Characterization Of Obubra Barite Ore For Industrial Applications

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***Abstract*— This study aimed to characterize barite ore sample from Iyamitet in Obubra of Cross River State, Nigeria and to determine its suitability for various industrial applications. A 3000g of the ore sample was obtained from the location, the sample was prepared into powder form 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 Obubra ore contains a Barium-Sulphur (Ba-S) content of 86.02% with other trace elements such as Titanium, Vanadium, Aluminium, Silicon, Lead and Iron amidst others of 13.98%. The ore was also observed to contain small percentage of heavy metals and soluble alkaline earth metals. The physicochemical properties analysis gave the following results: specific gravity (3.78), moisture content (0.31%) and oil absorption rate (11.48%). The results obtained, when compared to general industrial applications standards proved that the barite ore is not suitable for industrial applications and will require some simple beneficiation processes to upgrade it for industrial use.**

Keywords*— *Barite, Characterization, X-ray flourescence, Comminution, Beneficiation, Physicochemical properties.

I. INTRODUCTION

Barite is an important industrial mineral consisting of barium and sulphur, it is composed of barium sulphate ($BaSO_4$) [1,2]. It has been reported to be available in Nigeria but has either been left unexploited or has experienced very little exploitation [3]. Obubra ore deposit is one of many barite deposits that can be found in Nigeria. Nigeria currently holds the fourth largest deposit of barite in Africa with an estimated reserve of over 21 million metric tons. Ironically, the nation is not anywhere in the list of globally recognized producers of barite for drilling activities and other industrial applications [4].

Barite is generally easy to identify. It is one of just a few nonmetallic minerals with a specific gravity greater than four. When we combine the specific gravity with its low Mohs hardness and its three directions of right-angle cleavage; with just these three observations, barites can be reliably identified. Pure barite has a specific gravity (SG) of 4.5 g/cm^3, moisture content of below 1%, oil absorption rate of between 9 – 12% and hardness value between 3.0 to 3.5 Mohs. It is also chemically inert and is insoluble in water which makes it an ideal mineral for many industrial applications [3].

Barite is utilized primarily because of its high specific gravity in addition to its chemical and physical inertness, relative softness and very low solubility [5-8]. A major amount (69-77%) of the total barite found is used as weighting agent for drilling muds in oil and gas exploration to suppress high formation pressures and prevent blowouts. The non-magnetic properties of this material makes it more useful for this purpose as it does not interfere with the magnetic measurements of the borehole during the whole process [9]. Moreover, high inertness and exceptionally low oil absorption of barite make it a near-perfect weighting agent coupled with its high specific gravity.

Barite also has other industrial applications which include its use in the pharmaceutical industry. It is used in the manufacturing of barium meals. Barium meals are widely used to detect abnormalities in the gastrointestinal tract and the digestive system by using CT-scan and other radiography techniques. Barium meal highlights the inner lining of soft tissues in the intestine and the stomach, making them visible in scans and radiographs [10].

Barite is used in added-value applications which include filler in plastics and rubber. It imparts weight and firmness to rubber and plastics, making them heavy, stiff, and durable. It is also employed to produce bright colours in plastics [10]. Other applications include it use in paints and coatings, chemicals, pulp for paper, radiation shielding, glass, and ceramics. The paints and coatings industry primarily use barite as a filler material to add weight and sometimes maintain viscosity, for sound reduction in engine compartments, coat of automobile finishes for smoothness and corrosion resistance, friction products for automobiles and trucks. Barites are good at absorbing gamma radiations. Hence, they are used in the nuclear industry to manufacture high-density cement concrete for use as a shield around a nuclear reactor [9].

The demand for high-quality barite for the formulation of drilling mud in oil and gas well drilling and other industrial applications has been increasingly difficult to meet due to variation in the level and form of gangue minerals in the barite ore. For it usage, some physicochemical properties of the ore must be assessed which include: purity, content of heavy metals, soluble alkaline metals, iron content, moisture content, oil absorption rate and most importantly, the specific gravity of the barite ore.

