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Investigating the effects of elutriation in upgrading middle-grade barite ore from Obubra, Cross River State, Nigeria for drilling mud applications

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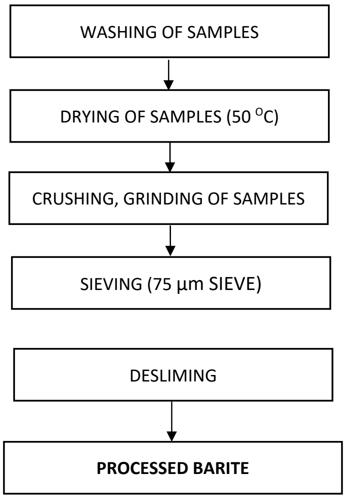
HIGHLIGHTS

- The upgrading of middle-grade barite ore by the process of elutration was studied.
- Elutriation of barite by desliming improves the physical properties for its applications in the oil and gas industries.
- The physical and mineralogical properties of the processed barite were investigated.

GRAPHICAL ABSTRACT



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ABSTRACT

Obubra's local government, located in the Cross River State of Nigeria, possesses commercial quantities of barite ores. These ore possess various forms of processing challenges. This paper presents research carried out to evaluate the efficacy of desliming to improve the physical properties of Obubra barite ore for its applications in the oil and gas industries. The desliming was carried out via elutriation using a floccumatic machine. The desliming parameters used in the experimental design were selected based on related literature; the factors and levels used included flowrates of 25.2 and 42.50 ml.s^{-1} and agitation times of 20 and 30 min. The physical and chemical responses were evaluated after each operation to evaluate the efficacy of each processing step. The optimal specific gravity after desliming was 4.18 g.cm^{-3}, up from the raw value of 4.14 g.cm^{-3}; the fineness or grain size remained the same at 75 µm; the moisture content was within the values of 0.01-0.02% before and after the desliming process; while the pH value decreased to 7.06, from the raw value of 7.3. These results showed that Obubra barite ore can be upgraded by elutriation to meet the required American Petroleum Industry (API) specifications for use in the formulation of drilling mud.

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1. Introduction

It is estimated that 80% to 90% of the world's barite is used as a weight additive to control the formation pressure during oil and gas exploration [1,2]. Nigeria possesses an enormous quantity of barite ore deposits, which are hosted in various rock types such as magmatite, limestone, sandstone, mudstone, shales, and granite [3]. As a major oil and gas producing nation, barite is thus an essential mineral for the success of oil and gas drilling operations in Nigeria. Nigeria's estimated reserve of over 22,298,843 tons exceeds its national demand of 10,000,000 tons per annum [3,4]. Significant barite deposits are present in the nine Nigerian states [5], including Adamawa, Benue, Cross River, Ebonyi, Gombe, Nasarawa, Plateau, Taraba, and Zamfara [3].

Despite Nigeria's surplus barite reserves, the country's multinationals oil companies (which operates mainly) in its Niger Delta region, are largely dependent on the importation of barite from other countries for drilling operations because of Nigeria's inability to exploit the use of globally tested technologies to improve its barite specifications to meet required global standards [6]. Thus, the importation of barite constitutes a drain on Nigeria's foreign reserve. According to the Association of Miners and Processors of Barite (AMAPOB), Nigeria loses an excess of over ₦5 billion ($3.5 trillion American dollars) yearly in foreign exchange as a result of barite importation [7].

Froth flotation, a versatile method for the physicochemical separation of valuable minerals from their associated minerals (gangue), is based on differences in the ability of air bubbles to selectively adhere to specific mineral surfaces in a mineral/water slurry.

The use of froth flotation is often associated with the production of fine gangue mineral particles, collectively referred to as "slimes". Slimes exert many detrimental effects on flotation efficiency, such as increases in reagent consumption and pulp viscosity. Slimes are equally liable to entrain into froth products [8].

The options for the treatment of slimes are partially based on the assumption that for a given particle size distribution, the ore contains certain minerals from which the valuable mineral needs to be liberated. When the unwanted minerals are, in fact, gangue minerals, a method referred to as desliming presents a viable and effective technique.

Desliming can be carried out with a hydrocyclone, by manual screening with sieves or using a digital sieve shaker, as well as by the process of elutriation. Elutriation is a process for separating a mixture of minerals into two or more products by utilizing the difference in particle settling velocity [9].

Previous works on the desliming of barite and other minerals [8,10-14] have mainly focused on the effects of desliming as a precursor to froth flotation. Little literature exists on the sole effect of desliming on minerals without recourse to froth flotation.

