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Original Research Article

Physico-Chemical and Mineralogical Characterization of Barite-Bearing Ores from Selected Locations in Nigeria

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ABSTRACT

Nigeria is naturally endowed with solid mineral reserves and must do everything necessary to harness and maximize these resources and unlock their potential. Hence, the research on Nigerian barites was examined in this study. Barite-bearing ores were sourced from Azare (Bauchi State), Obi (Nasarawa State), and Obubra (Cross River State) in Nigeria and characterized to determine their properties. The physico-chemical characteristics revealed specific gravity of 4.02, 4.14, and 3.95, moisture content of 0.94%, 0.68%, and 0.57%, density of 4.60 g/cm^3, 4.82 g/cm^3, and 5.32 g/cm^3, bulk density of 2.45 g/cm^3, 2.49 g/cm^3, and 2.38 g/cm^3, and water absorption capacity of 4.79%, 4.96%, and 2.85% respectively. For the barite samples, highly crystalline peaks with barite compositions of 98.93%, 92.26%, and 92.50% respectively were revealed using X-ray diffractometry (XRD). X-ray fluorescence (XRF) analysis revealed $BaSO_4$ as the primary component with minor gangue impurities while scanning electron microscopy (SEM) analysis offered quantitative information on the morphology and surface characteristics present. This research has shown that Azare barite is suitable for high purity applications such as those found in the pharmaceutical industry, whereas Obi and Obubra barite ores must be beneficiated to be suitable for potential applications such as drilling mud formulation, glass production, filler, or extender in paint and rubber manufacturing, according to the standards specified by the American petroleum institute (API), the Oil Company Mineral Association (OCMA), and the Indian standard (IS).

1. INTRODUCTION

Solid minerals are inorganic substances with unique chemical and atomic structures that occur naturally. An ore is usually depicted as a collection of minerals in sufficient quantity to make extraction economical

(Muriana, *et al.*, 2013; Maduaka, 2014). The standards for a mineral deposit to be classified as an ore vary depending on the mineral (Subba, 2011). In the long run, not all mineral discoveries will have sufficient reserves to appeal to mining companies. The deposit's location and size, as well as the ore feed grade, mineralogy, texture, mining costs, and ancillary services costs such as power, as well as the ore's amenability to cost-effectively viable treatment, the metal's value, and its concentrate, all limit the suitability of a deposit for exploration, exploitation, and processing technology for solid minerals such as barite ores (Wills , 1989).

West African countries including Burkina Faso, Ghana, Ivory Coast, and Niger have been active global mining hubs for a long time (Markwitz, *et al.,* 2016; Chuhan-Pole *et al.,* 2017). Nigeria has the potential to become a significant global centre for the processing of solid minerals. Nigeria's geology is comparable to those of countries where world-class reserves have been discovered (Adekaya, 2003; Ajaka and Oyathelemi, 2010). Over 40 solid minerals are available throughout Nigeria (NEITI, 2014; Omoh, 2015), with 34 of these solid minerals having been proven to exist in sufficient quantities in Nigeria's geology. Seven solid minerals have been strategically targeted and developed for commercial purposes and they include barites, limestone, gold, coal, lead/zinc, iron ore, and bitumen (Hanor, 2000; NEITI, 2014). These seven minerals are world-class and were chosen for development due to their abundance in large-scale quantities to support long-term mining operations.

Nigeria has a large barite reserve with the majority of it located in Benue, Nasarawa, Plateau, and the Cross-River States (Onwualu, *et al*., 2013a). Similarly, barites have been discovered in eight Nigerian states: Zamfara, Gombe, Plateau, Adamawa, Taraba, Cross River, Nasarawa, and Bauchi (Onwualu, et al., 2013b). According to a recent assessment by the Nigerian Geological Survey Agency, confirmed reserves in Benue and Nasarawa states are estimated to be 111,000 tonnes, with commercially viable resources estimated to be 21,123,913 metric tonnes throughout the country (Ministry of Mines and Steel Development (Okpanachi, 2004; MMSD), 2008; Obi, *et al*., 2014).