For barite to be used for industrial applications, it requires proper characterization of the ore. Characterization is simply done to ascertain the elemental composition and some physical characteristics of the ore. Characterization is fundamental in mineral processing which includes solid phase, surrounding environment (liquid/gas/second liquid phase), interface and interline, flow and force parameters. One of the characterization methods is X-ray Florescence (XRF) which determines the elemental composition the mineral with the use of a spectrometer [11].

An X-ray fluorescence (XRF) spectrometer is an x-ray instrument used for routine, relatively non-destructive chemical analyses of rocks, minerals, sediments and fluids. It works on wavelength-dispersive spectroscopic principles. The analysis of major and trace elements in geological materials by x-ray fluorescence is made possible by the behavior of atoms when they interact with X-radiation. When materials are excited with high-energy, short wavelength radiation, they can become ionized. If the energy of the radiation is sufficient to dislodge a tightly-held inner electron, the atom becomes unstable and an outer electron replaces the missing inner electron. The emitted radiation is of lower energy than the primary incident X-rays and is termed fluorescent radiation, the resulting fluorescent X-rays can be used to detect the abundance of elements that are present in the sample [11].

The American Petroleum Institute (API) and American Society for Testing Materials (ASTM) has set specifications for the barite used in the oil industry and other industrial applications [12,13]. These specifications primarily deal with specific gravity, moisture content, oil absorption and maximum quantities of some impurities. Most barite needs to be grounded to a small uniform size before it is used as a weighting agent in petroleum-well-drilling mud based on specifications set by the API or the former Oil Companies' Materials Association (OCMA). Table 1 shows the physical and chemical requirements of barite.

II. MATERIALS AND METHODS

Materials

The barite ore sample was obtained from Obubra in Cross River State. Other materials used for the analysis included: distilled water for washing of specimens, measurement of specific gravity and moisture content, engine oil for oil absorption test, filter papers for filtering of specimens. The equipment used during the course of this research work included: pulverizer, ball milling machine, set of sieves, digital weighing machine, laboratory oven, X-ray fluorescence spectrometer and pycnometer bottles.

Methods

The ore sample was characterized by determining the elemental composition and physicochemical properties. The elemental composition was determined using X-ray Fluorescence (XRF) to determine the elements and their various percentages present in the ore sample. Some physicochemical properties were also examined which included: specific gravity, moisture content and oil absorption test. These parameters where compared with American Petroleum Institute (API) and American Society for Testing Materials (ASTM) specifications for barite industrial applications to determine their suitabilities for various industrial applications.

Sample Preparation

3000g of barite was collected from the Obubra deposit. The ore sample was first crushed into smaller particle sizes with a Fritsch pulverizer (Type: 0130S and Serial No: 1060). The pulverizer was used to reduce the sizes to less than 1050 µm. A Jinpeng ball milling machine (Model MJG-3189 and Motor No: JR135-89) was used to further reduce the particle size and to achieve homogeneity in composition. Sample in powder form was collected and subjected to size analysis using a laboratory hand sieves of sizes of +180 µm and +63 µm to achieve particle sizes of -180 + 63 µm. The sample in powder form was collected for XRF composition analysis and physicochemical properties test. This test was carried out at the Mineral processing laboratory of Federal University of Technology, Akure, Ondo State.

Table 1: API and ASTM barite physical and chemical requirement, 2014.

Barite application	Barite (%)	SG	Oil absorption (%)	Moisture content (%)	Heavy Metals (Pb & As)%	Soluble alkaline (Ca & Sr)%	Fe content (%)
Drilling mud	90(Min)	4.20(min)	10 – 12	1(max)	N/A	2.5(max)	N/A
Paint coating	85 – 95	4.30(min)	10 - 12	0.5 (max)	N/A	2.0(max)	N/A
Asbestos production	90(min)	4.30(min)	10 - 12	N/A	N/A	-	N/A
Plastic filler	90(min)	N/A	N/A	0.5(max)	N/A	2.0(max)	N/A
Chemical industries	90 - 92	4.0	N/A	N/A	N/A	1(max)	2(max)
Glass Industries	90 – 96	N/A	N/A	0.5(max)	N/A	-	0.3(max)
Pharmaceutical	97(min)	N/A	N/A	N/A	<0.1	1(max)	0.1(max)

