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Assessment And Beneficiation Of Obubra Barite For Production Of Weighting Agent In Oil And Gas Well Drilling Mud

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***Abstract*— This study seeks to assess barite ore sample from Iyamitet in Obubra of Cross River State, Nigeria and to determine its suitability for its use as weighting agent in oil and gas well drilling and also produce an improved barite concentrate suitable for this application. The obtained sample was comminuted using a laboratory pulverizer and a ball milling machine. The ore sample was characterized by determining the elemental composition using X-ray Fluorescence (XRF) and physicochemical properties. The physicochemical properties determined included specific gravity, moisture content and oil absorption rate. The XRF analysis indicated that the ore contains a Barium-Sulphur (Ba-S) content of 84.79% with other trace elements such as Titanium, Vanadium, Aluminium, Silicon, Lead and Iron amidst others of 15.21%. The physicochemical properties analysis gave the following results: specific gravity (3.60), moisture content (0.30%) and oil absorption rate (11.03%). The results obtained, when compared to API and ASTM specifications proved that the barite ore is not suitable for application as weighting agent in oil and gas well drilling. The barite sample was subjected to beneficiation using Tabling, Froth flotation and Leaching processes. The combination of these beneficiation processes gave an improved barite concentrate of Ba-S of 90.92% and specific gravity of 4.39 which makes it suitable for use as weighting agent in oil and gas well drilling.**

Keywords*— *Barite, Characterization, X-ray flourescence, As-mined, Comminution, Beneficiation, Physicochemical properties, Drilling mud.

1.0 INTRODUCTION

Nigeria is often described as a country endowed with abundant mineral resources, including the occurrence of over 40 different solid minerals at approximately 450 different locations [1]. Nigeria's solid minerals industry, however, is severely underdeveloped, and for a variety of reasons, minerals that are easily produced domestically are being imported. One of such minerals is barite, a very important raw material for many industrial applications which include its use as pigment for making paints and paper coating, asbestos production, glass production, pharmaceutical industries and more importantly as a weighting ingredient in drilling for crude oil [2].

In Nigeria, barite deposits are found in veins and cavity fills that are supported by a variety of rocks. Principal areas of barite occurrences in Nigeria include; Nassarawa, Plateau, Taraba, Benue, Adamawa, Cross River, Gombe, Ebonyi, and Zamfara [3] as shown in Figure 1. The occurrence of barite in parts of Middle Benue trough was first reported by [4] in his memorandum, Geological Survey of Nigeria Report No. 1266 estimated reserve to be about 400,800 tons to 18 m depth. Nigerian Geological Survey Agency carried out an assessment of the barite located at Cross Rivers, Benue, Nassarawa, Ebonyi, Plateau and Taraba States, and the inferred deposit of barite for potential mining was estimated to be approximately, 21 million tons [5].

Figure 1: Map of Nigeria indicating states with barite deposit [6].

Barium sulphate occurs naturally as barite, sometimes referred to as "heavy spar" with a chemical formula of $BaSO_4$. It is an important industrial mineral characterized by its high specific gravity and chemical inertness which make it an ideal mineral for many applications. Barite ores of different grades vary from one location to the other and within deposits. It is also a common gangue in other ore deposits such as lead, fluorite, zinc, silicon, gold and rare earth minerals.

About 70% of the barite that is mined is used by the petroleum sector as a weighting agent in drilling mud for oil and gas well drilling. Due to its high specific gravity, it increases the hydrostatic pressure of the drilling mud allowing it to compensate for high-pressure zones experienced during drilling [7]. Also, it is preferred to all other weighing agents because of its unique physical and chemical properties.

The non-magnetic properties make it more useful for this purpose as it does not interfere with the magnetic measurements of the borehole during the whole drilling process [8]. Moreover, high inertness and exceptionally low oil absorption of barite make it a near-perfect weighting agent coupled with its high specific gravity. The softness of the mineral also prevents it from damaging drilling tools during drilling and enables it to serve as a lubricant [9].