Nigeria's mining industry is underdeveloped. As a result, the government is compelled to import minerals that could otherwise be produced in the country. As a result, the purpose of this research is to characterize the barite resources in a few selected Nigerian locations as feedstock for important industries. This is necessary in light of the country's significant solid mineral resources, as well as technical elements and potential actions to develop the country's solid mineral resource potential.

2. MATERIALS AND METHODS

2.1. Description of Study Areas

Geographic information system (GIS) was used to map the study regions of the locations in the mineral belts of Azare Local Government Area in Bauchi State (North-East), Obi Local Government Area in Nasarawa State (North-Central), and Obubra Local Government Area in Calabar, Cross River State (South-South). The map location of the research areas in Figures 1 to 3 were generated with global positioning system (GPS) satellite network coordinates carried out from the field studies. Azare is situated at an altitude of 436 meters in Bauchi State, Nigeria, at 11°40′27″N 10°11′28″E. The location of barite in the site's coordinate system is depicted in Figure 1 (Azare). It illustrates road networks as well as commercial areas. The brown shaded regions of the map with asterisks show barite locations, while dotted points indicate areas where barite was not present on the subject site. The dark brown shaded coloured part of the map is the development area of the site, where the residents of Azare live, which is shaded dark brown on the map. The red line depicts the road network, while the red dotted line denotes the study site's boundary. The map area or zone's coordinate system is located between 11°42′00″N, 10°10′00″E and 11°40′30″N, 10°13′300″ E in the northern part of the site, and 11°39′00″N, 10°10′30″E and 11°39′00″N 10°13′30″E in the southern part of the site, with a perimeter of 21642 meters and a land area of 27808036 meters square. Obi is a local government district in Nigeria's Nasarawa state that is rich in natural mineral resources such as barites (Geological survey of Nigeria, 2007). It covers 955.2 square kilometres and is at a height of 334 meters above sea level. Its

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coordinates are 8°23′30″N, 8°45′30″E, and 8°23′30″N, 8°47′30″E on the northern half of the study area (site), and 8°21′30″N, 8°47′30″E on the southern section of the site, with a perimeter of 15793 meters. Figure 2 represents a road network with red lines in terms of the coordinate system, asterisks on a brown backdrop for regions where barites are discovered, and dotted points for areas where there are no barites on the subject site. The boundary is denoted by red dotted lines, while the shaded region on the dark brown background denotes the residential/commercial area. Obubra is a Nigerian local government region in the Cross-River State. The Ibrahim Babangida College of Agriculture is sited in Obubra, which serves as the organization's headquarters (Ekwueme, 2003; Ekwueme and Akpeke, 2012) The coordinates in Figure 3 are 6°6′00″N, 8°19′00″E and 6°6′00″N 8°20′30″E for the Northern half of the site/location and 6°4′30″N, 8°19′00″E and 6°4′00″N 8°20′00″E for the Southern section. The map also depicts a road network with red lines, a water body with blue colour, a boundary with red dotted lines, and a development area with brown colour. With a radius of 13890 meters and an area of 7778072 square meters, asterisks and dotted points were utilized to illustrate places where Barites are discovered and not found, respectively.

