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Evaluation of Baryte Ores from Selected Sites in Obubra Mine of Cross River State, Nigeria, for Application as a Weighting Agent in Drilling Fluid: A Physical Characterization

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Abstract
Baryte, a non-metallic mineral, is used primarily for the formulation of drilling mud to control reservoir pressure during drilling. However, despite its abundant reserves in Nigeria, the international oil companies (IOC) operating in Nigeria still apply imported baryte for operations. Therefore, in this research, the physical properties (specific gravity (SG), hardness and moisture content (MC, and pH)), of baryte deposits from 5 selected sites in Obubra mine of Cross River State, were determined and compared, vis-à-vis the API specifications, to ascertain their suitability for use in oil drilling. The white powder obtained from a streak test on the 5 ores indicated that all were indeed baryte ores. Furthermore, the samples were separately washed, dried, crushed, ground, and milled. In addition, the ore's hardness of 3.5, and MC range of 0.00-0.01%, and pH range of 7-7.4, were all within the API specifications. Moreover, readings from the SG obtained were; site 1=4.25, site 2 = 4.28, site 3 = 4.32, site 4 = 4.12 and site 5 = 3.37. Thus, baryte in sites 3 and 5 recorded the highest and lowest SG, respectively. Ultimately, these results showed that ores from sites 1, 2, and 3 meet the API 4.20 specification for drilling purposes. At a SG of 4.12, site 4 sample, fell slightly below 4.20, while site 5 sample was unsuitable for drilling. Therefore, baryte from sites 4 and 5 should be further processed to meet the API SG specification for drilling. These findings prove that Obubra baryte possesses crucial criteria for drilling mud formulation.

Keywords: Baryte Ore, Specific Gravity, Hardness, Moisture Content, Obubra.

Introduction
Baryte is used as a weighting agent for the formation of drilling fluid in oil and gas operations. Baryte, which is also known as "heavy spar" or "spar", due to its relatively high density of 4.5 g/cm^3, is the basic mineral of barium with chemical formula $BaSO_4$, Nandi et al. [1]. Its high specific density ensures that it increases the hydrostatic pressure of the drilling mud, allowing it to compensate for high-pressure zones experienced during drilling. Moreover, the softness of the mineral also prevents it from damaging drilling tools during drilling and enables it to serve as a lubricant, Philips and Paul [2]. This high relative is the reason it is primarily used in oil drilling. According to Nandi et al. [1], over 88% of the worldwide baryte production is applied in the oil and gas sector. In comparison, the remaining 12% is applied in the production of chemical products, such as a pigment in paints and as a weighted filler for paper, cloth, and rubber. It is also used in the production of ceramics, pharmaceuticals, and explosives. It is the primary ore of barium, which is used to make a wide variety of barium compounds. Some of these are used for x-ray shielding. Furthermore, it can block x-ray and gamma-ray emissions.

According to the Nigerian Geological Survey Agency [3], baryte has been discovered in commercial quantities in Nigeria in over nine states: Adamawa, Benue, Cross River, Ebonyi, Gombe, Nasarawa, Plateau, Taraba, and Zamfara.

As earlier mentioned, Cross River State is one of the 9 states that possess baryte in commercial quantities. The estimated baryte reserves in Cross River State are roughly above nine million metric tons, as cited in Edem et al. [4], who referenced Obi et al. [5]. Moreover, this figure places Cross River as the State with the highest reserve of baryte in Nigeria. Alifokpa, Gabu, Obubra,

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Ogoja, and Osina are areas in Cross River State, Nigeria, that hold appreciable and commercial deposits of baryte reserves, as noted by Oladapo and Adeoye [6] and Jimoh and Adeleke [7]. Fig.s 1a and 1b represent baryte mines in the Osina and Alifokpa areas of Cross River State, Nigeria.

Fig. 1a Baryte mine at Osina, Innformed [8] Figure 1b: Alifokpa baryte mine, Oahimire et al. [9].

Currently, international oil companies (IOCs) operating in Nigeria utilize imported baryte in their operations, despite Nigeria possessing baryte in commercial quantities that could meet its operational needs. Several literatures on Nigeria's baryte include reasons for this external patronage by the IOCs. These reasons revolve around the allegation that Nigeria's baryte fails to meet critical specifications for use in oil drilling operations, one of which is its specific gravity, often reported as being below the API specification of 4.2. This argument persists despite recent research showing that several baryte mines in some States in Nigeria possess baryte ores within the API SG range of 4.2.

Thus, this research is aimed at proving that Baryte ore in the Obubra mine meets the API's physical properties for oil drilling and should be exploited for drilling operations in the country.