Drilling mud, also called drilling fluid is very crucial to the success and safety of oil extraction in petroleum engineering. Drilling mud is a heavy viscous fluid mixture that is used in oil and gas drilling operations to carry rock cuttings to the surface and also to lubricate and cool the drill bit. The drilling mud, by hydrostatic pressure, also helps prevent the collapse of unstable strata into the borehole and the intrusion of water from water-bearing strata that may be encountered. Drilling deeper, longer and more challenging wells have been made possible by improvements in drilling technologies, including more efficient and effective drilling muds. Drilling muds are added to the wellbore to facilitate the drilling process by suspending cuttings, controlling pressure, stabilizing exposed rock, providing buoyancy, cooling and lubricating.

During drilling, cuttings are obviously created, but they do not usually pose a problem until drilling stops because a drill bit requires replacement or stoppage when a problem arises. When this happens, and drilling fluids are not used, the cuttings then fill the hole again. Drilling fluids are used as a suspension tool to keep this from happening. The viscosity of the drilling fluid increases when movement decreases, allowing the fluid to have a liquid consistency when drilling and then turn into a more solid substance when drilling has stopped [8]. Cuttings are then suspended in the well until the drill is again inserted.

Generally, drilling muds are formulated to deliver a multitude of benefits, including: protection of the expensive drill shaft and bits, lubrication, corrosion control and cooling. Special additives are used to ensure that the drilling fluid is not absorbed by the rock formation in the well and that the pores of the rock formation are not clogged. Weighting agents are added to the drilling fluids to increase its density and, therefore, its pressure on the walls of the well.

For barium use in industries, it must meet some specifications as specified by The American Petroleum Institute (API) and American Society for Testing Materials (ASTM) [10]. The barite must meet a purity level of 90% minimum, specific gravity of 4.2, moisture content of less than 1% and oil absorption rate of between 10-12%. The use of barite for industrial applications requires proper characterization and beneficiation of the ore. Characterization is fundamental in mineral processing, it is important for making a correct assessment of reserves and resources and also for exploring novel processing strategies[11]. The characterization of solid is most important since the process selection is closely linked to the nature of minerals/ores resulting from geological formation. The mineralogical characterization may involve identification of minerals (crystal structure and chemistry), minerals fabric/association of minerals, quantification of phases, elemental associations and occurrence of minor/trace minerals/elements.

One of the major characterization methods applied is the X-ray florescence (XRF) which is used to determine the elemental composition of the mineral with the use of a spectrometer. The analysis of major and trace elements in geological materials by XRF is made possible by the behavior of atoms when they interact with X-radiation. An XRF spectrometer works in a way that if a sample is illuminated by an intense X-ray beam, known as the incident beam, some of the energy are scattered, but some are also absorbed within the sample in a manner that depends on its chemistry.

Since it is difficult to see pure barite in its crude form, most crude barite requires some upgrading to maximum purity in terms of content and specific gravity. For barite to meet drilling mud additive requirement, simple beneficiation methods can be used. The quality of the Nigerian barites is moderate to high. It is often associated with fluorite, calcite, dolomite, quartz, etc. The major impurities are quartz, iron oxide (goethite), carbonates of iron, calcium and magnesium. These impurities tend to increase the ore volume, suppress and reduce the specific gravity of the unprocessed barites to about 2.0 – 4.0 [12].

Physical processing techniques like jigging, grinding, washing, and crushing are used to extract gangue minerals and generate products with the proper size. The goal of mineral beneficiation is to separate the components of minerals using the least amount of energy possible. This can often be achieved by separating the minerals using physical processes as opposed to chemical ones, even though in most cases, chemical treatment methods will need to be used for the final stage of elemental separation. Froth flotation, heavy media separation, tabling, magnetic separation may be used to separate barite from those finely inter-grown gangue minerals. Leaching may also be used to remove iron oxide stains from white barite especially for coatings markets [13].

