMSD, 2010).

### **2.3.5 Barite in Cross River State, Nigeria**

Within the Lower Benue Trough, Cross River State in Nigeria has over 35 barite mineralized locations, of which 11 are in the sedimentary areas. Several geological mappings, geotechnical and geochemical analysis carried out by the Nigerian Geological Survey Agency and Nigerian Metallurgical Survey Agency revealed that the mineralized areas hosted both hard and soft rocks. These areas are classified into two; namely the south consisting of Yakkur and Biase LGA and the north comprising of Ikom, Yala and Obubra, LGA. Out of the seventeen mineralized locations obtainable in the south, barites is compered by sedimentary rock in nine locations, and by basement rocks in eight locations. Among the remaining eighteen mineralized locations in the north, only two locations is hosted by sedimentary rocks, and the other sixteen different locations are housed by the basement rocks (MMSD, 2010, MMSD, 2013; Fatoye et al., 2014).

The sedimentary rocks in the northern part of Cross River State host more of the barites mineralized areas and veins than in the south. However, resource extraction base in the northern zones is easier than those in the south as a result of the presence soft host rock as compared to the hard rocks. Inferred resource for the entire state is huge and promising, having a specific gravity ranging between 3.5 and 4.4, and a reserve base of over 8,612,880 metric tons distributed almost evenly between the north and south. This estimate is most likely to increase as new veins are discovered in the future, mining in the state is becoming more viable, and its proximity to the oil and gas industries than other locations in the country is of great advantage (MMSD, 2013, Ebechidi, 2014).

### **2.3.6 Barite in Benue State, Nigeria**

Barites mineralization in the Middle Benue Trough occurs in Gboko, Guma, Torkula, Hungwa, Gwer, Ushongo, Markudi, Konshisha. In Benue State, barite deposit is contained in both the Pre-Cambrian igneous-metamorphic rocks and the Benue trough sedimentary formations consisting of sandstones, mudstone, siltstone, limestone and shale formations. This barite deposits occur in the form of hydrothermal solutions filling fissures with barites, and are formed by the closing in of the Benue trough during the Santoninan (MMSD, 2010).

The barite mineral may occur as white, reddish-brown and clear varieties, with Specific Gravity (SG) varying between 3.7 and 4.4 (Fatoye et al., 2014; Ebechidi, 2014).

Geochemical analysis carried out by several agencies such as the Ministry of Mines and Steel Development (MMSD), Nigerian Geological Survey Agency (NGSA) and Nigerian Metallurgical Survey Agency (NMSA) revealed that most barite samples from various locations contain between 76 and 87% BaSO<sub>4</sub>, 5 and 21% Silica (SiO<sub>2</sub>) and up to 3% iron oxide (Fe<sub>2</sub>O<sub>3</sub>). Similarly, barite veins in these locations are usually 3m wide and 20m deep or more. Nevertheless when exposed, they do not persist over long distances. The huge market, quality materials with an average Specific Gravity of 4.0 and the estimated reserve base of 307,657 metric tons makes barite mining and processing a viable investment (MMSD, 2010, MMSD, 2013).

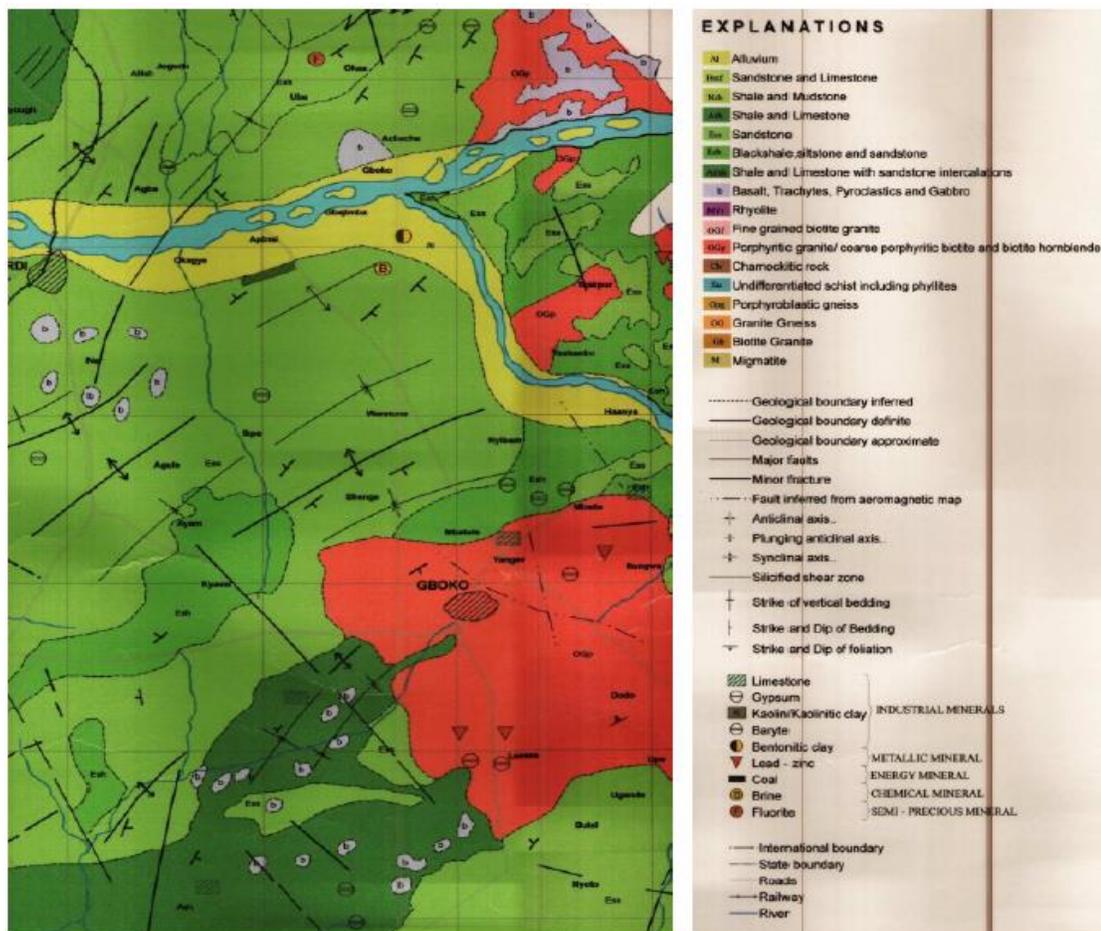


Figure 2.7: Barite occurrence in Benue State, Nigeria (Source: MMSD, 2010)



Figure 2.8: Barite samples from Hungwa, Turkula barite field (Source: Afolayan, 2017).

### **2.3.7 Barite in Gombe State, Nigeria**

Within the Upper Benue Trough, the mineralized areas in Gombe State such as Shongom, Daba Fulani, Dodin Kowa, Gombe and Liji hills has barite mineral being hosted in gneiss-migmatite complex and coarse sandstone. Similarly, the thicknesses of the barite veins do vary between 0.3 metres and 1.2 metres and some reaching 1.5 metres; while others can be drawn along the strike for up to 400 metres. Due to the presence of dust and other gangue minerals, the colour of the barites may be creamy to grey-white, with specific gravity between 4.09 and 5.3. These gangue minerals are fluorite, quartz and chalcopyrite. However, major element analysis estimates BaO content to be between 45.00% and 59.5%, and the inferred resource base for Gombe and Liji hills is 352,800 metric tonnes (MMSD, 2010, Fatoye et al., 2014).



into an uneven fractures and differences in colour, from white to the reddish brown due to the size of the granular and associated gangue minerals (MMSD, 2010).

Within the Upper Benue Trough, barite mineralization in Adamawa and Bauchi States occurs in Gban and Mayo-Kpoki; and Isimiya, Diji, Gidan Dari, Alkaleri and Gwana respectively. The geology of the barite mineral in Gban and Mayo-Kpoki among others is allied with fissure and cavities infilling by hydrothermal fluids, associated with faults and fracture zones of the Upper Benue trough. The colour of the mineral varies from white to pink, while some have vitreous luster with specific gravity values varying between 4.0 and 4.36. This is due to the presence quartz veins hosted by sandstone striking the N-W host of the mineralization at Isimiya, while the veins at Gidan Dari are hosted in grey shale member of the Yolde Formation. The projected resource base of the state is at 332,130 metric tonnes (Fatoye et al., 2014).

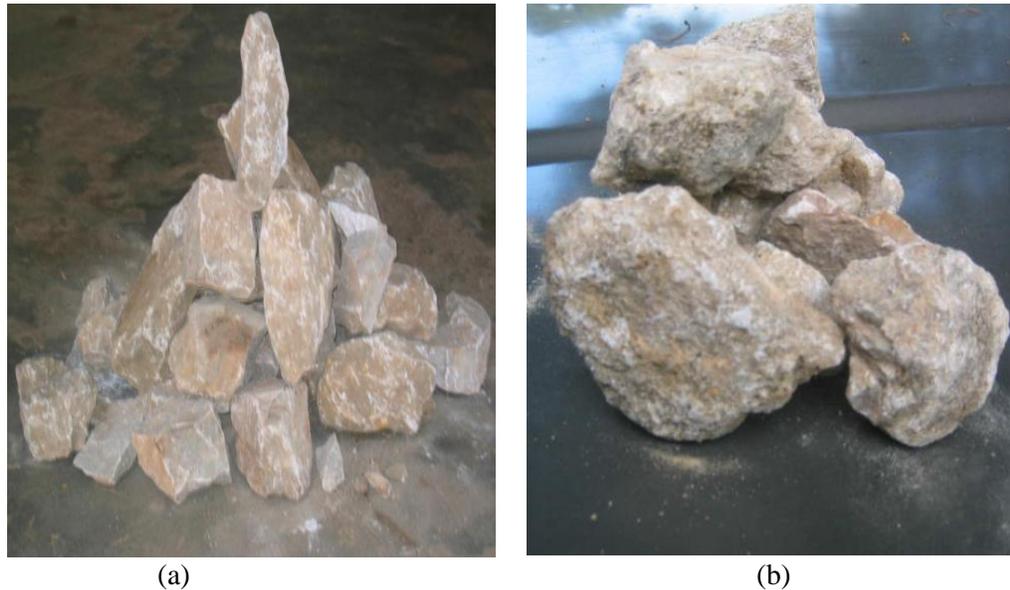


Figure 2.10: Massive barite and Granular barite from Deretta and Rekebu near Chafe

#### **2.4 Barite Mining and Characterization**

Barite and other solid minerals in the form of run-of-mine (ROM) exists primarily with at least one associated mineral either in the elemental or compound form. This is due to the presence of the dissolved minerals during leaching or movement of hydrothermal fluids. To determine the presence and the percentage composition of these minerals, the elemental

fraction, and the phase, structural and morphological analysis of the sample is critical prior to the beneficiation and application of the materials. In order to measure the level of hazardous and harmful gangue minerals, the nature of the metallic and non-metallic minerals, design and fabricate an optimal processing plant, the chemical compositions of the various deposits are to be characterized. These scientific processes provide the basis for what equipment is needed in the beneficiation train assembly, necessary to improve physical or chemical properties in preparation for further processing (Ebechidi, 2014; Grigorova et al., 2015).

However, the barite deposits and their constituent impurities in Nigeria vary from one place to another and the concentrations of BaSO<sub>4</sub> in hydrothermal fluids in the trough were conceivably low during the Cretaceous period. In many veins, there is barite quality contrast between the top part and the lower portions, and can either be the major or minor fractional component of the mineral. Such beneficiation techniques comprise of crushing, screening of particulates, enrichment, upgrading and materials handling. These can be carried out on the valuable minerals in micronized form. In many specialized operations, various distinguishing properties of the minerals such as magnetism, wettability and density are exploited to concentrate the desirable components. This solely hinge on the grade of the ore, the projected application of the minerals, nature of gangue and liberation size. Most importantly, heavy media separation and jigging are generally employed on high grade and coarsely liberated ores while gravity concentration techniques are adopted to produce intermediate concentrates. Similarly, flotation is followed where the ore is finely disseminated (Grigorova et al., 2015; Ofor and Nwoko, 1996).

#### **2.4.1 History and Background of Mining in Nigeria**

Organized mining in Nigeria started in 1903, and around 1939, private owned foreign companies were fully involved with the mining of minerals such as tantalite, columbite and coal. This progression in mining activities necessitated regulations, such as the Minerals Ordinance and Coal Ordinance, enacted in 1946 and 1950 respectively. These enactments provided the base for the establishment of various government agencies, leading to their reviews in 1999. The discovery of oil in 1958, the nationalization policy and energy crisis in the 1970s had resulted into the collapse of big mining companies and massive unemployment

of mine workers without any means of livelihood. Many of them went into illegal mining activities having seen no hope of diversifying the nation's economy. However in recent times, the government, in its pursuit to diversify the nation's economy, created the Ministry of Solid Mineral Development, which was later changed into the Ministry of Mines and Steel Development (MMSD). Similarly, to attract foreign direct investment to the solid minerals sector, the federal government has sought to increase exploration activities and global demand for solid minerals through the creation of a National Geological Survey Agency (NGSA) and Structural Adjustment Programme (SAP) (NGSA, 2010; MMSD, 2010).

The Mining Cadastral Office (MCO) was also created for the purposes of administering mining titles, to oversee the enactment and enforcement the Nigerian Minerals and Mining Act of 2007, National Mineral and Metals Policy of 2008, and the Minerals and Mining regulations of 2011. However, the official and prompt response to the menace in the mining industries was wild, yet not sustainable. Illegal mining activities have continued to flourish uncensored due to the ineffectiveness of the relevant agencies and lack of alternative gainful employment. Government of Nigeria can no longer ignore these illegal activities and other associated instabilities because of the huge revenue loss and the attendant environmental degradation. Ibrahim et al., (2010) and Warhurst, (1994) emphasized that mining involves the extraction of any non-renewable resources which covers all aspects of metal production, such as mine development, extraction and smelting, re-mining and waste management. It is a process through which man earns minerals from the earth and turns them into valuable goods for his use (Alokolaro, 2012).

#### **2.4.2 Artisanal and Small scale Mining Operation**

Coppin (2005) in the final report by Wardell Armstrong, emphasized that Artisanal and Small-Scale Mining (ASM) operations dominate mining in Nigeria, particularly from around 1903 up till 1940s. Similarly, it is very evident of artisanal mining characterizes barite mining in Nigeria. Generally speaking, artisanal and small-scale mining refers to mining by individuals, groups, families or cooperatives with minimal or no mechanization, often in the informal (uncensored) sector of the economy. However, this activity turned into a well-paid business prior to the abandonment of the mechanized method of mining in the 80s. Ever since

then and unto the present, the exploration and exploitation (extraction) of barite in Nigeria has been manually characterized by the use of primitive tools such as hammer, chisel, diggers and shovels usually on a small scale. Under these circumstances, certainly only the surface or near surface veins are exploited, while local miners work is highly risky, resulting in the loss of lives and property (Warhurst, 1994).

At the present, situation in the ASM sector in Nigeria has been described by some as utterly chaotic, with virtual breakdown in law and order in the mining areas since the dissolution of the mines field police force. Similarly, the exploitation of barite within the Benue Trough is done indiscriminately either by individuals or groups. Nevertheless, there appears to be some coordination in the manner in which each individual or groups go about the exploitation. The environmental impact and devastation of arable farmland are great, in addition to the presence of very large and deep pits left behind after exploitation which could form death traps (Chaanda et al., 2010). On a positive note, artisanal and small-scale mining in Nigeria is a significant sector with potentials to providing livelihood for millions of people and producing a sizeable proportion of the world's extractive commodities. It is an important sector to the international community, which has the potential to provide substantial benefits to efforts focused on reducing poverty and stimulating economic growth. This is necessary for political and economic stability especially to solid mineral endowed developing nations such as Nigeria and other Africa countries (Adiawe, 2011; Ibrahim et al., 2010).



Figure 2.11: Very large and deep pits (brine) left behind after exploitation at a mining site within the Benue Trough (Source: Afolayan, 2017).

### **2.4.3 Advance Mining Operations**

Barite deposits and minerals' distribution can be classified into four (4) categories namely; the layered type deposit, vein type deposit, transformed vein type deposit and accumulation type deposits. Similarly, commercial barite mining includes open-pit mining and underground mining with the former being the most popular method. In open-pit mining, barites are usually mined at residual talus deposits and outcrops and shallow depth ore deposits. In underground mining, barites are usually extracted through the shrinkage method, filling method and stage caving method. The application and use of explosives to extract and process the raw materials are now being adapted in advanced mining operations (Ebechidi, 2014).

These extraction processes determine the degree and level of environmental degradation and its effect to a large extent. In addition to these, such introduction of dynamites (explosives) had resulted into cracks on building, posed high security issues and concerns due to the illegal and un-licensed applications. The mining equipment used in underground mining consists of ore trucks, inclined shaft hoist, local exhaust ventilation fan and underground support. Machines used in open-pit mining include power shovels, draglines, excavators, loaders and dump trucks to haul to crushing sites. Similarly, the broken ore is trucked to the processing plant where it may be enriched and upgraded by log washer or trommel screen. These stages remove adhering clay and low-grade fines prior to the separation of the gangue minerals and subsequent size reduction by jaw crusher, as shown in Figure 2.12 (Adiawe, 2011; Afolayan, 2017).



(a)



(b)

Figure 2.12: Trommel Screen at a mining Site in Turkula barite field, Hungwa mining site (Afolayan, 2017).

## 2.5 Basics in Barite Processing

### 2.5.1 Introduction

The basis and practice of minerals processing is as old as human civilization itself. Minerals and products derived from minerals have formed our development cultures from the flints of the Stone Age man to the uranium ores of Atomic or Nuclear Age. It involves the reduction of the mine ore and removing the gangue accompanying the ore. This clearly describes the act of producing the ore to obtain a mineral, through the processes of prospecting, exploitation, development, exploration and reclamation with the application of the field of geochemistry, geobotany, geophysics and geotechnics. It forms the major division of extractive metallurgy referred to as beneficiation. However, beneficiation strategies largely depend on the grade of the ore, nature of the gangue minerals and liberation size. Barite is commonly beneficiated by using physical separation techniques like crushing, screening, log washing, jigging, heavy media separation, tabling and spiral concentration, magnetic separation and in some cases by selective flotation (Metso Mining and Construction, 2015).

Conversely, a mineral is natural components of chemical elements which comprises of rocks containing such minerals or metallic component that can be recovered with profit, under the

application of heat and pressure. This can be treated accordingly to prescribe recycling processes (Metso Mining and Construction, 2015; Afolayan, 2017). These are clearly distinguished as shown in Figure 12.

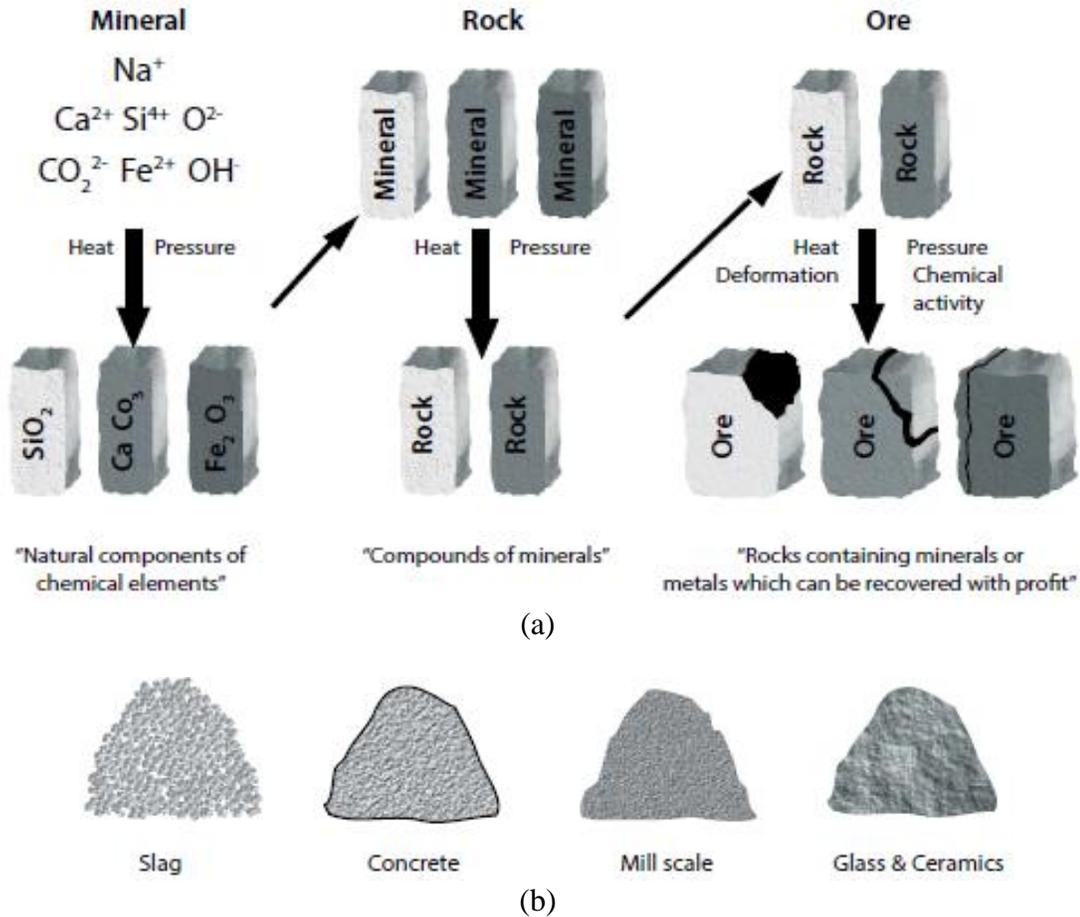


Figure 2.13: Transition between a mineral and its ore (Source: Metso Corporation, 2015).

Similarly, the goal in mineral processing is to recover and produce maximum value from a given raw material. This can be a crushed product with certain size and shape, or maximum recovery of barite mineral out of the overburden and other gangue minerals. The technologies and methods of achieving these goals are conventional, complementary, specific and well defined. The basis in the process Frame of Minerals is clearly classified according to their interrelations in product size and process environment. They are:

1. Blasting (drilling): This is targeted at achieving primary fragmentation of “in situ” minerals. It is the starting point of most indigenous barite mining and processing plant

within the Benue Trough. To achieve this, the use of dynamite (explosives) is being employed to blast the rock or stone formations. Similarly, the manual methods or medium of expanding and cracking rocks can as well be enhanced through the application of wood fuel, horn out tires or the combination of any of the processes to heat up the barite vein (Metso Mining and Construction, 2015; Afolayan, 2017). .

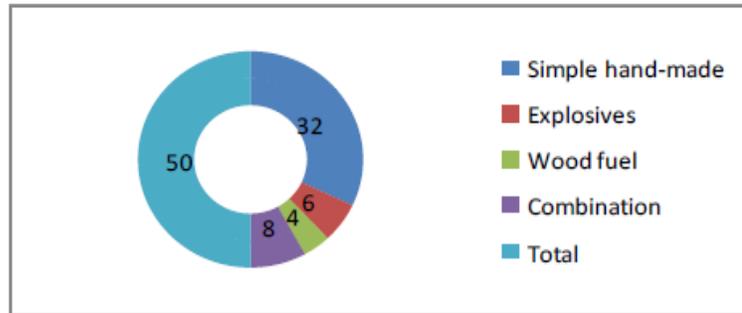


Figure 2.14: Application of explosives and other medium for blasting of barite mineral

1. Crushing and screening: This first controlled size reduction stage (FCSRS) controls other processes aggregate production and preparation process for further size reduction.
2. Grinding: The liberation sizes for barite and other associated gangue minerals are reached at the stage of size reduction where further size reduction filler (micronized form) is produced. This can be achieved for both wet and dry processing techniques.
3. Slurry and Pyro processing: These apply technologies for the wet processing of minerals, and for enrichment and upgrading of the mineral fractions by drying and calcining.
4. Materials handling and Compaction: The fully enriched and upgraded barite mineral are further processed prior to milling. To do these, technologies are employed in moving the process flow of materials via loading, transportation, storage and feeding. At the course of doing these, the minerals are densified, impacted and pressurized (Metso Mining and Construction, 2015; Afolayan, 2017).

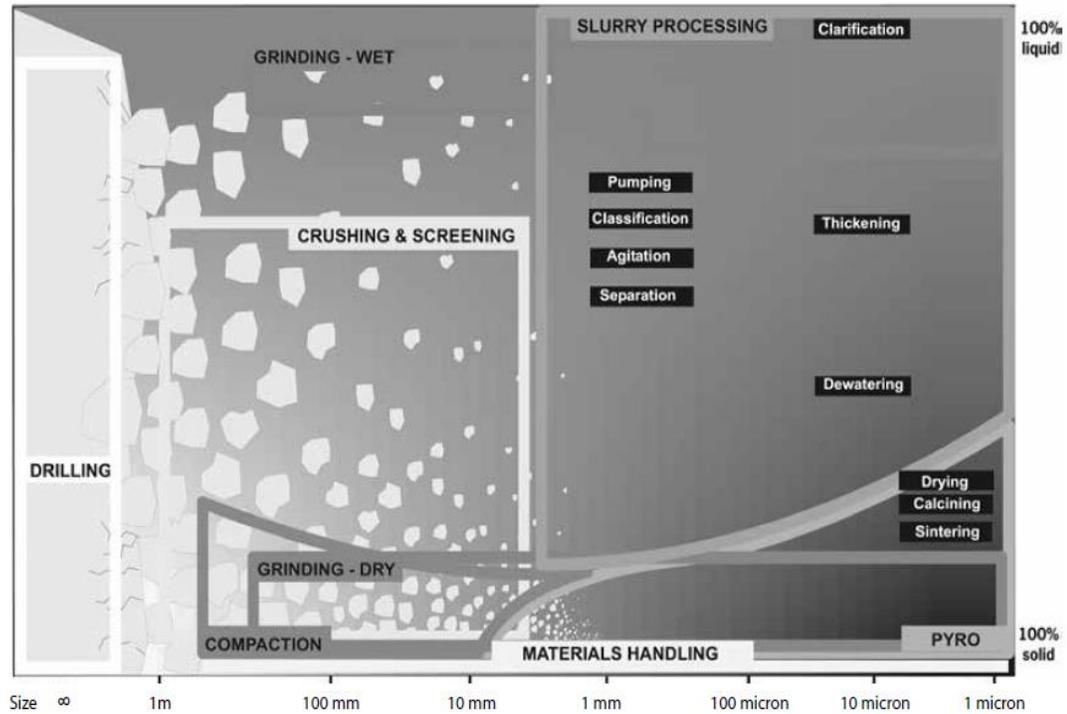
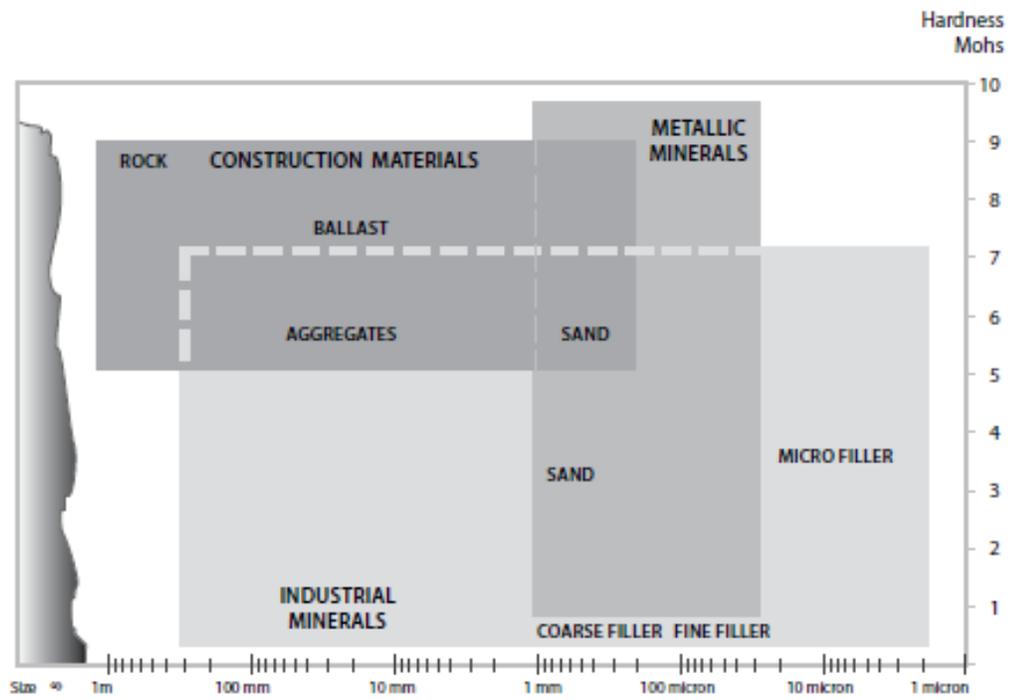
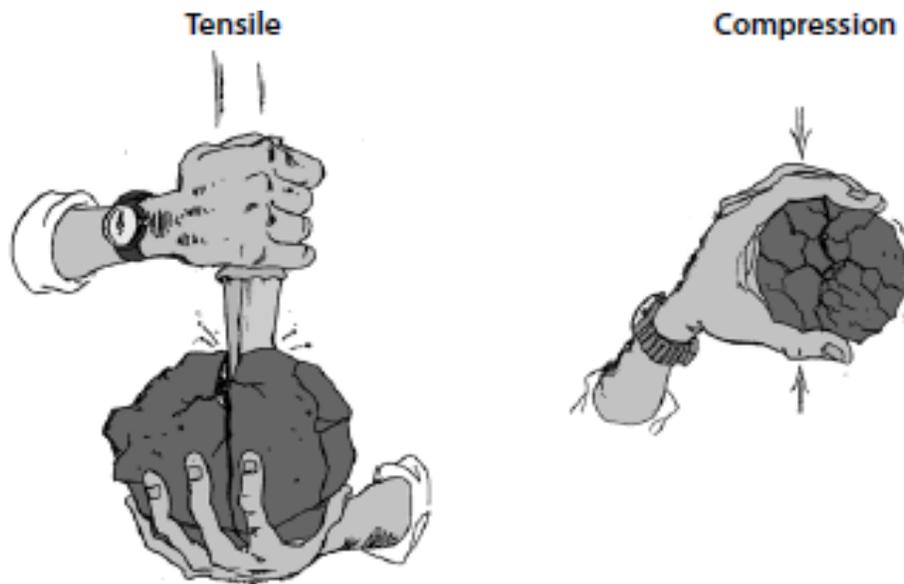


Figure 2.15: Stages in barite mining and processing (Source: Metso Corporation, 2015)

The hardness of barite depends largely on the chemical composition and geological environment. This is taken into consideration during size reduction and other operations within a controlled process environment. To do these, an efficient work and abrasion indexes are carefully chosen for a specific size range of the minerals in a way that limit destructive effects on the process parameters such as the wear rate, uptime and operation parameter. Beyond these processing parameters, the classical stress forces mechanics are most fundamental in barite and other minerals processing. A study on these forces is necessary and important to the equipment designs, system layout; wear resistance and protection designs as clearly revealed in Figure 2.16 (Adiawe, 2011).



(a)



(b)

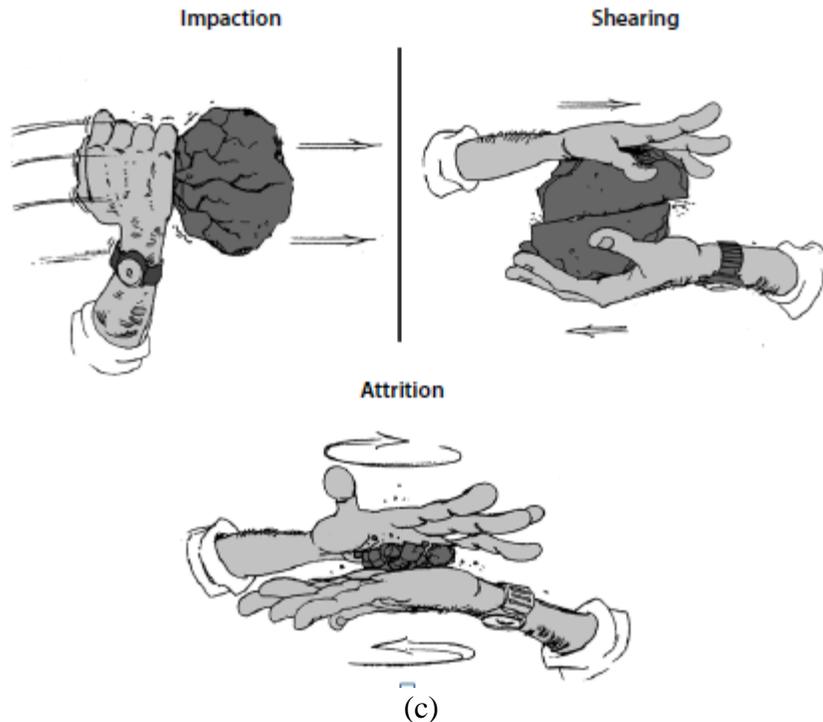
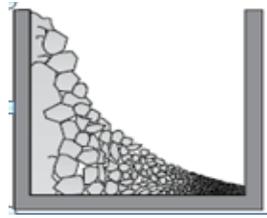


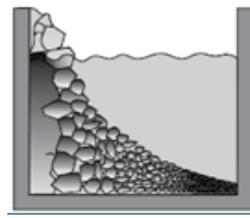
Figure 2.16: Relationship between hardness and size, and natures of forces acting on barite mineral during mining and processing (Source: Metso Corporation, 2015).

### 2.5.2 Barite and other Associated Minerals in Operation

The size fraction of barite mineral in operation determines the operating stages and pattern in minerals processing. This is very peculiar to the design of equipment and processes required to extract or recover maximum value out of hard, abrasive and inhomogeneous mineral crystals. Similarly, the operation pattern evaluates the front service, size reduction and control, enrichment, upgrading, materials handling and protection. In most barite mining and processing plants, wet processing operation is chosen over the dry processing. However, wet processing techniques offer for better efficiency, more compact installation and freedom from dust. Wear rate is generally higher in wet processing. The recovery of barite and other gangue minerals from the surface and underground deposits during the beneficiation process is largely dependent on the mining and quarry fronts. These are fully accomplished during the blasting, primary crushing and materials handling operations in open or underground pits, as clearly shown in Figure 2.17 (Metso Mining and Construction, 2015; Afolayan, 2017).



(a)



(b)

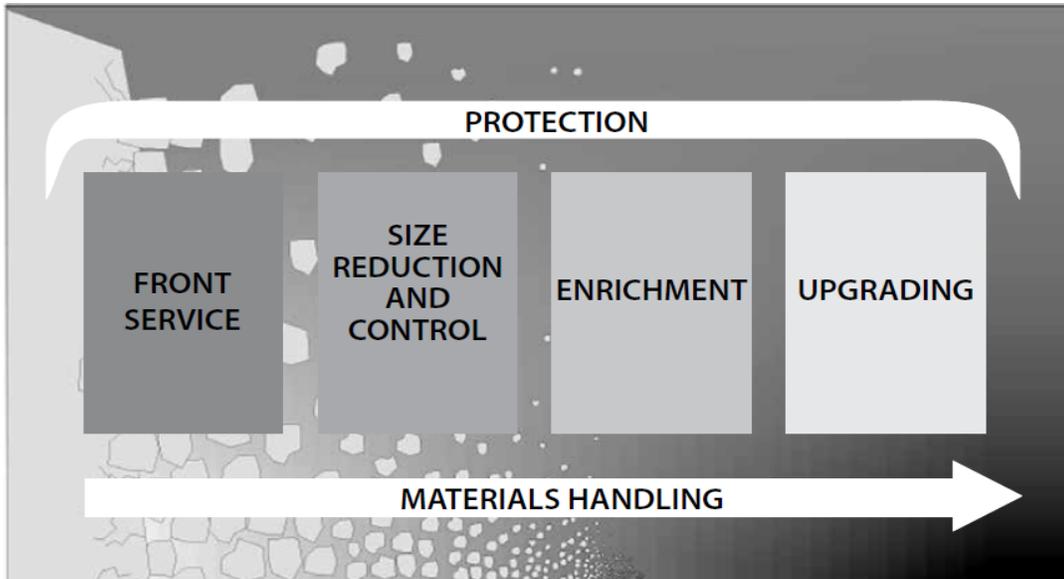


Open pit

Underground



(c)

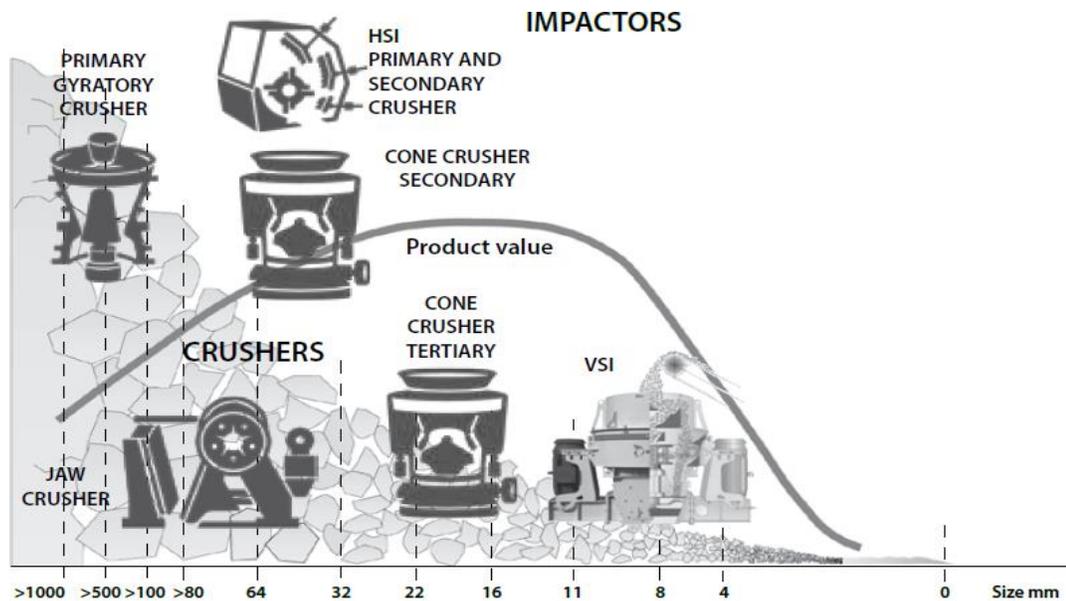


(d)

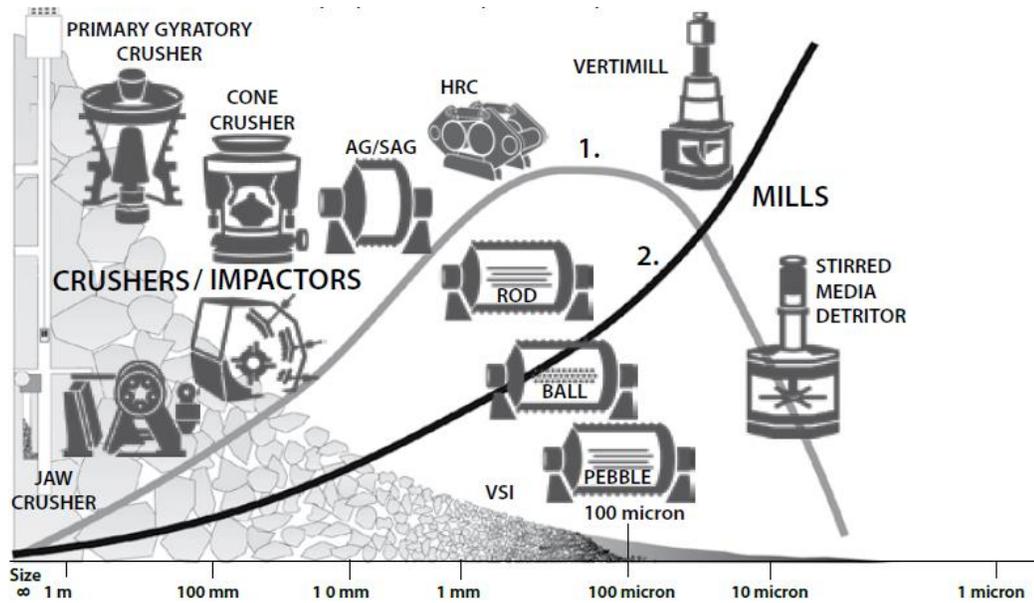
Figure 2.17: Processing techniques and mine pits (Source; Metso Corporation, 2015).