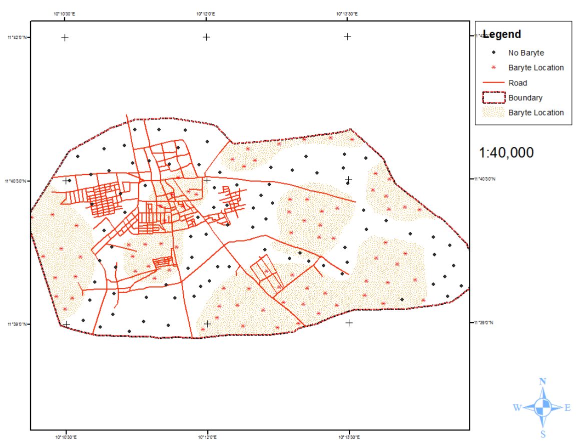

Figure 1: Geologic map of Azare area in Bauchi State showing barites deposit

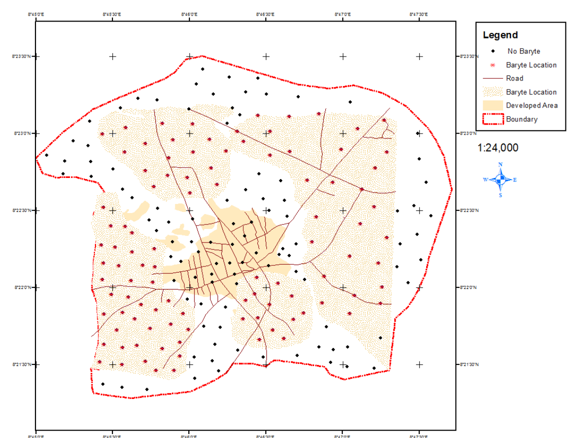

Figure 2: Geologic map of the Obi area of Nasarawa State showing barite deposit

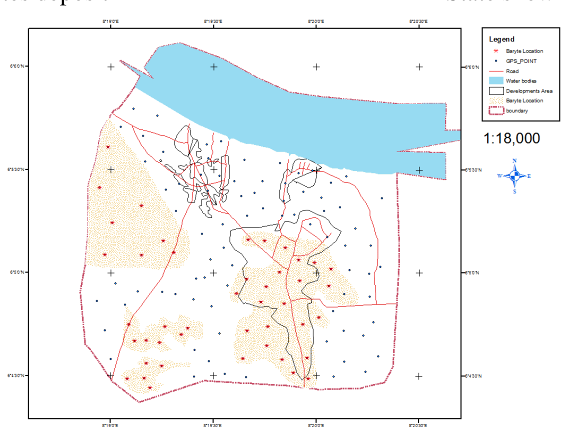

Figure 3: Geologic map of Obubra areas of Cross River State showing barites deposit

2.2. Sample Collection

Low-grade samples of 5 kg barite-bearing ores, the raw material, were collected at an interval of 0.6 m from the bottom to the top of the exposure, using a sledgehammer to disaggregate the amalgamated barite ores in

15 different locations of the selected locations. The stratified random sampling technique was used, which ensures that all possible barite-bearing ores were sampled.

2.3. Geochemical Characterisation Tests

The starting materials of barite-bearing ore samples (Azare, Obi, and Obubra) for the research work were characterised by X-Ray diffraction (XRD) (Thermo Scientific, Switzerland), scanning electron microscopy (SEM) (Phenom Prox, Netherland), X-ray fluorescence (XRF) (Model Xsupreme 8000 by Oxford instrument) to determine their mineralogical compositions and microstructural characteristics. These instruments were calibrated using certified reference barite samples materials supplied from the National Institute of Standards and Technology (NIST) in the United States via Kaduna's Geoscience Research Laboratory Centre. On the barite ore matrix, adequate sample preparation for the analysis was carried out using aqua regia and wet acid (i.e., HNO_3 + HCl) treatment by digestion and fusion.

2.4. Particle Size Analysis

Screening involves the distribution of the coarse barite into various mesh sizes. The sieves used in the experiment were stacked or nestled, with the coarsest sieve on top and the finest on the bottom. A pan was positioned underneath the bottom sieve to collect the finer undersize, and the top sieve was safeguarded with a lid to prevent samples from escaping during shaking. The feed material to be screened was put on the topmost coarsest sieve and closed with a lid, the nest was then shaken by hand and sieved for 10 mins. The samples were hand-panned to upgrade their barites' content. Particles cannot pass through the sieve unless they are offered in a favourable direction due to their uneven forms (Kolawole, *et al.,* 2021). Allowing the sample material to run through a succession of test sieves, where each mineral grain is practically free, was used to measure the percentage weight of closely sized particles. The analyses were conducted at the instrumentation and control laboratory of the Lagos State University of Science and Technology, Ikorodu, Lagos, Nigeria. Each sieve's contents were removed and weighed. The particle size of barite samples is significant because it influences a variety of properties, including surface area and sample solubility. It also aids in the screening or reduction of deleterious gangue materials that are capable of affecting the beneficiation properties (Egbe, *et al.,* 2013; Akpan, *et al.,* 2014). The particle size analysis determines the size and weight fractions of the various grains that make up the samples, which are referred to as particle size fractions.

2.5. Physico-chemical Characterisation

The barite-bearing ore samples were subjected to physicochemical tests/analyses in the laboratory to affirm the quality of the barite ores obtained from the study fields. Thus, the barite-bearing samples were subjected to some geotechnical (physical properties) tests such as bulk density, density, moisture content, specific gravity, and water absorption capacity in the laboratory to evaluate the grade of the samples.