XRF Characterization

The elemental composition of the barite ore sample was determined using Skyray EDX 3600B Energy Dispersed X-ray Fluorescence spectrometer. 20g sample pellet was loaded in the sample chamber of the spectrometer. A voltage of 40 Kv and current of 350 μA were applied to produce the X-rays to excite the sample for 10 minutes at a temperature of 20°C ± 0.2°C. The spectrum from the sample was analyzed to determine the concentration of the elements in the sample. This test was carried out at the Engineering Materials Development Institute (EMDI), Akure, Ondo State. The result of this study was also analyzed to identify the major and trace elements present, the percentage of heavy metals, soluble alkaline metal and iron present.

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.

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

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

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 (4) was used to calculate of the percentage of water and oil absorption (14):

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

Where:

Wi - weight after immersion and

Wo - weight before immersion

III RESULTS AND DISCUSSION

Results

The result of the characterization of Obubra barite ore which involved both chemical and physicochemical analyses aimed at identifying the various components present in the ore and also ascertain their physical and chemical properties are presented in this section. Also, the result of the chemical characterization of the elemental composition through X-ray Fluorescence and the physicochemical characterization which includes: specific gravity test, moisture content analysis and oil absorption test are equally presented in this section.

XRF elemental characterization

Table 2 shows the result of the elemental composition analysis for Obubra barite ore sample using X-ray Fluorescence. From the table, it was

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observed that the barite sample contains 50.14% Barium, 35.88% Sulphur, 3.75% Titanium, 3.51% Vanadium, 1.54% Aluminium, 2.56% Silicon and 0.16% Iron amidst others. The occurrence of Iron (0.16%) was observed. 0.20% Heavy metals of lead (Pb) and Arsenic (As) was also observed in the sample. Calcium a soluble alkaline metal of 0.18% was also found present. Figure 1 shows the spectra pattern of the ore sample at various energy level.

The peak height of various elements is directly related to the concentration of elements and intensity within the sampling volume. Sulphur, Barium, Titanium and Vanadium showed more distinct peak.

Table 2: XRF elemental composition of barite sample

Element	Mg	Al	Si	P	S	Ba	Ca	Ti	V	Mn	Fe	Ni	Cu	Zn
Sample (%)	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	0.07

Element	As	Pb	W	Au	Nb	Mo	Sn	Sb
Sample (%)	0.08	0.12	0.07	0.00	0.00	0.17	0.44	0.44

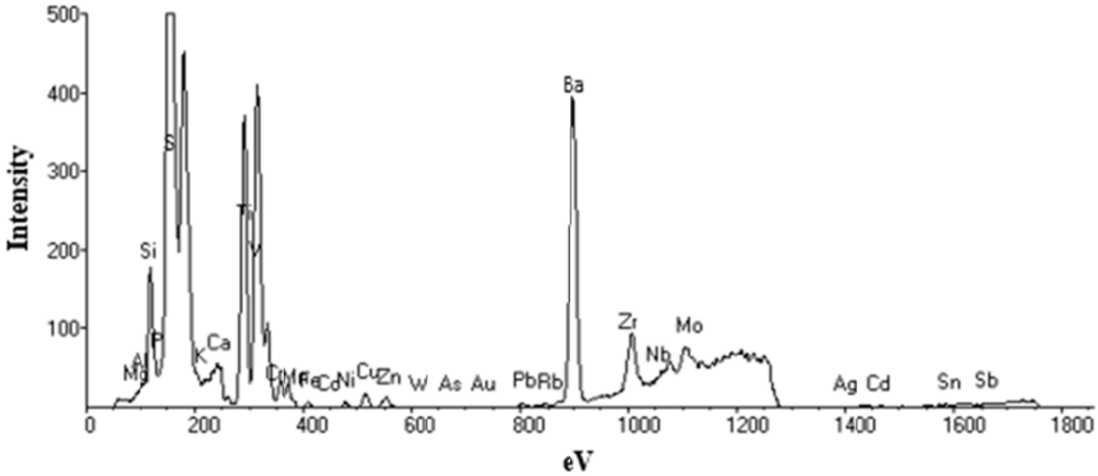

Figure 1: XRF elemental composition pattern for Obubra barite sample

Table 3: 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 4: 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 5: 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	