### 2.5.2.1 Operation Stages, Size Reduction and Control, Screening and Classification of (ROM) in Torkula Field

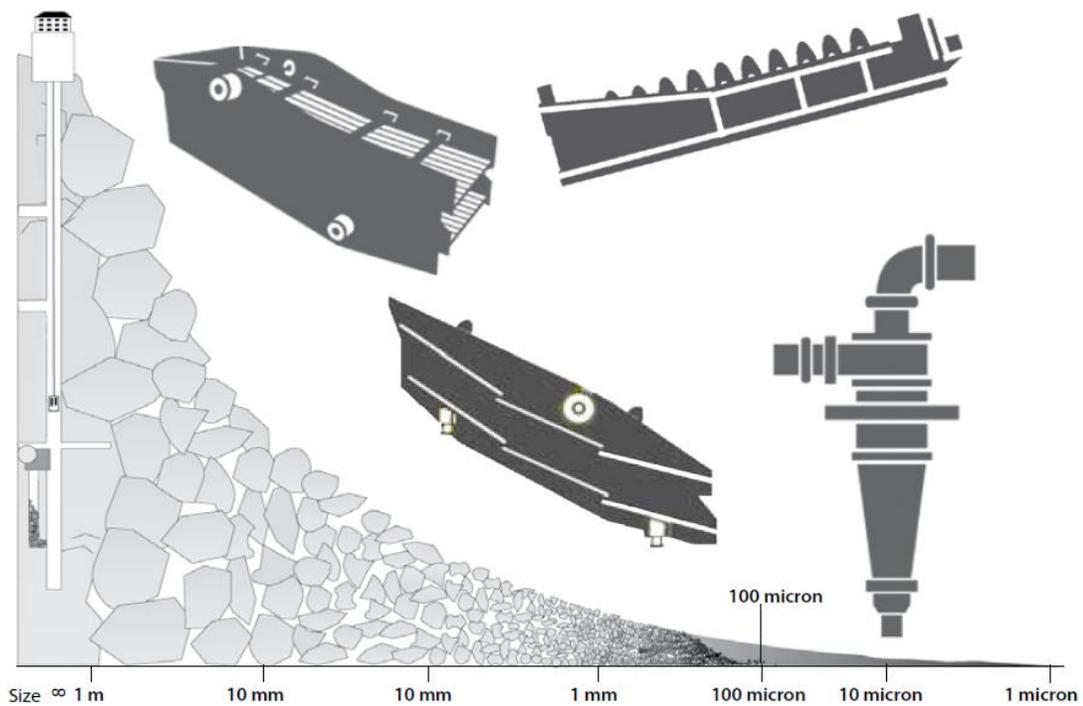
Operation Stages and Size Reduction of Turkula Barite: This is the largest process operation in the processing of barite mineral with the goal to produce mineral fractions that eases the liberation and recovery of the mineral from other gangue minerals. At this stage, the parameters that measure the quality of minerals are strength, size and shape. The appropriate liberation size of barite mineral from the host rock and dust is normally in the interval 100 – 10 micron. However, both crushing and grinding of the ROM or barite ore is necessary to maximize the value, either in situ or in vivo. These comminution techniques are combined to improve the size fraction of the minerals at the process stages and in the final products as shown in Figure 2.18. This is achieved through the application of screens and spiral classifier for the coarse and finer part (Metso Mining and Construction, 2015; Afolayan, 2017).



(a)



(b)



(c)

Figure 2.18: Stages of Size reduction in barite processing (Source: Metso Corporation, 2015)

The mixture of over and under sizes during size reduction of Torkula barite mineral do limit the tendency to break the lump into endless number of sizes and shape upon the application of energy. This may be controlled by the proper selection of equipment for size reduction, and optimum combination of crushing conditions such as reduction ratio, reduction

techniques and feed size. All operations in size reduction are controlled by the feed characteristics of the ore, the crushing parameters such as the crushability or grindability (work index) and the wear rate (abrasion index). Moreover, three crushing stages are required in barite processing to achieve a desired value at the fine end. However, this may be reduced depending on the size of the feed at the primary grinding stage as revealed in Figure 2.19 (Metso Mining and Construction, 2015; Afolayan, 2017).

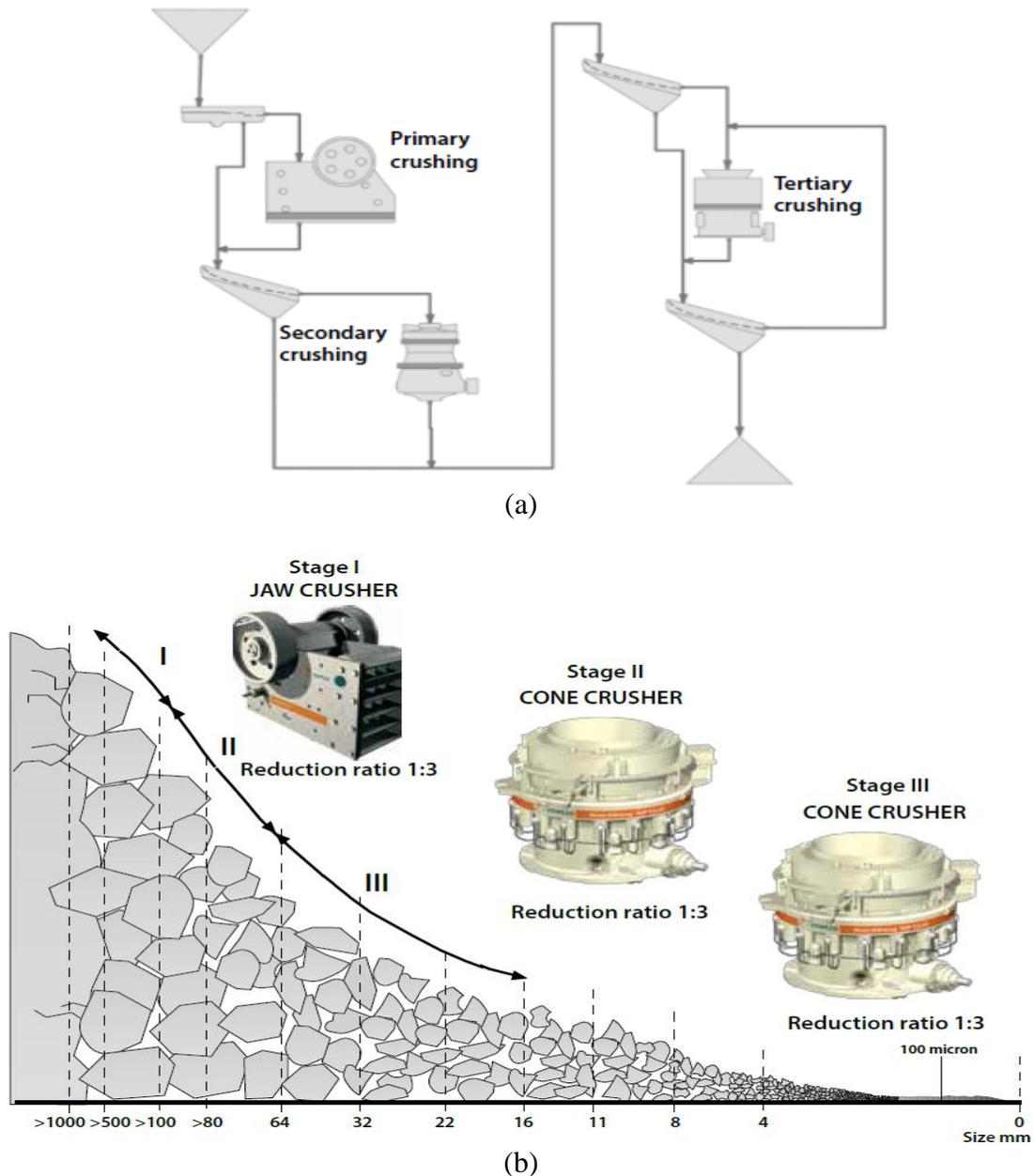


Figure 2.19: Three crushing stages in barite processing (Source: Metso Corporation, 2015)

Size control, Screening and Classification: Due to the low precision of the crushers and grinding mills, optimum size control is achieved through the process of separating barite mineral into two or more products on the basis of their size, capacity and shape. To do these, the undersize in the feed is prevented from blocking the next reduction stage, the over size from moving into the next reduction or operation stage and to prepare the sized product. Other size controlled methods such as screening and classification are achieved through a geometrical pattern and particle motion respectively. In a typical barite processing plant or mining site, the screening process is done by stratification. This is where the finer particles pass in-between the larger ones to enable a sharp separation. This was clearly described in Figure 2.20. However, a triple inclination screen type is commonly employed over other screen type due to its best combination of capacity, selectivity and advance product fractions (Metso Mining and Construction, 2015; Afolayan, 2017).

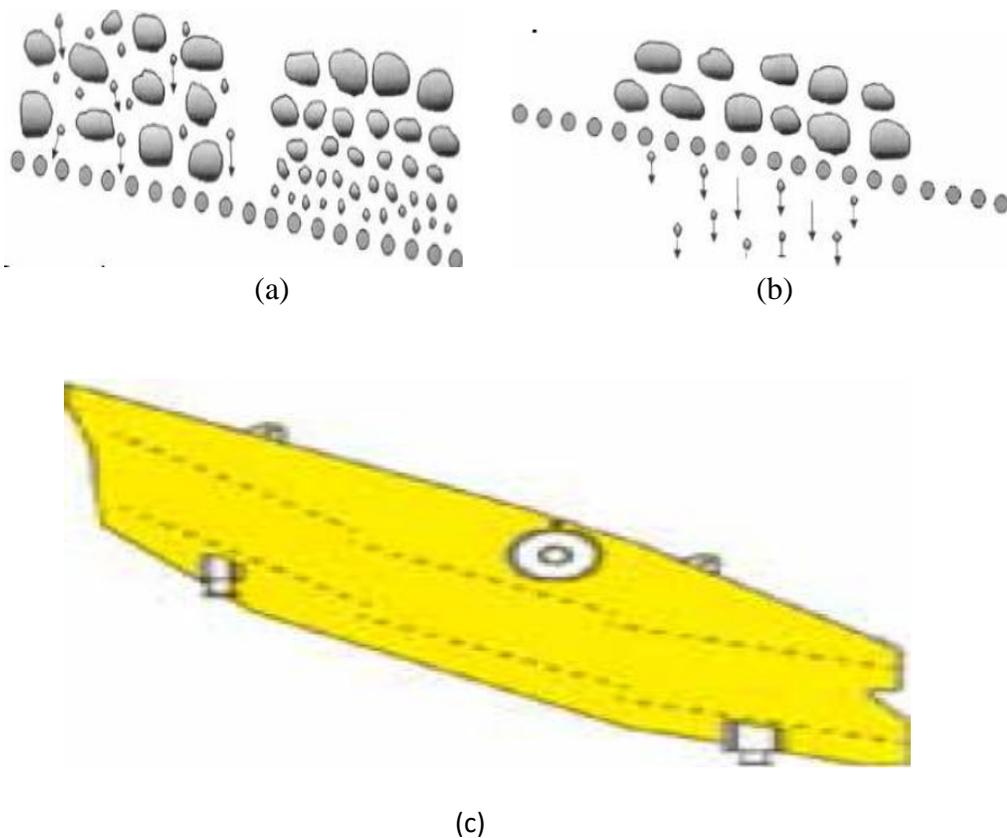
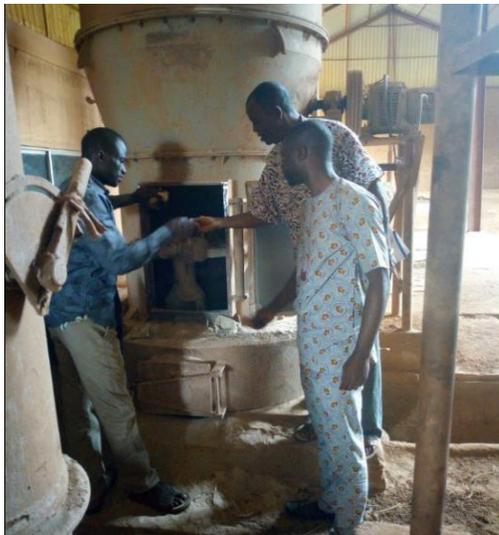


Figure 2.20: Screening by stratification, separation and screen type (Source: Metso Corporation, 2015).

Similarly, barite particles finer than 1mm are separated by sizes into two or more products on the basis of their behaviour in water. The hydrocyclones separates barite particles by the application of centrifugal force, covering the size range of 100 – 10 micron. In a typical barite processing plant, centrifugal classifier is most used over others for wet classification techniques due to high degree of separation accuracy and exceptionally low maintenance requirement. In addition to these, the high efficiency cyclones offer dry solution capabilities to ease the movement of the product from the pendulum pulverized miller, through the bucket elevator to the product silo, as shown in Figure 2.21 (Metso Mining and Construction, 2015; Afolayan, 2017).



(a)



(b)



(b)

(d)

Figure 2.21: Pendulum pulverized Mill and Jaw crusher in a crushing Site at Benue State  
(Source: Afolayan, 2017).

### **2.5.2.2 Enrichment, Upgrading and Mechanical Dewatering of Barite Mineral.**

Enrichment of barite Minerals: The removal of impurities (dust, clay) and other gangue minerals (galena, sphalerite, hematite and calcite) from barite mineral improves the value and the quality of the mineral. This is achieved through washing and separation of products either in solid form or liberation particle form. In a typical barite mining and processing plant (PIN Ltd), wet screen and tumbling scrubbers were considered for the enrichment process over the log washers, aquamator separator and attrition scrubbers. For the particle sizes, the leaching method of separation for wet processing techniques is mostly considered over gravity or magnetic separation and floatation. For wet screen, the water spraying washes barite on a screen regardless of the hole size in the screening media. On the mining site, tumbling scrubber or a speedy washing drum for scrubbing solids is also used to wash barites mineral which contain a high and sticky content of clay and dirt (Afolayan, 2017).

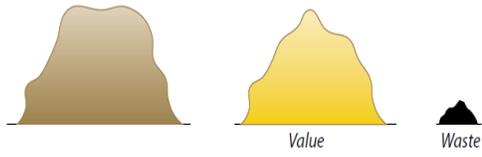
Barite mineral can be separated individually after the liberation of all individual mineral in the run-of-mine (ROM), either by grinding or by natural size reduction. In terms of particle density, separation by gravity (in water) is preferable over other separation techniques such as floatation, magnetic and leaching due to the difference in density between barite and other gangue minerals such as sandstone, galena, sphalerite and hematite. In barite processing, heaps of ROM mixed with the overburden are first leached prior to the separation by gravity. In addition to these techniques, dense media separation may be introduced to recover barite mineral from the mix of heavy gangue minerals such as galena, hematite with a density difference of 1.75 and above, for mineral size range of 100 micron and below (Metso Mining and Construction, 2015; Afolayan, 2017).



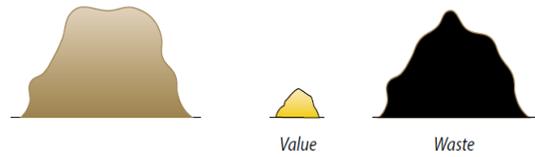
(a)



(b)



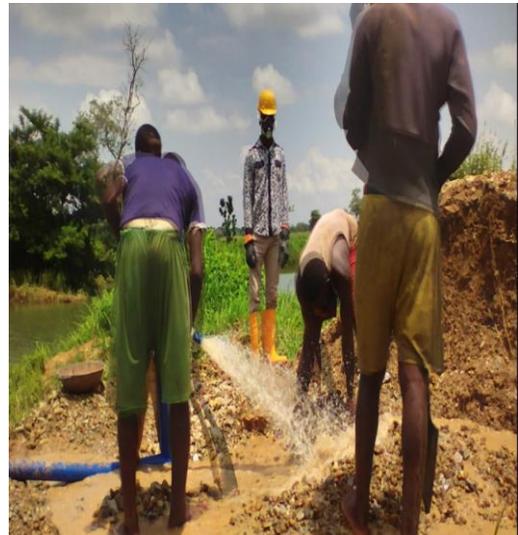
(c)



(d)



(e)



(f)

Figure 2.22: Heaps of Barite ROM, manual and mechanized enrichment techniques (Source: Afolayan, 2017; Metso Mining and Construction, 2015).

❖ Upgrading and Mechanical Dewatering of Barite Mineral:

Upgrading of final products serves as a post-enrichment stage in barite processing. It involves processes of improving product values of the concentrate. This is achieved by bringing the concentrate into a completely dry form, either through calcination or sintering in a way that enables the efficient recovery of valuable minerals from the tailings. Similarly, the valuable materials dissolved in the waste water and process effluent is properly taken care of through efficient recycling and recovery processes, in order to protect and preserve the environment. Upgrading of barite minerals involves combined processes of sedimentation, flocculation, clarification and thickening. After the enrichment of the minerals through screen washing and water leaching, the minerals are allowed to settle into layers by gravity. The flowing of water at lower pressure across the surface of the minerals and increased particle size improves the settling speed of solids by gravity, and enables the recovery of barite mineral from other associated gangue minerals.

Barite minerals of fine or small particle size are recoverable by means of mechanical removal of liquids from slurry to obtain solids in a suitable form. To do this, the fine particle mixtures of barite and other gangue minerals are packed with the shovel into the sack, which allows the water to drain off the slurry. The drained slurry is allowed to dry under the heat of the sun for few hours to enable the recovery of barite (Metso Mining and Construction, 2015; Afolayan, 2017).



(a)



(b)

Figure 2.23: Enrichment and upgrading of barite and other associated gangue minerals



(a)



(b)



(c)



(d)

Figure 2.24: Enrichment and upgrading of barite and other associated gangue minerals

### 2.5.2.3 Materials and Slurry Handling, Wear of Equipment and Protection in Barite Processing

#### ❖ Materials and Slurry Handling in Barite Processing:

This is the climax in barite processing where the process stages of size reduction, size control, enrichment and upgrading the core values of the minerals are brought to the attainable optimum. This stage requires technologies such as loading and unloading, storing, feeding and transportation, to achieve a complete and comprehensive processing with a minimum of disturbances in capacity and flow. Similarly, the focus of mineral mass flow when transporting dry barite material is the application of conveyor. The minerals are being transported by the conveyor belt, upon which the ROM prepared for enrichment moves. The materials are conveyed from the feeding plate of the trommel, through the washing drum and to the product plate. Barite and other gangue minerals such as galena and zinc are separated into bags for weighing, and transported into the store through a wheelbarrow (Metso Mining and Construction, 2015; Afolayan, 2017).

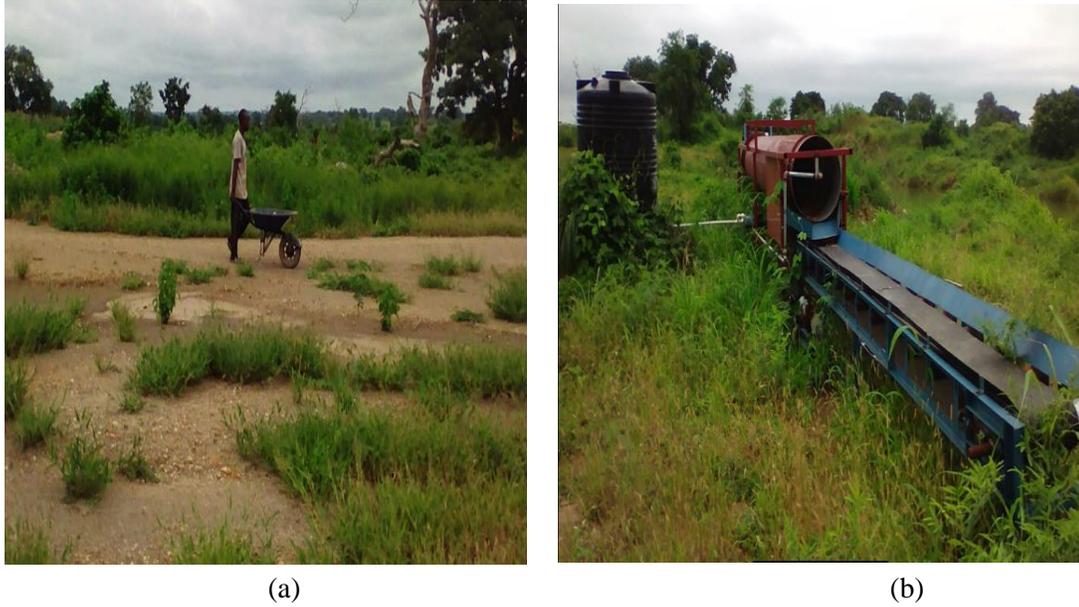


Figure 2.25: Materials handling in barite processing (Source: Afolayan, 2017).

❖ **Wear of Equipment and Protection in Barite Processing:**

The wear of mining and processing tools or equipment is comparatively high in wet process than in the presence of air. On a general note, mineral processing activities unavoidably result in wear, which often cost lots of money. Due to the abrasive and hard nature of barite, wear of tools results from normal rock stress forces in the form of compression, impact, shearing and attrition. All these in combination with mineral abrasion, hardness and energy result into the wear and degradation of many cutting and mining tools. Depending on the structure and texture of the minerals, mining and minerals processing equipment can be protected by a polymeric materials (rubber, polyurethane, UHMWPE), ceramics, steel and high chrome backings. These are applied in the form liners on equipment linings and trommel screen (Metso Mining and Construction, 2015; Afolayan, 2017).



(a)

(b)

Figure 2.26: Wear of tools due to the hardness of the host rocks, and processed product for storage (Source: Afolayan, 2017).

### 2.5.3 Emerging Investment Opportunities in Barite Processing

Nigeria as a well-endowed nation has in every of its local government huge deposits of mineral resources at different levels of exploration, and some are yet to be discovered. There are unparalleled opportunities for professionals, scientists and researchers with relevant skills to engage in mineral prospecting and exploration. However, barite minerals are being exported as raw materials in its crudest form without adding value through beneficiation or processing. This has resulted into loss of revenue for the country. There exist overwhelming abundant opportunities for mineral beneficiation in Nigeria. Similarly, efficient barite processing strategy is the surest route to self-sufficiency in oil drilling and massive industrialization of the entire petroleum industry. To do these, applying for mineral titles with a view of wholly owning the barite mining right is a promising way to start (MMSD, 2010; Alokolaro, 2012).

Mining is the foundation for modern industrialization. Industrial mineral such as barite can be used in their natural or processed states either as raw materials or as additives in a wide range of applications. Several foreign and indigenous mining industries use barite as basic raw materials. Investing in barite mining and processing in Nigeria is timely. Moreover, a lot of industry and employment are expected alongside the mining sector value chain. The

exploitation for barite will lead to the working of more oil rigs, creation of oil refinery, oil field and petrochemical industries. Partnership with the existing title holders for comprehensive exploration as consultants and specialists, and in joint venture agreement to explore, exploit and market the barite resources in the area of interest. Areas of opportunities also abound in equipment sales and leasing. Equipment used for mining and processing of barite is comparatively expensive, and as such, not everyone in the mining industry can afford to buy all the equipment need for the smooth running of the plant (Bonel, 2005; Fatoye et al., 2014).

There abound countless opportunities for practitioners from other sectors such as in legislation, engineering and business, communication and public analysis, to actively participate in the mining value chain. The Lawyers, Risk Analysts, and Business Service Consultants are required to support new companies entering into the market with robust and bankable business plans, realistic feasibility studies and market research. Community Relations and Strategic Communications experts are needed to skillfully engage stakeholders in the communities on behalf of companies, prior to minerals prospecting and exploration in the fields. Barite mining industry will welcome Universities, Research Institutions, Mining Specialist Blogs and Publications. Support services to address and resolve associated risk in mining requires Safety Experts for necessary Environmental Impact Assessment. The services of Medical Doctors, Paramedics and other health workers are needed on mines to respond to emergencies and welfare of mining workers through Catering and Hospitality (MMSD, 2010; <http://www.minesandsteel.gov.ng> 28/03/2017).

#### **2.5.4 Distinguishing Characteristics and Types of Barite**

##### **❖ Distinguishing Characteristics of Barite**

The unusually high specific gravity of this coloured mineral is the main distinguishing feature. It can also be identified by its insolubility in acid. It can be distinguished from anhydride and Celestine by its orange fluorescence and the green flames that result in flame test. Its crystal shape, perfect cleavages and lack of fluorite fluorescence differentiate it from fluorite (<http://geology.com/minerals/barite.shtml>. 26/11/2016).

This barium sulphate contains Barium oxide (BaO: 65.7%), sulphur trioxide (SO<sub>3</sub>): 34.3%. The chemical formula for barite is BaSO<sub>4</sub>. The basic properties of barites varies and ranges, these are that: it is transparent crystalline material with colour varying from colourless to white and light shades of yellow, blue, brown and grey. It has a molecular weight of 233.4 grams per mole, a density of 4.48g/cm<sup>3</sup> and almost soluble in water. It has orthorhombic crystal system, a tabular parallel to base, fibrous, modular to massive. It has irregular and uneven fracture. Its hardness ranges between 3 and 3.35 on Mohr scale (Omoniyi and Mubarak, 2014).

Barite crystals have vitreous pearly luster. The specific gravity of barite is between 4.3 and 5.0. It has white streak. The tenacity of its crystalline mineral is brittle. It has transparent to opaque diaphaneity, biaxial positive optical properties, 0.012 birefringence, fusibility of 4, produces yellow-green barium flames (<http://www.galleries.com/barite>. 26/11/2016). The crystals commonly appear as tabular or leaf-like aggregates with edges projecting into crest-like or rosette forms. Its habit is described as prismatic, grainy masses or compact, having the appearance of massive gypsum. It is chemically inert; it is not a toxic chemical under section 313 of the emergency Planning Community Right-to-know Act of 1986. It occurs as a vein filling and as a gangue mineral in silver, zinc, copper, nickel and lead ores (Ngukposu, 2015; Olokolaro, 2012)

#### ❖ **Types of Barite Deposits:**

Barite deposits are found in a variety of geological environments. The three major types of barite deposits are stratiform, vein and residual. Earliest mining activities were from veins, often associated with lead and zinc deposits, and from residual deposits. Most barite in Nigeria and around the world is produced from stratiform deposits (Oden, 2012).

1. **Stratiform Deposits:** These are formed by the precipitation of barite at or near the sea floor of sedimentary basins or exhalative (sedex) deposits. The brines are generated by the migration of reduced, saline fluids concentrated by major basin-controlling faults. They are often associated with base metal sulphide. They occur in

rocks varying in age from Precambrian to Cenozoic. They are very much less frequent and have so far been seen in Alifokpa in Cross River State, Gidanwaya and Gidan Waya in Taraba State. Stratiform barite deposits of Mangampet in Andhra Pradesh, India, having two stratiform lenses up to 1.2km long and 20m wide. It has huge deposits of over 74 million tonnes of barite with an annual production of 700,000 tonnes. It contributes about 90% of the barite production in Andhra Pradesh. Similarly in Cambrian black shales, Jiangnan region of south china and the Quilling region in the Yangtze valley, have a sedex deposits with almost 4 million tonnes per year. It contains some witherite and baritocalcite ( $\text{BaCa}(\text{CO}_3)_2$ ) deposits associated with the barite horizons. The deposit in the Nevada barite belt, western USA, extends over 500km from north to south and is about 125km wide. Its estimated reserve is around 90 million tonnes of barite in hundreds of small lenses in silicious sediments. Other significant stratiform deposits occur in late Proterozoic meta-sedimentary rocks near Aberfeldy in Scotland, Primorye region of far-eastern Russia, Devonian shales in Germany and in Mesozoic carbonates in a belt from Pakistan through Iran (Bonel, 2005; Oden, 2012; Ngukposu, 2015).

- 2. Vein and Replacement Deposits:** These are classified into the small and large vein-style barite deposits. They have been exploited prior to 1980s until large sedex deposits became more important. These deposits are formed by the precipitation from hot barium-enriched fluids in faults and fractures as a result of fluid mixing or reduced pressure and temperature. However, the fluids may dissolve the surrounding host-rocks (especially limestone and dolomite) to form irregular replacement deposits. Several veins and replacement deposits exist in Nigeria. The most impressive barite veins are in Azara area, Ribbi, Keana, Alosi, Ambua, Chita, Kuduku, Wuse and Akiri in Nasarawa State; Torkula, Kaseyo, Zanzan, Makurdi, Lessel area (Lessel Bunde, Lessel Mbato and Lessel Mbagwa), Gboko, Yandev and Ihugh barite field in Benue State; Didango, Ibi, and Kumar barite in Taraba State. It exists also in Yala and Gombe barite field. Vein deposits in Britain and many have been worked on in Morocco, USA, Germany and Slovakia. The Ballynoe vein deposits in Ireland has a production capacity of over 5 million tonnes of direct shipping barite, and occurred as a single

lens associated with replacement Pb-Zn mineralization in lower Carboniferous carbonate. The veins consist predominantly of barytes (80% BaSO<sub>4</sub> and less than 5% SrSO<sub>4</sub>), sulphides (galena, pyrrhotite [FeS<sub>2</sub> – magnetic pyrite]), iron, manganese oxides as well as fluorite, calcite and quartz. These types of deposits are generally smaller than the bedded deposits, with examples to include Dreislar and Rhineland-Palatinate vein deposits in Germany; Les Arcs deposit in France and Pennine ore fields in the United Kingdom (Lorenz and Gwosdz, 2003; Fatoye et al., 2014).

3. **Residual or Bedded Deposits:** These barite deposits are formed by the dissolution of the host rock of bedded deposits, leaving irregular masses of barite in a clay matrix. Such deposits are extremely variable in size and shape, but can extend over several kilometers. Individual beds are fine-grained, huge to laminate and may contain about 50 – 95% barite. They are the most valuable and economically most important deposit type because they are usually large and have higher grades (Lorenz and Gwosdz, 2003). Clark et al. (1990) further subdivided the bedded barite deposits into five groups based on its depositional environment and the presence of base-metal sulphides. Torkula barite field in Hungwa, Nigeria is characterized with bedded barite deposits. It is comprised of several veins stacked together to form a bed (Oden, 2012; Ngukposu, 2015).

#### 2.5.5. Uses, Pricing and Availability of Barite

❖ Specification and Uses:

1. The main and global use of barite is as a weighting agent in oil and gas well drilling mud. Barite in micronized form is added to the drilling mud to increase the density of the column of fluid above the drill bit to prevent a blowout. However, there are alternatives such as hematite and galena; barite is the preferred weighting agent. It is non-corrosive, non-abrasive, insoluble and non-toxic. It is also relatively cheap and easily available (Strachan, 2010).
2. Barites' outstanding qualities of high density, low solubility, high brightness and whiteness, chemical inertness, softness and relative cheapness also make it of a great value in many other applications. These unique uses include:

- As sustainable filler in paint and plastic, and as the major source of barium for the chemical industry. Processed barite in the form of barium metal can be used to remove traces of gases in vacuum tubes.  $\text{BaCO}_3$ ,  $\text{BaCl}_2$ ,  $\text{Ba}(\text{OH})$  are used to control efflorescence and scum from forming on brick and ceramic materials during its manufacture
- In the part-production of lithopone (high performance white pigment composed of a mixture of chemically precipitated and calcined zinc sulphide and barium sulphate).
- Barite may be added to concrete to increase its density for specialist application. It is also used to identify problems within the body such as in the absorber of gamma and X-ray radiation. Barite significantly blocks x-rays and gamma rays. It is used as a contrast medium in medical x-ray examination and an aggregate in high density concrete to shield against radiation.
- Used as a flux, which add up brilliance and clarity during glass manufacturing (Bonel, 2005; Ngukposu, 2015).

❖ Pricing and Availability of Barite:

There are relatively few major sources when considering barite reserves suitable for use in drilling fluids. They are China, India, the US and Morocco, Mexico and Turkey, accounting for about 80% of global production. China accounts for around 45% of world production with India, US, Morocco and Turkey, making up a further 35% of the global productivity. Barite productivity is directly linked to oil and gas-well drilling activities, which has increased from 5 million TPA in the 90s to about 8.5 million TPA in 2013. United States consumes nearly 3 million TPA and China, 1.1 million TPA, accounting for more than 56% of the annual shipment. Barite mineral reserves in Nigeria are huge, of higher quality and prospect. However, it is only available and exported in the crude form with little or no beneficiation. Moreover, the world's barite resources in all classifications are about 2 billion tonnes, but only about 740 million tonnes have been identified (USGS, 2016 on [ehughes@indmin.com](mailto:ehughes@indmin.com)).

Barite prices are directly linked to purity and grade. In the global market, there have been variations in the price of barite. This is largely due to the specific gravity and the source of the product. However, the prices of barite have remained relatively steady until 2006, comparative to the price of the same commodity in 1995. In recent years, it had experienced the consequences of rapid increase and slight falls. This unexpected inflation resulted from the rationalization and consolidation of the Chinese barite mine, highly intensified US onshore drilling for tight oil and gas suppliers (Scogings and Hughes, 2014; USGS, 2016).

## **2.6 Drilling Operation and Fluid Technology**

This operation involves the application of science and technology to bore hole through the earth surface by using drilling bit. This is done to pay zone or reservoir at the post seismic survey and prior to the drilling activities. Drilling in itself is the only provision to ascertain the presence of oil in the formation, produce reservoir fluid and as the major communication link with the reservoir or subsurface. To do these, hole is drilled many hundreds of meters into the rock formation to capture the oil and gas and bring them to the surface. This operation is held many meters in underground reservoir at enormous high pressure and temperature. In this process, pressure variations are closely monitored during drilling to prevent accident which could occur when sudden unusually high pressures are faced in the subsurface (Joel, 2013).

To control pressure in the oil well during drilling and prevent blowout, a mixture of clay, weighting materials and chemicals may be added with either water, oil or a mixture of oil and water. This is referred to as drilling mud and exerts pressure by its weight maintained slightly higher than the hydrostatic pressure of the overlying water. However, this pressure relationship creates an overbalance which prevent fluids from the reservoir rock (formation fluids) entering the well prematurely. On other hand, if the pressure on the fluid in the subsurface rock is higher than the pressure on the drilling mud, underbalance occurs, and the formation fluids flow out of the rock into the well. This phenomenon called kick results in a blowout if the flow of formation fluids from the well to the surface is not controlled.

Similarly, rotary drilling rigs are used for most drilling operation. To do this, hole is drilled by rotating a bit to which a downward force is applied to the bit by using the drill collars, in the drill string above the bit where the cuttings are separated from the drilling fluid at the surface (Baroid Training Manual, 1997; Strachan, 2010; Ukooa, 1996b).

The types and composition of drilling fluids used daily depend on the nature of the oil well and the hole. Drilling fluids are selected, maintained and related either directly or indirectly to most drilling problems. On the contrary, if the drilling fluid does not perform the functions adequately, it could become necessary to abandon the well. As a result of the cost of fluid additives, the fluid itself is expensive and often exceeds \$ 1 million on a single deep well. However, the cost of the drilling fluid is maintained in good condition at the lowest possible cost. In addition to these, the produced formation water injected into the reservoir to maintain pressure is usually discharged from the platform after separation from the oil. This consists of salt solutions with a highly complex mixture of organics, radionuclides and metals such as barium, cadmium, chromium, iron and lead. Similarly, the content and volume of heterogeneous drill cutting piles are difficult to forecast. They are brought up to the platform where it is separated from the drilling mud and then discharged into the sea. Moreover, the dumped drill cuttings still contain the insoluble portion of the drilling mud which coats the cuttings. This results into huge waste of the mud and incurred cost (Breuer et al., 2004; Holdway, 2002, Neff, 2005).