2.5.1. Moisture content test

After 30 minutes of oven drying, the samples were weighed on an electronic compact scale (600 g x 0.01 g). This method was continued until the weight remained consistent. Equation (1) was used to calculate the moisture content of the sample.

$$\%\ W = \frac{(A-B)}{B} \times 100 \quad (1)$$

where % W = Moisture content of the sample in percent, A = Initial weight of the sample (wet sample in kg), B = Final weight of the sample (dry sample in kg)

2.5.2. Specific gravity

The specific gravity of the barite collected for this study was determined by immersion. The immersion method is based on Archimedes' principle. The weight of the density bottle, the weight of the barite stored in the density bottle, and the pre-measured volume of water were all noted, as well as the increase in volume (V) of water. Equation 2 was then used to calculate the specific gravity (SG) of the samples:

$$\text{SG} = \frac{w}{v_2 - v_1} \quad (2)$$

Displaced water volume, $V = V_2 - V_1$ and w = weight of the sample

2.5.3. Density

The pycnometric technique was used to determine the sample density. This involved the use of a density bottle (a) whose volume (v) is 25 mL and a barite sample (b) with a mass of 25 mg, then reweighed. The density of the sample was then calculated using the formula in Equation (3).

$$\rho = \frac{b - a}{volume\ (V)} \quad (3)$$

Where a = density bottle weight, b = the sample's weight plus the density bottle's weight

2.5.4. Bulk density

After the barite sample had dried fully, 1 g of the sample was measured into a petri dish from the total weight initially recorded. Water (4 ml) was measured in a 10 mL measuring cylinder, and then 1 g of sample was submerged in it and the displaced volume was recorded. This was done for each sample, and the bulk density was calculated using Equation (4).

$$\text{Bulk density} = \frac{mass\ of\ sample\ in\ air}{volume} \quad (4)$$

2.5.5. Water absorption capacity

Each barite sample was dried in an oven at 320 °C for 4 hours before being placed in a desiccator to cool. It was then soaked in water for 24 hours at ambient room temperature (34 °C) and filtered and patted dry. For each of the three samples, the same technique was followed, and the water absorption capacity was calculated using Equation (5).

$$\%\ \text{Water absorption capacity} = \frac{wet\ weight - dry\ weight}{dry\ weight} \times 100 \quad (5)$$

3. RESULTS AND DISCUSSION

3.1. Particle Size Analysis

When considering any given mineral separation process, recognition is usually given to the range of particle sizes that may be treated which is often limited. Figure 4 shows the weight percent of the material retained vs the average or mean particle size and is termed a linear scale frequency plot. The apex points of the weight percent retained with the average size are more than 30.52 % at 5412.5 µm, 45.46% at 63125 µm, and 52.55% at 1990 for Azare, Obi, and Obubra respectively as revealed in Figure 4. Calculations of mean particle size, the specific surface area of the mixture, or the number of particles in the mixture may be based on either a frequency-size distribution plot or cumulative distribution curves obtained by plotting the fraction of the total weight particle having a size greater than or less than a given screen opening. In theory, screen analysis methods based on cumulative analysis are more exact and accurate than those based on frequency analysis, because cumulative analysis eliminates the requirement to assume that all particles in a particular fraction are the same size (McCabe, *et al.,* 1993; Badger and Banchero, 2005). Individual differential weight percentages from the collected screen analyses results were added cumulatively to generate the cumulative weight percentage retained from the differential analysis. This gives the quantitative picture of the relative distribution of the sample material over the entire size range, with the physicochemical analyses. In many cases, the data is more commonly plotted as cumulative weight percent passing versus mesh size, called a linear scale cumulative plot, as shown in Figure 5. The cumulative curve of Obubra deviated to the left while both the curves of the Azare and the Obi deviated to the right and they all converged at the terminal points. But the Azare, Obi, and Obubra weight percent retained to mean size were all inversely proportional. However, the cumulative curves indicated the particle sizes decreased faster from the Obubra, Azare, and Obi in that order when comparing the curves. Hence, average particle size has an impact more on the Obi and Azare than Obubra particle size distribution.