Geological Settings

The study area is located in Obubra Local Government Area of Cross River State. Obubra lies on latitude 06.08 0N and longitude 08.33 0E, with elevation 109 m, Kamalu et al. [10]. Moreover, the soil is practically Sandy loam in nature. In addition, the annual rainfall distribution of Obubra is within 2500mm to 3000 mm per annum, with an annual temperature range of 25 0C–27 0C, Adinya et al. [11]. Obubra lies beneath two key lithologic components, namely: Cretaceous sediments and crystalline basement. Its human population is 200,000 as recorded in the 2006 census. Moreover, it is bounded in the north by Ikom Local Government Area (LGA) and Iyala; in the south by Yakurr LGA; and in the west by Ebonyi State. Obubra is blessed with an abundant forest and mineral resources. Furthermore, these mineral resources include: gravel, lead ore, salt deposit, and, as mentioned earlier, baryte ore. Moreover, Fig. 2a and b give the location map of the study area within Cross River State, and the base map of Calabar South Local Government Area of Cross River State, identifying the study area.

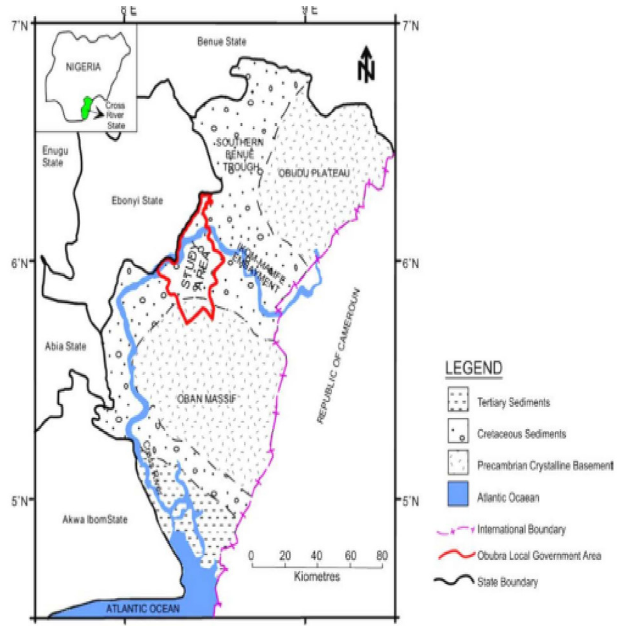

Fig. 2a: Location map of the study area within Cross River State. Modified after Kudamnya and Andongma [12] and Kudamnya et. al. [13].

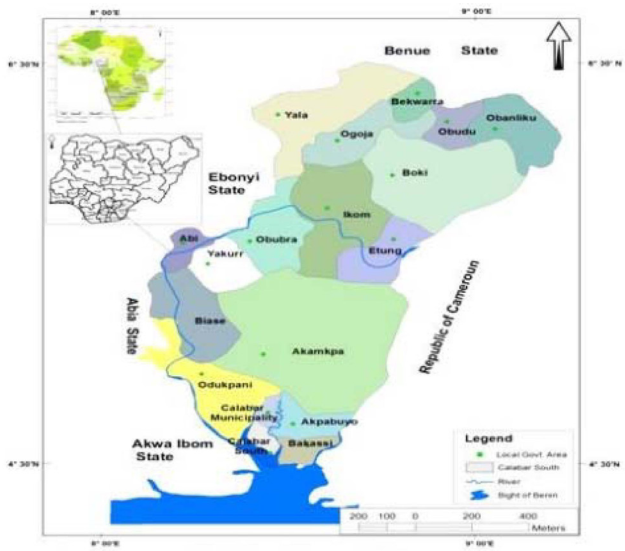

Fig. 2b: Base-map of Calabar South Local Government Area of Cross River State, Geography/GIS unit [14].

Materials and Methods

Sample Collection

Baryte ore samples from 5 sites of Obubra baryte mine were transported to Qualchem Global LTD. Furthermore, in Rivers State for the analyses, the samples were first washed clean with water, after which they were oven dried at 50 °C. The samples are then subjected to a series of comminution unit operations: crushing, grinding, and milling operations to reduce the ores from one average particle size to smaller particle sizes, which are less than 75mμ. Moreover, the crushing and grinding were carried out by a locally fabricated hammer and pan, while sieving was done using a digital sieve shaker with a 75μm sieve (no 200) sieve placed on the shaker. Each action, from washing to sieving, was handled separately for each sample.

Fig.s 3a- 3j show the 5 baryte samples before and after grinding. While Fig.s 3k-3m give the pictorial representation of the hammer/pan, the digital sieve shaker, and the zoom image of the 75μm sieve (no 200). Moreover, Fig. 3k: represents the diagram of the locally fabricated hammer/pan used in crushing and grinding the ores. In addition, Fig. 3L represents a digital sieve shaker, and Fig. 3m. represents the zoom image of the 75 μm sieve (as you seen below).