### **2.6.1 Types and Components of Drilling Fluid (Mud)**

#### **❖ Types of Drilling Fluid (Mud)**

- **Water-Based Drilling Mud (WBM):** This is an aqueous suspension of clay or polymeric substances with a higher viscosity than water and has its carrier fluid in most drilling operation as either freshwater or saltwater. It is classified by its specific shale drilling fluid formations, the effect of the mud system, swelling and pore pressure. Water-based drilling mud usually disperses drill-cutting particles into the drilling fluid, produces larger amount of drilling waste and contain significant levels of hydrocarbons from the drilling process in the reservoir. It consists of bentonite clay (gel or viscosifier) with additives such as

barium sulfate (barite), calcium carbonate ( $\text{CaCO}_3$ ) or hematite ( $\text{Fe}_2\text{O}_3$ ). Various thickeners used to influence the viscosity of the fluid, includes xanthan gum, guar gum, glycol, carboxymethylcellulose (CMC), polyanionic cellulose (PAC) or starch (Caenn and Chillinger, 1996; Ukooa, 1996a; Ukooa, 1996b).

On the other hand, deflocculants that are frequently used to reduce viscosity of clay-based muds includes anionic polyelectrolytes such as acrylates, polyphosphates, lignosulfonates (Lig) or tannic acid derivatives (Quebracho). Other components are added to provide various specific functional characteristics. Additives such as lubricants and shale inhibitors act to slow down or prevent the mechanical or chemical disaggregation of shale. These materials include inorganic salts, some organic compounds and fluid loss additives. Such are added to control the loss of drilling fluids into permeable formations. To achieve this blend, a weighting agent such as barite is added to increase the overall density of the drilling fluid so that sufficient bottom-hole pressure can be maintained (Ibe et al., 2016; Strachan, 2010).

- Diesel Oil-Based Mud (OBM): This drilling mud was introduced to replace water as the base fluid for mud for certain application and selected for their superior temperature stability, lubricity and hole stabilizing attributes. Similarly, it is employed for use in high temperature formations, and formations containing water sensitive minerals, clays or reactive gases in oil well where a high level of lubrication is required. The use of oil-based muds has special considerations which include its ability to withstand greater heat without failure, cost and environmental considerations (Davies et al., 1984). It is composed of various molecular-weight-range cuts refined from crude oil for fuel and may vary with the original crude composition and the distillation process. Moreover, it can be classified as either low fluid loss or relaxed fluid loss system on the basis of the additives and reaction conditions. However, application of diesel oil in drilling mud has been prohibited due to its high aromatic content. Oil-

based muds also withstand greater heat without breaking down (Gerrard et al., 1999; Strachan, 2010; Engelhardt et al., 1983).

- Low Toxicity Oil-Based Drilling Mud (LTM): This class of drilling mud was introduced to replace diesel oil-based drilling mud as a means of addressing the environmental concerns it poses, and to eliminate the high aromatic content. In order to reduce the environmental impact of the fluid, a discharge limit for oil cuttings was set to 1% (McCosh and Getliff, 2002; Neff, 2005; Wills, 2000; Strachan, 2010).
- Synthetic-Based Drilling Mud (SBM): This is water in oil emulsion designed and formulated to replace oil based drilling mud as a low toxicity and readily biodegradable alternatives to mineral oil-based muds used in offshore drilling activities. Synthetic-based drilling mud has a readily biodegradable synthetic material as the carrier fluid which contains lubricants synthesized from products such as ethylene. Such material also contains double bond or functional groups which promote rapid environmental breakdown in water (Wills, 2000; Breuer et al., 2004). However, its base fluid differs from other oil-based mud as a result of the replacement with esters, polyalphaolefins (PAOs) or linearalphaolefins (LAOs) and vegetable oils. These additives provide with the same drilling advantages but with the handling and disposal characteristics of water-based drilling mud. This makes the synthetic-based drilling mud less more toxic in comparison with other hydrocarbon-based oil and the associated drilling cuttings are either re-injected back into the oil well or taken ashore for treatment (Caenn and Chillingier, 1996; Strachan, 2010).
- Alternative Water-Based Drilling Mud (AWBM): This was specifically developed to reduce harm towards the marine environment. Several replacement mud systems currently in use as alternative to the existing OBM and SBM includes the fresh-water polymer mud, salt-polymer mud and cationic mud systems. These was primarily introduced to address current and advance drilling operations, minimize cost and control fluid loss, stabilize the shale

formation and reduces dispersal properties (Strachan, 2010, Caenn and Chillingar, 1996; Orszulik, 2008).

❖ Components of Drilling Fluid (Mud):

The formulation of drilling muds depends on the drilling job and the location of the oil well (Wills, 2000). Such fluids often contain a variety of chemical additives with varying formulation according to the required characteristics of the mud and the well to be drilled (Holdway, 2002). These additives are categorized according to the physico-chemical properties required, their functions and compositions (Terzaghi et al., 1998). Each category may contain several alternative materials with different properties (Neff, 2005), which includes:

- **Weighting Agents:** These are the most abundant component of the drilling mud (Neff, 2005). It is strategically used to prevent blowouts by maintaining the borehole pressure (Sadiq et al., 2003). To do these, the weighting agent is mixed with water and other materials, and then pumped into the drill hole. The weight of the mixtures of water, weighting agent and other materials counteracts the force of the oil and gas when it is released. Barite is the preferred weighting agent among other alternatives, due to its relatively high specific gravity of 4.5. These substitutes such as ilmenite ( $\text{FeTiO}_2$ ), haematite ( $\text{Fe}_2\text{O}_3$ ), siderite, dolomite ( $\text{CaMg}(\text{CO}_3)_2$ ) and calcium carbonate ( $\text{CaCO}_3$ ) are also used as weighting agents (Caenn and Chillingar, 1996). However, haematite or ilmenite may replace barite for use in deep drilling within a higher density mud (Neff, 2005).
- **Viscosifier:** These are mostly added in the form of clay and are used with all types of drilling mud. Viscosifiers build its viscosity through complex interactions with emulsion (Caenn and Chillingar, 1996). It forms a thick gel in the well bore preventing the settlement of drill cuttings and barite. The mostly used clay component in drilling mud is sodium montmorillonite (bentonite). It helps to prevent fluid loss by coating the wall of the bore hole (Neff, 2005). Another clay type commonly used is attapulgite (salt gel) (Caenn and

Chillingar, 1996). Combined with water-based drilling mud, organic polymers derived from cellulose and natural biopolymers may be used as a replacement for clay when drilling in soft formations (Neff, 2005).

- **Surfactants:** These can be used as defoamers, detergents, lubricant and emulsifiers. Surfactants in drilling process help to maintain wet ability, improves the lubricity, shale stability and formation return permeability of the mud (Wenger et al., 2004; Caenn and Chillingar, 1996). Modified vegetable or mineral oils and refined polyols have been used to lower the friction factor in water-based drilling mud and reduce the resistance of the drill string (Neff, 2005).
- **Shale Stabilization Agents:** It has been revealed that shale-fluid interactions can be manipulated to enhance cuttings and wellbore stabilization, and improving hole-making ability in shale formations (vanOort, 2003). Asphalt-based shale stabilizers and Polyalkylated glycols are added to aid well-bore proficiency, prevent sticking drill pipe and the formation of gas hydrate in the drilling mud (Wenger et al., 2004). To do these, soltex, a residue in petroleum refining and water-soluble asphalt is primarily used for shale control (Terzaghi et al., 1998).
- **Fluid Loss Control Agents:** These are fine particle size compounds added to the drilling mud to reduce the loss of fluid from the mud into the drilled formation. These agents such as bentonite, lignite and polymers (modified starch and polyanionic cellulosic polymer) act as an emulsifier and important additives for water-based drilling mud (Ukooa, 1999a; Caenn and Chillingar, 1996).

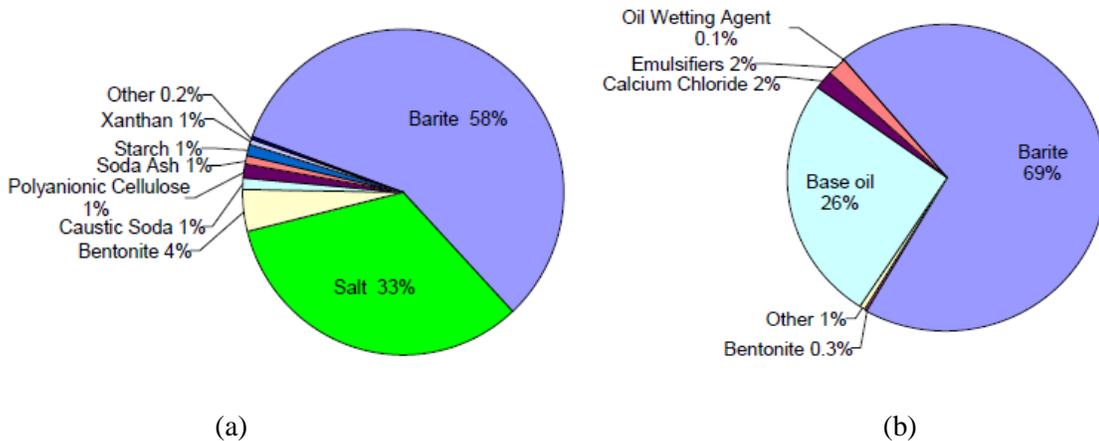


Figure 2.27: example of Typical Water and Oil based Mud Composition (% of weight)  
(Adapted from Ukooa drill cutting Programme, 1999a)

### 2.6.2 Function and Performance of Drilling Muds

Fluids or muds used in surface and offshore drilling activities range from simply water or oil (diesel, vegetable or mineral oil) to compressed air and pneumatic fluids to more multifaceted water-based or oil-based systems. Such additive that constitutes a typical drilling fluid includes weighting materials; viscosifier; filtration control additives; pH/alkalinity control chemicals; dispersants/deflocculants/thinners; surfactants and emulsifiers; shale inhibitors; corrosion inhibitors/oxygen scavengers/hydrogen sulphide (H<sub>2</sub>S) scavengers; lubricants; and bridging agents/ lost circulation materials (LCMs) (IADC Drilling Manual: Drilling Fluid, 2014; Caenn and Chillingar, 1996);



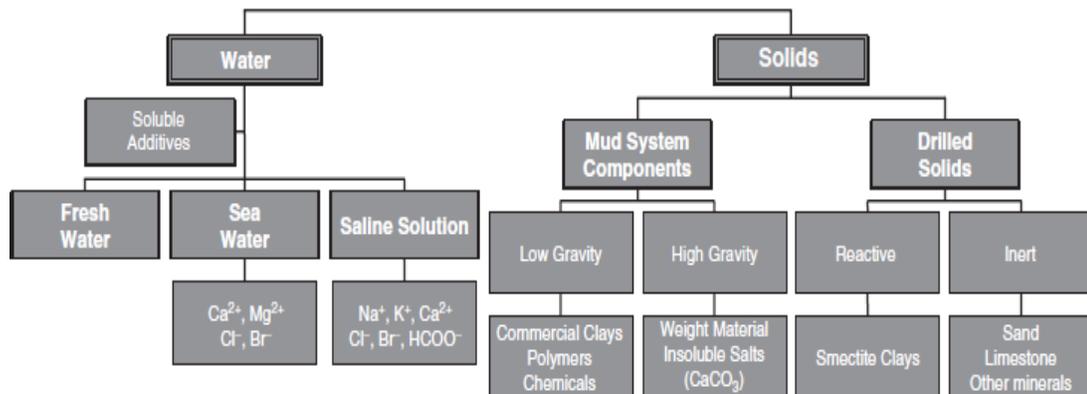
Figure 2.28: Drilling fluids are major factors in a successful drilling program. Courtesy MI-SWACO, a Schlumberger company

The performance and the function of fluids employed for drilling operations depend upon the type of formation being drilled and the unique properties of the selected fluids. However, compromises may be made often times to achieve a balance in the selection and design of a particular drilling fluid. Such consideration is required to tailor the fluid properties which depend on the complexity of the well, subsurface pressures and temperatures, logistics, cost of equipment and other local expenses. The properties of the drilling fluid are selected on the basis of the available hydraulics, whose horsepower at the bit is optimized to improve the rate of penetration (ROP) and the drilling bit life span. To do these, an adequate fluid flow rate is used with minimal overbalance. To optimize the drilling performance, the properties of drilling mud and circulation rate, density of the mud and the nature of the suspended solids are adequately selected and computed in-practice to balance the parasitic pressure losses in the drill string and the available pressure at the bits, and enhance the rate of penetration. This is achieved through adequate interpretation of the results obtained from regular and complete test, designed to control the mud properties (Strachan, 2010; IADC Drilling Manual: Drilling Fluid, 2014; Engelhardt et al., 1983).

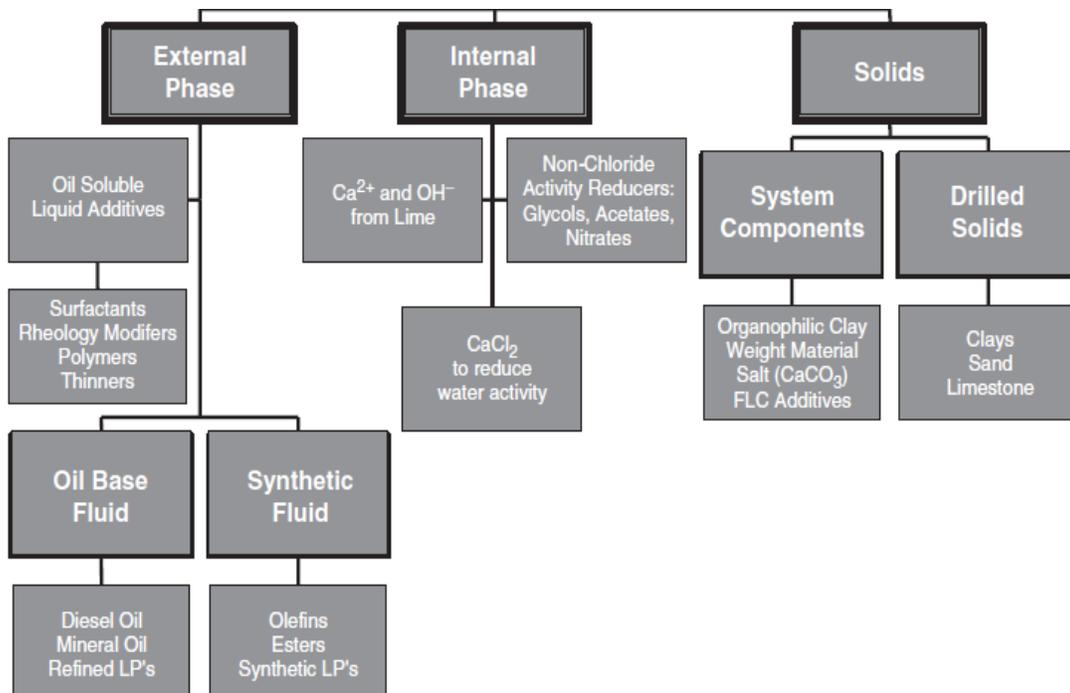
The most critical function a drilling fluid performs is to minimize the concentration of cuttings around the drill bit and throughout the well bore. To do these, the fluid assumes cuttings burden, cleans the hole and creates thick filter cakes. These functions are as follows:

- Cool, lubricate and support the drilling assembly (drill bit and drill string), transmit hydraulic energy to the drill bit and downhole tools
- Assist in removal of drill cuttings or dormant weight material from the well bore, beneath the bit and in the surface fluids processing system in order to allow them circulate to the surface.
- Control subsurface pressure, maintaining well control to prevent any blowouts from the well

- Maintain borehole or wellbore stability, mechanically and chemically by protecting produced formations by minimizing formation/fluid interactions and sealing the wall of the bore hole with an impermeable cake.
- Control corrosion of the metal components of the drilling tools, casing and rig facilities that are exposed to the corrosive marine environment
- Minimize reservoir damage and impact on the environment, inhibit gas hydrate formation and facilitate cementing, drilling penetration rate and completion.
- Permit adequate formation evaluation, seal permeable formations and form a low permeability, thin and tough filter cake across permeable formations to control formation pressure and maintain wellbore conditions.
- Suspend drilled cuttings, the weight of the drill string, casing, and caving-in the annulus when circulation is stopped (Barlow and Kingston, 2001; Caenn and Chillingar, 1996; Wills, 2000; Growcock, and Harvey, 2005)



(a)



(b)

Figure 2.29: Types of Water based and Invert-Emulsion Drilling Muds. (Growcock and Harvey, 2005)

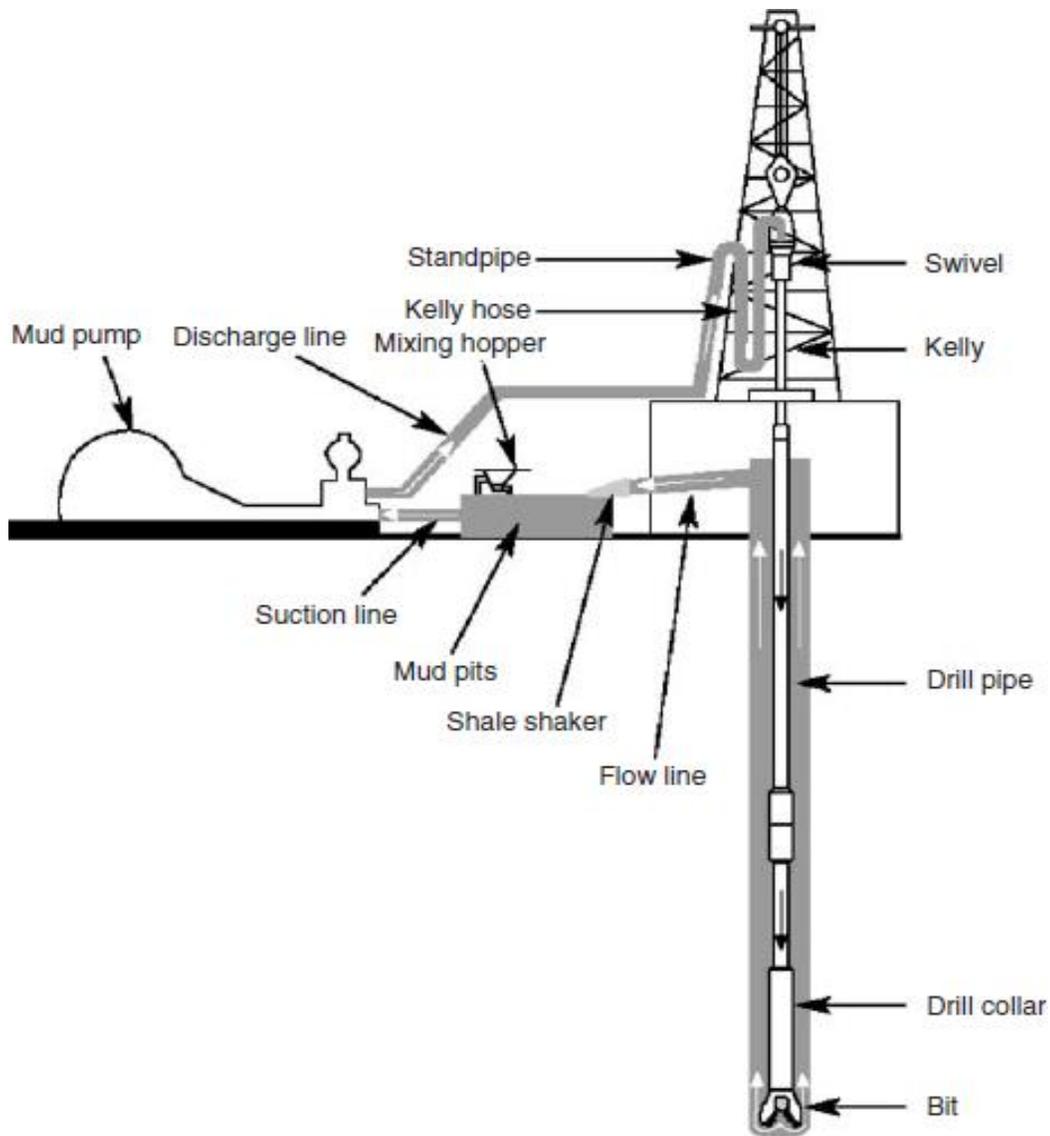


Figure 2.30: Drilling Fluid Circulating System. (Courtesy of M-I SWACO)

### 2.6.3 Testing of Drilling Mud Properties

The American Petroleum Institutes (API) has recommended and endorsed standard methods of conducting field and laboratory tests for drilling fluids. These detailed procedures may be found in the API publication, “Recommended Practice: Standard Procedure for Field Testing Water-Based (Oil-Based) Drilling Fluids,” API RP 13B-1, 13B-2 and supplements (13I for Laboratory Testing Drilling Fluids). The problem statement and research needs associated with drilling operations are better identified through regular and adequate monitoring and interpretation of any deviation from reality, qualitative and quantitative routine field testing. To examine the suitability of a weighting agent as candidate in drilling mud for oil drilling

mud application, the following test are conducted (Osokogwu et al., 2014; API, 2010; API, 2004):

- **Mud Weight (Density):** This is determined with a mud balance which consists of a cup with lid mounted at the end of a graduated arm. Mud weight is a pressure dependent property. The formation pressures of the oil well are contained by the hydrostatic pressure of the drilling fluid, which is a function of depth and mud density (Ibe et al., 2016; Joel, 2013). The experimental setup for such analysis is given in the Figure 31.

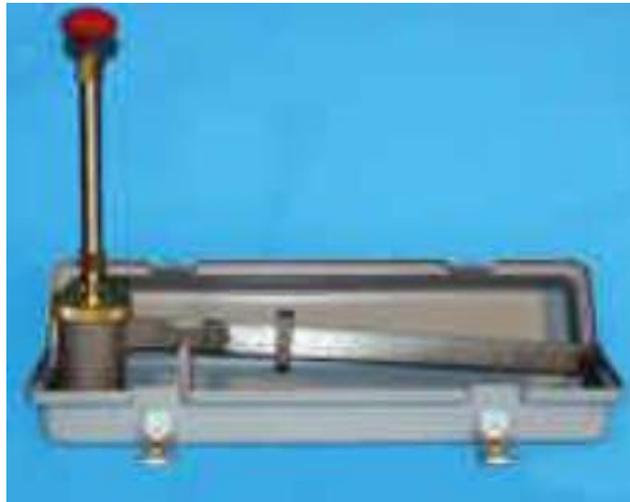


Figure 2.31: Pressurized mud balance (Source: Joel, 2013 at POCEMA)

- **Rheological Measurement:** The drilling mud rheological properties are from a direct-reading measurement. This is done using the Fann viscometer as shown in Figure 2.32. It is simple to use, and provides readings that are easily converted to the rheological parameter's plastic viscosity and yield point of the drilling mud (Joel, 2013; Osokogwu et al., 2014).



Figure 2.32: Fann Viscometer (Source: Petroleum Engineering Laboratory, Drilling Lab, AUST, Abuja; Joel, 2013 at POCEMA).

- Fluid Loss Determination: This involves the determination of the filtration or wall-building property of a drilling mud. The test consists of determining the rate at which a typical drilling mud or fluid is forced through the filter paper. The test is run under specified conditions of time, temperature and pressure (Ibe et al., 2016). Figure 2.33 revealed the experimental set up for the test.



Figure 2.33: Low and High-Pressure High-Temperature (HPHT) Filtration cell (Source: Petroleum Engineering Laboratory, Drilling Lab, AUST, Abuja; Joel, 2013 at POCEMA)

- Retort: The retort affords a method for measuring the percentage (%) of oil to the ratio of water. It is used for estimating both suspended and dissolved solids contained in sampled water-based, oil-based or any other types of drilling muds and cuttings (Joel, 2013). The laboratory-scale table top retort is shown in Figure 2.34.



Figure 2.34: Retort (Source: Petroleum Engineering Laboratory, Drilling Lab, AUST, Abuja; Joel, 2013 at POCEMA)

- Free Fluid and Sedimentation Determination: This is a free-water test designed to simulate a mixture of the solids in water, using a 250ml graduated cylinder. The slurry formed is left to stand for two hours under the simulated wellbore conditions. The volume of water extracted at the end of two hours is measured and expressed as a percentage by volume. This is done to mitigate channel formation and gas migration problems through a more critical test inclining the column at 45° (Joel, 2013).



(a)

- **Thickening Time:** This experiment determines the maximum available pumping time of slurry or typical drilling mud before the slurry reaches a phase transformation where the flow through the pump has become difficult, prior to setting. This stage is described as consistency. The drilling fluid or slurry viscosity (consistency) under the simulated well pressure and temperature is measured by the application of High temperature/High Pressure Consistometer (HPHT). The experimental set up for this analysis is shown in Figures 2.35 (Joel, 2013).



(b)

Figure 2.35: High-Pressure/High Temperature (HPHT) Consistometer (Source: Petroleum Engineering Laboratory, Drilling Lab, AUST, Abuja; Joel, 2013 at POCEMA).

- **Atmospheric Consistometer:** This laboratory equipment is used extensively in oil well cement testing applications as pre-conditioning cement slurry, prior to other tests such as the fluid loss, free water or rheological testing. To do this, the prepared slurry sample is conditioned for 20 minutes at specified test temperature of 190F (105.6°C) in an atmospheric Consistometer at atmospheric pressure before measurements are taken (Joel, 2013).



Figure 2.36: Atmospheric Consistometer (Source: Petroleum Engineering Laboratory, Drilling Lab, AUST, Abuja; Joel, 2013 at POCEMA).

- Fluid loss: The bottom hole circulating temperature (BHCT) measures the fluid loss of drilling fluid or mud and cement slurry. The sampled drilling fluid was conditioned for 20 minutes. To measure the fluid loss of the sample, conditioned slurry formed is placed in the fluid loss cell and 1000psi differential pressure is applied across the 325-mesh screen as shown in Figure 2.37 (Joel, 2013).

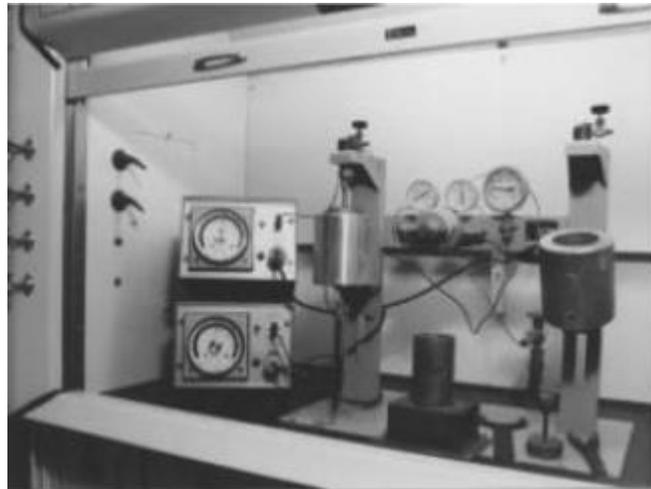


Figure 2.37: Static Fluid loss Cell (Source: Petroleum Engineering Laboratory, Drilling Lab, AUST, Abuja; Joel, 2013 at POCEMA).

- Compressive Strength: This is a pressure dependent measurement. The compressive strength measures the pressure it takes to crush the set cement made from drilling mud, using either the autoclave system or the ultrasonic analyzer (UCA). This

indicates how the cement sheath will withstand the differential pressures in the oil well (Joel, 2013; Ibe et al. 2016). The experimental set up is shown in Figures 38a and b.



(a)



(b)

Figure 2.38: An Autoclave System and Ultra Sonic Cement Analyzer (Source: Petroleum Engineering Laboratory, Drilling Lab, AUST, Abuja; Joel, 2013 at POCEMA).

#### 2.6.4 Petrology and Geochemistry of Barite from Torkula Field

This study measures or determines the mineralization potentials, the contents, distributions and textures of the various veins through petrographic studies and gravimetric method of analyses. Estimating the relative sizes of the gangue minerals inter-locked up with the barite enables the mineral processor to choose the right sizes to crush the ore, as required to liberate all locked up particles prior to beneficiation. The host rock to Torkula barite mineralization is the ubiquitous sandstone and shale within the prospect. Zones with high potassium and low thorium/potassium ratio irregularities indicated potassium enrichment associated with sericitic or K-feldspar alterations, regional and localized aeromagnetic irregularities with associated lineaments are the target for Barytes and Lead-Zinc (Udinmwen et al., 2016; Tanko et al., 2015).

Torkula barite has shown a predominance of BaO, PbO, ZnO, and SiO<sub>2</sub> in the samples (i.e. 50% and above). Other minerals also occur in recoverable grades (over 5%). This shows the richness of the site mineralization. There is high sulphur content in the barite and Zinc

mineralization. The S.G. test conducted by the National Metallurgical Development Centre (NMDC), Jos evidenced S.G. of 4.5 and 7.4 for barite and Lead respectively (PIN, 2017).

## 2.7 American Petroleum Institute (API) and other Oil Industry Standards

The American Petroleum Institutes (API) Standards recognizes the refining characteristics of crude petroleum and the effects of the application of the associated materials that constitutes the drilling fluid as it moves across the production, transportation and refining sectors of the oil industry. To do these, the API Task Force has been set up to enforce and maintain the integrity and quality of the entire drilling and refining processes. Here is the API Specification 13A for various classes of barite for oil drilling mud application (Birch, 1989).

Table 2.2: Specification for Drilling Fluids: Specifications and Testing (API, 2004; API, 2010)

Specification for Drilling Fluids:	Specifications and Testing
<b>Product:</b>	<b>Barite</b>
Grade:	Drilling grade
Product Description:	API Specification 13A – 7.1.1 Drilling-grade barite is produced from commercial barium sulfate-containing ores. It may be produced from a single ore or a blend of ores and may be a straight-mined product or processed by beneficiation methods, i.e. washing, tabling, jigging or flotation. It may contain accessory minerals in addition to the barium sulfate (BaSO <sub>4</sub> ) mineral. Because of the presence of gangue minerals, commercial barite can vary in colour from off-white to grey to red or brown.
Product Physical Requirements:	API Specification 13A – 7.1.2

	Drilling-grade barite shall be deemed to meet the requirements of this International Standard if a composite sample representing no more than one day's production conforms to the chemical and physical specifications, represents the product produced and is controlled by the manufacture
<b>Product:</b>	<b>Barite 4.1</b>
Grade:	Drilling grade
Product Description:	<p>API Specification 13A – 20.1.1</p> <p>Drilling grade barite is produced from commercial barium sulfate-containing ores. It may be produced from a single ore or a blend of ores and may be a straight-mined product or processed by beneficiation methods, i.e. washing, tabling, jigging, or flotation. It may contain accessory minerals other than the barium sulfate (BaSO<sub>4</sub>) mineral. Because of gangue minerals, commercial barite may vary in colour from off-white to grey to red or brown. These gangue minerals include silicates such as quartz and carbonate compounds such as siderite and dolomite, and metallic oxide and sulfide compounds. Although these minerals are normally insoluble, they can react with other components in some types of drilling fluids and cause adverse changes in the drilling fluid properties under certain conditions</p>
Product Physical Requirements:	<p>API Specification 13A – 20.1.5</p> <p>Barite 4.1 shall be deemed to meet the requirements of this International Standard if a composite sample</p>

	representing no more than one day's production conforms to the chemical and physical specifications, represents the product produced, and is controlled by the manufacturer.
<b>Testing Product:</b>	<b>Calibration barite</b>
Grade:	Calibration
Product Information:	API Specification 13A – Annex B.1.2  Calibration barite and test calibration Bentonite are available through the API offices (see 4.2.1) for use by laboratories to determine their test precision.

Table 2.3: Barite physical and chemical requirements (API, 2004; API, 2010)

<b>Requirement</b>	<b>Standards for Barite</b>	<b>Standards for Barite 4.1</b>
Density	4.20 g/ml, minimum	4.0 g/ml, minimum
Water-soluble alkaline earth metals, as calcium	250 mg/kg, maximum	250 mg/kg, maximum
Residue greater than 75 micron	Maximum mass fraction, 3.0 %	Maximum mass fraction, 3.0 %
Particles less than 6 micron in equivalent spherical diameter	Maximum mass fraction, 30 %	Maximum mass fraction, 30 %

Table 2.4: Combined API/Company Barite Specification (API, 2004; API, 2010)

<b>PROPERTY</b>	<b>UNITS</b>	<b>REQUIREMENTS</b>
		S

Density	g/cm <sup>3</sup>	4.20 minimum
Water Soluble Alkaline Earth Metals as Calcium	mg/kg	250 maximum
Residue greater than 75 micrometers	% w/w	3.0 maximum
Particles less than 6 micrometers in equivalent spherical diameter	% w/w	30 maximum
Particles less than 4 micrometers in equivalent spherical diameter	% w/w	20.0 maximum
C. E. C	meq/10gms	0.18 maximum
Extractable Carbonates	mg/kg	3000 maximum
Cadmium	mg/kg	5 maximum
Lead	Mg/kg	1000 maximum
Mercury	Mg/kg	5 maximum
Moisture Content	% w/w	1.0 maximum
Flotation Chemicals	% w/w	Zero
Hematite	% w/w	Zero
SAPP	% w/w	Zero
<p>Other:</p> <p>The presence of sand can be observed under a microscope.</p> <p>Additional properties may be determined according to API standardized test procedures (Reference RP 131, June 1, 1995, for the following controls:</p> <ul style="list-style-type: none"> <li>• Abrasive capacity of the weighting agent</li> <li>• Presence of mercury, cadmium and lead</li> </ul>		

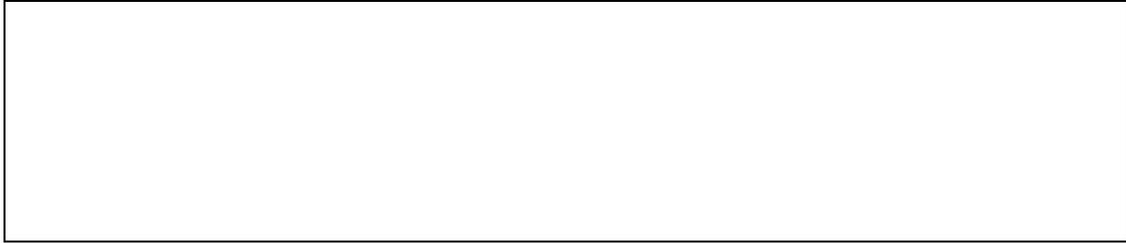


Table 2.5: Typical Physico-chemical properties of Barite for Drilling Mud Application (API, 2004; API, 2010)

<b>Typical Analyses of barite</b>	
Specific Gravity (g/cm <sup>3</sup> )	<b>4.35</b>
Specific Resistance (ohms/cm) (ASTM D-2448-85)	7750
Average Particle Diameter (MV)	11
Median Particle Diameter	10.5
325 Mesh Retention (%)	0.5
Hegman Fineness (ASTM D-1210)	4.5
Brightness (%)	91
Oil Absorption (g oil/100g) (ASTM D-281)	8.0
Weight/solid gallon (lb)	36.3
One Pound Bulk (gal)	0.0275
Bulk Density (lbs/ft.3) (ASTM C-110)-Loose Tapped 144	85
<b>Typical Chemical Analyses</b>	
BaSO <sub>4</sub>	95%
Fe <sub>2</sub> O <sub>3</sub>	0.03%

CaO	0.01%
MgO	0.04%
Al <sub>2</sub> O <sub>3</sub>	0.035%
Silicates	0.01%
Loss On Ignition (LOI)	0.5%
Total Heavy Metals	0.001%
Water Soluble Salts	0.1%
pH	7-9.5
Moisture (%)	0.1%

## 2.8 Selection of Weighting agents and other Drilling Fluid Additives

### ❖ Choice of Weighting Agent:

The choice of weighting agent required for oil drilling mud, solely depend on the entire properties of a material. Such properties include mechanical, chemical, thermal, electrical, physical, biological, optical and magnetic properties in relation to the nature of the well bore. For appropriate selection of materials that give the best combination or balance of these properties, candidate weighting agent for oil drilling fluids is chosen on the need to;

- Provide materials or fluids with low rheology (low plastic viscosity) particularly in high density fluids
- Demonstrate low settling tendency and providing low sag as a result of the solubility or miscibility.
- To be simply removable from the reservoir by mechanical or chemical means

- To be sufficiently hard enough not to create fines during drilling which may cause formation damage or high gel values.
- To have a good health, safety and environment (HSE) profile.
- Would contain a minimum of coarse particles to prevent abrasion.
- To be sustainable and readily available and in large quantities
- To be cost effective, locally beneficiated and process to high quality (Omoniyi and Mubarak, 2014; Al-Bagoury and Steele, 2012, API, 2005).

❖ Selection of Drilling Fluids:

The selection materials with best combination of properties required to meet anticipated conditions will minimize the well costs and reduce the risk of catastrophes, posed as a result of stuck drill pipe, loss of circulation and gas-kick. Most serious consideration should also be given to obtain more comprehensive formation evaluation and maximum productivity. Such considerations will affect the choice of a drilling mud. To do this, scientifically proven and tested synergy ought to be in place to meet specific conditions presented as follows (©Halliburton energy services company, US6548452, April, 2013; Haaland et al., 1976).

- Location: The availability of supplies must be considered as a priority in the scale of preference, i.e., in an offshore well, the possibility of using salt water should be considered on the basis of application.
- Mud-making shale: Thick shale sections containing dispersible clays result into sharp increase in viscosity as cuttings become incorporated in the mud. Moreover, it is easy to correct the situation of extreme viscosity when the drilling mud is yet to be weighed. However, when the mud is weighted, costly chemicals such as barium sulphate is used to restore the mud properties.
- Pressured formations: The density of the drilling mud is adjusted as pressurized formations are to be drilled. Conversely, high density muds increase the cost of drilling and incurred the risks of stuck pipe and loss circulation.
- High temperature: Most of the Drilling-grade mud additives degrade with time and at elevated temperatures higher than degradation temperature. Such special additives are used to make the drilling mud resistive to high temperatures.