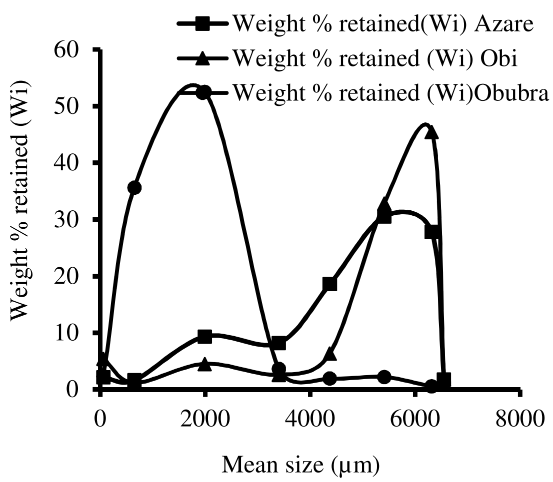

Figure 4: Linear scale frequency plots

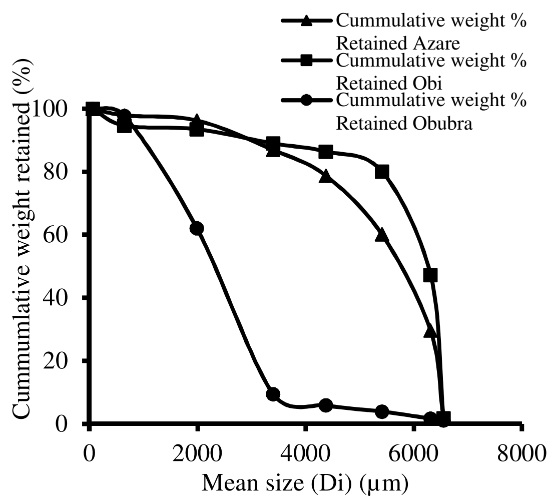

Figure 5: Linear scale cumulative plots

3.2. Physical Characteristics

The geotechnical properties of specific gravity, moisture content, density, bulk density, and water absorption capacity of the barite ores were determined as revealed in Table 1. The water absorption was measured to assess whether there was an increase in the barite configuration as a result of gangue components. The water absorption capacity was within the acceptance value of 1.2 maximum water absorption as noted by Ene, *et al.* (2016). The specific gravity (SG) of the barite samples Azare (4.02), Obi (4.14), and Obubra (3.95) were all within the recommended maximum range of 3.6 to 4.2 set by the American petroleum institute (API, 2010), and the American society for testing materials (ASTM, 2015) for various barite ores usage. A high SG value of barite is required to aid in pressure formation during drilling and also when used for other weighting agent materials (Rivera and Radovic, 2004; Mgbemere, *et al.,* 2018). However, all of these values were lower when compared to the minimum industrially accepted values of > 4.15 (Indian standard), > 4.2 (American petroleum institute), and > 4.2 (Oil company mineral Association) (Lowrenz and Gwosdz, 2003; Labe, *et al.,* 2018). The corresponding values of the moisture content of barite for the Azare, Obi, and Obubra were found to be 0.94, 0.68, and 0.57% respectively, which falls within the standard limits set by the API grade (1.0 wt.%).

Table 1: The results of the physical analysis of the raw barite sample from the study areas

Parameter	unit	Raw Azare barite	Raw Obi barite	Raw Obubra barite
SG	-	4.02	4.14	3.95
MC	%	0.94	0.68	0.57
Density	g/cm^3	4.60	4.82	5.32
BD	g/cm^3	2.45	2.49	2.38
WAC	%	4.79	4.96	2.85

SG = Specific gravity, MC = Moisture content, BD = Bulk density, WAC = water absorption capacity

3.3. X-ray Diffraction

XRD is a non-destructive analysis method. In this research work, it was used for both qualitative and quantitative analysis of the raw samples. The X-ray diffractograms of the untreated barites are presented in Figures 6 to 8 for Azare, Obi, and Obubra Ores respectively. They all indicate heterogeneous compositions at the peaks with some reasonable traces of the gangue components. Figure 6 shows the least impurities at the maximum peak point when compared to other lower peaks. Figure 7 revealed that the Obi barite sample contained thallium, hydroxide hydrate, and copper oxide. The XRD patterns of the crude Obubra barite presented in Figure 8 indicated heterogeneous compositions with impurities (gangue) of calcium, iron, aluminium, cobalt, silicate, tungsten, and carbide. A comparison of these results with others in the literature indicates that they are similar (Hanor, 2000; Falconer, 2003; Abubakare, 2011; Tanko, *et al.,* 2015; Mgbemere, *et al.,* 2018).