Figure 3a, Site 1: Baryte ore

Figure 3b, Site 1: Grinded baryte

Figure 3c, Site 2: Baryte ore

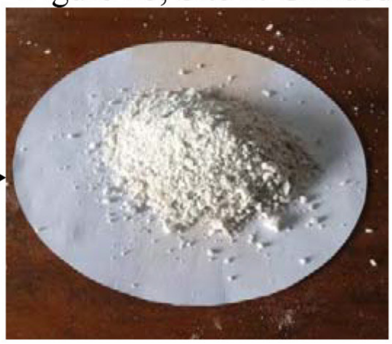

Figure 3d, Site 2: Grinded baryte

Figure 3e, Site 3: Baryte ore

Figure 3f, Site 3: Grinded baryte

Figure 3g, Site 4: Baryte ore

Figure 3h, Site 4: Grinded baryte

Figure 3L: A digital sieve shaker.

Figure 3m. Zoom image of the 75 µm sieve.

The Physical Appearances

The appearance of the unpulverized sample was assessed by visual inspection to determine its color and by tactile evaluation to assess its texture.

Determining the Streak

The streak test was carried out by dragging a chosen protrusion of the unpulverized baryte ore sample onto the backside of a glazed tile serving as the streak plate. The dragging resulted in the production of a small quantity of a powdery substance on the glazed tile. Moreover, this substance is described as a "streak". In addition, the hand and eyes were employed in establishing the texture and color of the streak. Moreover, this action was performed in triplets on each of the 5 baryte ores using different points of the baryte at each action.

Determining the Moisture Content

The milled baryte from each site was weighed using an analytical balance, and the five samples were oven-dried at a temperature of 110 °C for 30 minutes. At the end of the drying time, the samples were taken out from the oven and carefully placed inside a desiccator to cool for roughly an hour. Additionally, the cooled samples were measured for their dried weight. Moreover, the differential weights of each sample (before drying and after removal from the desiccator) were calculated using an analytical weighing balance with an accuracy of ±0.01 g. In addition, the whole process was repeated thrice until no further weight differential was observed, Ibe et al. [15].

The moisture content was calculated using Equation 1, as described by Philips and Paul [2].

$$\text{Moisture } (\%) = (w_i - w_f)/w_i \times 100\% \quad (1)$$

where W_i is the initial weight of the sample and W_f is the Final weight of the sample.

Determining the Hardness

The 5 fully unpulverized Obubra baryte samples were suitably cleaned, and each of them was scratched on the surface with the edge of a coin, resulting in the production of an etched line. In addition, the use of the fingernail determined the nature of the scratch. If the baryte 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 to relate the hardness of a mineral specimen to a number from Mohr's scale, Alan [16]. In addition, the scale was defined using an assembled set of common reference minerals of varying hardness, and these were labelled in order of increasing hardness from 1 to 10. Similarly, the samples were also scratched against a knife, a glass, and a fingernail. The observed results were evaluated on Mohr's scale of mineral hardness, Ibe et al. [15]; Omoniyi and Mubarak [17]. Moreover, Mohr's scale consists of a set of common reference minerals of varying hardness labelled in order of increasing hardness from 1 to 10.

The reference minerals of the Mohr's scale are as follows: Talc: 1, Gypsum: 2, Calcite or Penny 3, Fluorite: 4, Apatite: 5, Orthoclase: 6, Quartz: 7, Topaz: 8, Corundum: 9, Diamond: 10. Table 1 gives an expanded table of the Mohr's hardness scale.

Table 1 Mohr scale, Alan [16].

Hardness	Substance or mineral
1	Talc
2	Gypsum
2.5-3	Gold, Silver
3	Calcite, Copper penny
4	Fluorite
4-4.5	Platinum
5	Iron
6	Orthoclase
6.5	Iron pyrite
6-7	Glass, Vitreous, Pure silica
7	Quartz
7-7.5	Garnet
7-8	Hardened steel
8	Topaz
9	Corundum
11	Fused zirconia
12	Fused alumina
13	Silicon carbide
14	Boron carbide
15	Diamond

Determining the pH Values

A slurry was formed for each of the sieved samples by mixing 5 g with 10 ml of distilled water contained in a beaker. After standing the mixture for approximately 45 minutes, it was stirred for about 3 minutes to assess the pH value of the baryte, as reported by Ibe et al. [15]. Moreover, the pH value was then measured after a 24hrs as reported by Ibe et al. [15].

Determining the Specific Gravity

The Le Chatelier flask technique was used to determine the specific gravity (SG) of the Obubra sample. In addition, the procedure followed was API Specification 13A [18]. Also, 80 gr of each sample was used for each test.
The SG of the baryte (ρ) was calculated in grams per milliliter by applying Equation 2:

$$\rho = \frac{M}{V_{F - V_I}} \quad (2)$$

where m is the sample mass (g);
V_i represents the initial volume (ml);
V_f represents the final volume (ml).