- Hole instability: Hole contraction and hole enlargement are the most frequent forms of hole instability. In a situation when the imaginative earth stresses bearing on the walls of the hole surpass the yield strength of the formation, hole slowly contracts. The density of the drilling mud ought to be designed as higher, enough to resist contracting. Hole enlargement occurs at water sensitive shale zones. This is corrected or at least prevented through the application of shale stabilizers.
- Rock salt: The oil-base drilling mud (OBM) or saturated brine is often employed to prevent the mineralized salt from dissolving and consequently enlarging the hole.
- Hole inclination: In highly deviated holes, torque and drag are the major problem. The risk of pipe stuck is high due to the position of the pipe, lying against the low side of the hole. Proper and comprehensive selection of the drilling mud is a measure to prevent such problems and keep well cutting in proper position.
- Formation evaluation: The selected drilling mud should be suitable for logging tools and productivity impairment. Similarly, solids control or density adjustments should be considered properly to keep the formations free from being damaged or blocked (Omoniyi and Mubarak, 2014; Menzel et al., 1973; Al-Bagoury and Steele, 2012).

### 3.0 Materials and Methods

#### 3.1 Location of the study area, Sampling and Collection

❖ Location of the Study area:

The study area is strategically located in Hungwa, Torkula Village, Guma local government area, Benue State, Nigeria. Guma LGA in Benue State has a population of over 200,000 and the total land mass of about 28,882km<sup>2</sup>. It is surrounded with sedimentary rocks and the soil dominantly associated with mineral deposits like barite lead, salt, feldspar, and brown earth from volcanic eruption. Guma lies between latitude N7° 32” and N8° 51” and longitude E9° 35” and E9° 22” with an average high rainfall of about 1198 – 1798mm annually as shown in Figure 2.1(Ehirim et al., 2016). Relative humidity is between 43 – 86%, temperature ranges from 27° and 37°C. Similarly, Torkula village lies between latitude N7°12’54”/E9°0’22” (Sambo et al., 2016). It is characterized with large, interlocked, euhedral crystal growths, cocks-comb structure, and mineral inclusions. The formation consists of hard grey and black calcareous shale and ubiquitous sandstone within the prospect shown in Figure 2.1(Ehirim et al., 2016).

Baryte occur in veins-type and cavity filling open-space deposition within sediments associated with Lead-Zinc mineralization in the trough. Baryte mineralization in the Torkula area occurred as a hydrothermal event, directly connected with the Mesozoic Magmatism during the early Cretaceous. Possible sources to these are the leaching of barium out of country rock (ubiquitous sandstone) by hydrothermal brines. Convection cells of meteoritic water, with upward-moving hydrothermal fluid being concentrated along the line occupied by fractures, offer a possible mechanism (Ehirim et al., 2016). Many mineralized veins occupy NW-SE fracture within the prospect area. The E-W trends result to the strongest and most persistent veins and the N-S trend the weakest. Mineralization is structurally and strategically controlled and more or less confined to the limit of these faults (Chaanda et al., 2010).

❖ Sampling and Collection

The barite samples were randomly collected from the heaps of barite run-of-min as shown in the Figure 3.1. The blasted barite mineral were selected after several beneficiation

processes such as enrichment, upgrading and appropriate material handling. To do these, the pure barite was separated from the coloured barite mineral, collected into cellophane bags (black polythene bag) and appropriately labeled. Collected samples were labeled as A, B, C, D, E and F. Sample A is white coloured barite with relatively small portion of sandstones, brown coloured barite as sample B, different fractions of white and transparent barite samples as samples C and D. However, samples E and F are whitish but with some associated gangue minerals interlock within the barite vein. These samples were only randomly selected from various heaps of run-of-mines, from the second and third mines. For a comprehensive comparison and scientific criticism, research works on reference barite samples collected from Nigeria Petroleum Development Company (NPDC) Warri Field Office were cited.

### 3.2 Sample Preparation

The air-tight barite samples selected from the Hungwa mining site were crushed and milled using Jaw crusher combined with Pendulum Pulverized Milling machine. The crushed sample was then sieved and screened using vibrating screen to 0.600mm mesh size. This was achieved with the aid of an electric device which comprises up of an agitator coupled with a mesh of various sizes, ranging from 3mm to 45 micron (see Figure 3.1). The samples were poured gently into the upper sieve while lowering the samples across the various mesh designations, to collect the finest samples at the receiver plate. The sieving and screening is done to prevent the abrasion of casing during its potential usage by coarser grain particles during drilling (Ibe et al., 2016).



(a)



(b)

Figure 3.1: Arrangement of Sieves of Various Mesh Sizes (3mm-45 micron) (Source: Omoniyi and Mubarak, 2014).

### 3.2.1 Formulation of Drilling Mud Sample

The formulation and production of drilling mud and the determination of the rheological and allied properties of the mud were carried out based on the API drilling mud production standards (API, 1993; API, 2000; Ogbonna, 2010; Joel et al, 2012; Omotioma et al., 2015). The mixing method employed by Omotioma et al (2015) was adopted. The various quantities of the raw materials required to formulate globally recommended drilling mud were measured using the graduated cylinder for liquids. Spatula and electronic weighing balance were used to measure those materials in the powdery form. The raw materials were then poured simultaneously, within an interval of 5 minutes, into the steel cup of the single spindle mixer. As each component of the drilling fluid is being put into the mixer, it is powered to cause the spindle to rotate and mix the contents inside the steel cup held at a fixed position. At the end of the formulation and addition of these materials into the mixing medium, it was allowed to stir continuously for 30 minute in order to achieve finely formulated water based drilling mud (Omotioma et al., 2015).

Table 3.1: Different Formulations for Water-based Drilling Mud (Osokogwu et al., 2014; Omoniyi and Mubarak, 2014).

S/ N	Formulation for WBDM (1)		Formulation for WBDM (2 and 3)		
	Additive	Quantity	Additive	Quantity (2)	Quantity (3)
1	Distilled Water	1 quart	Water	340 ml	345 ml
2	Barite	20 g	Bentonite	8 g	4.0 g
3	CMC	20 g	Pac-R	1.0 g	0.5 g
4	Caustic soda (NaOH)	20 g	Pac-L	1.0 g	0.5 g
5	Bentonite	160 g	Xanthan gum	1.0 g	0.5 g

6			Caustic Soda	2.0 g	1.0 g
7			Potassium Chloride	10.0 g	5.0 g
8			Soda Ash	0.5 g	0.25 g
9			Barite	10.0 g	5.0 g

### 3.3 Characterization of Barite from Turkula Field

The micronized barite sample collected from Hungwa, Torkula Barite Field of Guma local government area, Mid-Benue Trough, Benue State, Nigeria was characterized. This was done through methods of instrumental characterization. The Empyrean diffractometer DY 674 (2010) for (XRD), Minipal 4 energy dispersive X-ray Fluorescence (XRF) and Scanning Electron microscope (SEM) for micrograph was used for phase composition, elemental and morphological analysis respectively.

#### 3.3.1 XRD Analysis (phase analysis) of the Sample

The micronized barite sample was analyzed using the reflection-transmission spinner stage using the Theta-Theta settings. The Two-Theta starting position starts from 15° and ends at 80° with a two-theta step of 0.0396 at 0.3 seconds per step. Tube current was 30mA and the tension was 40kV, as the energy specification for the instrument, at an incident beam of 1 mm. Fixed Divergent Slit (DS) size of 1° is used on a Position Sensitive Diode (PSD) detector and the goniometer radius is 240mm. The intensity of diffracted X-rays was continuously recorded as the sample and detector rotate through their respective angles. Similarly, a peak in intensity occurs when the mineral contains lattice planes with d-spacing suitable to diffract X-rays at that value of  $\theta$ , although each peak consists of two dispersed reflections ( $K\alpha_1$  and  $K\alpha_2$ ), at small values of  $2\theta$  the peak locations overlap with  $K\alpha_2$  appearing as a hump on the side of  $K\alpha_1$ , However, greater separation occurs at higher values of  $\theta$ . Typically these combined peaks are treated as one and the  $2\lambda$  position of the diffraction peak is typically measured as the center of the peak at 80% peak height.

Moreover, results are presented as peak positions at  $2\theta$  and X-ray counts as intensity in the form of a table, and x-y plot. For a clear-cut analysis, intensity (I) is reported as peak height intensity, either as above background, or as integrated intensity, within the area under the peak. Similarly, the relative intensity is recorded as the ratio of the peak intensity to that of the most intense peak (relative intensity =  $I/I_1 \times 100$ ). Thus, the d-spacing of each peak is then obtained by solution of the Bragg equation for the appropriate value of  $\lambda$ . Once all d-spacing have been determined appropriately, automated search or match routines compared the unknown values to those of known materials. Since each mineral has a unique set of d-spacing, matching these d-spacing provides an identification of the unknown sample. A systematic procedure was used by checking the d-spacing in terms of their intensity beginning with the most intense peak. Files of d-spacing for hundreds of thousands of inorganic compounds are liable from the International Centre for Diffraction Data as the Powder Diffraction File (PDF). The peaks obtained from the analyses were matched with the minerals from ICDD database which is attached to the software of the machine for proper and accurate interpretation of the results (Olusegu et al., 2015).

### **3.3.2 XRF Analysis (elemental analysis) of the Sample**

10g solid pelletized sample of barite was placed in the special "dekat" (rice cake) plastic cup sample holders. The sample was formerly loaded on the sample changer and the position label was noted appropriately. Beads used in the major oxide analysis were SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, TiO, MgO, CaO, K<sub>2</sub>O and P<sub>2</sub>O<sub>5</sub>, expressed in weight percent were prepared. 10g of pulverized sample was weighed and mixed exactly with 5 times of X-Ray flux consisting of 66.0% lithium Tetraborate (Li<sub>2</sub>B<sub>4</sub>O<sub>7</sub>) and 34.0% Lithium Metaborate, (LiBO<sub>2</sub>). This weighed mixture was mixed properly in a Platinum crucible and ignited in apre-set furnace at 1150°C for 10 min.

The weighed mixture in the crucible was immediately placed on the crucible holder, and the preparation process commenced as the start button was pressed on. The mixture was heated, mixed and automatically cast into a mold. During this process, a releasing agent was added (ammonium iodide tablet). Upon completion of cooling, the cold glass bead was unloading and made ready for the analysis. However, the glass bead was labeled and slotted into the

Minipal 4 energy dispersive XRF for major oxide analysis. For XRF Trace Elemental Analysis, the pellets were prepared by weighing 10g of oven dried pulverized sample and 5.0g cellulose powder (flux) added as a binder. This is mixed thoroughly with glass rod in a bowl. The well mixed was then compressed by applying a pressure of 1500kgm using manual compressor. The steps carried out for measurements were; Loading the sample, and opening the measure window for the required application (Olusegu et al., 2015).

### **3.4 Sample Analyses**

#### **3.4.1 Physical Parameter**

##### **3.4.1.1 Determination of Sample pH**

The pH-meter consists of a stirrer and a sensitive scale required in measuring the pH of the drilling mud and the temperature at which the pH was measured (Omoniyi and Mubarak, 2014). PH meter was calibrated with buffer solution of pH 4, 7 and 10 before using (Ibe et al., 2016). After calibrations, the meter (at the sensor) was rinsed properly, prior to the measurement. The electrode was clean and made ready for use. The meter was turn off and cover close to protect the instrument (Osokogwu et al., 2014). Twenty (20) grams of the sieved barite sample was placed in a beaker and 20ml distilled water was added to it. This mixture was stirred continuously until slurry was formed. The slurry was allowed to stand for seventy (70) minutes, and stirred for about 10 min before putting the electrode of the just calibrated pH meter into it (Ibe et al., 2016). The electrode remained immersed in the sample slurry until the meter stabilized, the reading was done and the results recorded respectively. This was repeated after twenty-four (24) hours and specifically at the temperature range of 21 and 27°C according to the pH manufacturer's instruction. To work within this limit, the barite slurry as prepared was analyzed at the temperature of 26.3°C. The pH meter electrode was cleaned and stored in accordance with manufacturer's instruction (Washington State Department of Transportation (WSDT), 2009; Ibe et al., 2016).

##### **3.4.1.2 Determination of Sample Specific Gravity**

This indicates the ratio of the weight of barite in water to the weight in other liquid. The specific gravity of the sample was determined in accordance with IS 2720-II standards specification, Singh and Chowdhary, 2009.

A mud balance (mud scale) consists of a graduated beam with a bubble level, a weight slider along its length and a cup with a lid on one end. The cup was used to hold a fixed amount of fluid for weighing. A slider weight can be moved along the beam and a bubble indicates when the beam was level. Density was read at the point where the slider-weight sits on the beam at level. Calibration was done using a liquid of known density, often water by adjusting the counter weight (Ibe et al., 2016; Henn, 2003).

A clean, dry 100ml beaker was weighed and recorded as  $M_1$  and a 15g air-dry sample was also weighed on the same balance and emptied into the 100ml beaker. The soil was made to pass through a 75 micron sieve. Both pulverized sample and the beaker were weighed and recorded as  $M_2$ . The sample was allowed to dry without heating (heating of the soil will change the natural specific gravity). The sample was covered with distilled water in the beaker and left for two hours. The beaker was then placed inside a vacuum desiccator connected to a stopper and a vacuum pressure was slowly applied on the sample using a vacuum pump. The de-gassing process continued until no bubble was observed on the surface of water. The sample was allowed to cool and weighed as  $M_3$ . The beaker was emptied, thoroughly drained and washed, refilled with distilled water and weighed immediately as  $M_4$ . The test was repeatedly done on other samples.

#### **3.4.1.3 Determination of Sample Moisture Content**

5, 10 and 15 grams of the micronized barite samples were weighed into a dry and clean crucible was placed. These were placed in an oven, working at an operating temperature of 250°C for 3hrs. At the end of the heating period, the samples were removed from the oven and kept in a Desiccator, to cool for about 3hrs. The cooled samples were weighed as the dried weight of the samples. The entire process was repeated after 3hrs until a constant weight was obtained (Ibe et al., 2016).

#### **3.4.1.4 Determination of Sample Mohr's Hardness**

Five (5) fully-enriched lumps (un-pulverized) barite sample was scratched on the surface with the edge of a penny and an etched line was noticed. This was observed with the aid of a

finger nail, to feel for a scratch and validate the nature of the scratch. If the barite is scratched, then it is softer than or equal in hardness to the test material, and if otherwise, it is harder than the test material. This method relies upon a scratch test in order to relate the hardness of a mineral specimen to a number from the Mohr's scale (Alan, 1997). The scale was defined using an assembled set of common reference minerals of varying hardness and labeled these in order of increasing hardness from 1 to 10.

These materials are:

Talc: 1, Gypsum: 2, Calcite or Penny 3, Fluorite: 4, Apatite: 5, Orthoclase: 6, Quartz: 7, Topaz: 8, Corundum: 9, Diamond: 10.

Similarly, the samples may also be scratched against either a knife, Coin, glass or finger nail. The observed results are evaluated on the Mohr's scale of minerals hardness (Ibe et al., 2016; Omoniyi and Mubarak, 2014).

### **3.4.2 Chemical Parameter**

#### **3.4.2.1 Determination of Metals**

0.5g of the sieved micronized barite sample was dissolved in 20ml of chloroform ( $\text{CHCl}_3$ ), and the resulting solution was poured into a separating funnel containing 20ml of 10% nitric acid ( $\text{HNO}_3$ ). The mixture was properly and thoroughly shaking for 10 minutes. The agitated medium was allowed to settle until the mixture is completely separated into layers. After thorough shaking and phase separation, the  $\text{CHCl}_3$  layer (denser phase) was separated from  $\text{HNO}_3$  layer (less dense phase) containing trace metals. The  $\text{HNO}_3$  layer was drained into a 50ml volumetric flask and made to mark with distilled water (Akpan, 2005). The blank was prepared similarly without the addition of the micronized barite sample. The metallic content in the digested samples were analyzed using atomic absorption spectrophotometer (AAS), Model: A-Analys 100. Liquid-liquid extraction method (LLEM) was employed in the absorption or digestion of the sample (Ibe et al., 2016).

#### **3.4.2.2. Determination of Sulphate**

25ml of the extracting solution (0.5g/L of  $\text{KH}_2\text{PO}_4$ ) was added to 5g of the micronized barite sample in a shaking bottle. The mixing was done on a mechanical shaker and subsequently filtered. Similarly, 10ml of the extract was pipetted into a 25ml volumetric flask and 10ml of

distilled water added to it. In addition to these, 1ml of gelatin–barium chloride was measured using a syringe, added and made up to volume with distilled water.

The content was mixed thoroughly and allowed to stand for 30 minutes. A little fraction of the mixture was measured into the cuvette and the absorbance was measured at 420nm using a direct reading spectrophotometer (Model T60 UV-Visible Spectrophotometer). A calibration curve was plotted and the concentrations of the samples were extrapolated from the calibrated graph. A plot of concentration vs. absorbance gives a straight line graph for the standard readings with molar absorptivity or extinction coefficient of 0.997. The method of IITA (1979) was adopted for the determination of the sulphate in the sample. This was done in-line with the recommended Specifications (Ibe *et al.*, 2016; IITA, 1979).

#### **3.4.2.3 Cation Exchange Capacity and Methylene Blue Absorption of Torkula Barite:**

These analyses suggest and validate the estimate of the total cation exchange capacity (CEC) of the solids (i.e. barite) in the drilling mud. It also indicates the amount or quantity in milliequivalent of the reactive clays and other additives (barite) present in the drilling mud. The analyses relate to the effect contributed by the presence of shale cuttings on the borehole stability, completion properties, rheology enhancement and other numerous mud making properties. Hence, the amount of the methylene blue dye absorbed is an indication of the clays Base Exchange Capacity.

To do this, the prepared drilling mud was manually stirred with a glass rod continuously for 5 minutes in order to break the gel. With the aid of 2ml syringe, little portion of the fluid was quickly drawn, with the inlet/outlet a little centimeters above the fluid. In the same vein, the fluid was slowly discharged into the remains of the fluid to prevent the formation of air bubbles. Similarly, exactly 2ml of the fluid was drawn again into Erlenmeyer flask. 10ml of deionized water and 15ml of 3% Hydrogen peroxide ( $H_2O_2$ ) were added to the drilling mud in the flask, and a mixture was formed. Subsequently, 0.5ml of 5N  $H_2SO_4$  was added into the mixture. This was heated through the heating mantle and allowed to boil gently for 10 minutes. Moreover, the mixture was diluted with deionized water to 50ml mark. Methylene blue solution was further added to the mixture in drops. The content was swirled manually for few seconds, and while the solids are still suspended, the glass rod was inserted into the solution (the content). The tip of the inserted glass rod was placed on the filter paper. The

drop on the filter paper was carefully observed. The swirling, observation and addition of more drops of methylene blue solution was continuous until a turquoise ring was seen on the filter paper (Instruction Manual by OFITI Equipment Inc: Methylene Blue Analysis (2010)).

The Methylene Blue Capacity and Cation Exchange Capacity of the drilling mud were calculated and analyzed using the formula in equation 1

$$\text{Methylene Blue Capacity} = \frac{\text{Methylene Blue (ml)}}{\text{Drilling Mud (ml)}} \dots \dots \dots (1)$$

Similarly, the Methylene Blue Capacity was also analyzed on the basis of Bentonite equivalent and a cation exchange capacity of 70 meq/100 grams in equation 2

$$\text{C. E. C} = \left[ \frac{100}{f^*} \right] \times (V_{cc}) \times \text{Normality; in } \frac{\text{meq}}{100 \text{ g}} \dots \dots \dots (2)$$

$$\text{Bentonite equivalent (kg/m}^3\text{)} = 2.85 \text{ X Bentonite equivalent (lb/bbl)}$$

Where  $V_{cc}$  : Volume of Methylene blue solution injected

**3.4.2.4 Phenolphthalein Alkalinity, Total Alkalinity and Hardness of Drilling Fluid**

The degree of alkalinity measures the acid neutralizing capacity of water. This is largely due to the hydroxyl, carbonate and bicarbonate ions present in water (ASTM, 1987). This property determines the quantity of lime in a drilling mud. Similarly, phenolphthalein alkalinity is the measure of the quantity of acid required to reduce the pH to the phenolphthalein endpoint, which is 8.3. At pH = 8.3, the (H<sub>2</sub>CO<sub>3</sub>) equals the (CO<sub>3</sub><sup>2-</sup>). This is the endpoint for carbonate alkalinity and CO<sub>2</sub> acidity titrations ((Instruction Manual by OFITI Equipment Inc: Water Analysis (2010)).

In the alkalinity titration, virtually all of the CO<sub>3</sub><sup>2-</sup> and half of the HCO<sub>3</sub><sup>2-</sup> has reacted at the endpoint (Snoeyink and Jenkins, 1980). This value of drilling mud or mud filtrate is designated as P<sub>m</sub> or P<sub>f</sub>. However, the amount of acid required to further reduce the pH of the drilling mud to 4.3 is the methyl orange alkalinity at the methyl orange endpoint. Similarly at pH = 4.3, the [H<sup>+</sup>] equals the [HCO<sub>3</sub><sup>-</sup>]. This is the endpoint for mineral acidity and total alkalinity titrations. These are designated as M<sub>m</sub> and M<sub>f</sub>. The total alkalinity of the drilling

mud is an expression of the quantity of acid (H<sub>2</sub>SO<sub>4</sub>) required to reducing the pH of the mud to 4.3 from the actual pH value of the drilling mud. The quantity of bicarbonate or carbonate salt is measured to determine the total hardness of the mud filtrate (Snoeyink and Jenkins, 1980).

3ml of filtrate was measured into the titrating dish. Three drops of phenolphthalein solution was added to the mud filtrate in the dish. Pink colour was formed. This was titrated against N/50 H<sub>2</sub>SO<sub>4</sub> from a pipette while stirring until the pink colour disappear, that is, until the content turns colourless. The volume of N/50 H<sub>2</sub>SO<sub>4</sub> used was taken and recorded as P<sub>f</sub>. Three drops of methyl orange was further added to the clear solution. An orange colour was obtained. The orange colour solution was titrated simultaneously against N/50 H<sub>2</sub>SO<sub>4</sub> from a pipette as the stirring continues, until the orange colour change to pink. The total volume of N/50 H<sub>2</sub>SO<sub>4</sub> per milliliter of filtrate used throughout the test (i. e. the quantity required to reach the final end point was taken and recorded as M<sub>f</sub>.

In the same vein, 3ml of filtrate was measured into the titrating dish. The content was diluted with deionized water to the 50ml mark to determine the hardness of the drilling mud. Moreover, 3ml hardness buffer solution and 6 drops of hardness indicator solution were measured into the mixture, mixed simultaneously and thoroughly. A light reddish colour was observed. The titrating solution from a pipette was added intermittently as stirring continues, until the colour changes to blue. An approximate volume of titrating solution used from the start to the end point was taken and recorded respectively. The phenolphthalein, total alkalinity and hardness of the drilling mud can be calculated using equations 8-10 below.

Phenolphthalein Alkalinity

$$= \frac{\text{Volume of H}_2\text{SO}_4 \times \text{Normality} \times 50 \times 1000}{\text{Volume of sample taken}} \dots \dots \dots (8)$$

$$\text{Total Alkalinity} = \frac{\text{Total volume of H}_2\text{SO}_4 \times \text{Normality} \times 50 \times 1000}{\text{Volume of sample taken}} \dots \dots \dots (9)$$

$$\text{Total Hardness as Calcium } \left( \frac{\text{mg}}{\text{L}} \right) = 400 \times \frac{\text{Volume of Titrating Solution}}{\text{Volume of Filtrate}} \dots \dots \dots (10)$$

### 3.4.2.5 Chloride ion and Total Calcium Sulphate in Drilling Mud

This indicates the migration of negatively charged chlorine ions  $[Cl^-]$  resulting in an accelerated dissolution of metals  $[M^+]$ . This is referred to as crevice corrosion. The presence of iron in this situation may act as a scavenger, reacting to displace  $H^+$  from sulphur in hydrogen sulphide. Chloride content in most situation is been analyzed in the reaction with silver nitrate. The excess silver present after all Chlorine has been consumed in the solution results in a white precipitate. An end point was obtained at titration using a potassium chromate indicator. The orange-red precipitate obtained at the end point indicates the chemical reaction that results in the formation of  $Ag_2CrO_4$ . The American Petroleum Institute (API) Specifications has an approximate limit for  $Cl^-$ ,  $Fe^{2+}$ ,  $Fe^{3+}$  in barite mineral recommended for drilling operations. Similarly, the amount of the dissolved salts in the drilling mud is measured to estimate the potential risk of the mud to the water bodies, when exposed to the aquatic and terrestrial organism (Brevier et al., 2002; Sterner, 2002).

6ml filtrate was measured into a clean titrating dish. Three drops of phenolphthalein was also added to the content in the dish. A pinkish colour was observed, and later disappeared on the addition of N/50  $H_2SO_4$  in drops. Similarly, 25ml of deionized water and 5 drops of  $K_2CrO_4$  were added to the clear solution. The mixture was stirred intermittently as 0.282N  $AgNO_3$  is added from a pipette, to the mixture. A deep-reddish colour was observed in the solution as an indication of the end point. The quantity of 0.282N  $AgNO_3$  used for the analysis was read and recorded respectively. However, 1ml of the drilling fluid was diluted in 50ml of deionized water. The mixture was stirred for 15 minutes. This was filtered to obtain the clear solution from the cloudy portion. 10ml of clear filtrate was titrated against ethylene diamine tetra acetic acid (EDTA) to the pH of 7 (EDTA end point). Similarly, 1ml of the initial solution was also titrated against EDTA to the end point. The volume of EDTA used up to the end point was read and recorded respectively.

To determine the iron count in the drilling mud, an acid solution of 3 drops HCl in 100ml distilled water was prepared. The pH of the solution was measured and recorded. The addition of 0.5ml  $H_2O_2$  solution results in a pale yellow end point, and that of 1.0ml iron indicator solution in a purple colour end point. 0.5ml of iron buffer solution was further added, and the pH of the solution was measured. Finally, the total hardness (EDTA) solution (20 EPM Ca

& Mg) was titrated with the mixture and certain ml of the filtrate until the colour changes from purple colour to the pale yellow end point. The chlorine and iron count, and the quantity of the dissolved sulphate in the drilling mud were calculated using equations 11-13 below.

$$\text{Chloride } \left(\frac{\text{mg}}{\text{L}}\right) = \frac{\text{ml Silver Nitrate} \times 1000}{\text{ml Filtrate}} \dots \dots \dots (11)$$

$$\text{Total Calcium Sulphate } \left(\frac{\text{Kg}}{\text{m}^3}\right) = 6.79 \times V_t \dots \dots \dots (12)$$

$$\text{ppm Fe}^{3+} = \text{Total Hardness Titrating Solution, 20 EPM, ml} \times 5.6 \dots \dots \dots (13)$$

### 3.4.3 Rheological Parameter

#### 3.4.3.1 Determination of Sample Density (Mud Weight)

The required weight of the mud column establishes the density of the mud for any specific case. This is required as the starting point of pressure control of mud density and the balance of the formation pressure (Osokogwu et al., 2014). The mud balance comprises up of a small vessel, filled with the mud and a metering section which was adjusted gently to account for the mud weight. This balance has two (2) different calibrations on it. The upper calibration gives the mud weight in (g/cm<sup>3</sup>) while the calibration below indicated the mud weight in (lb/gal) (Omoniyi and Mubarak, 2014). To start with, the temperature of the mud sample to be tested was measured; mud balance base was placed on a flat, level surface and a clean, dry mud balance cup was filled with the sample of mud to be tested. The cup's cap was rotated until it was firmly seated. However, some mud was ejected through the hole in the cap to remove any trapped air or gas. The balance arm was positioned on the support base and was adjusted by moving the rider along the graduated scale until the level bubble was centered under the center line. The density of the mud (mud weight) was measured and recorded appropriately. The cup outside was made cleaned and dry (Osokogwu et al., 2014).

### **3.4.3.2 Determination of Sample Gel Strength, Plastic Viscosity (PV) and Yield Point (YP)**

In a mud circulating system, flow occurs at different rates in pipes of various sizes and shapes. Limited number of measurement was made to identify and predict effects of the drilling mud flow behavior at various points of interest in the circulating system. The shear stress is the frictional drag on the surface of the pipe exerted by the components of the fluid during flow. The magnitude of the shear stress depends on the frictional drag between adjacent “layers” of fluid traveling at different velocities, and the difference in velocities of adjacent layers next to the wall of the pipe. The difference in velocities between adjacent layers is referred to as the shear rate. This analysis is focused on identifying the effect of the flow at the wall where both shear rate and shear stress are at maximum (Osokogwu et al., 2014).

To do this, the formulated drilling mud was measured into the thermal cup up to approximately 70%, and positioned on Viscometer stand. The cup was raised and stands until rotary sleeve was completely immersed to scribe lie on Sleeve Locked into place by turning locking mechanism (Omoniyi and Mubarak, 2014). With the sleeve rotating at 600 rpm, dial reading in the top window of Vann viscometer was delayed for 20 seconds to stabilize. The dial reading at 600 revolutions per minute (rpm) was observed, noted and recorded respectively. With red knob in bottom position, Vann viscometer (VG meter) toggle switch was flipped to a lower position by pushing the toggle switch away. Dial reading was delayed or otherwise paused for 20 seconds to stabilize. The process was repeated for 300, 6 and 3rpm respectively. Finally, the dial readings were observed and recorded respectively (Osokogwu et al., 2014).

### **3.4.3.3 Determination of Sample Electrical and Emulsion Stability**

This is one of the commonest routine field and laboratory measurement of the relative electrical strength of drilling fluids based on the continuous oil phase. This is the dielectric breakdown voltage of the drilling mud. At this point, the drilling fluid becomes electrically conductive and the emulsion stability of the fluid is measured. The value obtained is controlled by the chemical composition and the shear history of the drilling mud. This was to examine the possibility for a differential mud weight at the top and bottom of the drilling

mud as a result of barite sag, insufficient emulsifier, water kick or leakages from the reservoir, exposure to highly concentrated calcium chloride (CaCl) and defects due to mixing. This relates the barite test with the emulsifier test, and the water-in-oil emulsion stability. This is a function of viscosity, the types and concentrations of solids, aqueous fluid and emulsifier.

To do this, the entire electrode gap, the connector and entire electrode body were thoroughly cleaned. This was calibrated with distilled water (for water based drilling mud). The drilling mud was pre-heated to about 50°C, stirred with the electrode probe for about 10 minutes and the mud temperature was measured while the electrode surface was completely submerged by the sample. The voltage ramp button was pushed and held while the electrode is stationary, and until the ES value is obtained simultaneously within 5% difference. The average of the two ES measurement of the drilling mud is calculated and recorded respectively. ES Value indicates the number stability of the emulsion mud and certifies the conductivity of the mud. However, the low ES value indicates a material that is not conductive other than the water phase. This determines the interface between water and oil based mud while displacing water with oil based fluid. Such fluid has a bad emulsion. On the other hand, high ES value indicates a mud with good emulsion stability and hence a good emulsion drilling mud. The essence of the analysis is to ascertain the role of barite during the normal dielectric breakdown process. The resistivity of barite is  $10.2M\Omega \cdot m$ . Although, barite itself is a poor conductor and the aqueous phase is absorbed onto the barite, so that the solids, serving as carriers of water, are attracted to the bridge and not as active participant in its formation. This state can be improved by the addition of emulsifier.

#### **3.4.3.4 Determination of Fluid Loss**

This is carried out at bottom hole temperatures, under static conditions to determine condition of emulsion, the filtrate volume and filter cake quality. The presence of water in the filtrate indicates emulsion weakening while thick filter cakes and high fluid loss indicate excessive drill solids content. Lowering the filtration rate is achieved with filtration control agents, emulsifiers and limes to increase the fluid loss by dilution with base oil. The High Pressure High Temperature Consistometer at a gas pressure of 100psi was bled through the lid and inlet of the pump. The lid was removed from the bottom of clean and dry cell and the O-ring

placed in an undamaged groove in order to seal the inlet with a finger and prevent any mechanical damaged.

The cell was filled with the mud sample up to one-fourth ( $\frac{1}{4}$ ) of the O-ring groove and a filter paper (Whatman No. 50 or equivalent) placed on the top of the O-ring. The lid was placed on the filter paper with the flanges of the cell and turned clockwise until hand tight. A suitable graduated cylinder was placed under the filtrate opening to receive the filtrate. At the end of the test, the valve was closed to shut off the pressure automatically, at the source bleeding and to remove the cell. This was done for 30 minutes and within the API test Specification. The cell was disassembled, the mud discarded and extreme care used to save filter paper with a minimum of disturbance of the cake. The cake was washed gently to remove excess mud, the thickness of the filter cake are measured (Osokogwu et al., 2014).

The primary function of thickening time is to determine the maximum available pumping time of the drilling mud before the slurry become molten and solidifies. This test indicates how the cement sheath will withstand the differential pressures in the well. To measure compressive strength, the slurry or mud sample was cured at bottom-hole temperatures and pressures and the results reported in psi. The compressive strength can be measured using either the autoclave system or the ultrasonic cement analyzer (UCA) respectively (Joel, 2013).

#### **3.4.3.5 Determination of the Lubricity Value:**

This is the measure of resistance to rotation of the drill in a deviation hole. This measures the lubricating quality of the drilling fluids, suggest and evaluate the type and quality of lubricating additives required in a drilling mud and predict wear rates of mechanical parts in a given system. The fluid resistance of various lubricating additives indicates the lubricity value of a fluid (Aston et al., 2013; Cameroon, 2001).

The OFITE extreme pressure (EP) and lubricity tester was used to measure the lubricity value of the drilling mud. The ring, block, block handle and cup for the sample were rinsed thoroughly and softly with acetone. The parts were properly coupled to the tester, with the test block in the holder and a reasonable gap between the ring and block. The machine was

powered, turned on and ran for 15 minutes. Similarly, the fluid was stirred gently for 10 minutes, the motor speed set to 60rpm and the filled cup with the fluid was placed on it stand. The cup was raised until the ring and the entire block assembly was fully submerged in the fluid. The thumb screw was tightened to support the sample holder (Redburn et al., 2013; Mamat et al., 2013).

The torque reading was stabilized and zeroed on the control panel to positioned the torque arm in the clamp and adjust the torque handle until the gauge reads 150in-lb. Similarly, the time was zeroed and the lubricity tester was run for 5 minutes. The torque reading on the display screen was read and recorded. The pressure on the ring and block was released to adjust the motor speed back to zero, lower the cup and discard the fluid. The tester was disassembled to remove and thoroughly washed the ring, block, block holder and the cup holding the sample, with water. The Lubricity coefficient was calculated using equations 3 and 4 below

$$\text{Lubricity Coefficient} = \frac{\text{Meter Reading X Correction factor}}{100} \dots \dots \dots (3)$$

$$\text{Coefficient of Friction} = \frac{F}{W} \dots \dots \dots (4)$$

The load acting on the test block can be expressed in equation 5 below

$$W = \frac{150 \text{ Inch-pounds (Torque Wrench Reading)}}{1.5 \text{ Inches (Torque Shaft Lever Arm)}} = 100 \text{ pounds} \dots \dots \dots (5)$$

From equation (4) above, we have that,

$$\text{Coefficient of Friction} = \frac{F}{W} = \frac{\text{Torque Reading}}{100} \dots \dots \dots (6)$$

The correction factor in equation (3) can be expressed in equation 7 below,

$$\begin{aligned} & \frac{\text{Standard Meter Reading for Deionized Water}}{\text{Meter Reading Obtained in Deionized Water Calibration}} \\ & = \frac{34}{\text{Meter Reading (32 to 36)}} \dots \dots (7) \end{aligned}$$

### 3.4.3.6 Solid and Water Content Analysis

The Retort analysis measures the percentage (%) water and solids (both suspended and dissolved) contained in the water-based drilling fluids and cuttings. This is necessary and as



water-based drilling mud is measure and controlled to stimulate better evaluation characteristics from electrical logs as recorded on the wire line. This measurement is necessary to monitor the process changes and control mechanism such as in pressure, temperature and geology formations. All these are to prevent certain type formation or pressure resulting in the crack of rocks and massive loss of fluid into the formation. This is instrumental to monitor the performance of the drilling mud during the drilling operations, and to prevent kicks.

The prepared drilling fluid was thoroughly stirred to achieve homogeneity. Moreover, the lucite cell connected to the suction bulb, to pull the sample, was used to refilled the cell and wet the cylindrical walls. Two terminal posts on the meter were connected to their assemblies and was set for the analysis at the temperature within the room temperature. The black button was depressed until the meter needle reaches a stable end. Similarly, this meter needle was adjusted to the ADJ position at the rear end, using an adjusting control knob. Such set up was examined to be completely correct. Both the black and red buttons were depressed at once, and the reading for the resistivity was read from the meter in ohms-meter. The temperature of the fluid was also read and recorded (Instruction Manual by OFITI Equipment Inc: Electrical stability meter (2010)).

## **4.0 Chapter Four**

### **4.1 Sample Coding**

Collected samples were labeled as A, B, C, D, E and F. Sample A is white coloured barite with relatively small portion of sandstones, brown coloured barite as sample B, different fractions of white and transparent barite samples as samples C and D. However, samples E and F are whitish in colour, containing some associated gangue minerals interlock within the barite vein. Similarly, the on-site processed Torkula barite was labeled as milled sample. This was compared with the American Petroleum Institute (API) and Nigerian Petroleum Development Company (NPDC) specifications, labeled as “API Spec” and “NPDC Ref. Samp” respectively.