This research focuses on investigating the effects of desliming through elutriation on Obubra barite ore located in Cross River State, Nigeria, to upgrade Nigeria's barite ore to globally acceptable specifications to attract both local and foreign investment.

2. Experimental

2.1. Materials and Methods

2.1.1. Features of the study area

Fig. 1 gives the map of Cross River State with the highlighted study area, the Obubra local government area (LGA). The Obubra LGA is located in the central senatorial district of Cross River State, Nigeria, with a land mass of 1115 square kilometers. The 2006 census puts its population at 200,000. It is bounded in the north by Iyala and Ikom LGA, in the south by Yakurr LGA, and in the west by Ebonyi State. Together with its forest resources, Obubra is blessed with a great deal of mineral resources such as lead ore, gravel, salt deposits, and, as mentioned earlier, barite ore. The barite ore reserves at Obubra are about 9,660,306 metric tons [15].

2.1.2. Sample collection

2003.2 g of middle-grade Obubra barite ore sample with a tested specific gravity of 4.14 g.cm^{-3} was collected from Qualchem Global Nigeria Ltd. The sample was bagged and transported to the PTDF Bio-oil research laboratory ETF Building, Abuja campus, Uniport, Rivers State.

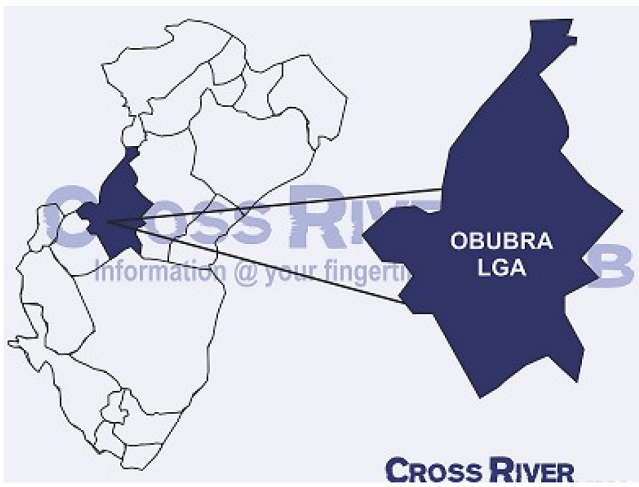

Fig. 1. Map of Cross River State of Nigeria highlighting the study area, The Obubra LGA [16].

2.1.3. Sample preparation

The sample ore was washed with water and then oven-dried at a constant temperature of 50 °C for a week [17]. The ores were then reweighed until a constant weight of approximately 2000 g was obtained. The dry barite ore was then crushed into smaller pieces using a clean, locally fabricated hammer crusher. The ores were further ground to particles ranging from sizes 300 μm to 1mm using the face of a locally fabricated hammer. The barite ores were then hand-milled by a combination of scrubbing and grinding actions using the cheek of the same hammer. The comminution exercises reduced the quantity of the Obubra barites to 1630.20 g. After milling, the barite ore was manually sieved using a 75 μm sieve (No. 200 mesh). The milling and sieving actions continued until 1500 g of the ore with grain sizes of less than 75 μm were obtained. Fig. 2 shows a sample of the sieved barite of 75 μm obtained after milling.

Elutriation was selected to upgrade the ore samples. Elutriation was conducted with two factors at two levels (25.20 and 42.50 ml.s^{-1}; agitation time: 20 and 30 min) as shown in Table 1, and the factorial method (using Design expert software, version 13) was used for the desliming experiments. The desliming experiment consisted of eight runs performed in duplicates, which were conducted using a floccumetric machine, two desliming buckets, and two tubes of varying lengths and widths. The flow rates were equated to filling up a 500 ml beaker in 19.8 and 12 sec to obtain a flow rate of 25.2 and 42.5 ml.s^{-1}, respectively. The flow rate selection was based on a study on how desliming affects the flotation response of Kansanshi mixed copper ore [14].

In each of the (8) runs, a 110 g test sample was placed in a plastic bucket containing an opening at the bottom through which water was injected at predetermined flow rates of 25.2 and 42.50 ml.s^{-1}. The upward flow of water, allowed to run for 30 min, transported a portion of the barite solids into the overflow fraction. The water flow was shut off after 20 and 30 min (as seen in the experimental design), and the underflow fractions from both fractions were allowed to settle (on the bottom) for 20 min before decantation. The decanted barite from both buckets was added to arrive at a single weight for each run. For each run, the deslimed barite was dried in a muffle furnace at 110 ^{o}C and weighed at 20 min intervals until a constant weight was observed. The dried caked slurry (in the form of lumps) formed after drying in the muffle furnace was milled to obtain fine particles of barite, as it was after the initial milling. Finally, a 75 μm sieve (No. 200 mesh) was used to obtain barite particles of less than 75 μm in size. At the end of the desliming operation, each run's responses were evaluated, including the specific gravity, fineness (grain size), moisture content, pH, and hardness. Fig. 3 shows the experimental setup for desliming the Obubra barite sample.