2.0. MATERIALS AND METHODS

2.1 Materials

3000g of the barite sample used for this research was obtained from Iyamitet in Obubra Local Government area of Cross River State, Nigeria. Other materials used included: distilled water, engine oil, pine oil, oleic acid, hydrogen chloride acid (HCl), sodium hydroxide (NaOH), Whatman filter paper of 24.0 cm. The equipment used during the course of this research work included: labouratory pulverizer, ball milling machine, set of sieves, digital weighing machine, laboratory oven, pH meter, hot plate stirrer with magnetic bar, X-ray spectrometer, shaking table, flotation cell, pycnometer bottles and conical flask.

2.2 Methods

The sample was first subjected to comminution using a labouratory pulverizer and a ball milling machine. Some of the comminuted sample were subjected to both chemical and physical characterization. The comminuted sample were further subjected to beneficiation processes. This study utilized the combination of two gravity separation techniques and one chemical separation technique. The gravity separation technique used are tabling and froth flotation. Leaching chemical separation was applied to the products of froth flotation analysis. Chemical composition analysis using X-ray Fluorescence was carried out on the samples before and after beneficiation. This helped to determine the elemental composition of the ore sample. Physicochemical properties analysis was also examined on the samples which included: specific gravity, moisture content and oil absorption rate. These parameters where compared with American Petroleum Institute (API) specifications for barite application as weighting agent in Oil and gas well drilling mud.

2.2.1 Elemental Characterization

The elemental composition of the barite ore samples were determined using Skyray EDX 3600B Energy Dispersed X-ray Fluorescence spectrometer. 20 g sample pellet was loaded in the sample chamber of the spectrometer at voltage (40 KV maximum) and a current (350 μA maximum) was applied to produce the X-rays to excite the sample for 12 minutes at 20°C ± 0.2°C. The spectra from the sample were analyzed to determine the concentration of the elements in the samples. This process was also performed after the beneficiation of the barite sample. These tests were carried out at the Engineering Materials Development Institute (EMDI), Akure, Ondo State.

2.2.2 Physicochemical Characterization

The physicochemical characterization was determined by assessing the: specific gravity, moisture content and oil absorption rate. The obtained results were compared with the standard industrial application requirements. The barite ore samples were also beneficiated using a combination of two gravity separation techniques and one chemical separation technique. The gravity separation techniques employed were tabling and froth flotation. While chemical separation technique employed was leaching. The physicochemical characterization was also performed on the beneficiated concentrates.

2.2.3 Specific Gravity

The specific gravity of the ore sample was measured by weighing an empty pycnometer bottle and the weight recorded. The sample was filled into the pycnometer bottle to about one-third and re-weighed. The remaining space inside the bottle was filled with distilled water and weighed. The content of the bottle was emptied and refilled with distilled water and their weights recorded.

Equation (1) was used to calculate the specific gravity of the barite grains.

$$\text{Specific gravity of barite grains} = \frac{B-A}{(D-A)-(C-B)} \quad (1)$$

Where:

A - weight of empty pycnometer,

B - weight of pycnometer + mineral grain.

C - weight of pycnometer + mineral gain + liquid,

D - weight of pycnometer + liquid

2.2.4 Moisture Content Analysis

Moisture content analysis was aimed at determining the level of moisture in the barite sample. The analysis utilized a direct measurement approach where water content was determined by removing moisture and then by measuring weight loss. The direct measurement approach was used to remove all the moisture in the barite sample. The steps involved in this analysis included the weighing and recording of an empty container. 10 g barite sample was placed in the empty container and weighed. The sample + container was dried in an oven at 100^0C for 3 hours. After 3 hours, the barite + container was removed, weighed and recorded. Mathematically, moisture content analysis was calculated using the formula in equation (2);

Moisture content (%) = $\frac{W2-W3}{W2-W1}$ x 100 (2)

Where:

W1 - Weight of empty container;

W2 - Weight of container and sample before drying; and

W3 - Weight of container and sample after drying.