#### **4.1.1 Phase and Morphological Analysis of Clay Minerals**

The X-ray diffratograms in Figures 4.1 and 4.2 revealed the crystallinity, structural properties, the atomic arrangement, the topography, morphology, composition and the crystallographic information of crude barite and clay minerals. This was validated with an extensive comparison of the d-values obtained from the ICPDS card number 00-024-0020, and JCPDS (Joint Committee on Powder Diffraction Standards) data of card number: 39-222. It indicated peaks corresponding to elemental constituents of barium sulphate and aluminosilicates and other clay minerals. Figure 4.2 evidently indicated a significant proportion of barite mineral which dominates at different crystallographic orientations. This simply describes the arrangement of atoms with respect to materials properties. However, the most intense X-ray peak indicated the present of quartz ( $\text{SiO}_2$ ). This revealed the crude nature of the minerals, and the structural defects inherent in the crushed sample. Most obviously, barite minerals have been identified by its characteristic X-ray diffraction peaks at  $20^\circ$ ,  $20.8^\circ$ ,  $22.7^\circ$ ,  $23.7^\circ$ ,  $24.9^\circ$ ,  $25.8^\circ$ ,  $28.7^\circ$ ,  $31.5^\circ$ ,  $32.8^\circ$ ,  $36.5^\circ$ ,  $38.7^\circ$ ,  $41.6^\circ$ ,  $42.6^\circ$ ,  $43.0^\circ$ ,  $44^\circ$ ,  $47^\circ$ ,  $49^\circ$ ,  $51^\circ$ ,  $52^\circ$ ,  $53^\circ$ ,  $54.5^\circ$ ,  $55^\circ$ ,  $56^\circ$ ,  $60.4^\circ$ ,  $60.8^\circ$ ,  $63^\circ$ ,  $64^\circ$ , and  $65^\circ$  in accordance to the PDF files. In addition to these, the defects structure constitute of the relative amount of sand (Quartz,  $\text{SiO}_2$ ), calcite ( $\text{CaCO}_3$ ) and hematite ( $\text{Fe}_2\text{O}_3$ ). They are classified as gangue minerals interlocked in the barite mineral, and occurred in a relatively small quantity. This validates the crystallinity of the mineral, structural properties and topography.

The crystallographic planes and directions of the barite mineral clearly revealed the arrangement of atoms in the crystal system and describe the crystal structure of barium sulphate. This is highly significant to explain the nature of the slip system that must be tailored to strengthen and toughen, and to enhance the catalytical activation of distinctive phase in barite mineral. To do this, the barite phase of inter-axial side [123] at the 2theta value of  $52^\circ$  is one of the most critical slip planes wherein more atoms are arranged. However, the plasticity of barite mineral in both water and oil-based drilling mud is enhanced along the closed-packed plane as the major determinant of the effect of any lattice vibration or distortion. Hence, the plasticity of barite in the drilling mud is better controlled on these major slip systems. Similarly, the XRD diffratograms clearly revealed the predominance of barite mineral over the relatively dispersed minerals of silica, calcium, carbonate, iron and oxides. In addition to these, the occurrence of the gangue minerals in barite sample is only in traces and interlocked within the host mineral (Barite). Calcite, hematite and quartz exist in the association of others within similar barite phase.

The XRD peaks for the de-aluminized kaolinite in Figure 4.1 clearly revealed the reduction of aluminum which was balanced with the silica content (Gougazeh and Buhl, 2014). This greatly reduced the kaolinite minerals; support the introduction of calcite and feldspar at the 2theta value of  $30^\circ$  and the disappearance of kaolinite at 2theta values of  $35^\circ$ ,  $36^\circ$ ,  $38^\circ$  and  $39^\circ$  respectively. The disappearance was replaced with sodium Montmorillonite minerals. After the de-alumilization process, the XRD patterns exhibit a significant change when compared to the raw kaolin. However, the highest diffraction peak in the two materials corresponds to the presence of silica (quartz). The process initiated a change in the structural and tribological properties of the clay minerals required for enhanced plasticity and improved performance in service. Hence, the de-aluminized kaolinite will sufficiently substitute the sodium Montmorillonite in the oil drilling application.

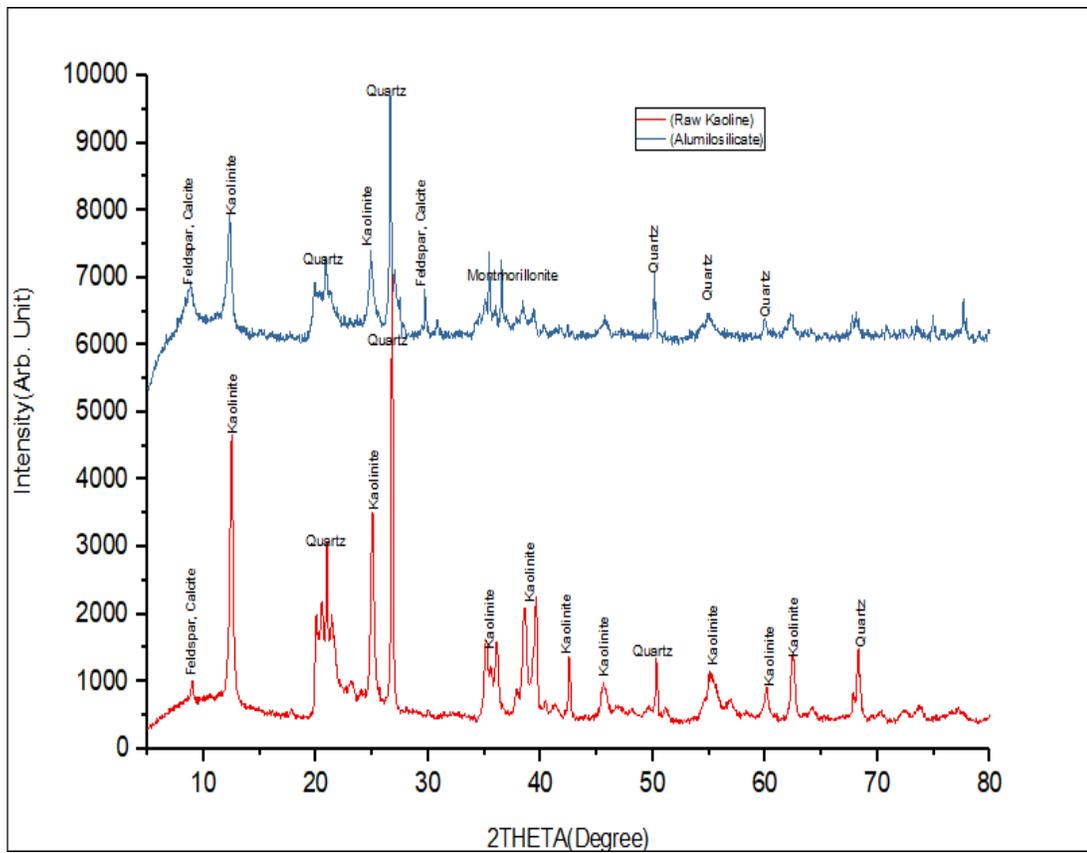


Figure 4.1: XRD patterns for de-aluminized and raw kaoline from Kankara, Katsina State

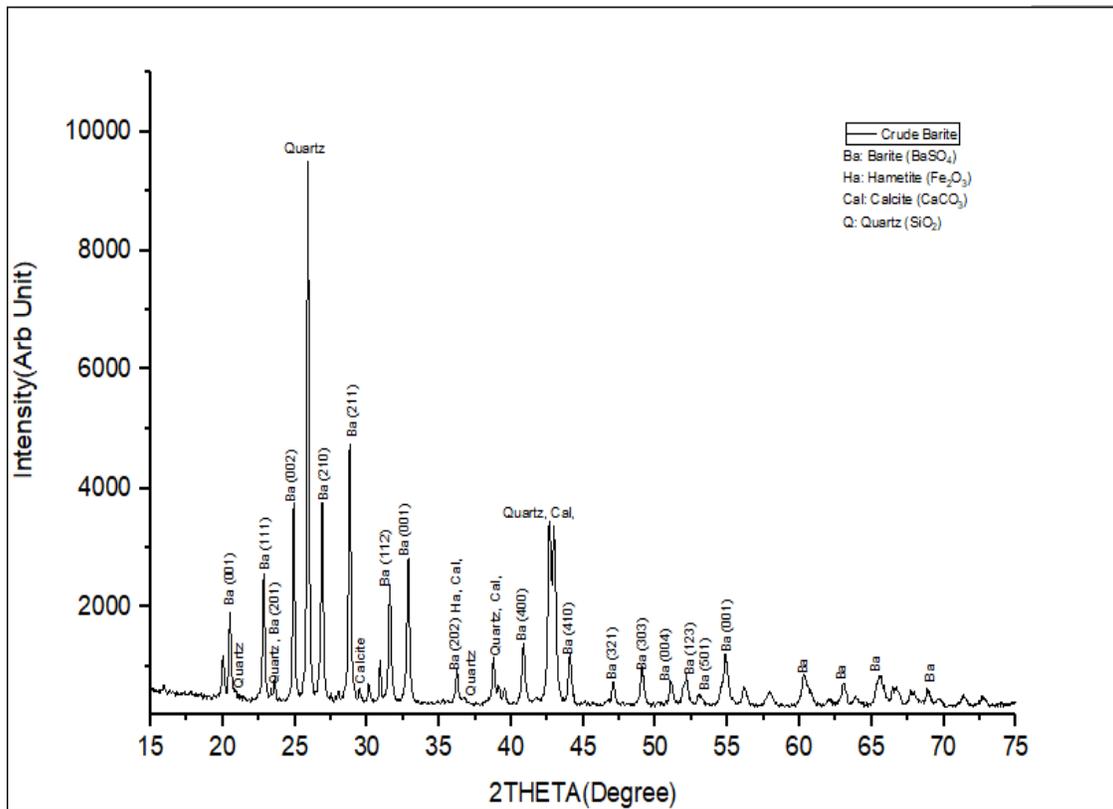


Figure 4.2: XRD pattern for Torkula barite from Guma LGA, Benue State

#### 4.1.2 Elemental Analysis of Torkula Barite

The mineralogy of Torkula barite was completely described in Table 4.1. The crude barite has high quantity of sandstone, iron oxide and lead oxide. However, the mineral is rich in barium oxide and sulphur trioxide, which is the candidate material for sulphate. Torkula barite has

87.79 %  $\text{BaSO}_4$ , 1.39 %  $\text{Fe}_2\text{O}_3$ , 6.66 % silicate salts, 1.603 % total heavy metals, 0.589 %  $\text{CaO}$ , 0.086 %  $\text{MgO}$ , 0.75 %  $\text{Al}_2\text{O}_3$  and 1.10 % LOI. The quantity of metallic ions such as potassium and sodium required to enhance the mud rheology was quite low and the mass loss on heating was highly significant. Similarly, the elemental composition of Torkula barite was compared with the American Petroleum Institute (API) set limits. It was observed in Table 4.2 that, the quantity of barium sulphate and water soluble salt was lower than the API set limits. The percentage of iron oxide and sandstone was reasonably high. The associated minerals at the mining site are galena, hematite, calcite, brucite, halite, teriorite, muscovite, sphalerite and few others in traces. There may therefore be a health risk and environmental

degradation inevitability due to the huge deposits of harmful heavy metals such as lead, copper, strontium, zinc and tungsten.

Table 4.1: Result of XRF Elemental Composition of Torkula Barite

SiO <sub>2</sub> (%)	Al <sub>2</sub> O <sub>3</sub> (%)	CaO (%)	Na <sub>2</sub> O (%)	TiO <sub>2</sub> (%)	MgO (%)	Fe <sub>2</sub> O <sub>3</sub> (%)	K <sub>2</sub> O (%)
6.66	0.75	0.589	0.01	<0.001	0.086	1.39	0.02
CuO (%)	ZnO (%)	PbO (%)	WO <sub>3</sub> (%)	SO <sub>3</sub> (%)	BaO (%)	SrO (%)	LOI (%)
0.053	0.079	1.03	0.02	21.80	65.99	0.421	1.10

Table 4.2: Elemental Evaluation of Torkula Barite

Composition	Percentage (%)	
	API Specification	Torkula barite
BaSO <sub>4</sub>	95.00	<b>87.79</b>
Fe <sub>2</sub> O <sub>3</sub>	0.03	<b>1.39</b>
CaO	0.01	<b>0.589</b>
MgO	0.04	<b>0.086</b>
Al <sub>2</sub> O <sub>3</sub>	0.035	<b>0.75</b>
Silicate	0.01	<b>6.66</b>
Total Heavy Metals	0.001	<b>1.603</b>
Water Soluble Salt	0.1	<b>0.0301</b>
Loss on Ignition (LOI)	0.5	<b>1.10</b>

## 4.2 Analysis of the Physical Properties of Torkula Barite

### 4.2.1 Hydrogen Potentials of Torkula Barite

The Potential of Hydrogen (pH) as a function of electromotive force (e.m.f.) was measured at a given temperature and for a period of 24 hours. Figure 4.3 clearly revealed an increase in the hydrogen concentration and the dissociation of the ions possessed by barite in water. The slurry barite sample was slightly alkaline after the first 70 minutes, but its alkalinity (hydroxyl ion,

OH<sup>-</sup>) at 28.3°C was 7.936. Hence, the result obtained was at a temperature outside the range recommended by the manufacturer. However, the temperature of slurry as prepared was

lowered at to 26.3°C and a pH reading of 8.015 was obtained. The pH of the sample is within the operational pH and the standard range of drilling weighting agent, as recommended by the American Petroleum Institute (pH between 7 and 12). At these pH values, the solubility of calcium is very limited, and the drilling mud is suitable for use in the drilling of carbonate formations. This addresses the associated uncertainty and enhances the control measures to erosion and dissolution of freshwater mud. However, the active clays in the mud are easily disintegrated at high potential of hydrogen (Azar & Samuel, 2007; Christ, 2006; Ibe et al., 2016).

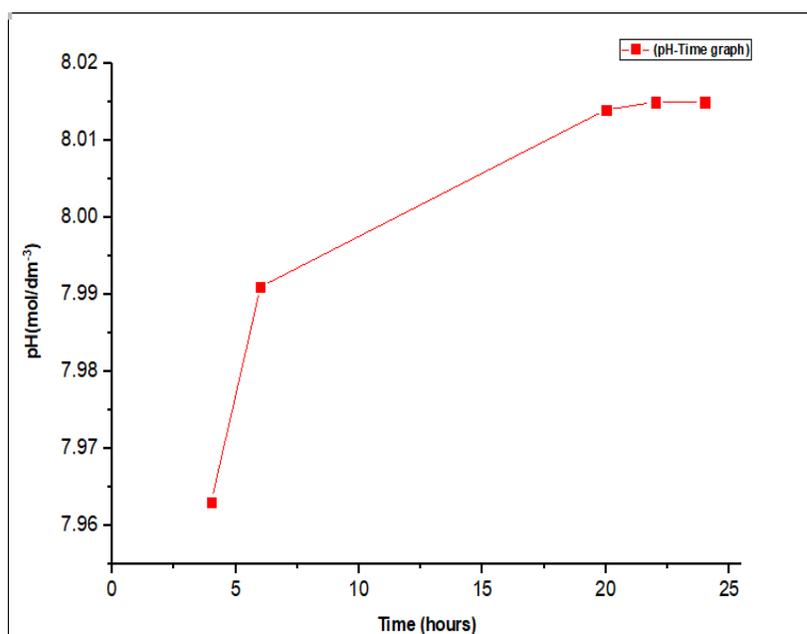


Figure 4.3: pH-Time graph for Turkula barite from Guma LGA, Benue State, Nigeria

In addition to these, its storage does not have any operational adverse effect on the drilling fluid. However, at the pH of less than 9.5, the fatigue life of the drill stem is reduced drastically. This is corrected through the addition of 7% sodium chloride brine or lime to raise the pH to about 10, to reduce the diffusion of oxygen to the metal surface and corrosion rate, and to increase its performance in service (Committee, 2001; Ibe et al., 2016). Water contamination, carbonate or bicarbonate, carbondioxide (CO<sub>2</sub>), hydrogen sulphide (acid gas) and anhydrite neutralize hydrogen ions in the drilling mud.

It is therefore noteworthy to increase the pH of the drilling mud while drilling near any of the formations that could cause changes in the pH. Similarly, at the pH=10 and above, the

plastic viscosity, apparent viscosity, yield point and gel strength are suddenly increased (Committee, 2001; Philip, 2012; Caenn, 2011; Huang et al., 2016). Hence, more of the additives are required to achieve these rheological properties stipulated by the American Petroleum Institute (API) standards. However, checking the pH reduces waste, manages the product and money at the long run (Ghassem et al., 2007; McCoremich, 2015). The complete dissociation of the hydrogen ion or potential of barite minerals occurred within 24 hours or a day.

#### **4.2.2 Moisture Content of Torkula Barite**

As clearly observed from the Figures 4 (a) – 4 (c) above, the weight loss during the over drying of the barite samples results into the calculated moisture content of the samples for 5, 10 and 15 grams. The percentage moisture content measured and calculated for these samples were

0.0704%, 0.1225% and 0.1610% respectively. The mean of these values was obtained as 0.118%. In comparison to the API standards, the moisture content of the barite sample under analysis is within the standards and specification of 1% at maximum (Ken and Clifford, 2011). Changes in the moisture content of weighting agent used in the drilling application greatly affect the mud viscosity. This results in the collapse of the borehole due to several technical faults that may arise from the varying downhole conditions. The implication of this to the drilling operation may be that the hydration potentials of the clay, swelling and lifting ability are enhanced to counter the effect of excess moisture content during the mud formation. Similarly, the results clearly describe the nature and the structure of the barite-water interface. The volume of the liquid or moisture is very small and as such, not sufficient to cause the splitting of the barium and sulphate. This may result in the formation of scale deposits that restrict the flow of the drilling mud in pipes during the drilling operation (Ibrahim et al., 2017; Osokogwu et al., 2014; Dhiman, 2012).

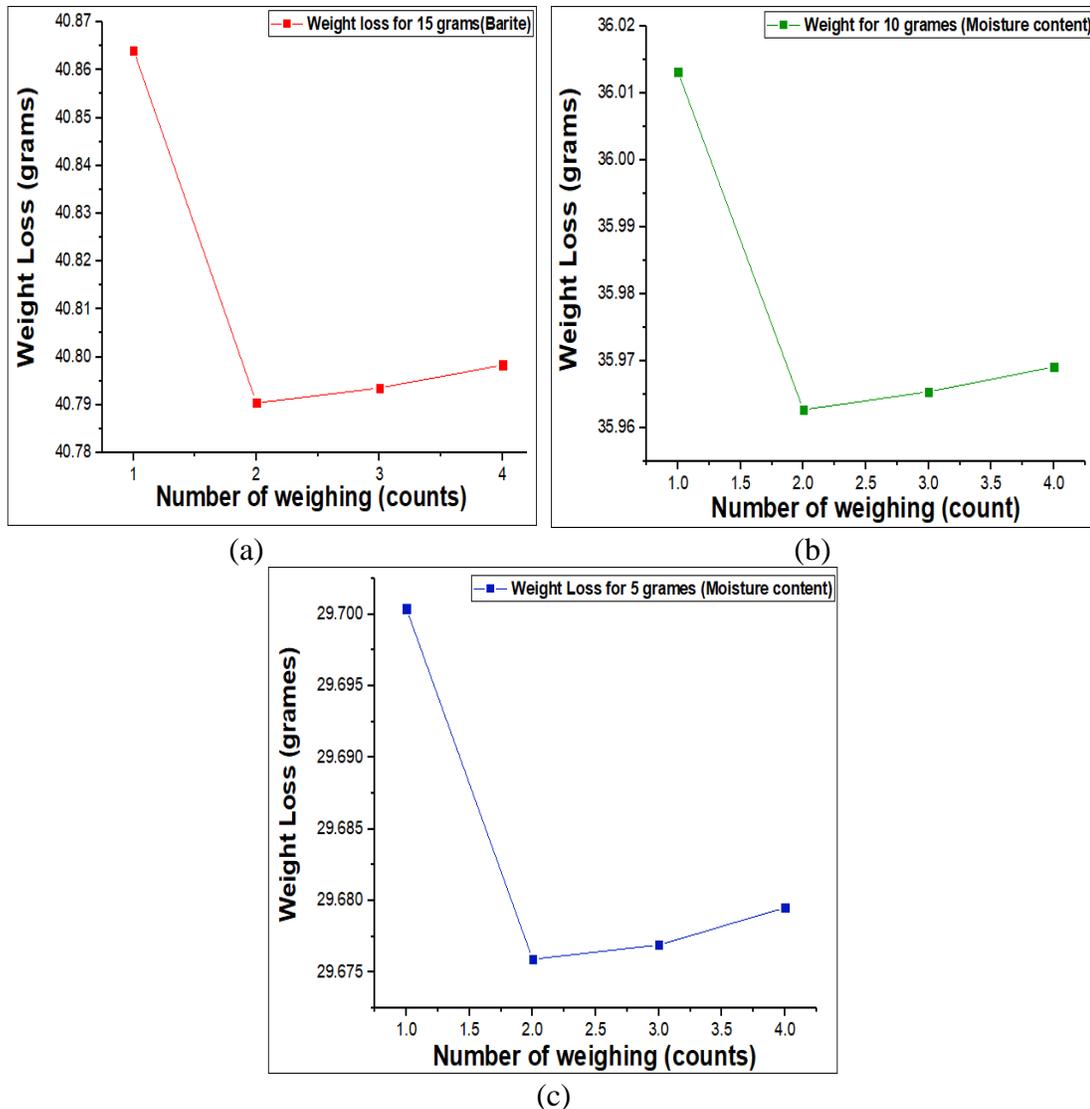


Figure 4.4: Weight loss due to barite mineral moisture content (Plotted using Origin Software, 2017).

### 4.2.3 Specific Gravity of Barite Samples

The distinctive specific gravity for each barite sample is clearly given in the Figure 4.5 above. Samples B and F have specific gravities of 3.85 and 4.078 respectively. These are less than the acceptable standards specified by the American Petroleum Institute (API). This fall in the quality of the barite samples may be as result the associated gangue minerals (impurities), with a lesser weight when compared to the weight of a barite mineral. In the real sense, sample B is a coloured barite (brownish coloured due to the impurities) and sample F has some reasonable content of sandstone, pyrite, calcite, etc. These associated minerals are not

as heavy when compared to barite mineral. Hence, there is a critical need to separate the gangue mineral from the host materials in order to increase the specific gravity.

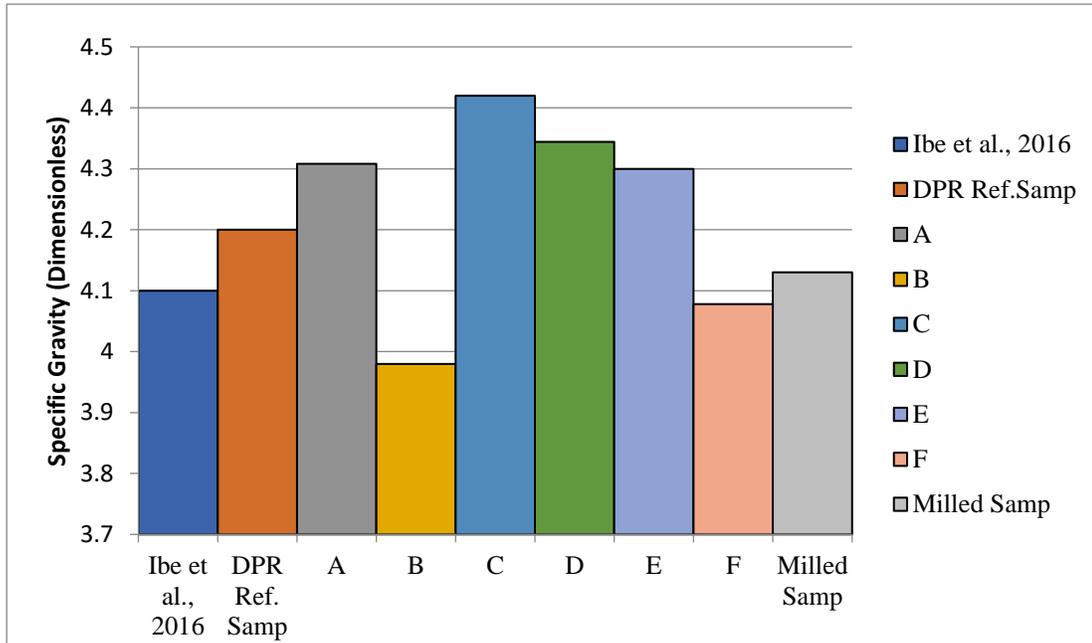


Figure 4.5: Specific gravity of barite samples from Torkula Barite Field, Guma LGA, Benue State, Nigeria

However, samples A, C, D and E have specific gravities that are reasonably higher than the globally acceptable standards of 4.21 minimum. Sample C has the highest specific gravity of 4.42. This suggested that the barite sample is crystal clear and neat, having a higher percentage composition of  $BaSO_4$  among the samples under analysis from Torkula Barite Field. Sample D was a fraction broken from the same vein with sample C. Its specific gravity is 4.344. Similarly, sample E has a specific gravity of 4.300. It is also in the pure state but with a little lower fall in specific gravity. This correctly justifies that the quality of barite mineral differs from point to point even in the same vein. It is therefore scientifically true that the quality of the mineral varies with depth. Hence, the quality assessment of solid minerals can only be correctly examined by the analysis of samples randomly selected from multiple points. The average specific gravity of all these fractions will likely give the most accurate and precise value (Ken and Clifford, 2001).

Samples E and F were barite lumps from the same vein with some associated minerals (gangue minerals) interlocked in the structure of the host mineral. This was broken into fraction in order to examine the diversity and physical distribution of these gangue minerals (fondly called impurities). Sample E has a specific gravity of 4.300 while sample F has 4.078 SG. This clearly suggests that the gangue minerals occur in different volume ratio across the barite vein. It is also possible to assume that this disparity in the SG is due to the different weight of the associated minerals. These minerals could be calcite, pyrite, magnetite, sandstone and other minerals associated with barite mineralization in Torkula Barite Field. Sample G is a milled or micronized barite powder obtained from the collection of the different forms of barite lumps. It has a specific gravity of 4.13 which is quite low compared to white or pure barite lumps, the values obtained by Ibe et al, (2016) and Department of Petroleum Resources (DPR) respectively. This is traceable to poor enrichment techniques without a significant separation of the barite lumps according to their grades. The coloured and those composed of some associated minerals (impure, or gangue) ought to be separated appropriately according to their qualities. Proper separation prior to milling will enhance the qualities and specific gravity of the mineral, as well as the mud weight of the drilling fluid.

On the basis of the SG, samples A, C, D and E can be used as weight control additives in drilling operations to increase the mud density and mud weight up to 21lb/gal ( $2.5\text{g/cm}^3$ ). These can also be employed to increase the wellbore pressure (i. e. the pressure drop encountered in lifting the reservoir fluid to the surface) ahead of the formation pressure (sub-surface pressure acting on a fluid regime within and between formations caused by high stratigraphic permeability). This will be achieved by the addition of barite to the drilling mud to increase the hydrostatic pressure as the depth increases. This prevents the inflow of the formation fluids into the wellbore, and it's sealing with a thin, low-permeability filter cake. The samples under analysis can sufficiently increase the mud density without exceeding the formation pressure (Dhiman, 2012; Chee et al., 2007).

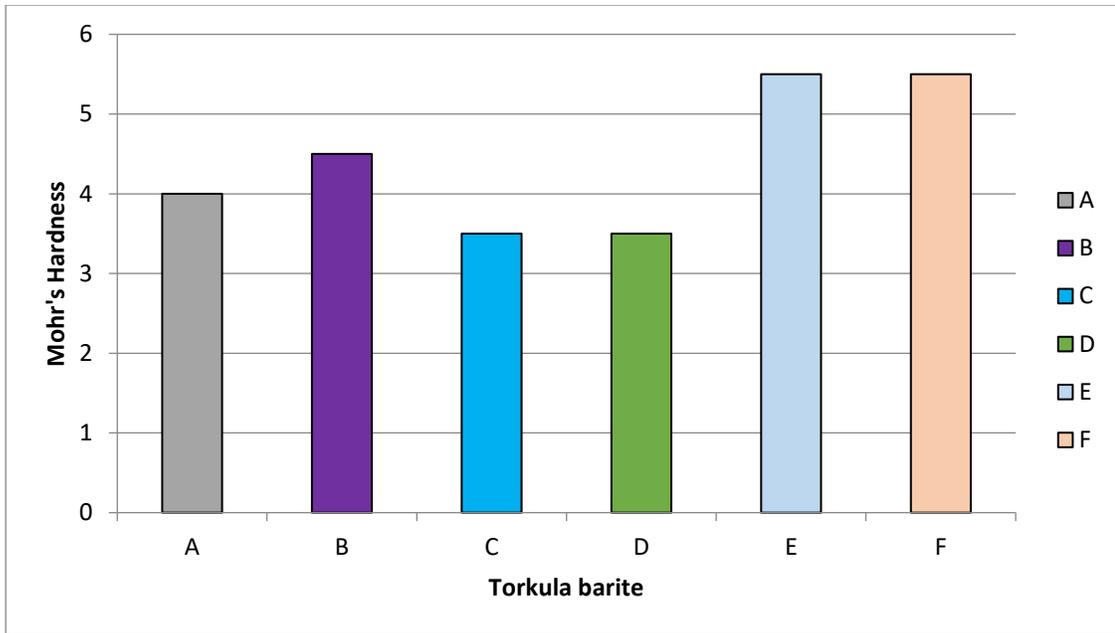


Figure 4.6: Mohr's hardness of the Barite samples from Torkula barite field, Benue State, Nigeria

Three white (pure) and three coloured (impure) barite were sampled and the hardness of each material was measured using the scratch method. Samples A, C and D have a mean value of Mohr's hardness between 3.5 and 4. However, samples B, E and F were within the range of 4.5 and 5 Mohr' hardness. Furthermore, samples C and D are the softest among all the samples. These samples are very pure, transparent and smooth. Sample A is also relatively pure and rough at the surface despite the colour. The texture is quite cloudy. Similarly, sample B has a brownish colour which may be traceable to the nature of the associated or gangue minerals. This is relatively harder when compared to samples A, C and D. The increase in the Mohr's hardness of sample B is due to the presence of such associated minerals having a Mohr's hardness value higher than barite.

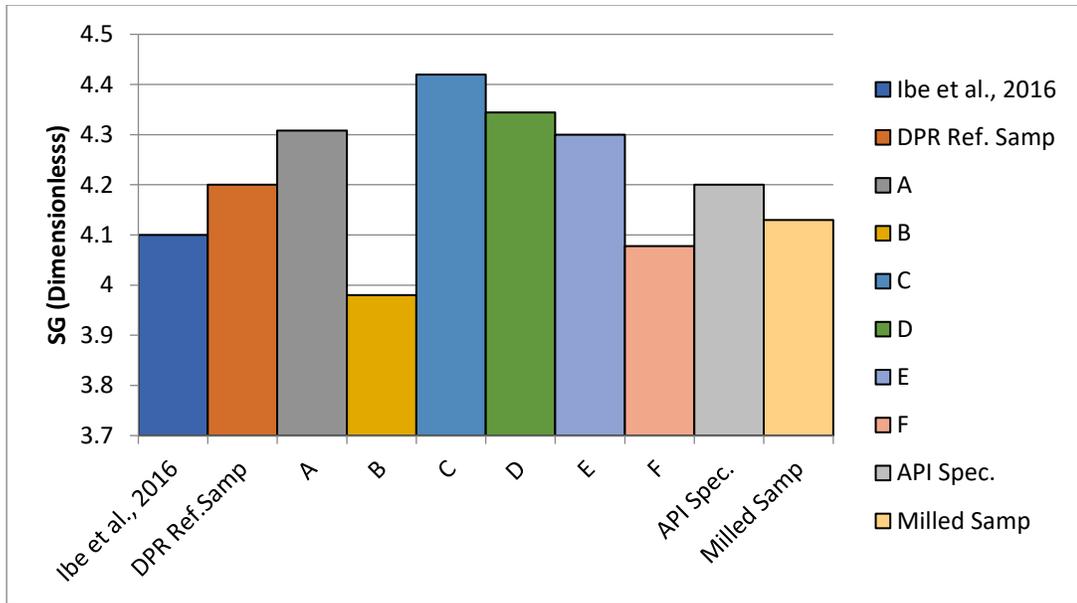
Samples E and F have a very rough surface due to the presence of gangue minerals (impurity minerals). The hardness of some associated minerals such as lead, pyrite and sandstones in the host mineral has increased the Mohr's hardness value of the barite samples. Hence, the presence of gangue minerals can increase the hardness of barite or reduce some of the physical properties. Most importantly, the values obtained for samples A, C and D clearly indicate a relatively soft texture material which may be recommended as candidate materials for oil drilling operation. Weighting agents within the Mohr's hardness of 3 and 4 can

sufficiently protect the drilling bit from being damaged when circulating in the system. Beneficiation of samples A, B, E and F to separate or extract the gangue minerals from the host will enhance their rheological properties and control other physical properties such as hardness and specific gravity. However, when such is not addressed prior to the drilling or cementing operation, the wear rate of the bore and the wear debris will be massive. This has a potential to change the quality of the drilling fluid and alter the rheological properties.

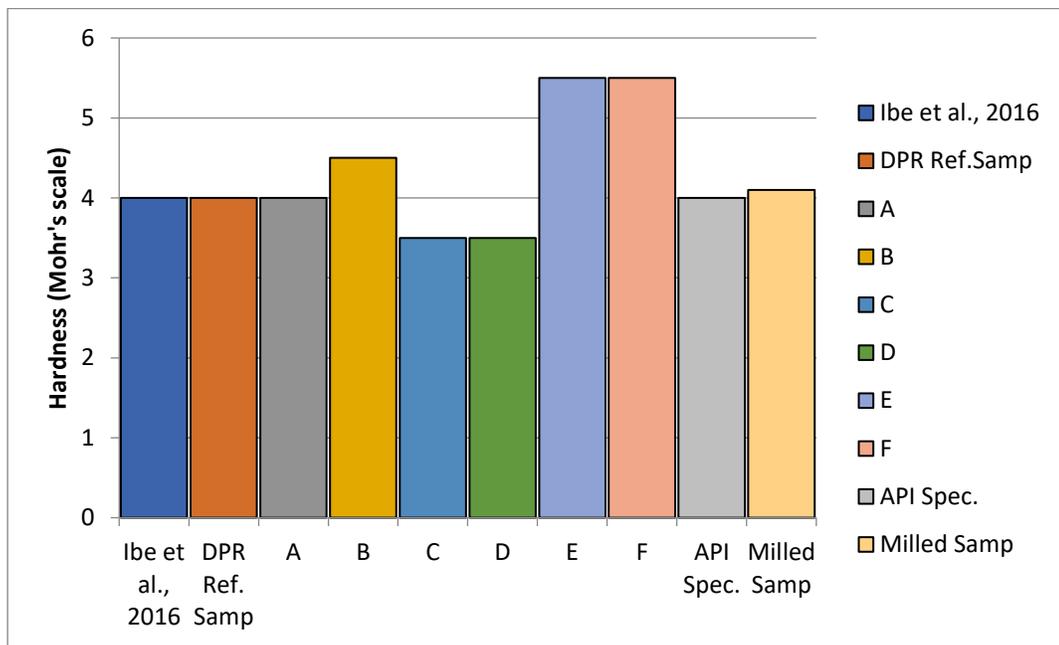
#### **4.2.5 Correlation between the Specific Gravity and Mohr's Hardness of Torkula**

##### **Barite**

The qualities of Torkula barite was evaluated in Figure 4.7 on the basis of their specific gravities and Mohr's hardness. These was validated through the side-by-side comparism with the globally recognized America Petroleum Institute (API) standards, the reference sample commonly used in many of Nigeria's indigenous oil and gas industries and other existing work. Four (4) out of seven barite samples from Torkula barite field in Benue State have SG greater than the API 4.2 SG. These can be classified as high grade barite while two (2) from the remaining three (3) is within the medium grade. The only sample in the lower grade class is a coloured barite. Six (6) among the seven (7) samples have a little higher SG when compared to the samples analyzed by Ibe et al., 2016. Experimental barite sample 2 (coloured barite) has a specific gravity of 3.85 which is below the API standard of 4.2 SG. This could be attributable to the gangue minerals such as calcite, pyrite, galena, hematite, Mg and Zn compounds. However, torkula barite mineral is rich, of higher qualities and heavy. It will sufficiently increase the density, mud weight and fluid density in all types of drilling fluid system. These properties qualify the barite mineral to be used in both ordinary and specialized drilling operations. Beneficiation of the coloured samples and others containing gangue minerals, will improve the quality and increase the specific gravity of the mineral (API, 1993; 2000).