Results and Discussion

Physical Appearance

A close observation of the baryte ores shows that baryte from site 3 is bright and practically white, except for a small brown patch on one of the pulverized portions. Baryte from sites 1, 2, and 4 exhibits a combination of white and ash/gray colors with a bright appearance. On the other hand, samples from site 5, though they appeared bright, were largely ash in colour. Moreover, pure baryte is plain white to milky white in color, Philips and Paul [2]. Thus, the deviation of the color from its natural pure state can be attributed to varying levels of associated minerals (in the form of impurities) with the baryte ore (Nzeh and Popoola, [19]). According to [19], these impurities tend to reduce the specific gravity (SG) of the unprocessed baryte. Thus, this can explain the low level of SG (3.37) of sample 5. When the samples were ground, the texture of the samples ranged from fine–grained to very coarse-grained.

Streak Test

This test produced a white streak. An ore that gives a white color streak is an indication that the substance is possibly baryte, as noted by Imasuen et al. [20]. The streak test resulted in a baryte powder with a white color for all 5 samples. Moreover, this agreed with similar results obtained by Imasuen et. al. [20] on baryte analysis from Guma Local Government Area, of Benue State, Nigeria.

Moisture Content

At the end of the moisture content experiment, the computed differential weights of all the samples ranged from 0.01%-0.02%. Also, this value was within the API moisture content specification of ≤ 1%, Afolayan et al. [21].

Hardness

The sample recorded a mean value of 3.5 using Mohr's hardness scale. The hardness value can be attributed to the nature of the gangue or associated minerals in the bartye sample (as reflected in the varying colors and textures of samples). The practical implication of the baryte samples having a Mohr's hardness of 3.5 means that it can satisfactorily give protection to the drilling bit as well as safeguard the circulation system during oil and gas drilling operations, Abdou et al. [22]; Afolayan, et al. [21]; Obot, and Oyeade [23]; Philips and Paul, [2].
Table 2 presents the results of the average specific gravity (SG) of each of the five baryte samples.

pH

Table 2 shows that the 5 samples were all within the pH range of 7.0-7.4, indicating the basicity of the samples. These values indicate that the 5 samples were within the API specification range (7-12.5) for use as a weighting agent for drilling, as reported by Osokogwu et al. [24] and Ádewale and Salihu [25]. These results were equally similar to those obtained by Oahimire et al. [9] in assessing some baryte ores from Northern Cross-River, Nigeria, for use as oilfield drilling fluid enhancement.

Specific Gravity

Table 2 presents the specific gravity (SG) of each of the 5 baryte samples. As shown in Table 1, sample 3 recorded the highest SG at 4.32, while the lowest SG was recorded by sample 5 at 3.37. Overall, the order of SG, from highest to lowest, is sample 3 > sample 2 > sample 1 > sample 4 > sample 5. Pure baryte is plain white to milky white in color, Philips and Paul [2]. Moreover, a close observation of the baryte samples shows that sample 3 is the closest to this color range (excluding the single brown patch). Hence, it recorded the highest SG. Moreover, this relationship between the coloration and the SG can be extended to another ore sample in the selected sites. Also, the grain color of site 5 is practically ash in color. In addition, this can be attributed to a high presence of several associated minerals in the form of impurities. According to Nzeh and Popoola [19], these impurities tend to reduce the SG of the unprocessed baryte. Thus, for sample 5, the lowest SG of 3.37 has been recorded.

Table 2 Results of the physical properties of each of the 5 baryte samples.

No.	Baryte Sites	Ore Color	Grain Texture	MC (%).	Hardness	pH	SG
1	Site 1	White-ash	Fine–grained	0.00	3.5	7.2	4.25
2	Site 2	White-Ash-brown	Coarse-grained	0.01	3.5	7.4	4.28
3	Site 3	White with a small brown patch	Coarse-grained	0.01	3.5	7.0	4.32
4	Site 4	White-Ash-brown	Very-coarse grained	0.00	3.5	7.2	4.12
5	Site 5	Ash-white	Fine–grained	0.00	3.5	7.4	3.37

Conclusions

This research demonstrates that the baryte ore in the Obubra mine meets the API's physical specifications for oil and gas drilling operations in Nigeria.

The use of Obubra baryte by oil and gas companies operating within Nigeria will ultimately reduce Nigeria's overreliance on imported barite by oil companies, resulting in a significant increase in its foreign exchange.

Recommendations

1. More research should be conducted at other baryte mines identified within Cross River State to fully ascertain their physical properties.
2. Since specific properties, such as SG, are a function of depth, these experiments should be conducted at varying depths.
3. In cases where these properties fall below the API requirement, various processing techniques should be investigated to bring them up to the API standard.

These studies should be extended to other identified baryte mines in Nigeria that have yet to be explored.

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