2.2.5 Oil Absorption Test

The 48 hours oil soak test was performed to determine the oil absorption behavior of the barite sample and the effect of the absorbed oil on its dimensions. After drying the specimen in open air for 48 hours, its weight was measured and recorded. Subsequently, the weight was measured and recorded after 48 hours of submersion in engine oil, SEA 20/50 at room temperature 27°C. The specimen was weighed and recorded after the excess oil had drained off. Equation (3) was used to calculate the percentage of water and oil absorption [1]:

$$Liquid\ Absorption\ (\%) = \frac{Wi-Wo}{Wo}\ x\ 100\% \quad (3)$$

Where:

Wi - weight after immersion and

Wo - weight before immersion

2.2.6 Tabling Analysis

The process of separating barite from its ore commenced with the tabling operation using a MTS laboratory shaking table. 2000 g of the ore sample with particle sizes – 180 to + 63μm was used to produce the pulp ores of 20% solid ratio for the tabling experiment. The pulps were used as shaking table feed test at table frequency 5 Hz and at feed rate of 500 g/min. The concentrates, the middlings and the tailings were collected into the collecting pans and were allowed to settle and dewatered. The outputs were dried in a Techmel laboratory oven (Model: TT-9053) at a temperature of 110°C for 5 hours and their weights measured and recorded. The outputs from the tabling analysis were subjected to specific gravity analysis, moisture content analysis and oil absorption test and the results recorded. This analysis was carried out at the Mineral Processing Laboratory, Federal University of Technology, Akure, Ondo state.

2.2.7 Froth Flotation Analysis

The concentrate from tabling operation was subjected to froth flotation analysis by mixing 200 g with distilled water to produce a pulp form of 20% solid ratio. These froth flotation analysis was performed in a Denver flotation cell (Serial no: BPAE22449) operating at a measured impeller speed of 18 rev/min to obtain proper suspension of solid particles within the pulp. Pine Oil as frother, corn starch as depressant, oleic acid as collector, HCl and NaOH as pH regulator were the reagents used for the froth flotation experiment.

Four drops of the oleic acid were added to the pulp and conditioned for about 5mins. Corn starch acting as a depressant was added and conditioning in vacuum were carried out for 10mins. The pine oil was added 2mins before the expiration of the conditioning time while allowing air to be introduced at a reasonable rate. The froth was skimmed off the flotation cell into the collecting pan until there was no visible froth.

The Froth and depressed minerals were the products from froth flotation operations. The froth contains the desired mineral while the depressed mineral contains both the impurities and some of the desired minerals. These procedure were repeated five times for varying pH values of 4, 6, 8, 10 and 12 to increase froths. The froths were allowed to settle, dewatered by filtration with Whatman filter papers, washed and dried in the laboratory oven at 110°C for 2 hours. The weights of the concentrate from the froth flotation experiments were measured, the specific gravity and other physical properties were analyzed. These tests were carried out at the Mineral Processing Lab, Federal University of Technology, Akure.

2.2.8 Leaching

The concentrate from the froth flotation analysis was subjected to chemical leaching to get a more pure barite concentrate. 50 g of each of the concentrate was poured into a conical flask mixed with 200 ml of 0.2M HCl using hotplate stirrer (Model H4000-H3) and magnetic bar for 60 mins at 27°C. The mixtures were gently washed and weights recorded. The products were filtered and dried in a laboratory oven. These concentrate was collected for chemical composition analysis and physical properties test.

3.0 RESULTS AND DISCUSSION

3.1 Results

3.1.1 Elemental Composition Analysis

The result of XRF elemental composition of the barite samples before and after beneficiation are presented in Table 1. From the table, it was observed that the sample before beneficiation (Sample A1) contains 3.51% Vanadium, 1.54% Aluminium, 2.56% Silicon and 0.16% Iron amidst others. The table also shows that after beneficiation, the barite (Sample A2) contains 53.59% Barium, 37.33% Sulphur, 3.77% Titanium, 3.81% Vanadium, 2.29% Aluminium, 50.14% Barium, 35.88% Sulphur, 3.75% Titanium,

2.11% Silicon and 0.23% Iron amidst others. Figure 2 & 3 shows the spectra pattern of the ore sample at various energy level before and after beneficiation respectively.