(a)



(b)

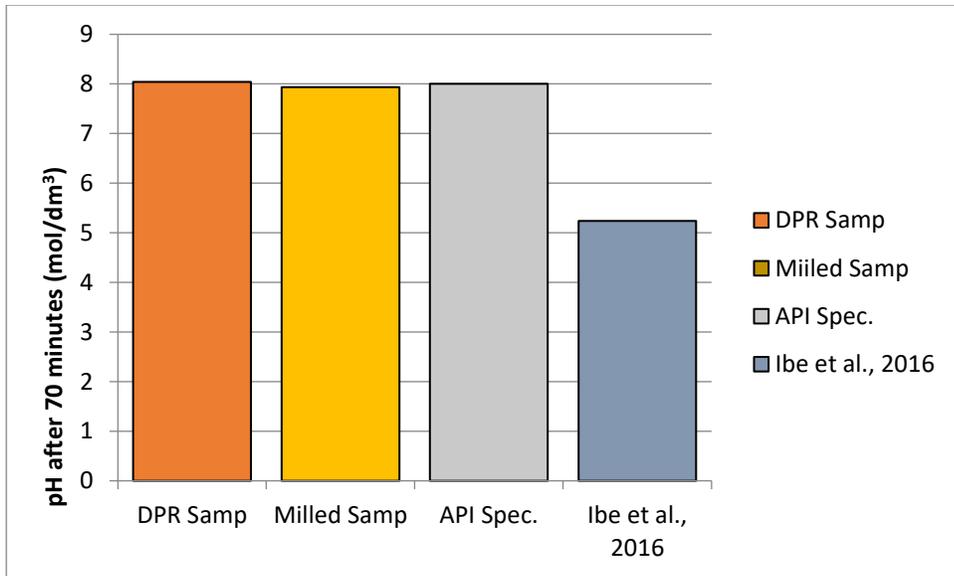
Figure 4.7: Comparison of SG and Mohr's hardness for Torkula barite deposits

Similarly, the wear rate of the bore in drilling operations is largely traceable to the hardness of the barite mineral. Samples A, C and D are soft while B, E and F are hard barite minerals. These were classified based on the API standards. The acceptable Mohr's hardness for barite minerals used in all drilling operations is within 3 and 4 Mohr's. However, the three (3) barite samples classified as hard minerals have several gangue minerals interlocked within the

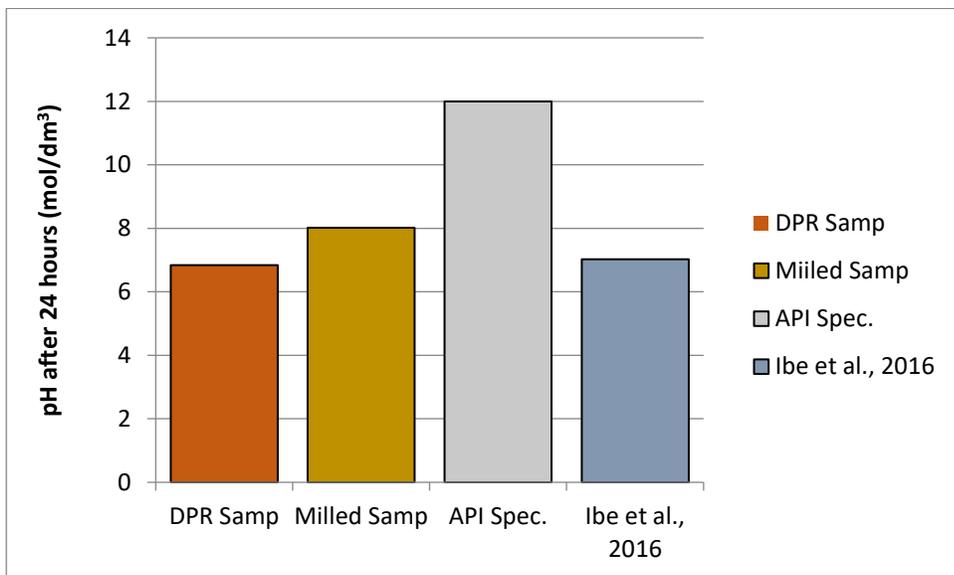
barite veins. The increase in hardness is due to the hardness of these minerals such as galena and hematite, whose Mohr's hardness is reasonably higher than that of barite. Moreover, inappropriate enrichment strategies resulted in the slight increase in the Mohr's hardness of the pulverized sample. This can be sufficiently corrected through the adoption of a well-designed washing bay and efficient separation mechanism. Barite samples B and C are softer than the imported barite (reference sample), and the two barite samples analyzed by Ibe et al., 2016 (Ibe et al., 2016; API, 1993; API, 2000; IADC, 2014).

#### **4.2.6 Contribution of Barite and other Gangue Minerals to Hydrogen Dissociation**

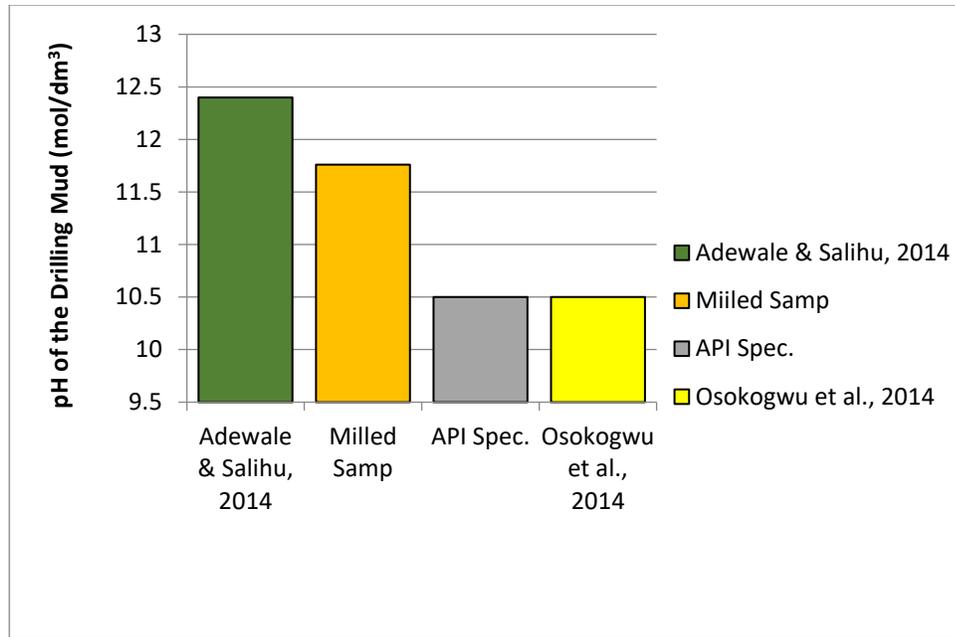
The pH of Torkula barite is within the American Petroleum Institute (API) Standards and almost the same as the quality of the reference sample imported by the Nigeria Petroleum Development Company (NPDC) Warri Field Office. This was clearly revealed in Figures 4.8 (a) – (c) respectively. The dissociation of the barite mineral after 24 hours was minimal when compared with the reference sample and other Nigeria barite analyzed by Ibe et al., (2016), Osokogwu et al., (2014), and Adewale and Salihu, (2014), as shown in Figure 4.8. This shows that the difference in the pH of Torkula barite after 24 hours is not as significant, and as such is within the API recommendations of  $pH=7-\leq 12.5$  (Christ, 2006, Ibe et al., 2016, Osokogwu et al., 2014 and Adewale and Salihu, 2014). Similarly, its application as a weighting agent in water drilling mud enhances the moderation of hydrogen potentials of the drilling mud within the acceptable limits stated by API. Hence, the pH of torkula barite fell within the operational pH regime and standards range of drilling weighting agent.



(a)



(b)



(c)

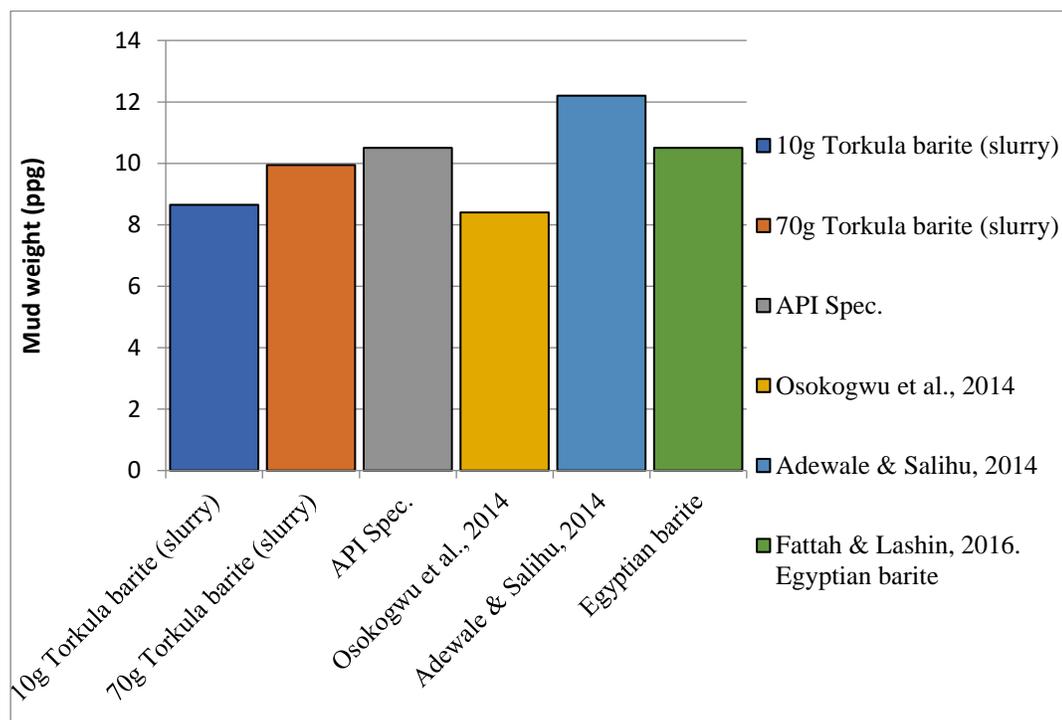
Figure 4.8: Comparison of Hydrogen Potentials for Torkula barite deposits

The long time storage does not have any operational adverse effect on the barite blend. Torkula barite can sufficiently detect and treat drilling cuttings such as cement and soluble carbonates. It will also enhance the solubility calcium and magnesium, and the flocculation of Bentonite clay (Osokogwu et al., 2014). The corrosion fatigue life expectancy of the drill pipe is partly dependent on the pH of barite. The pH of barite is strategic to the determination of the mud required for most drilling operations. Within the pH of the barite samples, the solubility of calcium is controlled and is limited. High pH mud is more suitable in the drilling of carbonate formations and reduces the susceptibility of the drill bits to erosion corrosion, sweet corrosion of casing and drill pipes. This is also instrumental in the disintegration of Bentonite (Azar and Samuel, 2007; Committee, 2011).

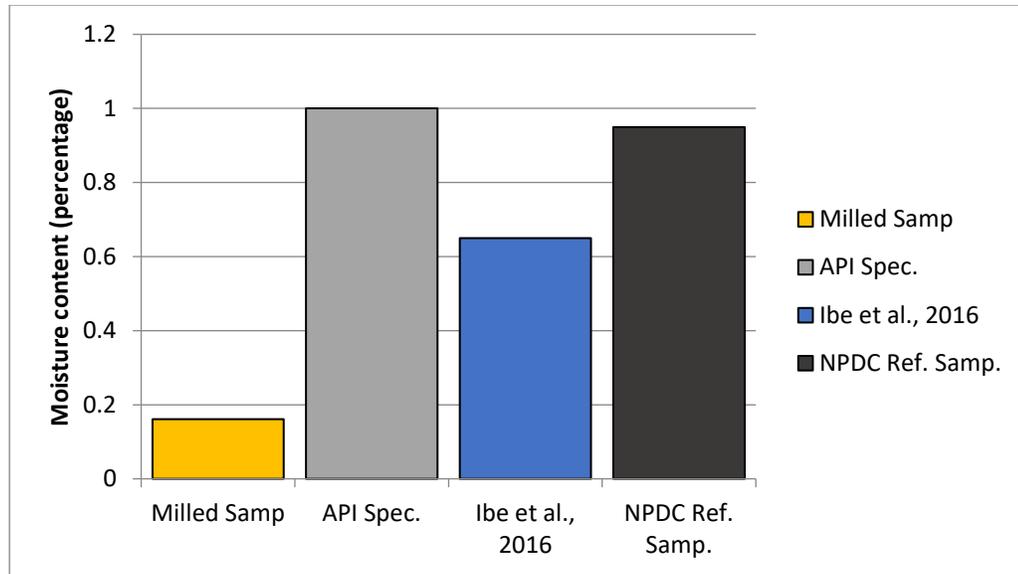
#### 4.2.7 Relevance of the Moisture Content of Torkula Barite on the Mud Weight

The mud weight of the barite samples under analysis revealed an average or mean value of the recommendations given by the API Standards and a balance of properties between the reference sample (imported barite), Egyptian barite and others as proposed by Adewale and Salihu, (2014) and Osokogwu et al., (2016). Beyond this, torkula barite has a mean moisture content of 0.118 % as shown in Figure 4.9 (b). This is quite low compare to the value given

in the API specifications and results of relevant literatures. The moisture contents of Torkula barite fell within the range of API standard of moisture content, 1% maximum. The 5g, 10g and 15g barite under analysis met the moisture content requirements for drilling mud additive as revealed in Figure 4.9 (b). This indicated that Torkula barite can be stored for a long time without compromising any of the physical, chemical and rheological potentials when employed in several drilling and cementing operations. The mud weight is completely controlled by the weight of barite added into the drilling mud. American Petroleum Institute (API) Standards has it that the acceptable specification for a drilling mud is between 9 and 12 parts per gallon. This is required to prevent the flow of the formation fluid into the wellbore during drilling (Ken and Clifford, 2011, Bourgoyne et al., 1986).



(a)



(b)

Figure 4.9: Comparison of the Mud Weight and Moisture Content of Torkula barite deposits

On the addition of 10g Torkula barite, the mud weight of 8.65 parts per gallon was obtained while with the addition of 70g Torkula barite, the mud weight was increased to 9.95 parts per gallon. This implies that Torkula barite can sufficiently prevent the flow of the formation fluid when the weight of barite is increased. However, the drilling fluid with formulation consisting of 10g barite may be efficient for drilling at the surface or at some few meters below the earth. Hence, torkula barite at a weight higher than 10 grams can sufficiently increase the wellbore pressure, and then the hydrostatic pressure of the fluid. All these are largely dependent on the mud weight or density of the drilling fluid. Similarly, for an efficient and optimum design, the mud weight of 8.65 and 9.95ppg may be considered. This is on the need for a drilling mud with a balance of moderate designs for the wellbore, at a reduced relative cost and controlled formation fracture. Summarily, torkula barite has a moderate weight per unit volume of the drilling mud that can supply sufficient hydrostatic pressure to the wellbore without causing any loss of circulation, or damage to the drilled formation (Osokogwu et al., 2014, <http://www.glossary.oilfield.slb.com/Display.cfm?Term=retort>, 23/11/2017. 9.30 am).

### 4.3 Assessment of the Chemical Composition and Properties of Torkula Barite

#### 4.3.1 Alkalinity and Hardness of the Drilling Mud as a Function of Torkula Barite

Alkalinity and hardness of water measure water's buffering capacity and ability to resist changes in its hydrogen potentials upon the addition of acids or base (acid rain and other acid waste). These are traceable to the partitioning of CO<sub>2</sub> from the atmosphere, weathering of rock, minerals and soil. The quantity of dissolved soluble salts of Torkula barite mineral in the drilling fluid was shown in Table 5. These were quantified and labeled as it relates to the global quality and environmental standards and systems (ISO 9001:2015; ISO 14001: 2015). The phenolphthalein alkalinity of the drilling fluid formulated based on Torkula barite and other additives, was at 1061mg/L. However, total alkalinity added up to 1658mg/L, indicating the total amount of calcium hydroxide (lime). Similarly, the chloride content of 1716mg/L and calcium sulphate salts of 1.6675kg/m<sup>3</sup> were obtained.

Table 4.3: Soluble Salts of Sulphate, Chlorides and Carbonate

Alkalinity	Remarks
Phenolphthalein Alkalinity	1061mg/L
Total Alkalinity	1658mg/L
Total Hardness as Calcium	Remarks
10g Torkula barite	373.3mg/L
70g Torkula barite	143.5mg/L
Chloride and Sulphate Salts	Remarks
Chloride Content	1716mg/L
Total Calcium Sulphate	1.6675kg/m <sup>3</sup> (0.5751b/lbbl)

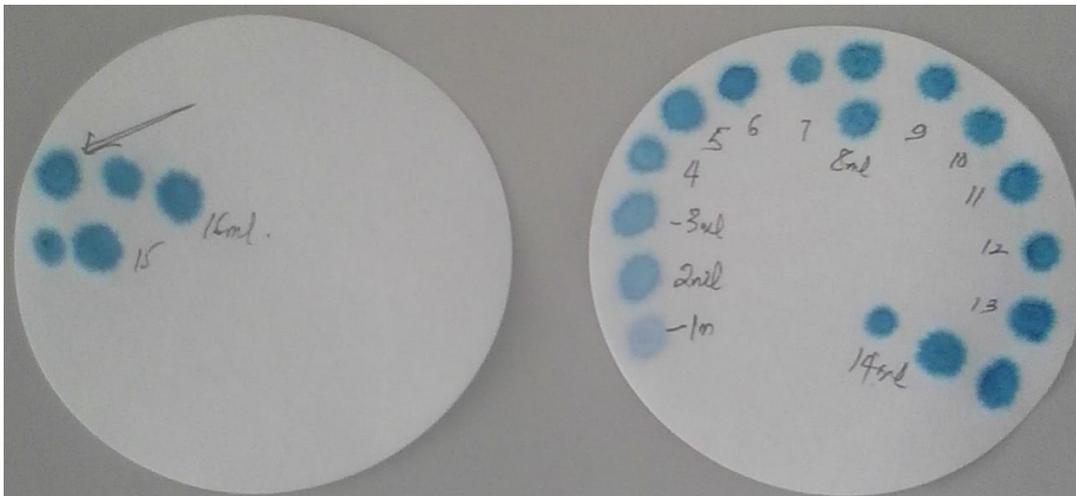
The carbonate alkalinity and barite mineral acidity are relatively higher when compared with the American Petroleum Institute (API) Standards. However, the quantity of extractable carbonates in Torkula barite is 1061mg/L. This is within the API specification of 3000mg/kg. Hence, the carbonates in Torkula barite when combined with other compound may alter toxicity. The chloride count of Torkula barite is very high. This may be detrimental to health except a scavenging element is introduced to reduce the health risk. Most importantly, high alkalinity of Torkula barite on the other hand may act to resist major change in the pH, and to control corrosion effectively (Snoeyink and Jenkins, 1980; SMEW, 1992).

### 4.3.2 Cation Exchange Capacity and Absorption of Torkula Barite Mineral

Methylene Blue Analysis studies to determine the cation exchange capacity of the solids present in a drilling mud. It focuses on the reactive portions of the clay present in media. Similarly, Cation Exchange Capacity (CEC) is the exact quantity of positively charged ion expressed in milliequivalents per 100g of fully dehydrated clay mineral (Schlumberger Oilfield Glossary; Burrafato and Miano, 1993). Table 8 and Figure 4.10 clearly examine the reactivity and absorption capacity of Torkula barite. The volume of methylene blue absorbed at different quantities of Torkula barite is 14ml. This also gave rise to similar values of methylene blue capacity and absorption, and CEC in both experiments. Hence, the active positively charged ions in Torkula barite such as  $\text{Na}^+$ ,  $\text{K}^+$ ,  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ , have been rendered passive due to the inertness of the clay mineral. And so, the basis for the indirect measured parameters was taken to be bentonite (API RP 131, 2004; API RP, 2005).

Table 4.4: Ionic Character of Clay Minerals

Quantity of Barite	M. B. C.	M. B. A.	C. E.C. (meq/10g)
10 g	7.0	12.5	0.112
70 g	7.0	12.5	0.112



70 g Torkula Barite

10 g Torkula Barite

Figure 4.10: Experimental Result for Methylene Blue Capacity, Absorption and Cation Exchange Capacity

#### 4.3.3 Mean Metallic Content of Torkula Barite

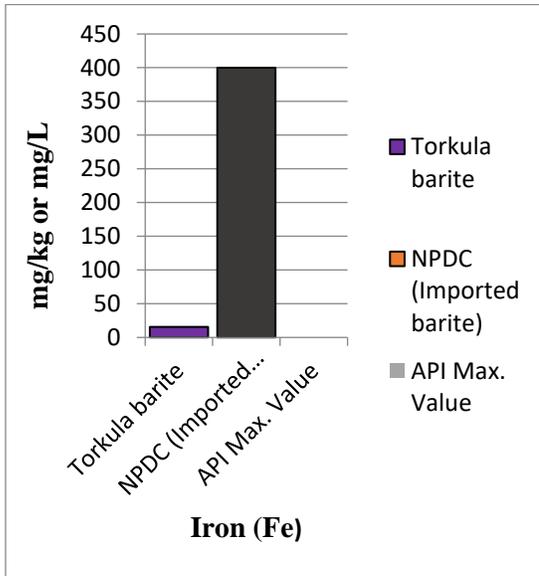
The chemical, physical and rheological responses of Torkula barite largely depend on the mean values of chemical parameter shown in Figures 4.11 (a) to (g) respectively. Similarly, these chemical parameters were compared with the American Petroleum Institute (API) and the Nigerian Department of Petroleum Resources (DPR) set limits. It was clearly shown in Table 9, Figures 4.11 (a), (b) and (d) that lead (Pb) has the highest concentration in Torkula barite, and is followed with calcium and iron minerals. This indicated the dominance of galena, calcite and hematite as gangue minerals, interlocked within the barite vein. Magnesium and zinc also occurred as gangue minerals in a relatively dispersed fraction. Similarly, copper and cadmium minerals are in traces. It can be summarized that Torkula Mining Site has a blend of high and medium grade barite mineral with deposits of galena, calcite, hematite, Pyrite, magnetite, sphalerite, brucite, chalcocopyrite and chalcocite as possible associated minerals (Osokogwu et al., 2014; Ibe et al., 2016).

Moreover, Figures 4.11 (b) to (g) show the exact value of the chemical parameters. The calcium (Ca), lead (Pb), zinc (Zn), magnesium (Mg), copper (Cu) and cadmium (Cd) minerals in Torkula barite are below the limits set by the American Petroleum Institute (API) and Nigerian Department of Petroleum Resources (DPR). The values for the minerals composition in the reference sample was evaluated in the work of Ibe et al., (2016). However, the quantity of iron mineral in Torkula barite is higher than the API set limits but lower when compared with the DPR set limit. Hence, Torkula barite needs to be beneficiated further to remove the iron content. This will be done through appropriate indigenous enrichment strategies (Ibe et al., 2016).

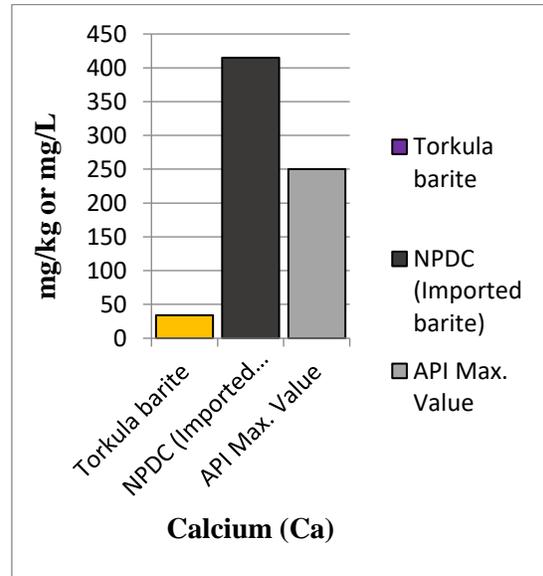
Table 4.5: Metallic Content of the Barite Samples

S/N	Metals	Concentration (mg/L)/(mg/kg)			API Max. Limit
		Torkula Barite	Imported Barite	(Ibe et al, 2016)	
1.	Ca	34.0135	415.00	709.00	250
2.	Zn	3.9051	44.60	1.60	140

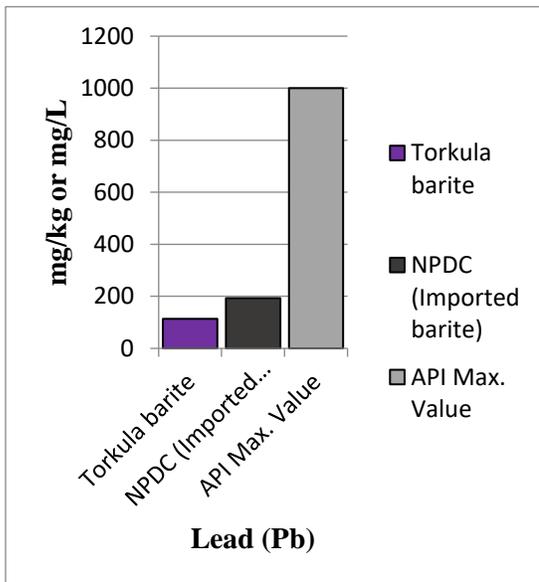
3.	Mg	8.5726	65.00	64.20	250
4.	Pb	113.8127	193.00	6.00	1000
5.	Cd	0.0008	1.60	1.60	5
6.	Fe	15.6094	400.00	62.20	Zero
7.	Cu	0.3024	1.60	160.00	36



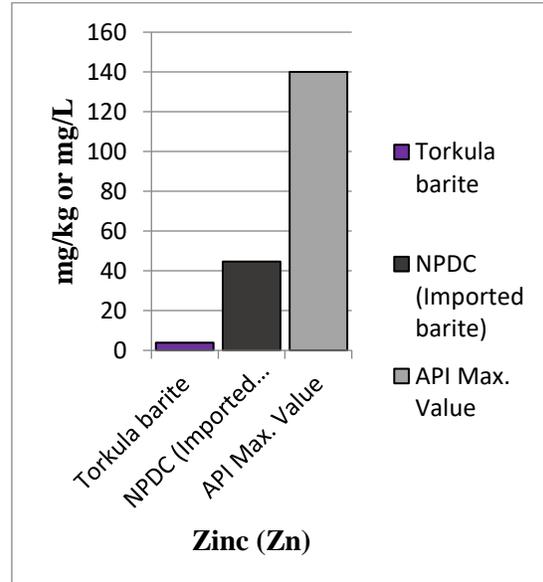
(a)



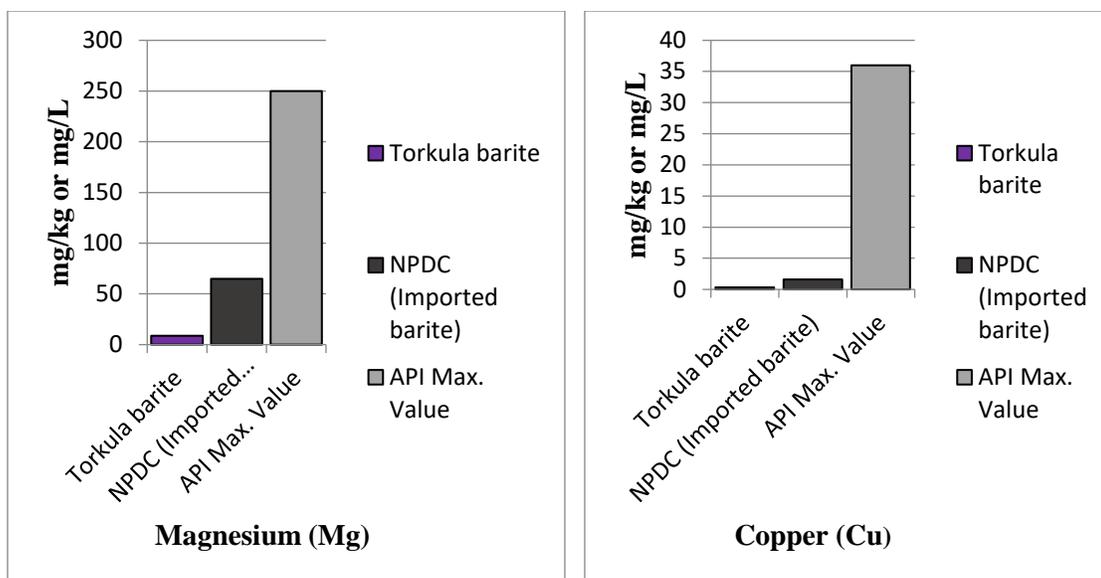
(b)



(c)

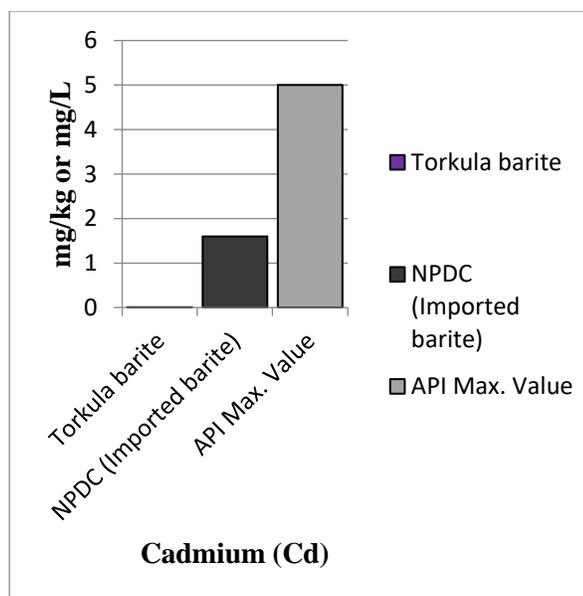


(d)



(e)

(f)



(g)

Figure 4.11a: Analysis of the Iron Mean values of most dominant Chemical Parameters

#### 4.3.4 Barium and Sulphate Content

The Fourier Transform Infrared Spectroscopy (FTIR) analytical method was used to investigate Torkula barite in the solid, liquid and gaseous states. This was aimed at establishing framework for the sample size, its state of aggregation and molecular interaction through qualitative and quantitative strategies. The Infrared (IR) spectrum of Torkula barite

has four (4) most fundamental vibration modes, exhibiting several significance bands as shown in Figure 4.12. The first two (2) strong bands (intense peaks) correspond to the asymmetric stretching and bending, as observed at  $608.43\text{cm}^{-1}$  and  $620\text{cm}^{-1}$  respectively. Similarly, the other two (2) bands are weaker ones at  $982.07\text{cm}^{-1}$  and  $1074.6\text{cm}^{-1}$ , corresponding to symmetric stretching and bending. Most of the barite spectra studied in relevant literatures emphasized on the dominance of the intense symmetric stretching of  $\text{SO}_4$  tetrahedral at  $982\text{cm}^{-1}$  respectively (Holtzer et al., 2011).

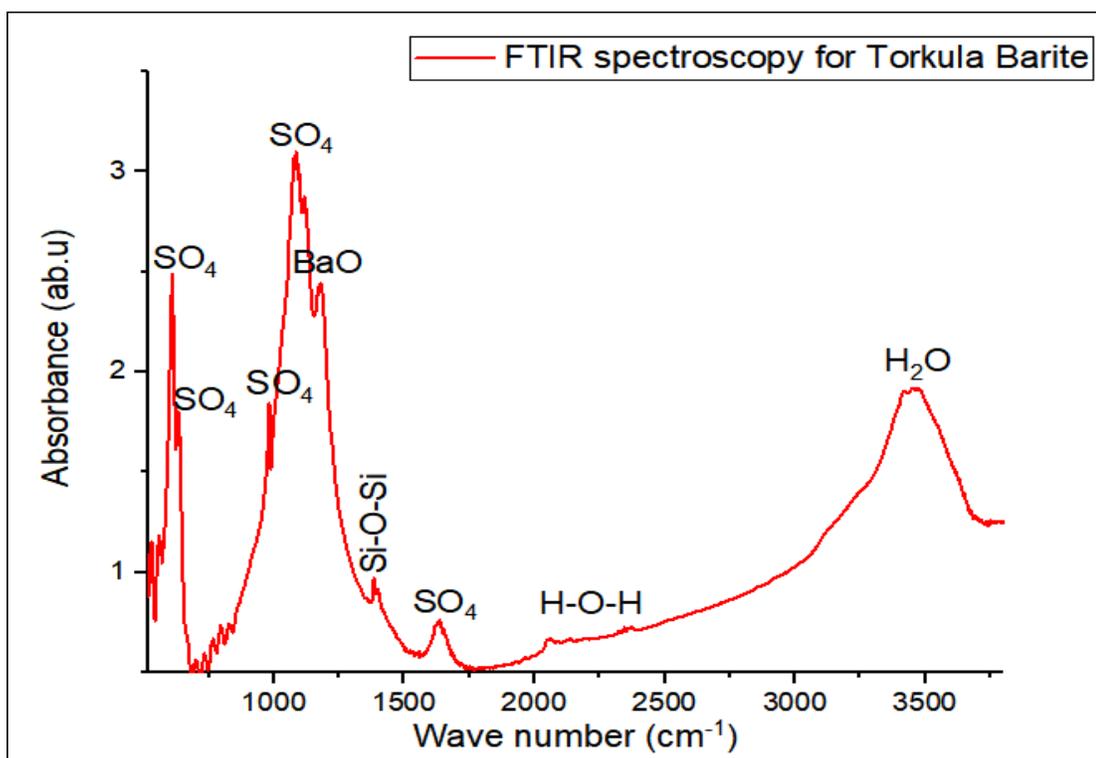


Figure 4.12: FTIR spectroscopy for Torkula barite

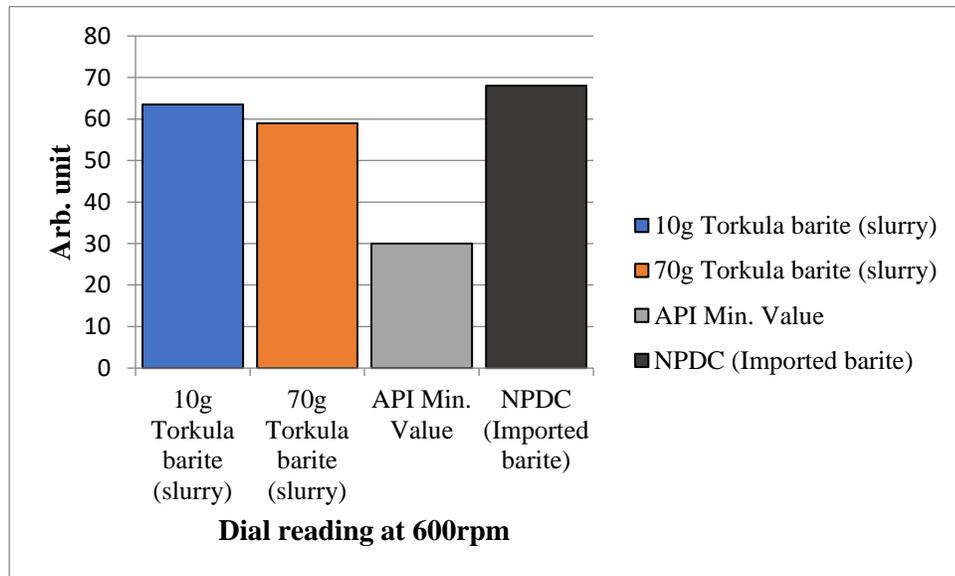
Further studies on relevant works confirmed the traces of calcite at  $1074.6\text{cm}^{-1}$  which is coinciding with other  $\text{SO}_4$  groups and the bands at  $620\text{cm}^{-1}$ . The free  $\text{SO}_4$  molecules at  $1626.6\text{cm}^{-1}$  corresponds to vibrations in the crystalline state. This is in agreement to the modification of the tetrahedral  $T_d$  group to a monoclinic local symmetry  $C_s$ . The liquid state of matter in Torkula barite was observed in the IR spectra. The occurrence was simply characterized by weak bands at  $3430$  and  $3435\text{cm}^{-1}$  respectively. However, the two (2) weak bands have strongly pleochroic absorption bands, assigned to both anti-symmetric and

symmetric stretching vibrations of H<sub>2</sub>O molecules localized on the vacant barium site at 1178.8cm<sup>-1</sup>(Prameena et al., 2013; Dimova et al., 2006).

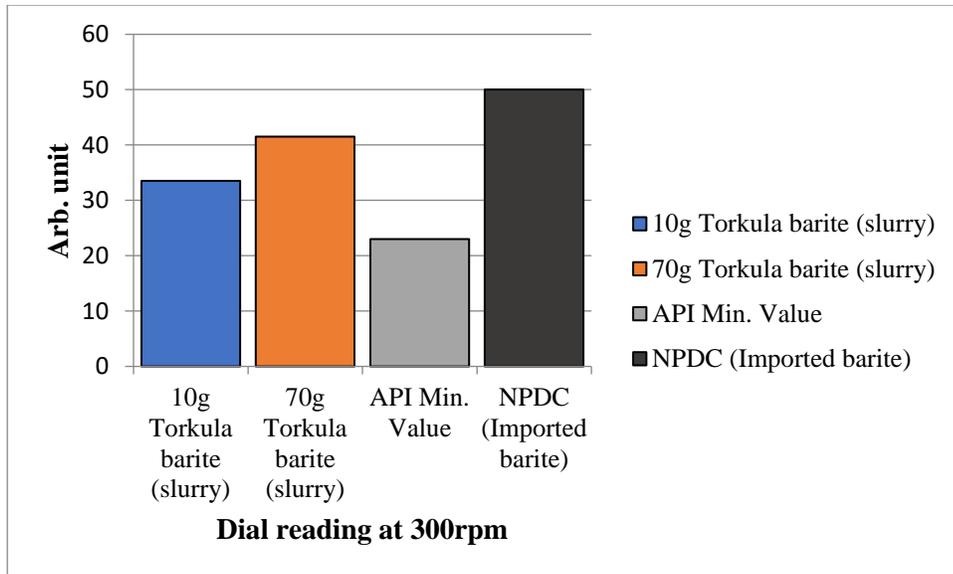
#### 4.4 Rheological Properties of Torkula Barite mineral

##### 4.4.1 Contributions of Torkula Barite to the Fluid Properties

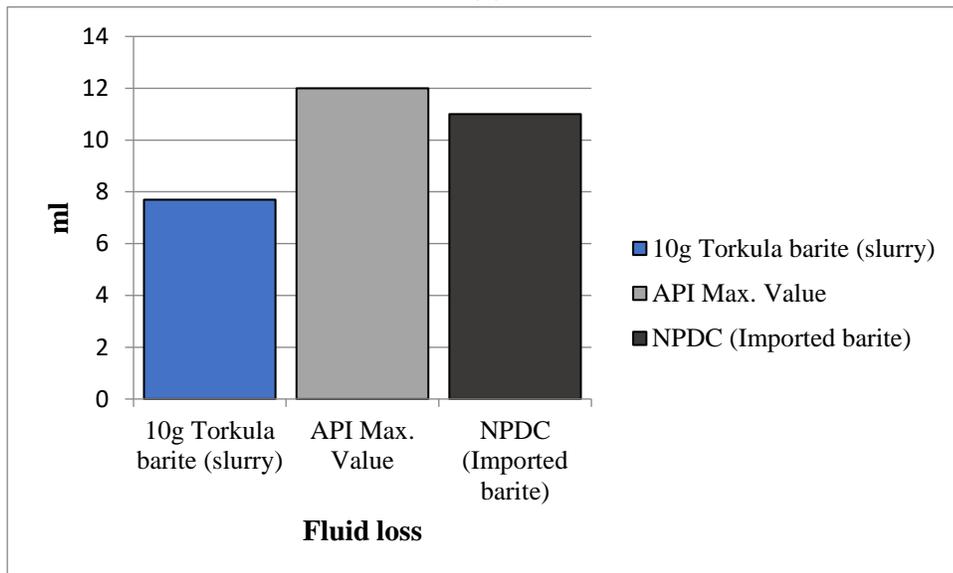
Torkula barite water-based drilling mud (WBM) can be described efficiently in relation to its behavioural change at different dial readings. Figures 4.14 (a) – (c) clearly reveal the response of the fluid at different mixing speed. The figure also shows an increase in the dial reading at 600 revolutions per minute (RPM) and a decrease in the dial reading at 300RPM, when the quantity of barite is increased from 10g to 70g. However, the dial readings at both mixing speed were higher than the minimum specification recommended by the American Petroleum Institute (API). The information obtained at different speed suggested that the drilling mud belongs to a class of Non-Newtonian fluids. Such reflects a higher viscosity at low-shear rate (RPM X 1.703) and a lower viscosity at high-shear rate. From the information in Figure 4.13, the shear stress at each mixing speed, plastic viscosity (PV) and the yield point can be calculated. Hence, Torkula barite can be used to enhance the fluid properties required for both ordinary and specialized drilling operations.