2.1.4. Determination of the physical properties of the Obubra barite sample

2.1.4.1. Determining the specific gravity

The specific gravity was calculated using the Le Chatelier method. The Le Chatelier method is performed using a Le Chatelier flask, see Fig. 4. Briefly, about 200 g of the

Table 1. Desliming condition by factorial experimental designs.

Runs	Factor 1 Flow rate (ml.s^{-1})	Factor 2 Agitation time (min)
1	25.20	30
2	42.50	30
3	42.50	30
4	42.50	20
5	25.20	30
6	25.20	20
7	25.20	20
8	42.50	20

Fig. 2. A sample of the Obubra barite (size 75 μm) obtained after milling.

Fig. 3. Experimental setup for desliming the Obubra barite sample.

Fig. 4. Diagram of the Le Chatelier flask.

pulverized barite (<75 μm) was dried in a muffle furnace for an initial period of 30 min. The sample was taken out intermittently after 30 min and weighed with a digital balance until a constant weight was achieved. The Le Chatelier container was then filled with kerosene to the 0.1 mark, corked, and placed in a water bath at a stable temperature of 31.6 ^{o}C to register the maximum kerosene expansion. After half an hour, the Le Chatelier flask was removed from the water bath, the kerosene volume was observed, and the graduated flask was read and recorded. This volume is referred to as the primary volume (V_1). Then, 80 g of the dried barite is poured into the Le Chatelier flask. The flask was closed by inserting the bulb into the flask, and the flask was again placed in the water bath for a period of 30 min. After removing the flask from the bath, the height of the kerosene was reobserved and recorded as the secondary volume (V_2) in millimeters.

The barite specific density (ρ) was calculated in grams per milliliter, according to Eq. (1):

$$\rho = m / (V_2 - V_1) \quad (1)$$

where m is the sample mass (g); V_1 is the initial volume (ml); and V_2 is the final volume (ml).

2.1.4.2. Determining the fineness (grain size)

The grain size of the pulverized 75 μm barite sample was obtained using a 75 μm sieve with a No. 200 mesh. The pulverized barite sample was gently agitated to allow barite of 75 μm and less to pass through the sieve onto a clean pan, after which it was collected and weighted.

2.1.4.3. Determining the moisture content of the Obubra barite sample

The deslimed barite (in the form of slurries) was put into three empty, dry, and clean crucibles and placed in a muffle furnace at 110 °C for 45 min. At the end of the heating period, the crucibles (housing the barite) were removed from the muffle furnace and kept in a desiccator to cool for about an hour. After removal from the dissector, the cooled sample was measured, and that value was considered the dried weight of the samples. The differential weights of the samples were compared and computed using an analytical weighing balance. The process was repeated until a net weight loss of 0.00-0.02 g was obtained. The whole process was repeated for each of the eight runs. An additional experiment to examine the effect of long-time storage on the moisture content was conducted by leaving an unpulverized ore sample in a desiccator for 14 days.

2.1.4.4. Determining the pH value

20 g of the barite sample was mixed with 350 ml of water, and its pH was measured using a Hanna instrument (HI98129 Model) pH meter by placing the probe of the meter into the solution and taking the reading.

3. Results and discussion

3.1. Percentage of each sample run recovered after desliming

Each of the eight runs was carried out with 120 g of each Obubra sample. Table 2 shows the percentage of each sample recovered after desliming. From Table 2, run 5 recorded the highest barite recovery (80.12%) after desliming, with the lowest recovery (69.17%) recorded in run 1. Overall, the order of runs, starting from the highest to the lowest, is:

Table 2. Percentage of each sample run recovered after desliming of the Obubra barite samples.

Runs	Quantity of recovered sample per run (g)	Percentage of recovered sample per run (%)
1	83.00	69.17
2	89.90	74.92
3	90.10	75.10
4	87.00	72.50
5	96.20	80.12
6	83.10	69.25
7	94.70	78.92
8	94.70	78.98

run 5 > run 8 > run 7 > run 9 > run 2 > run 4 > run 6 > run 1

This result showed that roughly one-third of the barite was lost during elutriation of barite at size 75 μm.