Table 1: XRF elemental composition of barite sample

Elements	Mg	Al	Si	P	S	Ba	Ca	Ti	V	Mn	Fe	Ni	Cu
Sample A1 (%)	0.05	1.54	2.56	0.70	35.88	50.14	0.18	3.75	3.51	0.18	0.16	0.05	0.09
Sample A2 (%)	0.00	2.29	2.21	0.56	37.33	53.59	0.11	3.77	3.81	0.18	0.23	0.02	0.03

Elements	Zn	As	Pb	W	Au	Nb	Mo	Sn	Sb
Sample A1 (%)	0.07	0.08	0.12	0.07	0.00	0.00	0.17	0.44	0.44
Sample A2 (%)	0.06	0.02	0.10	0.00	0.00	0.00	0.18	0.39	0.34

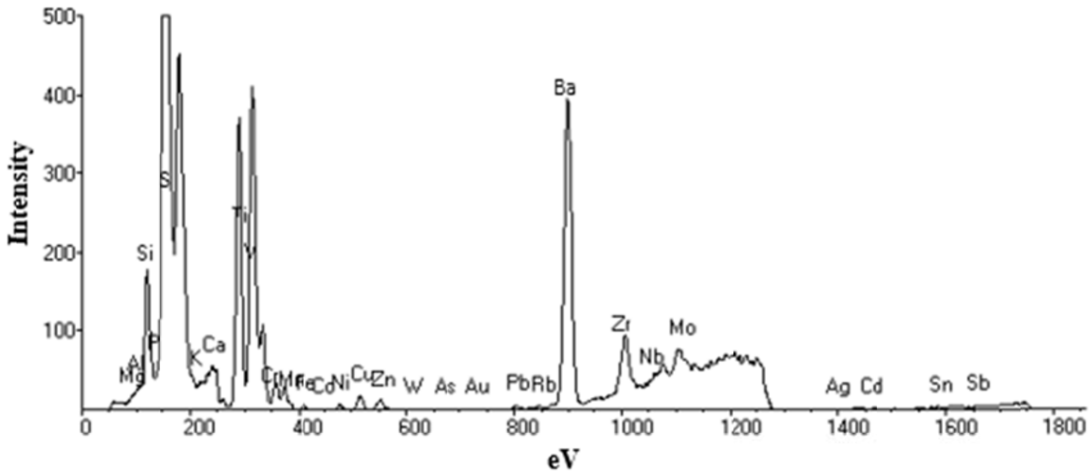

Figure 2: XRF elemental composition pattern for As-mined Obubra barite sample (Sample A1)

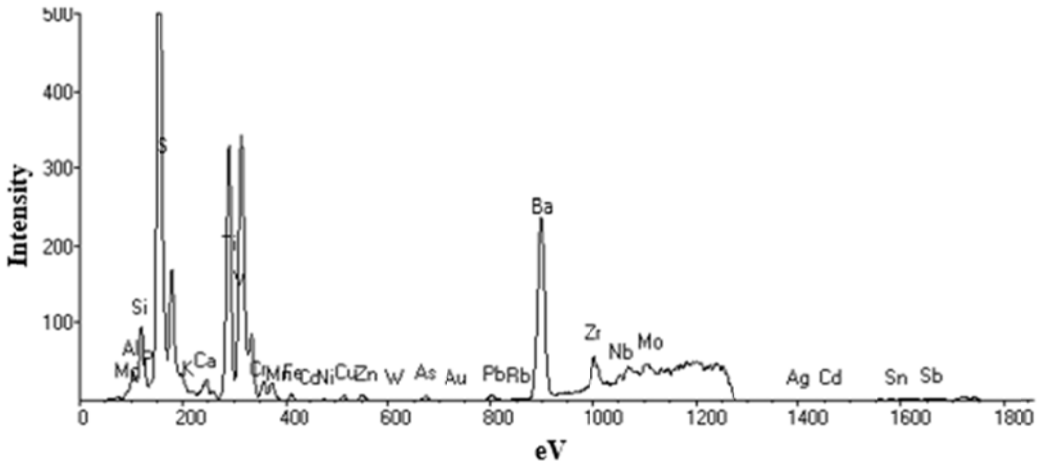

Figure 3: XRF elemental composition pattern for Obubra barite sample after beneficiation (Sample A2).