(a)



(b)



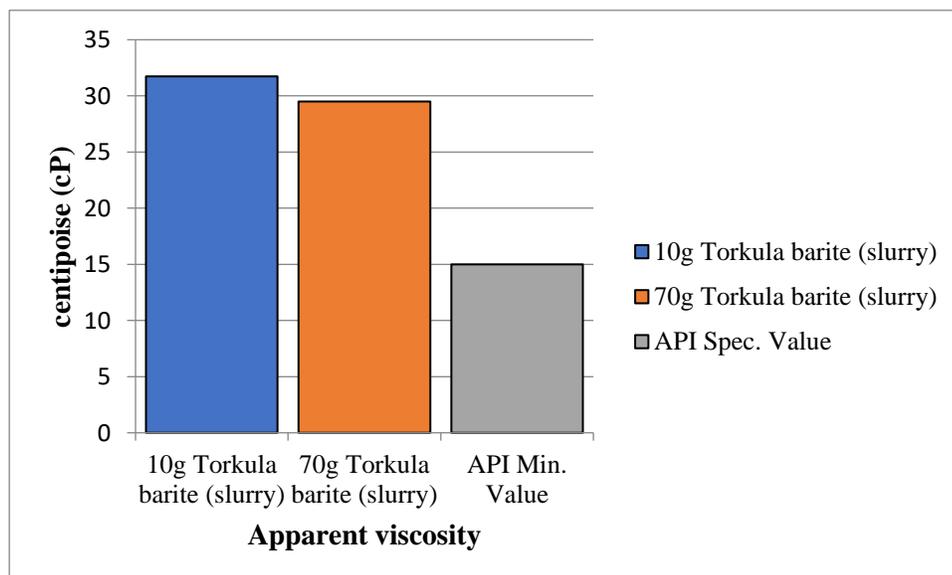
(c)

Figure 4.13: Comparison of the Fluid's Composition and Rheological behaviour of Water-Based Drilling Mud using Torkula barite

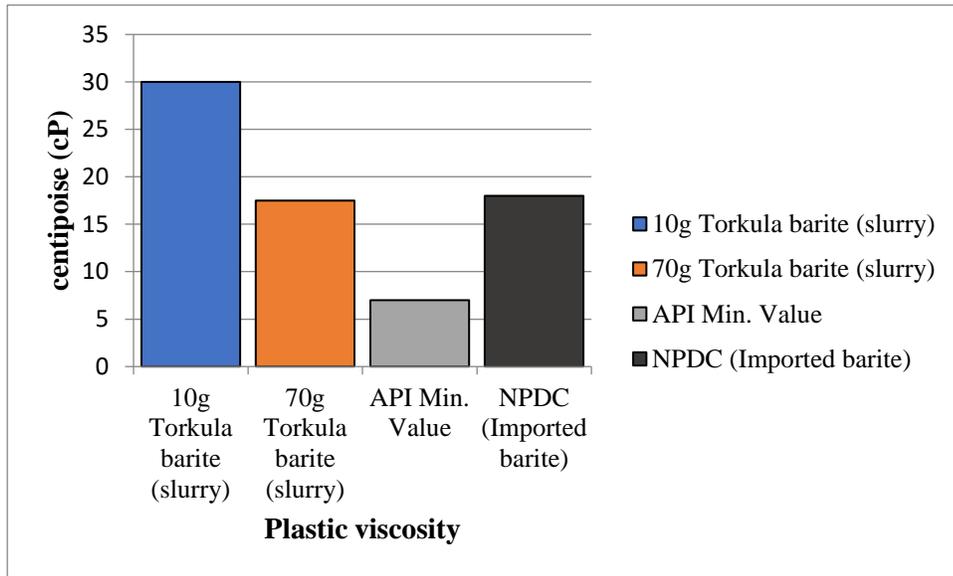
The filtrate or fluid loss from the drilling mud prepared with Torkula barite is lower when compared with the API specification and other drilling mud formulated using the imported barite. However, it is within the acceptable standards. The low value obtained is a function of the amount and particle-size of solids (barite and Bentonite) present in the mud. Hence, the field-scale beneficiation strategies employed on Torkula barite have greatly improved the quality of the mineral and its acceptance on the basis of the API regulations. The filtrate loss within thirty (30) minutes for Torkula barite is 7.7ml as it was shown in Figure 4.13 (c)

above, while that of the reference sample is 11ml and the API Specification, which is at 12ml. This attested that Torkula barite can sufficiently conserve the liquid content of the drilling mud due to its ability to form a good blend with other additives (IADC, 2014, Abduo et al., 2015; Al-Bagoury and Steele, 2012).

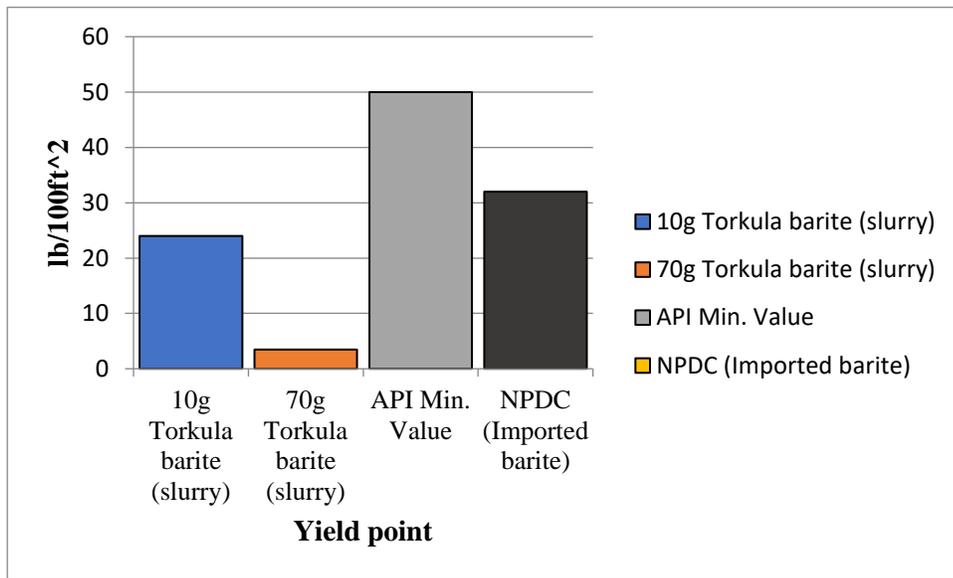
The calculated values that correctly described the measure of the internal resistance to the flow of formation fluid into the borehole during the drilling operations are clearly presented in the Figure 4.14. This shows the relationship between the fluid flow parameters. The apparent and plastic viscosities of the drilling fluid consisting of Torkula barite are higher than the API minimum values. However, these values were observed to be at the extreme end, and as such may be considered most appropriate even when there is an unexpected dilution of the fluid from a water ways. Similarly, the yield point of the fluid composed of 10g and 70g barite is lower when compared with the API Specification and the value obtained with the imported barite. Furthermore, the yield point observed on the addition of 10g barite is too low, while an average value was obtained when the amount of the weighting agent (Torkula barite) was increased from 10g to 70g. Hence, higher amount of Torkula barite is required to raise the yield point of the drilling mud. On the other hand, these velocities were measured at different shear rates to determine the rheology model coefficients. They indicated the shear stress as a dial unit or degree at a given shear rate (IADC, 2004, API, 2003; API, 2010; AMSE, 2005).



(d)



(e)

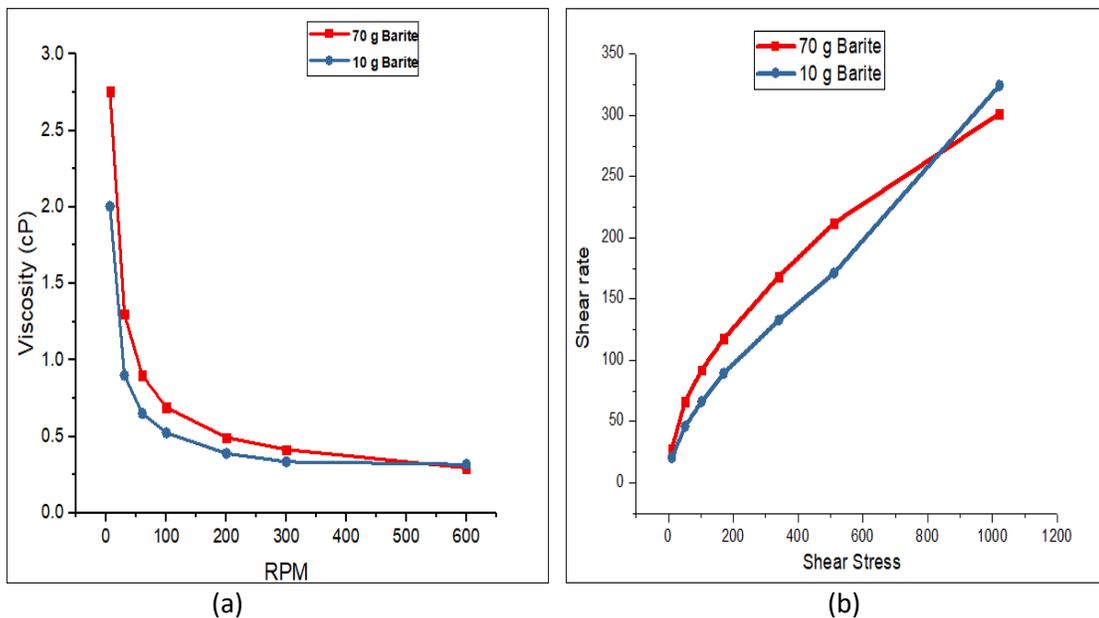


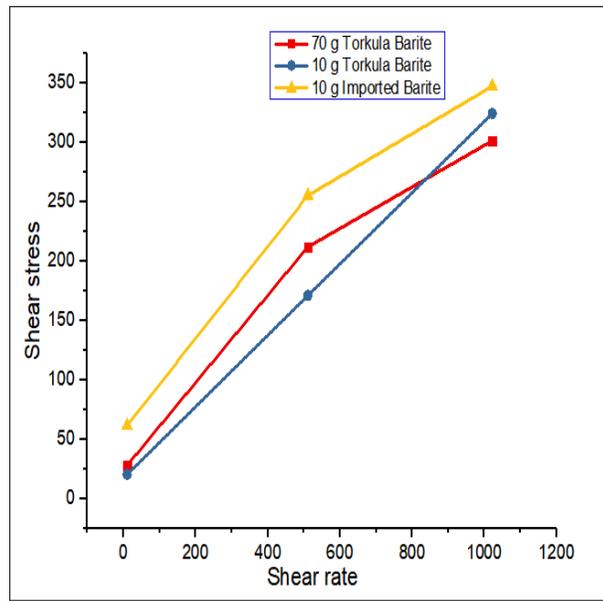
(f)

Figure 4.14: Comparison of the Rheological Properties and Behaviour of the Water-Based Drilling Mud consisting of Torkula barite

#### 4.4.2 Responses of Torkula Barite to Mud Rheology and Viscous Behaviour of the Water-Based Drilling Mud

It was obviously revealed in Figure 4.15 (a) that the viscosity experienced a sudden fall as the mixing speed was increased infinitesimally from 300RPM to 600RPM for the water-based drilling mud formulated using 10g and 70g Torkula barite. However, the viscosity was almost constant for both drilling mud between the mixing speed of 300 and 600RPM. This suggested that the fluid with 10g barite tends towards the Newtonian fluid while as a non-Newtonian fluid with the addition of 70g Torkula barite. Similarly, the emulsion gel was enhanced and significantly formed for the two fluids when the mixing speed was reduced from 300RPM until the final gel point. The gap at the elbow signifies the deviation of the drilling fluid from an ideal fluid (Newtonian fluid). Hence, Torkula barite can be used to enhance and sustain emulsion gel strength of a drilling mud. Furthermore, Figures 4.15 (b) and (c) validate the variation of the mud rheology for different weights of Torkula barite.





(c)

Figure 4.15: Variation of the Mud Rheology and Viscous Behaviour of the Water-Based Drilling Mud consisting of Torkula barite

The response of the drilling mud formulated with 10g imported and 70g Torkula barite was quite different from such consisting of 10g Torkula barite. This theoretical framework was consolidated with the low viscosity at high-stress rates, whereas a higher viscosity at low-stress rates. Hence, the rheology of the drilling mud can be improved with a slight increase in the quantity of Torkula barite, as clearly revealed in Figure 4.16. This will be desirable and appropriate where minimum pressure losses are wanted for the high-shear conditions inside the narrow bore of the drill string, and the large annulus (API, 2004; API, 2010, Ukooa, 1999a).

The qualities of the drilling mud and the permeability of the filter cake produced from Torkula barite as a weighting agent were clearly represented in the Figures 4.16 (a) & (b) respectively. These have been significantly justified in Figure 4.16 (a). The mud weight increases from 8.30ppg to 8.40ppg when the quantity of the imported barite was increased from 5g to 10g. However, the mud weight at 5g Torkula barite was higher and the differential increase experienced between 5g and 10g Torkula barite was 0.15g as compared to 0.1g in the imported barite. Similarly, the quality and permeability of the filter cake was a function

of the API Filtrate loss. The response of the water-based drilling mud formulated using 10g Torkula barite was within the API Standards. Despite the addition of the fluid loss enhancer to the reference sample, the filtrate loss was still beyond the API Standards. Hence, pulverized Torkula barite has no need for an enhancer to lower the filtrate loss. Although, this may be due to the quality of bentonite used in formulating the drilling mud. However, colloidal-sized solid particles should be introduced to minimize the drill solids content of the drilling mud (API, 2004; API, 2010, IADC, 2014, Osokugwu et al., 2014).

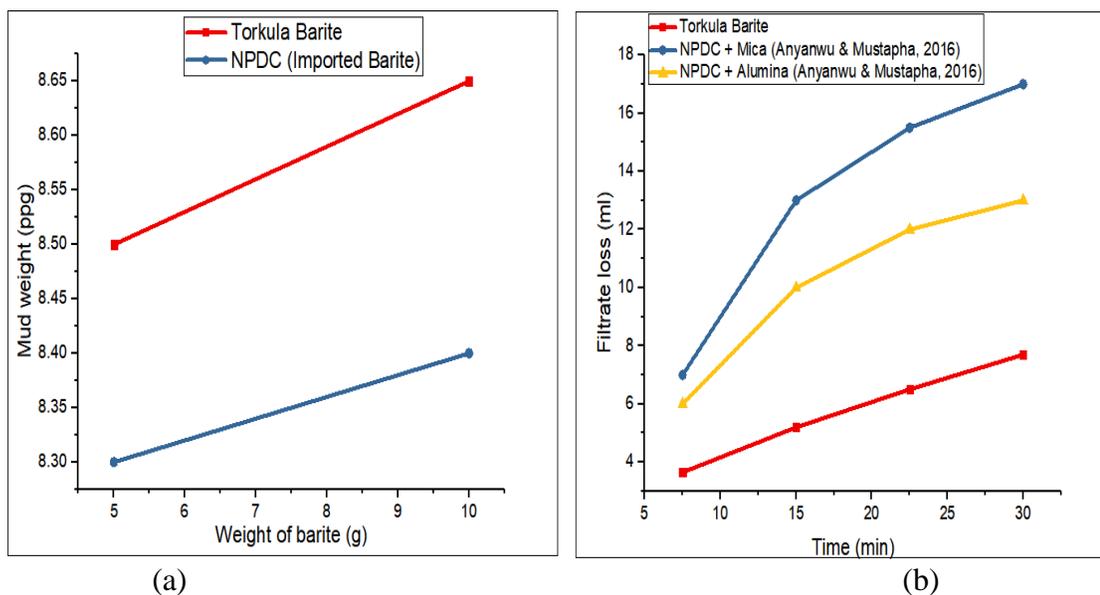


Figure 4.16: Variation of the Mud Rheology and API Filtrate Loss- Time graph for the Water-Based Drilling Mud, consisting of Torkula barite

#### 4.4.3 Correlation among Drilling Fluid Properties as a Function of Torkula Barite

##### Yield

##### Gel Strength:

This clearly distinguishes the shear stress measured at low shear rate. Such fluid property can be measured after the drilling fluid has set quiescently, and within the period of time stipulated by the API procedure. To sufficiently classify the drilling fluid according to the gel strength, the experiment may be done within 10 seconds, 10 or 30 minutes, and 16 hours. Torkula barite is capable of forming gelled structures few minutes after mixing. The

emulsion gel structure formed with 5g barite was more stable, which increases as the quantity of barite added into the drilling fluid increases. Hence, the strength of the attractive forces (gelation) in the drilling mud or fluid under static conditions is a function of the quantity of the barite added as a weighting agent. Above certain quantity of barite, high solid concentration may result into flocculation. However, Figure 4.17 clearly indicates a low-flat gel curve. This implies that the addition of Torkula barite into the drilling mud enhances a desirable condition, improved magnitude of gelation with time, the general performance of the drilling mud and well bore. Torkula barite produces a fluid with both flat and thixotropic gels. This can be related to the static and dynamic settling (Herzhaft et al., 2006; Marsh, 1931; Abduo et al., 2015; Anyanwu and Mustapha, 2016, Werner et al., 2017).

#### **Hydrogen Potentials of the Drilling Mud (pH):**

This relates the power of hydrogen or hydroxyl ion on the mud rheology. As one of the classical components of drilling mud required to enhance the corrosion inhibiting potentials, the fatigue life of the drill bore and bits is improved (Azar and Samuel, 2007; Committee, 2011). Torkula barite is a weak alkaline material with a pH of 8.015 as shown in Figure 4.17. Hence, the pH of the drilling mud can be gradually reduced from the excess of 12 to 10.5. Similarly, Torkula barite reduces the pH of drilling mud from the 11.973 to 10.65 when it is increased from 5g to 10g of Torkula barite. However, specialized drilling and cementing operations require the hydrogen potential between 10.00 and 10.50. On this basis, Torkula barite can be applied and used to formulate drilling mud for both ordinary and specialized operations with a guarantee of most promising fatigue life. This is achievable due to the low chloride content in Torkula barite. In addition to these, hydrogen potential controls the mud rheology and determines the yield stresses in a drilling mud. Similarly, Torkula barite increases the metallic ionization, enhances Ca and Mg dissociation required to improve the mud rheology, when added as a weighting agent. It gradually reduces the pH to normalize the inter-particle force of attraction and increases the viscosity at a pH of 8.5 (Huang et al., 2016).

#### **Plastic Viscosity:**

Figure 4.17 clearly distinguishes the relationship between the shear rate and shear stress induced in a drilling mud. It is the frictional resistance to the flow, until the yield stress is attained and an applied force reaches the induced frictional resistance. Such resistance is as a result of the inter-particle forces of attraction and continuous shear rate at and above the yield stress. It is also the slope of the shear stress-shear rate line above the yield point (Schlumberger Oilfield Glossary, [www.petropedia.com/definition/2920/plastic-viscosity-pv](http://www.petropedia.com/definition/2920/plastic-viscosity-pv) 25/11/2017, 00:05 am). Plastic viscosity varies with the quantity of Torkula barite added as weighting agent. It increases from 7 to 30cP when the weight of Torkula barite was increased from 5 to 10g. Similarly, the plastic viscosity falls to 17.5cP at the increase of 70g Torkula barite. This clearly revealed that plastic viscosity increases with the amount of solid, and might also decrease when the solid content is in excess. Torkula barite can be used to increase the plastic viscosity of drilling mud to an acceptable limit. However, such drilling operation will be done at an intermediate rate of penetration (ROP). Hence, drilling mud formulated with Torkula barite is capable of rapid drilling (Schlumberger Oilfield Glossary, <http://dragon.unideb.hu/~kolloid/colloid/lectures/chembsc/lecture%2009.pdf> 25/11/2017, 4.00pm)

### **Yield Point:**

This is an exact value of yield stress extrapolated to a shear rate of zero. It is defined as the initial resistance to flow induced by electrochemical forces between the particles of the solid content constituting a drilling fluid. Such parameter is graphically obtained from the Bingham plastic fluid plot as the zero-shear-rate intercept on the shear stress axis (Abduo et al., 2015). Contrary to the response of the drilling fluid in terms of the plastic viscosity, the additional increase in Torkula barite from 5g to 10g reflected a slight fall in the yield point. However, the increase in yield point from 3.5lb/100 ft<sup>3</sup> to 24lb/100ft<sup>3</sup> was as a result of the additional 60g barite. The contribution of Torkula barite as a weighting agent in a drilling mud is largely dependent on the surface properties and volume concentration of the solid components as correlated in Figure 4.17. This was evaluated on the basis of the cuttings and solids-carrying ability. Hence, increasing the quantity of Torkula barite in a drilling mud will increase the ability of such non-Newtonian fluid to lift cutting and clean the wellbore hole. Similarly, the yield point observed at 10g barite can be further improved through the addition

of freshly dispersed kaolin clay or calcium hydroxide (Schlumberger Oilfield Glossary, Abduo et al., 2015).

**Apparent Viscosity:**

The International Union of Pure and Applied Chemistry (IUPAC) defined plastic viscosity as the ratio of stress to rate of strain. It is calculated from the measurement of forces and velocities of flow of a liquid superimposed as a Newtonian fluid. Apparent viscosity is measured on the basis of the shear rate at a fixed temperature. The scale is modeled as one-half of the dial reading at 600RPM, and specified by the American Petroleum Institute (API) Standards (API RP 13B-1, ISO 9001:2015). The responses of the drilling fluids in correlation to the quantity of Torkula barite was clearly analyzed in Figure 4.17. The apparent viscosity of the drilling mud increases from 11lb/100ft<sup>3</sup> to 31.75lb/100ft<sup>3</sup> when the quantity of Torkula barite was increased by 5g. However, the plastic viscosity begins to experience a slight fall when an additional 60g of local barite was added into the drilling mud. This is as a result of the increasing solid content of the drilling mud. Torkula barite is compactible and suitable for specialized drilling operations within a control volume of liquid and solid content (Robert et al., 2012, <http://dragon.unideb.hu/~kolloid/colloid/lectures/chembsc/lecture%2009.pdf> 25/11/2017, 00:05 am)

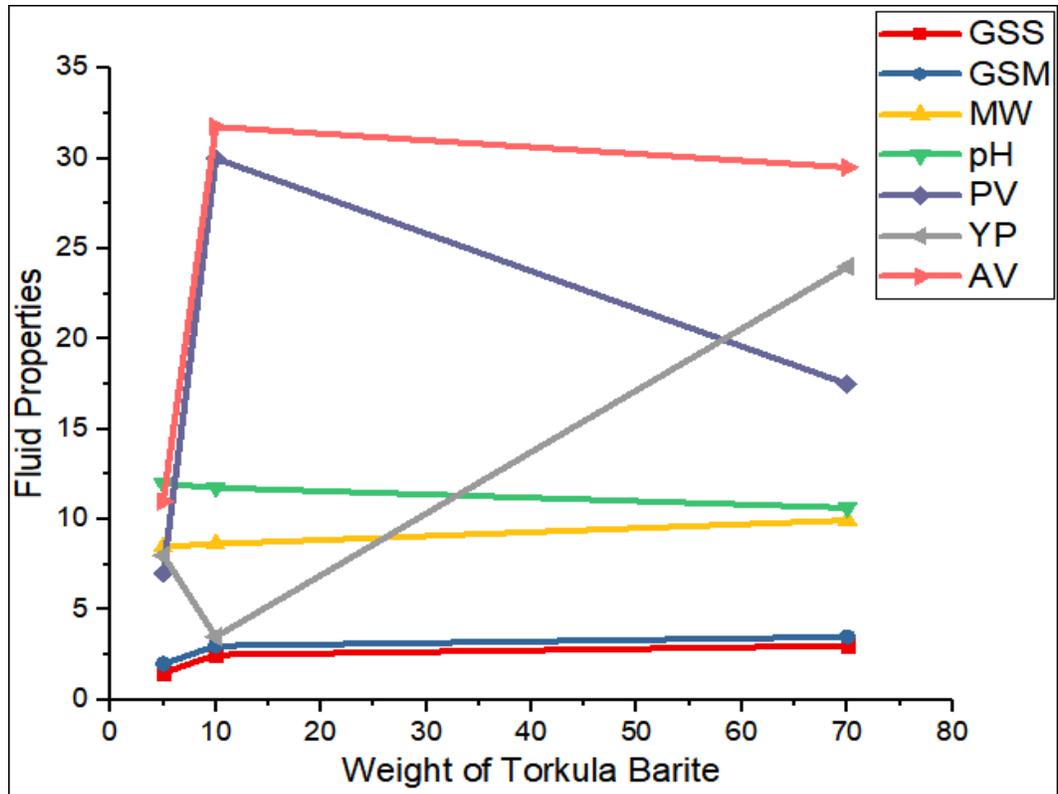


Figure 4.17: Effect of Torkula Barite on the Water-Based Drilling Mud Rheological and Physical Properties (Plotted using Origin Software).

#### 4.4.4 Mud Weight Control Parameter for Torkula Barite

##### 4.4.4.1 Torkula Barite Feed Rate

This is the exact mass per unit volume of an API drilling mud. It is measured to determine the hydrostatic pressure of the mud column required to enhance pore pressure and control the formation pressure. Such parameter can be measured based on the field or laboratory scale (API, 1993; API, 2004; API, 2010, IADC, 2014). The significance of the specific gravity of Torkula barite was clearly defined in Figures 4.18 and 4.19. Infinitesimal addition of the barite raised the weight of the drilling fluid. Increasing the quantity of Torkula barite from 5g to 100g reflects an increase in the mud weight from 8.49ppg to 10.5ppg. Similarly, the Torkula Mud Weight test as shown in Figure 4.19 resulted into the formulation of a mathematical expression to guild the industrial application of the weighting agent. Hence, such graphical analysis will serve as a solid control expression for an indigenous drilling mud. Torkula barite can be blended with Bentonite to increase the weight of the drilling mud

efficiently. It is rarely an indigenous mineral with potentials un-explored required to replace imported weighting agent. Torkula barite will sufficiently compete in functions, applications and performance without resulting into an excess weight of the solids. It is suspected to avoid settling down and fracturing of the formation gradient (Joel, 2013; Bourgoyne et al., 1986; Haaland et al., 1976).

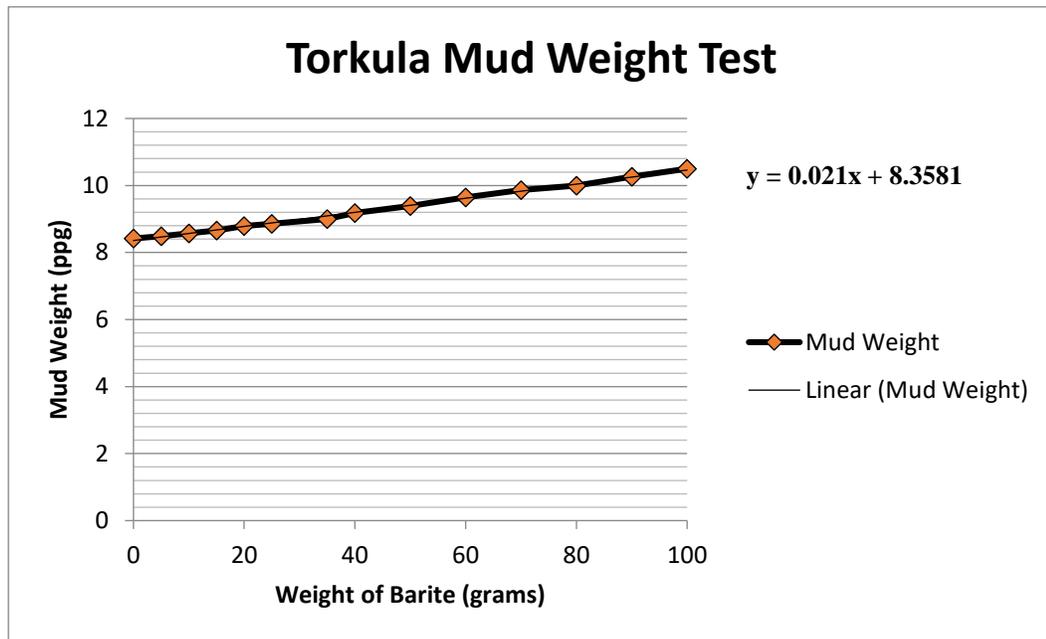


Figure 4.18: Mud Weight Control Parameter for Torkula Barite

#### 4.4.4.2 Percentage Volume of Liquid and Solids (Retort Test)

Improving and controlling the density of drilling fluid using Torkula barite requires a comprehensive understanding of the different classes of particle-sizes and the types of solid involved. This is essential to determine the fluid density, gel strength, filter cake quantity, filtration control, chemical and mechanical properties (Kanna et al., 2017). Figure 4.19 and Table 4.6 clearly show an increase in the quantity of solids from 2% to 19% and decrease in the volume of liquid from 81% to 98% as the added weight of Torkula barite increases from 5g to 70g. Hence, Torkula barite contributes to the increase in the solid content and a decrease in the volume of water. Similarly, it can be used to control the mud rheology and properties, quality of the filter cake and hole stability. Increasing the quantity of Torkula barite increases the surface area of the solid in the mud, as well the viscosity. Excess solid content in 70g

barite may result into wear of drill bits. However, relatively soft texture Torkula barite will reduce the rate of wear of wellbore parts (Ismail and Wai Meng, 2000).

Table 4.6: Composition of the drilling mud

S/N	Sample	Percentage Volume	
		Solid (%)	Liquid (%)
1.	5 g Torkula barite	2.00	98.00
2.	10 g Torkula barite	4.00	96.00
3.	70 g Torkula barite	19.00	81.00

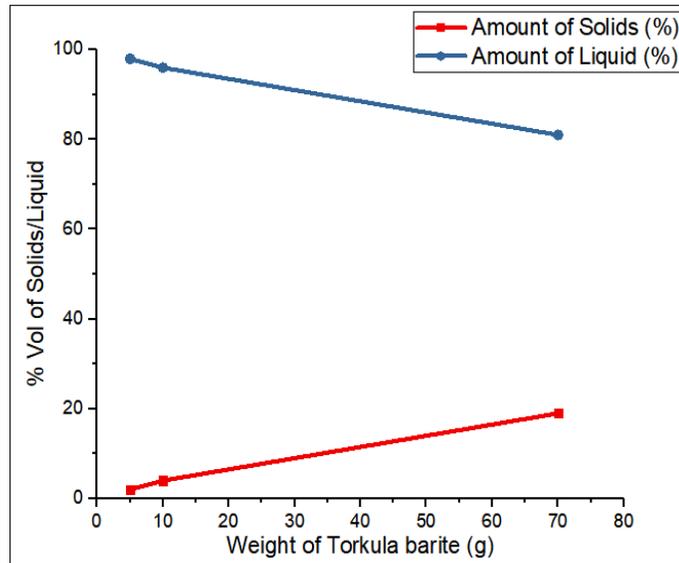


Figure 4.19: Physical Composition of the formulated Drilling Mud

#### 4.4.4.3 Quality of the Filter Cake as a Function of API Filter Loss

The analysis on the quality of filter cake was shown in Figures 4.21 (a & b). It was very obvious that the cumulative filtrate volume increases with time function. However, the volume of filtrate collected at the discrete time steps decreases gradually with time. This was justified with the quadratic nature of the filtration curve. Similarly, the volume of the filtrate lost increase with the square of time. The significance of the spurt loss was evident in Figures 4.20 (a) and 4.20 (b) respectively. The filtrate loss of 3ml was observed as the sporadic rise between 0 and 5 minutes, and measured on the API scale to be 0.0056. The negative (minus sign) symbol in the API filter loss expression clearly describes the initial formation processes

which gave rise to the filter cake. A thin, firm filter cake was obtained with 10g Torkula barite as shown in Table 4.7. Increasing the quantity of Torkula barite in the drilling mud reduces the spurt loss. However, the thickness of the filter cake was increased, became firm and flexible. Hence, 10g Torkula barite was used to enhance the quality of the filter cake. This is fundamental to the sealing of the formation pore by the restriction to influx and further invasion of the fluid (API RP 13B-1; Anyanwu and Mustapha, 2016; chee et al., 2007, ISO 9001, 2005; ISO 14001, 2015).

Table 4.7: Analysis of the Filter Cake

Description	Remark	
	10 g Torkula barite	70 g Torkula barite
Weight of the filter cake before drying	(1.50 ±0.02) g	19.86 g
Thickness of the Filter cake	1/32 inches	4/32 inches
Quality of the cake	Thin and Firm	Thick, Firm and Flexible

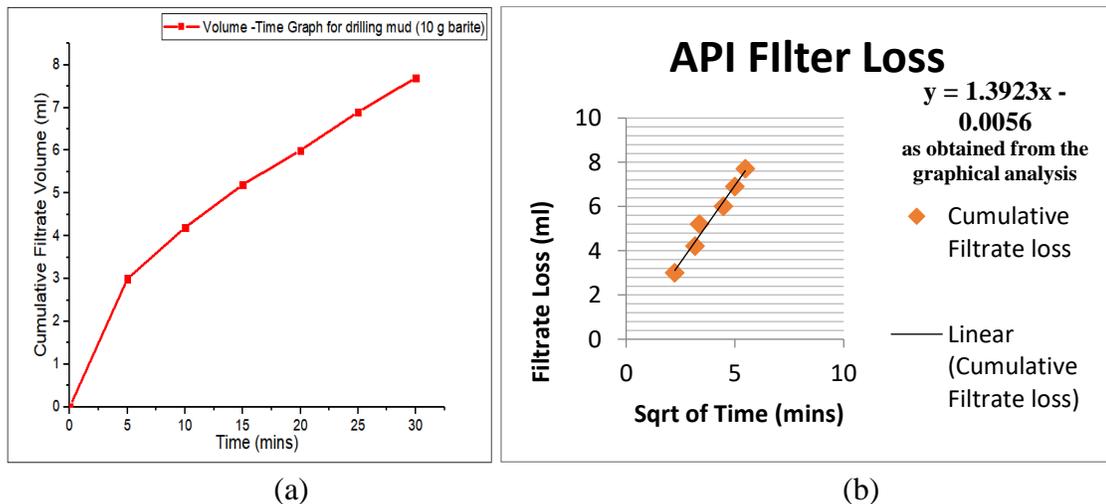


Figure 4.20: Filtrate loss Analysis as a Function of Torkula barite

#### 4.4.4.4 Analysis of Mud Lubricity

Mud lubricity measures the effect of torque and drag in extended reach drilling (ERD). It is a coefficient that relates the lubricating ability of a drilling mud on the basis of the reference liquid. The associated parameters are being measured in terms of the coefficient of friction (CoF) and reference correction factor. The response of the drilling mud in terms of the CoF

and lubricity coefficient was indicated as a function of the quantity of Torkula barite (Mamat et al., 2013; Aston et al., 2013; Cameroon, 2001). Table 4.8 further revealed that the solid particles of Torkula barite acted as a lubricating agent. The coefficient of friction and lubricity coefficient decreases from 0.46 to 0.323 and 0.46 to 0.24 respectively as the quantity of Torkula barite increases from 5g to 70g. However, this torque reading was unstable, and suddenly decreases as a function of time as shown in Figure 4.21 (b). Similarly, the lubricity index was sustained and increases infinitesimally for 10g Torkula barite, as shown in Figure 4.21 (a) accordingly. It clearly indicated an effective wetting the surface by the drilling mud but the film strength was insufficient to withstand the larger forces exerted between the drill strings. The film strength of the mud consisting of 5g Torkula barite was too weak to resist excessive emulsification while the high fluid density of the formulated drilling mud reduces the coefficient of friction after the first five (5) minutes. However, all the drilling mud at different composition of Torkula barite has their CoF within the general range of 0.15 to 0.45, but outside the typical range of 0.20 to 0.28 (Redburn et al., 2013; Schamp and Estes, 2006).