3.2. Physical responses as shown in the design of experiment (DOE) after desliming

Table 3 shows the measured physical responses to the experimental design after desliming of the Obubra barite samples.

3.2.1. Specific gravity

From Table 3, the optimal response value in specific gravity for the deslimed Obubra barite samples, as indicated by the highest specific gravity value of 4.18 g.cm^{-3}, occurs at a flow rate of 42.50 ml.s^{-1} and an agitation time of 30min. This indicates a rise in specific gravity of 0.04 g.cm^{-3} from the initial specific gravity of the unprocessed ore of 4.14 g.cm^{-3}.

3.2.2. Fineness (grain size)

The fineness results in Table 3 show that the grain size remained at 75 μm after the desliming processes, indicating that desliming has no effect on the grain size even after the earlier milling and sieving processes.

3.2.3. Moisture content

After drying each sample from each run at 110 ^{o}C for a cumulative time of 12 h, a range of 0.00 to 0.02 g values in moisture content were obtained, as seen in Table 3, signifying the near absence of moisture in the sample [18]. Compared with API standards, the moisture content values of the study's barite samples are within the API maximum value of 1%. A moisture content above the API standard of 1% will affect the mud viscosity negatively and may result in the collapse of the borehole due to varying downhole conditions [19]. Similar experimental observations have been recorded [20-22]. Edem *et al.* obtained a moisture content of 0.01g in their analysis of Azara barite [23]. Another experiment examining the effect of long-time storage on barite samples' moisture content showed a negligible effect between the moisture content and long-time storage of the pulverized barite. This was probably due to storing the dried deslimed barite particles in a desiccator, thus preventing the moisture content in the atmosphere from coming in contact with the barite. This result agrees with research work by Afolayan *et al.* [19], which showed that long-term storage has no significant effect on the quality of processed barites.

3.2.4. pH value

The results in Table 3 show that the deslimed Obubra barite sample was approximately alkaline, ranging from 6.81 to 7.06 pH. The pH of the optimal value of the deslimed process (i.e., the value corresponding to the highest specific gravity) was 7.06, which is well within the API standard range for drilling weighting agents (7 or ≤ 12.5) [24]. The results show that all eight desliming runs were within the standard API range of a drilling weighting agent. Therefore, it can be concluded that the effect of flow rate and agitation time of the spindle on the pH of the Obubra barite sample is negligible.

3.3. Application of the initial optimal desliming variables on four middle-grade samples

Applying the optimal variables (flow rate: 42.50 ml,

Table 3. Results of the design of experiment (DOE) for desliming of the Obubra barite samples.

Runs	Factor 1	Factor 2	Response 1	Response 2	Response 3	Response 4
	Flowrate (ml.s^{-1})	Agitation time (min)	Specific gravity (g.cm^{-3})	Fineness (μm)	Moisture content (%)	pH
1	25.20	30	4.14		0.02	6.96
2	25.20	30	4.14		0.01	6.89
3	25.20	20	4.14		0.00	6.97
4	25.20	20	4.14	<75	0.02	6.98
5	42.50	30	4.18		0.01	7.06
6	42.50	20	4.16		0.00	6.81
7	42.50	30	4.14		0.02	7.02
8	42.50	20	4.18		0.01	7.02

agitation time: 30 min) on the four other low-grade Obubra barite samples produced results validating the use of the optimal variables. The results in Table 4 signify an increase in the specific gravity within the margins obtained in the experimental design.

Results from Table 4 validated elutriation's capacity to increase the specific gravity of the Obubra barite samples at a flow rate and agitation time of 42.50 ml.s^{-1} and 30 min, respectively. The highest differential rise (of 0.08) in specific gravity was recorded by sample 3 (4.00-4.08).

Table 4. Application of the initial optimal desliming variables on four middle-grade barite samples.

S/N	Specific gravity before desliming (g.cm^{-3})	Specific gravity after desliming
1	3.95	3.99
2	3.98	3.98
3	4.00	4.08
4	4.04	4.05

3.4. Mineralogical analysis

The X-ray diffract gram (XRD) of the deslimed Obubra barite sample, as shown in Fig. 5, reveals the composition and crystallographic information of the deslimed Obubra barite sample, respectively. From the analysis of the XRD patterns of the barite sample, several peak values were observed, with quartz having the highest peak value at 27.20, 2θ at an intensity of 3000 cps. The quantitative result of the deslimed Obubra barite sample shown in Fig. 6 indicate that barite and quartz are still the most dominant compounds after desliming.