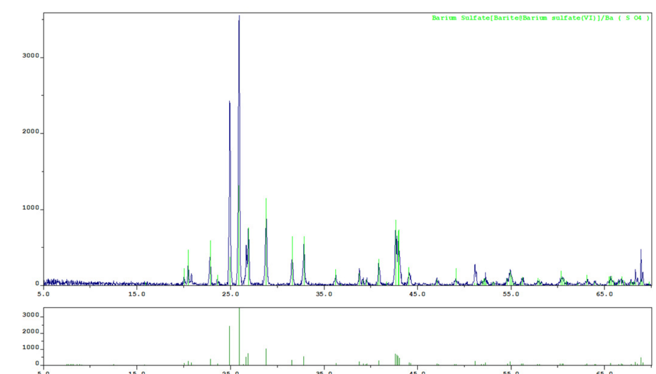

Figure 6: XRD patterns of untreated Azare barite-bearing ore

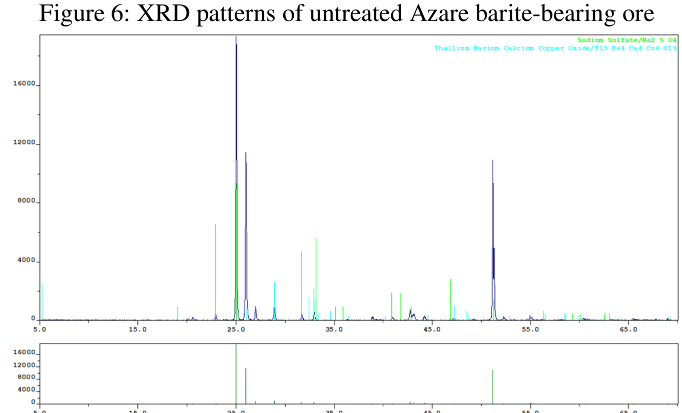

Figure 7: XRD patterns of untreated Obi barite-bearing ore

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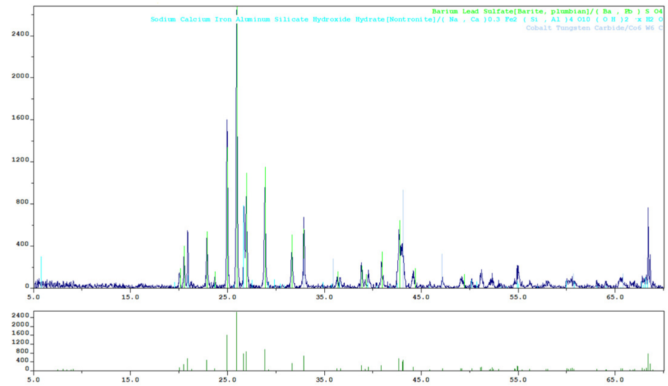

Figure 8: XRD patterns of untreated Obubra barite-bearing ore

3.4. Surface Morphology Characterisation

SEM images were used to analyse the surface morphology of barite samples. The morphological analysis of the samples revealed that the ore minerals are predominantly in a free state, resulting in neck expansion between deposited particles and the formation of a sticky deposit surface impacted particles. Figures 9 to 11 are typical SEM micrographs of barites that portray crystal grits-like structures.