Table 4.8: Mud Lubricity

Quantity of barite	Torque Reading @ 60RPM, 5 mins
5 g	43.7
10 g	42.0
70 g	32.3
Coefficient of friction	Lubricity Coefficient
0.437	0.46
0.420	0.45
0.323	0.34

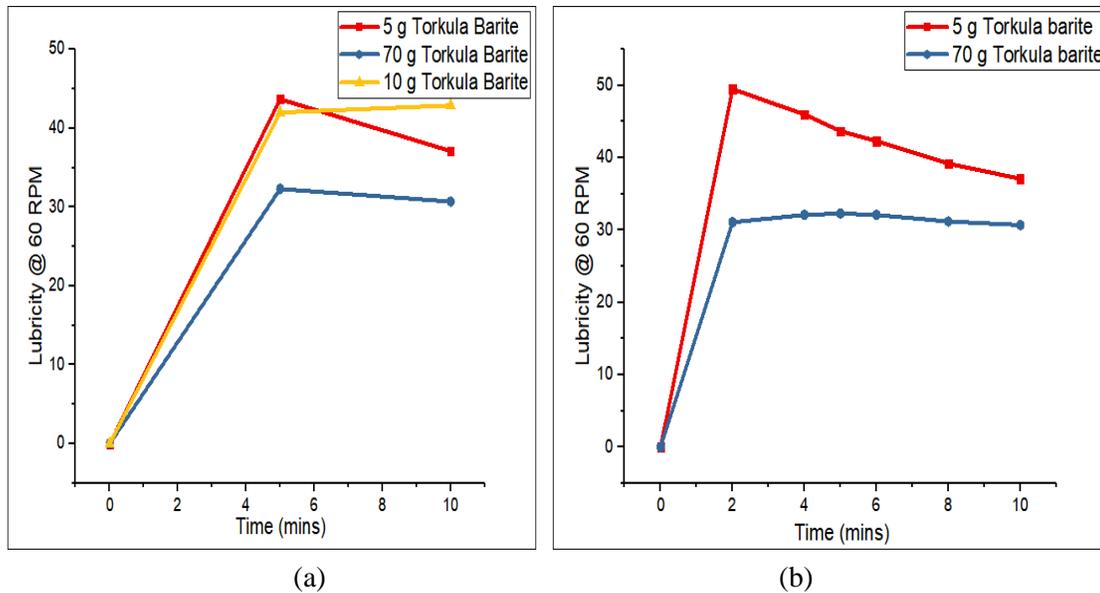


Figure 4.21: Mud Lubricity as a function of time and the quantity of Torkula barite

#### 4.4.5 Electrical resistivity and Emulsion Stability of Drilling Mud

Tables 4.9 and 4.10 clearly described the ionic character of the solid particles in the drilling fluid. This character and composition gave rise to different responses in terms of the cutting cleaning ability and mud rheology. Increasing the quantity of Torkula barite in the mud alternates the electrical charges in the solid particles such as the  $\text{Na}^+/\text{Ca}^{2+}$  ions. At an increase of 60g Torkula barite, the electrical resistivity increases from  $0.15\Omega m$  to  $0.59\Omega m$ , and decrease the temperature from 84F to 72F. Introducing more ionic character is required for mud-emulsion stability. The electrical conductivity was at zero because of the type of drilling mud. This implies that there was no formation of a conductive bridge due to the absence of an aqueous fluid and particulate matter. The voltage required to form the bridge was insufficient to conduct carriers of the water. However, the viscosity, types and concentrations of solids, aqueous fluid and emulsifier were a determining factor for considerations (Mohammed, 2017; Abd-Jdayil, 2011; Growcock, 1994).

Table 4.9: Electrical Resistivity

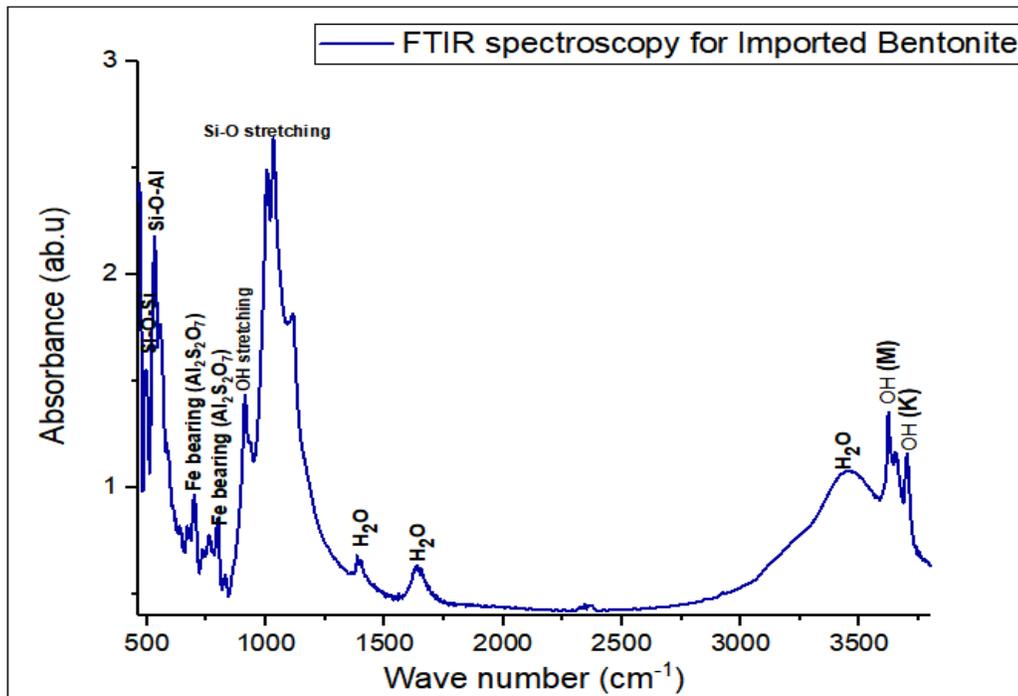
Composition	Resistivity ( $\Omega m$ )	Temperature (F)
5 g barite	0.15	84.00
10 g barite	0.27	74.00
70 g barite	0.59	72.00

Table 4.10: Electrical Conductivity

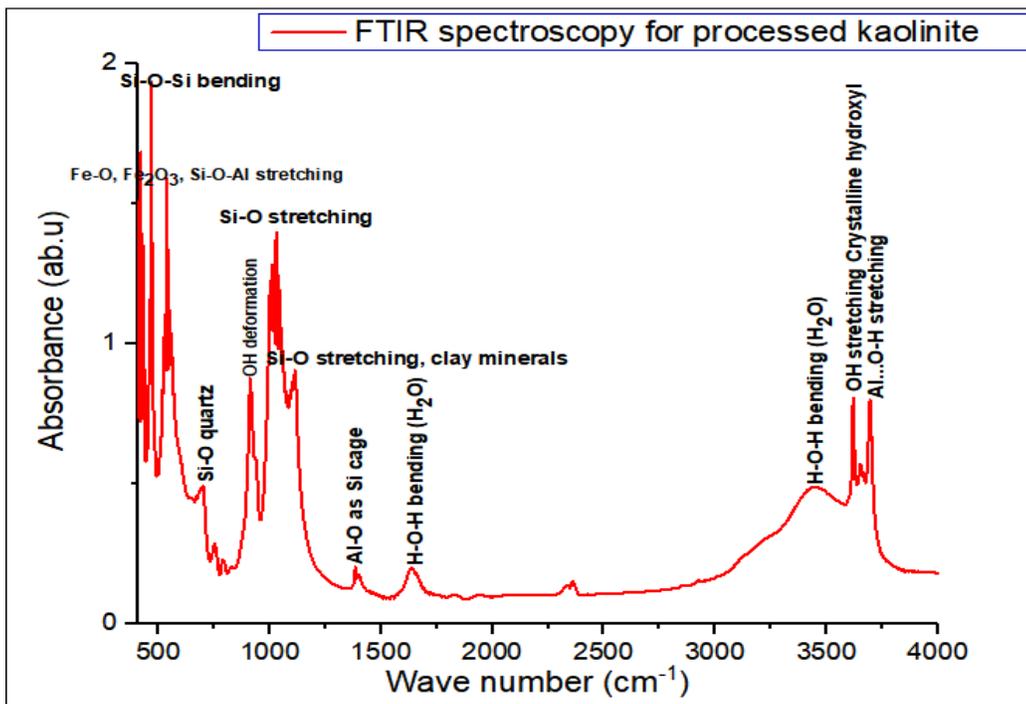
Composition	Conductivity (Volts)
10 g barite	0.00
70 g barite	0.00

#### 4.5 Materials Characteristics and Composition of Imported Bentonite and Locally Processed Nigeria Kaolinite

The pulverized imported bentonite used as a viscosity and filtration control agent was analyzed and compared with the locally processed kaolinite, to examine the inherent potentials of Nigeria kaolinite, and consider possible substitution of imported bentonite. Samples of the two minerals were prepared by the pellet techniques with potassium bromide (KBr). Investigations and analysis were carried out within the wave number range of 475 and 4000 $\text{cm}^{-1}$  to observe and record bands of absorbance function. The most dominant characteristics of Montmorillonite were observed in both spectra of imported Bentonite and Nigeria Kaolinite at 1034 $\text{cm}^{-1}$  respectively. Si-O asymmetric stretching at similar peak was also observed in relevant literatures (Holtzer et al., 2009). Similarly, the vibration of OH groups for both Montmorillonite and kaolinite was observed in imported bentonite at 3625 $\text{cm}^{-1}$  and 3696 $\text{cm}^{-1}$  respectively. However, the separated Fe ions attached to the kaolinite at 865 $\text{cm}^{-1}$  and 875 $\text{cm}^{-1}$  were replaced with sandstone (silica) as impurities in kaolinite spectra as revealed in Figures 4.22. Hence, further analysis and ionic substitution can be considered to examine the response of locally processed kaolinite in viscosity and filtration control (Holtzer et al., 2011; Holtzer et al., 2009).



(a)

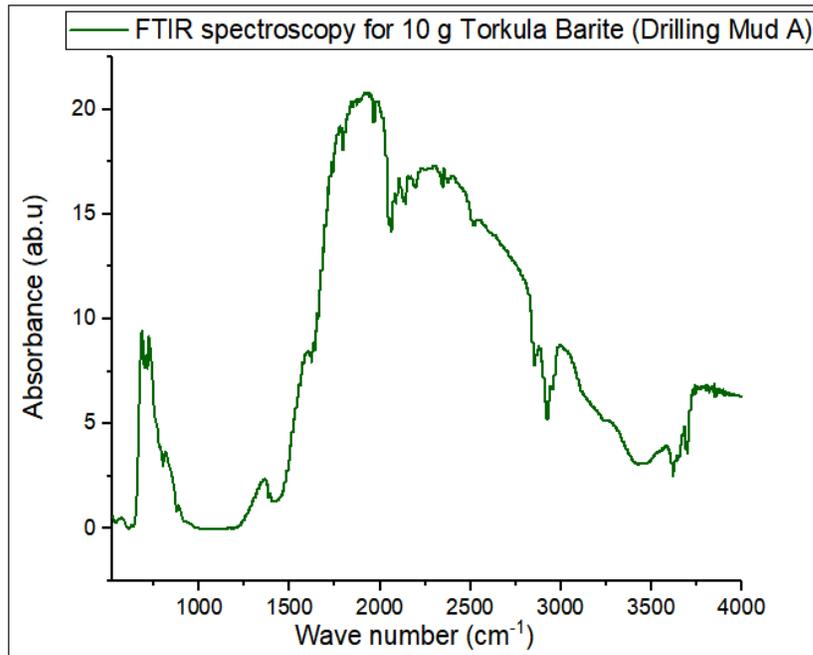


(b)

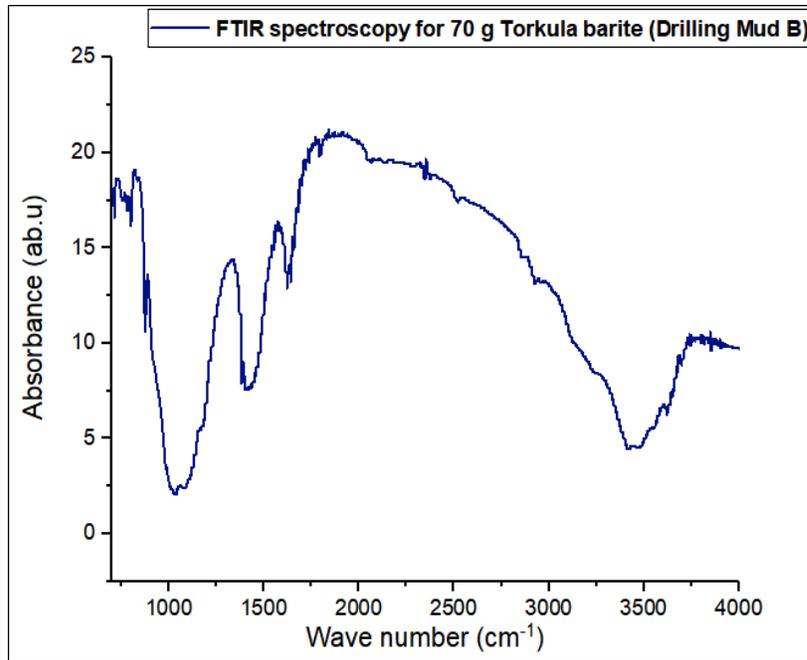
Figure 4.22: FTIR spectroscopy for imported bentonite and locally processed Nigerian kaolinite.

#### 4.6 Microstructural Variations of a Water-based Drilling Mud as a Function of Torkula Barite

The increase in the quantity of Torkula barite added as weighting agent in drilling mud resulted into significant microstructural transformation as shown in Figures 4.23 (a) and (b) respectively. The FTIR spectroscopy for 70g Torkula barite shows an increase in the absorbed functional group and the disappearance of the peaks found at  $329\text{cm}^{-1}$  and  $460\text{cm}^{-1}$ . This implies that such increase had led to the introduction of the amorphous character into the structure. Hence, the reactivity of the drilling mud in response to the dissolved salts may gradually diminish as result of the solid disparity (Redburn et al., 2013; Mamat et al., 2013).



(a)



(b)

Figure 4.23: FTIR spectroscopy for Torkula Barite at 10g and 70g respectively

## 5.0 Conclusions, Recommendations and Future Work

### 5.1. Conclusions

The phase and elemental analysis of Torkula barite mineral indicated the presence of sandstones, clay minerals of aluminosilicate, calcite, hematite, galena, brucite, halite, teriorite, muscovite, and sphalerite as gangue minerals. These associated minerals were interlocked within the Torkula barite vein. The on-site beneficiation processes reduce the quantity of the gangue minerals. This also increases the quantity of barium sulphate and enhances the quality of Torkula barite minerals. The barite mineral is rich in barium oxide and sulphur trioxide, which is the candidate material for sulphate. Torkula barite has 87.79% BaSO<sub>4</sub>, 1.39% Fe<sub>2</sub>O<sub>3</sub>, 6.66% silicate salts, less than 0.04% of water soluble salts, 1.603% total heavy metals and 1.10% LOI. However, the quantity of barium sulphate and water soluble salt was lower than the API set limits. Health risk and environmental degradation is inevitable due to the huge deposits of harmful heavy metals such as lead, copper, strontium, zinc and tungsten.

The pH of the Torkula barite is within the operational pH and the standard range for a weighting agent, as recommended by the American Petroleum Institute (API). It is appropriate for long-time storage without any operational adverse effect on the drilling mud. Similarly, the moisture content of 0.118% is within the API standards and specification of 1% at maximum. Moreover, the specific gravity of Torkula barite was increased from 3.85 to 4.13, 4.078, 4.344 and 4.42 respectively. In line with the API standards, Torkula barite mineral was classified as low, medium and high grade, soft and relatively hard on the basis of utilization as drilling mud additives. These properties qualify the barite mineral to be used in both ordinary and specialized drilling operations. Hence, it is more suitable in the drilling of carbonate formations and reduces the susceptibility of the drill bits to erosion corrosion, sweet corrosion of casing and drill pipes. This is also instrumental in the disintegration of Bentonite.

The carbonate alkalinity and barite mineral acidity are relatively high when compared with the American Petroleum Institute (API) Standards, while the quantity of extractable carbonates is within the API specification of 3000mg/kg. This may result in some health

risk. However, the major change in the pH and corrosion can as well be prevented. The metallic content such as Ca, Pb, Zn, Mg, Cu and Cd minerals in Torkula barite are below the maximum limits set by the American Petroleum Institute (API) and Nigerian Department of Petroleum Resources (DPR). However, the quantity of iron mineral in Torkula barite is higher than the API set limits but low when compare with the DPR set limit.

Torkula barite reflects a higher viscosity at low-shear rate (RPM X 1.703) and a lower viscosity at high-shear rate. Similarly, the filtrate loss within thirty (30) minutes is 7.7 ml. Moreover, it can sufficiently conserve the liquid content of the drilling mud due to its ability to form a good blend with other additives. The apparent and plastic viscosities of the drilling fluid consisting of Torkula barite are higher than the API and DPR minimum values. However, higher amount of Torkula barite is required to raise the yield point and sustain emulsion gel strength of a drilling mud. This is traceable to the increased metallic ionization, high rate of Ca and Mg dissociation required to improve the mud rheology, moderate mud lubricity, coefficient of friction, emulsion and electrical stability.

Hence, increasing the addition of Torkula barite as weighting agents without control, in water-based drilling mud introduces an amorphous character and reduces the reactivity of the dissolved salts responsible for mud rheology.

## **5.2 Recommendations**

The upgrading processes which include transportation and storage ought to be revisited in the future work. This will reduce the amount of sandstones in Torkula barite mineral. Moreover, on-the site beneficiation processes should be monitored and extended to the tailings found along the water ways to improve on the quality of assay and increase the recovered water soluble salts responsible for the mud rheology. However, there is a need for a laboratory-scale beneficiation process to reduce the quantity of the heavy metals and upgrade the quantity of barium sulphate in Torkula barite. Similarly, the beneficiation of the coloured samples and others containing gangue minerals, will improve the quality and increase the specific gravity of the mineral. The wear rate of the bore in during the drilling operations is largely traceable to the hardness of the barite mineral. This can be sufficiently

corrected through the adoption of a well-designed washing bay and efficient separation mechanisms.

Varying the mixing or void ratio of Torkula barite as weighting agent in a drilling mud improves the quality of drilling operation which is traceable to the mud rheology. This is desirable and appropriate where minimum pressure losses and high-shear conditions inside the narrow bore of the drill string, and the large annulus are most demanded. However, Torkula barite ought to be beneficiated to remove the iron content. This will be done through appropriate indigenous enrichment strategies. Moreover, the locally processed kaolinite mineral can serve as a potential substitute to the imported bentonite.

### **5.3 Future Works**

The enrichment and upgrading processes adopted in this work have significantly reduced the silica content, percentage of the heavy metals and water soluble salts. However, the proportion of barium sulphate and water soluble salts required in a drilling mud are below the American Petroleum Institute (API), Department of Petroleum Resources (DPR), Certified Quality and Environmental System Standards (ISO 9001 and 14001) respectively. This is due to the deficiency of relevant techniques in the adopted or indigenous processing strategies. The requirement for drilling and cementing operations has not been met by the beneficiated Torkula barite despite the on-site separation mechanism. This is due to the nature of the associated gangue minerals, the grade of the ore and their liberation sizes. To achieve the physico-chemical and rheological requirements for barite as a weighting agent in drilling mud, these laboratory techniques are to be examined and evaluated for a best balance of quality, purity, materials properties and cost.

➤ Introduction and examination of major physical separation techniques on Torkula barite while evaluating the performance of each stage prior to other beneficiation strategies. These physical techniques include crushing in addition to the application of explosives to reduce the liberation sizes of the gangue minerals, screening, log washing, jigging, heavy medial separation, tabling and spiral concentration, magnetic separation and selective floatation.

- Conditioning of the separation media at varying pH to achieve a condition with a balance of purity and performance in service.
- Heavy media separation and jigging combined with gravity concentration for coarsely liberated ore.
- The use of Zeta Potential measurement with varying cationic and anionic collectors and modifiers for selective floatation.

The analyses are to be done to achieve the following in-line with the API, DPR, ISO 9001 and ISO 14001 specifications:

- Attainment of product quality within 95 and 98 % Barium Sulphate
- Upgrading of the specific gravity of Torkula barite from 4.13 to 4.21, 4.42 and 4.5 SG
- Reduction of heavy metals of lead, iron, tungsten, strontium and cadmium to 0.5% maximum.
- Upgrading of the water soluble salts of sodium, calcium, titanium and potassium to 2% minimum

## Appendices

Table of Results 1

S/N	Rheological Properties	Drilling Mud(1)	Drilling Mud(2)	Drilling Mud(3)
1.	600 RPM	59.0	63.5	22
2.	300 RPM	41.5	33.5	15
3.	200 RPM	33.0	26.0	12
4.	100 RPM	23.0	17.5	9
5.	60 RPM	18.0	13.0	7
6.	30 RPM	13.0	9.0	5
7.	6 RPM	5.5	4.0	3
8.	Gel strength @ 10 seconds	3.0	2.5	1.5
9.	Gel strength @ 10 minutes	3.5	3.0	2.0
10.	Mud weight (ppg)	9.95	8.65	8.5
11.	pH	10.65	11.76	11.97
12.	Plastic Viscosity (PV)	17.50	30.00	7.00
13.	Yield Point (YP)	24.00	3.5	8.00
14.	Apparent Viscosity (AV)	29.50	31.75	11.00

Calculated Results for Formulation 1

S/N	Rotational Speed (RPM)	Shear rate (RPM X 1.703)	Dial Reading	Shear Stress (Dial reading X 5.11)	Viscosity (cP)
1.	600	1021.8	59.0	301.5	0.295
2.	300	510.9	41.5	212.1	0.415
3.	200	340.6	33.0	168.6	0.495
4.	100	170.3	23.0	117.5	0.690
5.	60	102.2	18.0	92.0	0.900
6.	30	51.1	13.0	66.4	1.299
7.	6	10.2	5.5	28.1	2.755

Calculated Result for Formulation 2

S/N	Rotational Speed (RPM)	Shear rate (RPM X 1.703)	Dial Reading	Shear Stress (Dial reading X 5.11)	Viscosity (cP)
1.	600	1021.8	63.5	324.5	0.318
2.	300	510.9	33.5	171.2	0.335
3.	200	340.6	26.0	132.9	0.390
4.	100	170.3	17.5	89.4	0.525
5.	60	102.2	13.0	66.4	0.650
6.	30	51.1	9.0	46.0	0.900
7.	6	10.2	4.0	20.4	2.004

Calculated Result for Formulation 3

S/N	Rotational Speed (RPM)	Shear rate (RPM X 1.703)	Dial Reading	Shear Stress (Dial reading X 5.11)	Viscosity (cP)
1.	600	1021.8	22	112.42	0.110
2.	300	510.9	15	76.65	0.150
3.	200	340.6	12	61.32	0.180
4.	100	170.3	9	45.99	0.270
5.	60	102.2	7	35.77	0.350
6.	30	51.1	5	25.55	0.500
7.	6	10.2	3	15.33	1.502

**Formulation #1**

Plastic viscosity (PV), centipoise:

600 RPM – 300 RPM

59.0 – 41.5 = 17.5 centipoise

Yield point (YP),  $lb/100 ft^2$

300 RPM – PV

41.5 – 17.5 = 24  $lb/100 ft^2$

Apparent viscosity (AV), centipoise:

$$\frac{600 \text{ RPM}}{2}$$

$$\frac{59.0}{2} = 29.5 \text{ Centipoise}$$

Gel strength for 10 seconds: The maximum deflection after 10 seconds:  $4.0 - 1.0 = 2.5 lb/100 ft^2$

Gel strength for 10 minutes: The maximum deflection after 10 minutes:  $3.5 - 0.5 = 3 lb/100 ft^2$

Specific gravity of the drilling mud:

$$\frac{\text{Mud weight}}{8.33}$$

$$\frac{9.95}{8.33} = 1.194$$

## Formulation #2

Plastic viscosity (PV), centipoise:

$$600 \text{ RPM} - 300 \text{ RPM}$$

$$63.5 - 33.5 = 30 \text{ centipoise}$$

Yield point (YP),  $lb/100 \text{ ft}^2$

$$300 \text{ RPM} - \text{PV}$$

$$33.5 - 30 = 3.5 \text{ lb}/100 \text{ ft}^2$$

Apparent viscosity (AV), centipoise:

$$\frac{600 \text{ RPM}}{2}$$

$$\frac{63.5}{2} = 31.75 \text{ centipoise}$$

Gel strength for 10 seconds: The maximum deflection after 10 seconds:  $3.5 - 1 = 2.5 \text{ lb}/100 \text{ ft}^2$

Gel strength for 10 minutes: The maximum deflection after 10 minutes:  $3.5 - 0.5 = 3 \text{ lb}/100 \text{ ft}^2$

Specific gravity of the drilling mud:

$$\frac{\text{Mud weight}}{8.33}$$

$$\frac{8.65}{8.33} = 1.038$$

## Formulation #3

Plastic viscosity (PV), centipoise:

$$600 \text{ RPM} - 300 \text{ RPM}$$

$$22 - 15 = 7 \text{ centipoise}$$

Yield point (YP),  $lb/100 \text{ ft}^2$

$$300 \text{ RPM} - \text{PV}$$

$$15 - 7 = 8 \text{ lb}/100 \text{ ft}^2$$

Apparent viscosity (AV), centipoise:

$$\frac{600 \text{ RPM}}{2}$$

$$\frac{22}{2} = 11 \text{ centipoise}$$

Gel strength for 10 seconds: The maximum deflection after 10 seconds:  $2.5 - 1 = 1.5 \text{ lb}/100 \text{ ft}^2$

Gel strength for 10 minutes: The maximum deflection after 10 minutes:  $2.5 - 0.5 = 2.0 \text{ lb}/100 \text{ ft}^2$

Specific gravity of the drilling mud:

$$\frac{\text{Mud weight}}{8.33}$$

$$\frac{8.50}{8.33} = 1.020$$

### Moisture Content

Initial weighing: Weight of the empty crucible + Weight of the raw sample

A:  $24.7004 \pm 0.002 \text{ g} + 5 \text{ g} = 29.7004 \text{ g}$

B:  $26.0132 \pm 0.002 \text{ g} + 10 \text{ g} = 36.0132 \text{ g}$

C:  $25.8641 \pm 0.001 \text{ g} + 15 \text{ g} = 40.8641 \text{ g}$

First Weighing

A:  $29.6759 \pm 0.002 \text{ g}$

B:  $35.9627 \pm 0.001 \text{ g}$

C:  $40.7904$

Second Weighing

A:  $29.6769 \text{ g}$

B: 35.9654 g

C: 40.7935 g

Third Weighing

A: 29.6795 g

B: 35.9691 g

C: 40.7983 g

Hence, the moisture content of the barite samples were calculated thus in percentage as

$$\text{For 5 g: } \frac{29.7004 - 29.6795}{29.7004} \times 100 \% = 0.0704 \%$$

$$\text{For 10 g: } \frac{36.0132 - 35.9691}{36.0132} \times 100 \% = 0.1225 \%$$

$$\text{For 15 g: } \frac{40.8641 - 40.7983}{40.8641} \times 100 \% = 0.161 \%$$

### Specific Gravity:

Sample A: Grade 2 White barite

$$\text{SG: } \frac{\text{Density of the sample in water}}{\text{Density of water}} = \frac{4.308 \frac{\text{g}}{\text{cm}^3}}{1.00 \frac{\text{g}}{\text{cm}^3}} = 4.308$$

Sample B: Coloured barite

$$\text{SG: } \frac{\text{Density of the sample in water}}{\text{Density of water}} = \frac{3.85 \frac{\text{g}}{\text{cm}^3}}{1.00 \frac{\text{g}}{\text{cm}^3}} = 3.85$$

Sample C: Grade 1 White barite

$$\text{SG: } \frac{\text{Density of the sample in water}}{\text{Density of water}} = \frac{4.42 \frac{\text{g}}{\text{cm}^3}}{1.00 \frac{\text{g}}{\text{cm}^3}} = 4.42$$

Sample D: Grade 1 White barite

$$\text{SG: } \frac{\text{Density of the sample in water}}{\text{Density of water}} = \frac{4.344 \frac{\text{g}}{\text{cm}^3}}{1.00 \frac{\text{g}}{\text{cm}^3}} = 4.344$$

Sample E: White barite with gangue minerals

$$\text{SG: } \frac{\text{Density of the sample in water}}{\text{Density of water}} = \frac{4.30 \frac{\text{g}}{\text{cm}^3}}{1.00 \frac{\text{g}}{\text{cm}^3}} = 4.30$$

Sample F: White barite with gangue minerals

$$\text{SG: } \frac{\text{Density of the sample in water}}{\text{Density of water}} = \frac{4.078 \frac{\text{g}}{\text{cm}^3}}{1.00 \frac{\text{g}}{\text{cm}^3}} = 4.078$$

**Samples' Hardness:**

S/N	Description	Scratch Mechanism			Remark
		Finger	Blade (Knife edges)	Hardness	
1.	Grade 2 White barite	No	SOFT	(4.0)	Relatively rough
2.	Coloured barite	No	HARDER	(4.5)	Difficult to cut, rough edges
3.	Grade 1 White barite	No	SOFTER	(3.5)	Smooth surface and edges
4.	Grade 1 White barite	No	SOFTER	(3.5)	Smooth surface and edges
5.	White barite (gangue minerals)	No	HARDER	(5.0)	Difficult to cut, very rough
6.	White barite (gangue minerals)	No	HARDER	(5.0)	Difficult to cut, very rough

**pH of the Barite Samples:**

No of Experiment	pH	Temperature (degrees)
1.	7.936	28.3
2.	7.963	28.1
3.	7.991	28.3
4.	8.016	26.7
5.	8.015	27.0
6.	8.015	26.3

**Retort Test:**

Aim: To determine the amount of solids and liquid in the drilling mud

**Table 1: Composition of the drilling mud**

S/N	Sample	Percentage Volume	
		Solid (%)	Liquid (%)
1	5 g Torkula barite	2.00	98.00
2.	10 g Torkula barite	4.00	96.00
3.	70 g Torkula barite	19.00	81.00

**Alkalinity Test:**

Phenolphthalein Alkalinity of the filtrate:

$$\frac{\text{Volume of H}_2\text{SO}_4 \times \text{Normality} \times 50 \times 1000}{\text{Vol. of sample taken}}$$

$$\frac{3.2 \text{ ml} \times 0.0199 \text{ N} \times 50 \times 1000}{3 \text{ ml}} = 1061 \text{ mg/Lit}$$

Total alkalinity as calcium:

$$\frac{\text{Total volume of H}_2\text{SO}_4 \times \text{Normality} \times 50 \times 1000}{\text{Vol. of sample taken}}$$

$$\frac{5.0 \text{ ml} \times 0.0199 \text{ N} \times 50 \times 1000}{3 \text{ ml}} = 1658 \text{ mg/Lit}$$

**Total Hardness as Calcium:**

Total hardness as calcium (mg/L) for drilling Mud A (10 g barite):

$$400 \times \frac{\text{Vol. u.me of titrating solution}}{\text{Volume of filtrate}}$$

$$400 \times \frac{2.8 \text{ ml}}{3 \text{ ml}} = 373.3 \text{ mg/L}$$

Total hardness as calcium (mg/L) for drilling Mud B (70 g barite):

$$400 \times \frac{\text{Vol. u.me of titrating solution}}{\text{Volume of filtrate}}$$

$$400 \times \frac{1.4 \text{ ml}}{3.9 \text{ ml}} = 143.59 \text{ mg/L}$$

**Ion Counts of Drilling Muds:**

$$\text{Chloride} \left( \frac{\text{mg}}{\text{L}} \right) = \frac{\text{ml Silver Nitrate} \times 1000}{\text{ml Filtrate}}$$

$$= \frac{10.3 \text{ ml} \times 1000}{6 \text{ ml}} = 1716 \text{ mg/L}$$

**Total Calcium Sulphate:**

$$\text{Total Calcium Sulphate} \left( \frac{\text{Kg}}{\text{m}^3} \right) = 6.79 \times V_t$$

$$6.79 \times 0.25 = 1.6675 \frac{\text{kg}}{\text{m}^3} = 0.575 \text{ lb/lbbl}$$

**Total Chloride Content:**

$$\begin{aligned} \text{Chloride Ion Count} &= \frac{\text{ml Silver Nitrate} \times 1,000}{\text{ml Filtrate}} \\ &= \frac{10.3 \text{ ml} \times 1,000}{6 \text{ ml}} = 1716 \text{ mg/lit} \end{aligned}$$

**Methylene Blue Test:**

$$\text{Methylene Blue Capacity: } \frac{\text{Methylene blue (ml)}}{\text{Drilling fluid (ml)}}$$

$$\text{Drilling mud A (10 g barite): } \frac{14 \text{ ml}}{2 \text{ ml}} = 7.0$$

$$\text{Drilling mud b (70 g barite): } \frac{14 \text{ ml}}{2 \text{ ml}} = 7.0$$

$$\text{Methylene Blue Absorption Value (MBA): } \frac{100}{f^*}$$

$$\text{MBA for Drilling mud A} = \frac{100}{8} = 12.5$$

$$\text{MBA for Drilling mud B} = \frac{100}{8} = 12.5$$

**Cation Exchange Capacity (C. E. C.):**

$$\begin{aligned} \text{C. E. C} &= \left[ \frac{100}{f^*} \right] \times \text{Volume of Methylene blue solution injected (V}_{cc}) \\ &\times \text{Normality; in } \frac{\text{meq}}{100 \text{ g}} \end{aligned}$$

$$\begin{aligned} \text{C. E. C for 10 g barite} &= \left[ \frac{100}{10} \right] \times 14 \text{ ml} \times 0.01 \text{N } \frac{\text{meq}}{100 \text{ g}} \\ &= [8] \times 14 \text{ ml} \times 0.01 \text{N } \frac{\text{meq}}{100 \text{ g}} \\ &= 1.12 \frac{\text{meq}}{100 \text{ g}} = 0.112 \frac{\text{meq}}{10 \text{ g}} \end{aligned}$$

$$\begin{aligned} \text{C. E. C for 70 g barite} &= \left[ \frac{100}{70} \right] \times 16 \text{ ml} \times 0.01 \text{N } \frac{\text{meq}}{100 \text{ g}} \\ &= [8] \times 14 \text{ ml} \times 0.01 \text{N } \frac{\text{meq}}{100 \text{ g}} \\ &= 1.12 \frac{\text{meq}}{100 \text{ g}} = 0.112 \frac{\text{meq}}{10 \text{ g}} \end{aligned}$$

**Metallic Content of the Barite Samples:**

S/N	Metals	Concentration (mg/L)/mg/kg			API maximum limit
		Torkula Barite	Imported Barite	Ibe et al, 2016	
1.	Ca	34.0135	415.00	709.00	250
2.	Zn	3.9051	44.60	1.60	140
3.	Mg	8.5726	65.00	64.20	250
4.	Pb	113.8127	193.00	6.00	1000
5.	Cd	0.0008	1.60	1.60	5
6.	Fe	15.6094	400.00	62.20	Zero
7.	Cu	0.3024	1.60	160.00	36

**Filtration Analysis:**

Time (t) (mins)	$\sqrt{t}$ (min)	Cumulative Filtrate Volume (ml)
5.0	2.236	3.0
10.0	3.162	4.2
15.0	3.378	5.2
20.0	4.472	6.0
25.0	5.000	6.9
30.0	5.477	7.7

**Calculation for Lubricity:**

The load or force acting on the torque shaft is given as:

$$\text{Load} = \frac{150 \text{ in} - \text{lb (Torque wrench reading)}}{1.5 \text{ in (Torque shaft level arm)}} = 100 \text{ lb}$$

Correction factor

$$= \frac{\text{Standard Meter Reading for Deionized Water}}{\text{Meter Reading Obtained in Deionized Water Calibration}} = \frac{34}{32.2}$$

$$= 1.056$$

$$\text{Coefficient of friction} = \mu = \frac{\text{Meter reading}}{\text{Load or force}}$$

$$\text{Lubricity Coefficient} = \frac{\text{Meter Reading} \times \text{Correction factor}}{100 \text{ lb}}$$

**Lubricity Test:**

Time (mins)	Torque reading @ 60 RPM	
	10 g barite	70 g barite
5.0	42.0	32.3
10.0	42.9	30.7

**Lubricity Test for barite**

Time (mins)	Torque reading @ 60 RPM	
	5g barite	70 g barite
0.0	0.0	0.0
2.0	49.5	31.1
4.0	46.0	32.1
5.0	43.7	32.3
6.0	42.3	32.1
8.0	39.2	31.2
10.0	37.1	30.7

**Table 3: Mud Lubricity:**

Qty of barite	Torque Reading @ 60RPM, 5 mins	Coefficient of friction	Lubricity
Coefficient			
5 g	43.7	0.437	0.46
10 g	42.0	0.420	0.45
70 g	32.3	0.323	0.34

**Analysis of the Filter Cake:**

Description	Remark
Weight of the filter cake before drying	19.86 g
Thickness of the Filter cake	4/32 inches
Quality of the cake	Firm and Flexible

**Electrical Properties of the Drilling Mud:**

**Electrical Resistivity:**

Composition	Resistivity ( $\Omega m$ )	Temperature (F)
5 g barite	0.15	84.00
10 g barite	0.27	74.00
70 g barite	0.59	72.00

**Electrical Conductivity:**

Composition	Conductivity (Volts)
10 g barite	0.00
70 g barite	0.00

**Mud Weight Evaluation:**

Composition	Cumulative Mud Weight (ppg)
8 g Bentonite only	8.42
5 g barite	8.49
10 g barite	8.57
15 g barite	8.66
20 g barite	8.79
25 g barite	8.86
35 g barite	9.00
40 g barite	9.18
50 g barite	9.39
60 g barite	9.65
70 g barite	9.86
80 g barite	10.00
90 g barite	10.26
100 g barite	10.50

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