IV.RESULTS AND DISCUSSION

Discussion

From the result of the XRF elemental composition of the barite sample as shown in Table 2, it was observed that the barite ore was associated with a lot of impurities. The study indicated that the sample contain two major elements which are barium and sulphur with 20 other trace elements as shown in table 2. The ore contains 50.14% Barium, 35.88% Sulphur, 3.75% Titanium, 3.51% Vanadium, 2.56% Silicon, 1.54% Aluminium and 0.12% lead amidst others. This result gave 86.02% Barium-Sulphur (Ba-S) empirical composition which does not meet most of the Industrial application requirements except for paint coating which requires 85 – 95% Ba-S content as shown in table 6.

Heavy metals like lead (Pb) and Arsenic (As) was also observed in the sample. A total percentage of 0.20% was observed which makes it unsuitable for pharmaceutical application that requires less than 0.1% heavy metal. 0.18% soluble alkaline element of calcium was also observed to be present. When compared to the application requirement as shown in Table 6, it was observed to meet all the industrial application specifications which requires maximum of 1.0%. The sample was also observed to contain Iron (fe) of 0.16%. This value when also compared to API and ASTM general specification, it was observed to be suitable for industrial applications except for its use in pharmaceutical industries which requires maximum of 0.1%.

The specific gravity which is also an important parameter for industrial application was determined. The specific gravity experiment was performed three times for better accuracy, the sample had an average specific gravity value of 3.78 as shown in Table 3. When this value was compared to the API and ASTM general specifications, it was observed that the barite sample is not suitable for industrial applications. This specific gravity is low compared to the minimum industrially accepted value of 4.2 for drilling mud additive grade and other industrial applications of between 4.0 to 4.3 except for it use in glass industries, pharmaceutical industries and as plastic filler which does not have any specific gravity specifications. The reason for this low specific gravity can be traced to the high level of impurities observed from the chemical analysis.

The moisture content analysis was performed three times on the sample and an average moisture content of 0.31% was obtained as shown in Table 4. This value was compared to the API and ASTM specification, it was observed that the barite sample meet all industrial application requirements as seen in table 6. The oil absorption test was also performed three times and an average of 11.48% was obtained. This value, when compared to the API and ASTM specification, it was observed that the barite sample meet all industrial application requirements which requires between the range of 10 – 12% for all industrial application requirements as seen in table 6.

Table 6: American Petroleum Institute (API) and American Society for Testing and Materials (ASTM) general specification standards for various uses of barite ores for different industrial applications.

Barite application	Barite (%)	SG	Oil absorption (%)	Moisture content (%)	Heavy Metals (Pb & As)%	Soluble alkaline (Ca)%	Fe content (%)	Suitability for applications
Drilling mud	90(Min)	4.2(min)	10 – 12	1(max)	N/A	2.0(max)	-	No
Paint coating	85 – 95	4.30(min)	10 -12	0.5 (max)	N/A	2.0(max)	-	No
Asbestos production	90(min)	4.30(min)	10 - 12	N/A	N/A	-	-	No
Plastic filler	90(min)	N/A	N/A	0.5(max)	N/A	2.0(max)	-	No
Chemical industries	90 - 92	4.0	N/A	N/A	N/A	1(max)	2(max)	No
Glass industries	90 – 96	N/A	N/A	0.5(max)	N/A	-	0.3(max)	No
Pharmaceutical	97(min)	N/A	N/A	N/A	<0.1	1(max)	0.1(max)	No