3.1.2 Physicochemical Analysis

Tables 3, 4 and 5 show the result of specific gravity test, moisture content analysis and oil absorption test performed on the as-mined barite sample before beneficiation.

Table 2: Specific gravity test

		Experiment 1	Experiment 2	Experiment 3
Wt. of pycnometer (g)	A	52.00	38.10	42.00
Wt. of pycnometer + mineral (g)	B	86.00	76.01	76.5
Wt. pycnometer + mineral + liquid (g)	C	132.00	94.00	122.01
Wt. pycnometer + liquid (g)	D	106.10	68.00	96.70
Specific gravity	$\frac{B-A}{(D-A)-(C-B)}$	3.78	38.0	3.76
Ave Specific gravity			3.78	

Table 3: Moisture content analysis

		Experiment 1	Experiment 2	Experiment 3
Wt. of container	W1	43.70	43.70	43.70
Wt. of container + sample before dryness	W2	53.85	51.30	53.82
Wt. of container + sample after dryness	W3	53.55	51.10	53.54
Moisture content (%)	$\frac{W2-W3}{W2-W1}$ x 100	0.31	0.31	0.31
Ave. Moisture content (%)			0.31	

Table 4: Oil absorption test @ 48hrs

		Experiment 1	Experiment 2	Experiment 3
Wt. after immersion	Wi	34.88	26.50	17.39
Wt. before immersion	Wo	34.84	26.20	17.37
Oil absorption (%)	$\frac{Wi-Wo}{Wo}$ x 100	11.48	11.45	11.51

Ave. oil absorption (%)	11.48

3.1.3 Tabling Analysis

Table 5 shows the result obtained from gravity separation concentrates of the barite sample using tabling operation. The tabling operation gave three outputs: concentrates, middlings and tailings as shown in the Table 5.

Table 5: Result of gravity concentration using tabling method

Net weight(g)	Output	Weight (g)	Recovery(%)	SG
	Concentrate	1502.0	75.1	4.02
2000	Middlings	452.0	22.6	2.83
	Tailings	6.0	0.3	1.81

3.1.4 Froth Flotation Analysis

Table 6 shows the results obtained from the froth flotation experiment performed on the concentrate obtained from tabling operation. The froth flotation experiment gave two products: froth and depressed minerals at varying pH of 4, 6, 8, 10 and 12 respectively. The recovery and SG gave varying value of different pH as shown in the Tablet.

Table 6 Result of concentration of Obubra and Yala barite ore using froth flotation method.

pH level	Net weight	Weight of froth	SG	Weight of Depressed	SG
pH 4	200	164.3 (82.2%)	4.07	20.9(10.5%)	3.65
pH 6	200	166.6 (83.3%)	4.10	16.3(8.2%)	3.36
pH 8	200	188.7 (94.4%)	4.14	6.7(3.3%)	3.30
pH 10	200	182.0 (91.0%)	4.11	12.9(6.3%)	3.33
pH 12	200	169.3 (84.5%)	4.09	16.9(8.5%)	3.51

3.1.5 Leaching

The leaching of pH 8 froth concentrate gave recovery rate of 85.9%, SG of 4.39, moisture content of 0.28% and oil absorption rate of 11.42%.

Table 7 shows the result of specific gravity test, moisture content analysis and oil absorption test performed on the barite sample before and after each beneficiation experiment.

Table 7: Result of specific gravity (SG), moisture content (MC) and oil absorption (OA) rate of the barite samples.