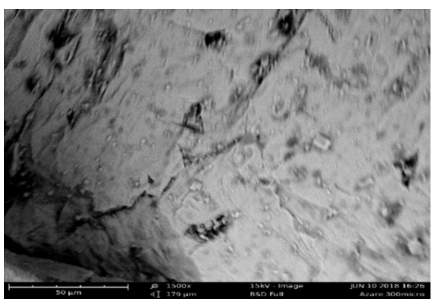

Figure 9: SEM images representing untreated (raw) barite ore of Azare

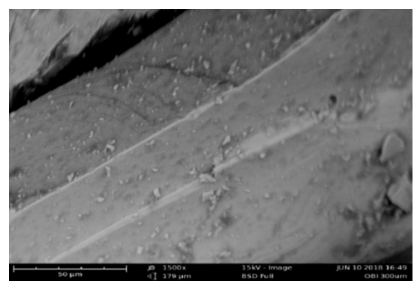

Figure 10: SEM images representing untreated (raw) barite ore of Obi

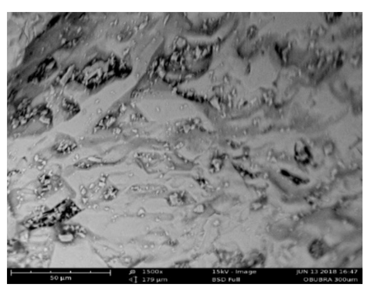

Figure 11: SEM images representing untreated (raw) barite ore of Obubra

3.5. Chemical Composition Analysis

The results of the XRF analysis of barite-bearing samples from the research locations are shown in Table 1. The major impurities were SiO_2, Fe_2O_3, Na_2O, and Al_2O_3, which must have influenced the appearance of the final samples. From Table 1 the barite ($BaSO_4$) samples were compared with API grade standards for the barite composition. The combined BaO and SO_3 give a $BaSO_4$ value of 98.93 wt.% placing Azare barite well above the minimum value of > 92% and 96% maximum value of API grade specifications range which is required for general industrial applications usage for barite reserves (Mgbemere , *et al.,* 2018). The results also showed that the barite samples of Obi and Obubra fell substantially lower than the maximum value of 96% and > 94% for Indian standard (IS) for standards barite (Lowrenz and Gwosdz, 2003; Labe, et al., 2018) with the combined untreated $BaSO_4$ values of 92.26% and 92.44 wt.% for Obi and Obubra barites respectively but were higher than minimum specification standards of > 92% for oil company material association (OCMA). Therefore, the run-of-mine ore of Azare barite bearing ore is a potential $BaSO_4$ source of the refined barite with minimal impurity. While the Obi and Obubra findings show that barite-bearing ores are below the range for use in glass production and as a filler or extender in paint and rubber production, they will need to be treated before they can be used as components in high-quality feedstock products. Hence, all the raw samples were above the 88 % requirement for the white rate production of wallpaper and white paint as reported by Otoijamun, *et al.* (2021).

Table 1: General specification of the raw and refined barite content compared with API grade

Element (wt.%)	API grade standards for barite (%)	Raw Azare barite (%)	Raw Obi barite (%)	Raw Obubra barite (%)
BaO	65.830	74.259	72.216	75.207
SO_3	34.070	24.667	20.044	17.229
Na_2O	0.000	0.028	0.084	0.054
K_2O	< 0.010	0.005	0.008	0.009
MgO	0.000	0.001	0.002	0.003
CaO	0.027	0.014	0.026	0.020
Al_2O_3	< 0.010	0.062	2.012	2.038
Fe_2O_3	0.050	0.028	4.027	4.066
SiO_2	3.500	0.931	1.845	1.549
$BaSO_4$	> 92- 96 max	98.926	92.260	92.436

4. CONCLUSION

The following conclusions can be drawn from the study's findings:

- The barite ore samples collected from the three selected sites were excellent prospects for beneficiation and eventual industrial application. This was demonstrated by their qualities, as determined from the characterization results obtained.
- The physical and geochemical characteristics of the Azare, Obi, and Obubra barite ores have been determined, and the government may now use them to develop mining and exploitation methods.
- Azare barite has been proven to be acceptable for applications requiring high purity, such as those found in the pharmaceutical industry, according to this research. As a result, the low-grade barite-bearing ores from Obi and Obubra can be enriched by mixing their crude samples with a high-grade barite ore sample from Azare, as described by Abraham et al. (2021), where low-grade barite quality was improved by a high grade.
- Nigeria is well-positioned to take advantage of the barite-bearing ores mineral as a means of economic diversification because these minerals are available in commercial quantities in the selected locations of the research.

5. ACKNOWLEDGMENT

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6. CONFLICT OF INTEREST

There is no conflict of interest associated with this work.

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