V.CONCLUSION AND RECOMMENDATION

In conclusion, the barite ore sample contain high quality of barite of 86.02%. The barite contain low heavy metals suitable for industrial applications except its use in pharmaceutical industries which requires less than 0.1%. The samples also contains low iron and soluble alkaline metals in ranges suitable for industrial applications. The specific gravities are low for any industrial application except for glass industries and pharmaceutical industries which does not have any required specification. The moisture content and oil absorption rate fell in between the required industrial application specification. The Obubra barite ore needs to be subjected to combination of some simple beneficiation processes such as tabling, jigging, floatation, leaching etc to improve the ore in other to make it suitable for industrial applications in production of drilling mud additive, paint coating, Asbestos production, plastic filler, glass production, chemical industries and its use in pharmaceutical industries.

REFERENCES

1. Arrifan, S.K. (2003). EBS 425—Mineral Perindustrian: Penang, Malaysia, School of Materials and Mineral Resources Engineering, University of Science, Malaysia, p. 14. Retrieved from http://mineral.eng.usm.my/web%20halaman%20mineral/EBS%20425 %20industrial% 20minerals.pdf.
2. Brock-Hon, A., & Johnston, S. E. (2014). Separation and characterization of pedogenic barite crystals from petrocalcic horizon materials for future isotopic and geochronological applications. Geoderma, 217-218, 129– 134. doi:10.1016/j.geoderma.2013.11.006
3. Raw Materials Research and Development Council. (2010) "Non-Metallic Mineral Endowments in Nigeria," Abuja: Federal Ministry of Science and Technology, pp. 1-113.
4. Garside M (2020) Global barite production from 2011 to 2019. https://www.statista.com/statistics/799487/global-barite-production/
5. Bosbach, D., Hall, C., & Putnis, A. (1998). Mineral precipitation and dissolution in aqueous solution: in-situ microscopic observations on barite (001) with atomic force microscopy. Chemical Geology, 151(1-4), 143–160. doi:10.1016/s0009-2541(98)00076-x
6. Crecelius E, Trefry J, McKinley J, Lasorsa B, Trocine R. (2007). Study of barite solubility and the release of trace components to the marine environment. New Journal Pre-proof 26 Orleans (LA): US Department of the Interior, Minerals Management Service, Gulf of Mexico Region. OCS Study MMS 2007–061. p 1–147
7. Ulusoy, U. (2019). Quantifying of particle shape differences of differently milled barite using a novel technique: Dynamic image analysis. Materialia, 100434. doi:10.1016/j.mtla.2019.100434.
8. Das, S., Essilfie-Dughan, J., & Hendry, M. J. (2020). Characterization and environmental implications of selenate co-precipitation with barite. Environmental Research, 109607. doi:10.1016/j.envres.2020.109607.
9. Michael Miller Barite. (2009) Minerals Yearbook.
10. Boamah, S. K. (2019). Comparative analysis of barite production in Africa. A thesis presented to the Department of Petroleum Engineering African University of Science and Technology, Abuja In partial fulfillment of the requirements for the degree of Master of Science.
11. Fitton, G. (1997). X-Ray Fluorescence spectrometry; an introduction to quantitative chemical analysis of earth, environmental and material scientist; Addison Wesley Longman UK.
12. U.S. Geological Survey (USGS) (2017) Critical mineral resources of the United States, Barite: mineral commodity summaries, professional paper 1802, U.S. Geological Survey, Reston, VA, pp 30–31. ISBM: 978-1-4113-3991-0. https://doi.org/10.3133/ pp1802A
13. U.S. Geological Survey (USGS) (2020) Barite: mineral commodity summaries, prepared by Michele E. McRae, U.S. Geological Survey, VA, pp 28–19
14. Dagwa, I. M and Ibhadode, A. O. A. (2006): Development of Asbestos-Free Friction Lining Material from Palm Kernel Shell. J of the Braz. Soc. of Mech. Sci.& Eng. (02):166-173