Sample condition			S G	M C(%)	O A(%)
As mined			3.78	0.31	11.48
After tabling		Concentrate	4.02	0.30	11.32
		Middling	2.83	0.31	11.51
After flotation	pH 4	Froth	4.07	0.28	11.39
		Depressed	3.31	-	-
	pH 6	Froth	4.10	0.27	11.42
		Depressed	3.28	-	-

	pH 8	Froth	4.14	0.28	11.37
		Depressed	3.21	-	-
	pH 10	Froth	4.11	0.29	11.43
		Depressed	3.27	-	-
	pH 12	Froth	4.09	0.31	11.37
		Depressed	3.33	-	-
After leaching		HCL	4.39	0.28	11.42

3.2 Discussion

From the result of the XRF elemental composition of the barite samples as shown in table 7, it was observed that the as-mined barite contains: barium

50.14% and sulphur 35.88% to give Barium and sulphur (Ba-S) content 86.02% with 18 other trace elements which amounts to 13.98%. The trace elements include: 3.75% Titanium, 3.51% Vanadium, 1.54% Aluminium, 2.56% Silicon, 0.12% Lead, 0.07% Zinc and 0.16% Iron amidst others. When the Ba-S content was compared to API and ASTM specifications for drilling mud additive use, it was observed that the Obubra Ba-s content was not suitable for drilling mud which requires minimum of 90%. The as-mine barite was also observed to have specific gravity of 3.78 which does not also meet the API and ASTM specification of minimum of 4.2. It was also observed that the moisture content and the oil absorption rate meet the API and ASTM specification.

The test gave a moisture content of 0.31% which fall between the requirement of 1% maximum. The oil absorption test gave 11.48% which meet application requirement of 10-12%. The Ba-S content improved after beneficiation with the combination of tabling, froth flotation and leaching processes to 90.92% which makes it suitable for drilling mud additive which requires minimum of 90% Ba-S content. It was also observed that the specific gravitiy increased after application of the various beneficiation process. The specific gravity increased from 3.78 to 4.02 after tabling and from 4.02 to 4.14 after flotation at pH 8 and from 4.14 to 4.39 after leaching as seen as shown in figure 4.

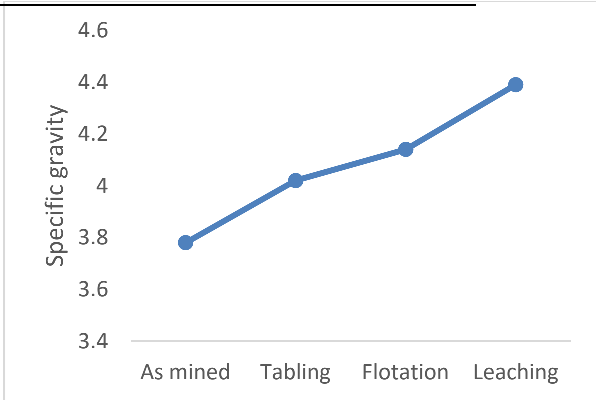

Figure 4: Specific gravity of Obubra barite beneficiation

Calcium, a soluble alkaline element of 0.18% was observed present which meets the ASTM application specification of 2.0% maximum. It was observed that during flotation experiment, as the pH of the flotation process increased, the amount of froth obtained increased while the depressed minerals decreased but the froths started decreasing after pH 8 while the depressed increased. The same can also be observed of the specific gravity. Flotation at pH 8 gave the best specific gravity and recovery yield. It was also observed that the beneficiation processes had no much impacts on the values of the moisture content and oil absorption rate.

4.0 CONCLUSION AND RECOMMENDATION

In conclusion, the barite ore sample was observed to contain Barium-Sulphur content of 86.02%. This value is low for its application as weighting agent in drilling mud for oil and gas well drilling which requires minimum of 90%. The specific gravity was also observed to be low for this application. The moisture content and oil absorption rate fell in between the required industrial application specification. The ore sample was upgraded by subjecting it to combination of three beneficiation processes. Beneficiation with the combination of Tabling, Froth flotation and Leaching processes gave an improved barite ore of Barium-Sulphur of 90.92% and specific gravity of 4.39 which makes suitable for use as weighting agent in oil and gas well drilling mud. More beneficiation techniques can be combined to get an even more improved barite concentrate.

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