When compared to the XRD analysis of the unprocessed Obubra barite, we can see that there was a slight reduction in the kaolinite content from 1% to 0.9%; muscovite reduced from 2.6% to 1.2%; hematite reduced from 7% to 5%, while illite and garnet dropped from 0.1% to 0.002% and 10% to 1%, respectively [23]. Summarily, the concentration of the gangue minerals in the sample decreased slightly after the desliming process.

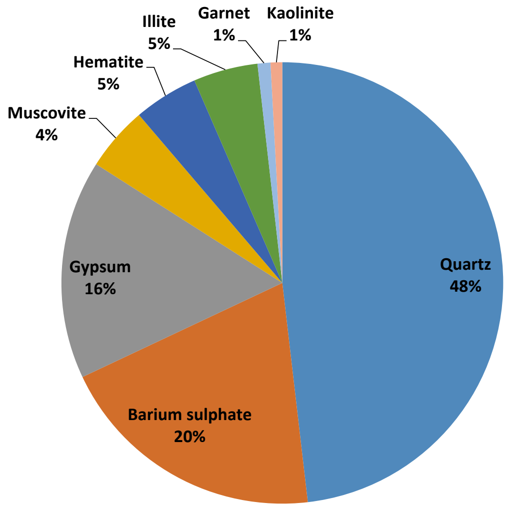

Fig. 6. Pie chart representation of the quantitative result of the deslimed Obubra barite samples.

The pie chart representation of the quantitative result of the deslimed Obubra barite sample indicates that quartz or silica oxide (SiO_2) and sulfate of barium ($BaSO_4$) are the most dominant compounds at 48% and 20%, respectively; other gangue minerals in traces included Gypsum ($CaSO_4 \cdot 2H_2O$) (16%); Muscovite ($KAl_2(Si_3Al)O_{10}(OH,F)_2$) (4%); Hematite (5%); Illite ($K(AlFe)_2AlSi_3O_{10}(OH)$) (5%); Garnet 3(Ca,Fe, Mg)O(Al,Fe) at 1%; Kaolinite (1%).

4. Conclusion

The results of the elutriation of Obubra barite shows that the obtained optimal specific gravity of 4.18 g.cm^{-3} (from an initial value of 4.14 g.cm^{-3}) was marginally below the API standard of 4.2 g.cm^{-3}, but well above Nigeria's Department of Resources (DPR) standard of 4.0 g.cm^{-3}, making it suitable as a drilling mud additive in Nigeria's oil and gas industry.

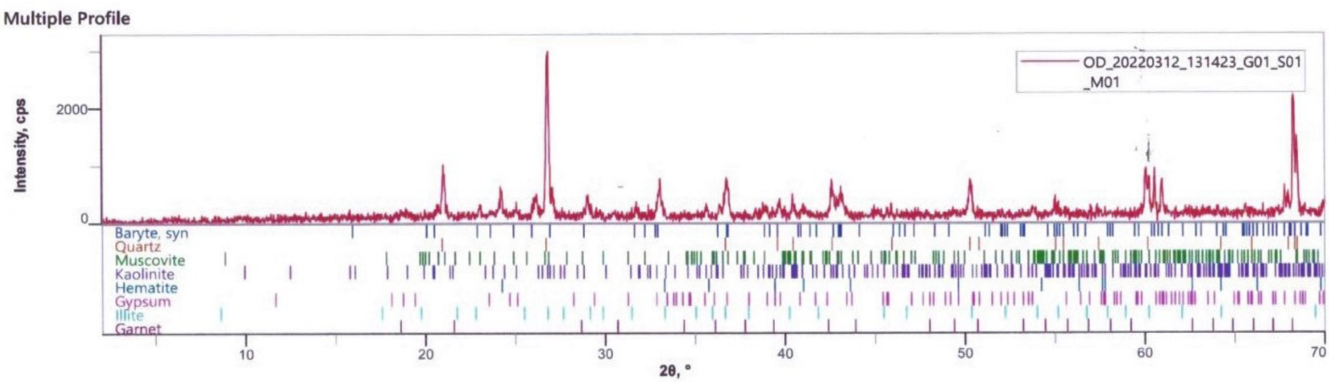

Fig. 5. XRD analysis of the Obubra barite sample after elutriation.

In terms of the moisture content, pH value and hardness tests carried out, Obubra barite is also of good grade and quality for a host of products such as paint, paper, as well as the preparation of several barium compounds.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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Additional information

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