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A Comparative Study on the Effects of Desliming on the Physical Properties of Barite Ore from Azara and Obubra areas of Nigeria, for Drilling Applications

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Abstract
This research compares the effects of desliming of barite ore from Azara and Obubra local government areas of Nasarawa and Cross river states of Nigeria respectively. Desliming was conducted with a view of separating the valuable barite from its unwanted or associated minerals in order to bring it (the barite) to the required specifications for use in oil and gas drilling operations. Desliming was carried out using a floccumatic machine. The factors and levels used in the desliming operation were flowrates at 25.2 ml.s^{-1} and 42.50 ml.s^{-1} and agitation times at 20 and 30 min respectively. The Obubra baryte and Azara desliming experiment consist of (8) runs each. The physical and chemical responses were evaluated after each run. The highest specific gravity value after desliming of Obubra barite was 4.18, up from the raw value of 4.14; while that of Azara was 3.80, up from an initial value of 3.45. The grain size remained the same at 75µm for both samples after desliming, while the moisture content of both samples was within the range of 0.01% before and after the desliming. The pH of Obubra decreased to an average value of 7.06, from the initial value of 7.3, while that of Azara experienced a 0.1 increase to 7.5 from 7.4. These results showed that while the deslimed Obubra barite practically meet the API physical requirements for drilling, the Azara barite, failed to meet the API specific gravity specification for oil drilling. Hence, for the Azara barite can be used in the production of paint fillers, ceramics, asbestos, glass, as well as a component for several other industries applications where barium compounds are required.

***Keywords:** Barite, Oil drilling, Desliming, Azara, Obubra, Specific gravity*

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1. Introduction

Nigeria is often classified as the leading crude oil producer in Africa, with an abundance of oil and gas reserves (Olujobi, 2021; Dhali et al., 2023). Nigeria is greatly dependent on its oil and gas sector for its socio-economic development. Studies revealed that Nigeria's oil and gas sector, generated 95% of the country's foreign exchange income (FOREX), and roughly about 80% of its budget revenue (Nwuke, 2023).

Barite is one of the primary sources and foremost ore of barium and its compounds. It's named from the Greek word 'barus' which means 'heavy'. Studies ascribe its name to its high SG of 4.0-4.5 (Nzeh, and Popoola, 2023), which is unique for a non-metallic mineral. Barite has several global industrial applications. However, it is very crucial in the oil and gas sector, where, roughly 80% of the global production of 44 million tons is being utilized by the petroleum industry in the form of a weighting (or heavy) agent for drilling operations. The remaining 20% is primarily utilized in the manufacture of barium chemicals (Nzeh and Hassan, 2018; Nzeh, and Popoola, 2023).

However, its primary usage is in the oil and gas sector where it is used in the formulation of drilling mud for oil and gas exploration. Nigeria possesses a huge reserve of barite scattered in different states. However, it is largely left unused or unprocessed when in used. In other cases, it is processed using unconventional means, which reduces its efficiency in drilling operations. The use of such unprocessed or poorly processed barite in drilling operations can result in several issues such as: corrosion of the drill bits, enormous rise in the wear debris as a result of an increase in the wear rate during drilling operations, etc. Consequently, the International Oil Companies (IOCs) operating in the country import over 440,000 metric tons of its barite for drilling operation. This however, results in a loss of over $96 million in Forex (Afolayan et al. 2021). Desliming ranks among one of the modern methods for processing mineral ores such as barite. Generally, ores constitute a collection of minerals, among which the mineral of interest lies. It is thus necessary to disassociate the mineral of interest from the ore, because quite often, these associated minerals lower the valuable content of the mineral of interest. Naturally occurring deposits of minerals hardly meet the specification required for various industrial applications. The process of desliming involves the separation of the valuable mineral from its associated mineral by the use of agitated water. The process facilitates the valuable mineral separation from deleterious materials. The mixture is left to settle for a chosen time, and then the unwanted mineral flushed away by the simple process of decantation - thus increasing the value of the mineral of interest.

Several chemical and physical techniques abound in processing of barite. These includes; Table shaking, jigging, desliming, magnetic separation, electrostatic separation, froth flotation, leaching, etc. However, only jigging, froth flotation and leaching, are often employed in the processing of Nigerian barite (Mgbemere et al. 2018; Mgbemere et al. 2019; Philips and Paul 2023). Mgbemere et al. (2018; 2019) and Philips and Paul (2023) conducted a laboratory-based studied using jigging, froth flotation, and acidic leaching, in the processing of barite from Azara. The results obtained, meet the required specifications for oil drilling operations. Thus, the objective of the research is to apply desliming as a barite processing technique on both Azara and Obubra barite, with a view of carrying out a comparative analysis on the physical properties of the deslimed barite from these two locations. Results of the physical properties were used to determine their suitability for drilling operations.

2. Materials and methods

2.1. Study area

Fig. 1 and 2 give the study areas of the research; Namely, Azara Development Area in Awe Local Government Council of Nasarawa State and Obubra in Obubra Local Government Area of Cross River State-both in Nigeria. A brief discussion of these study areas follows.

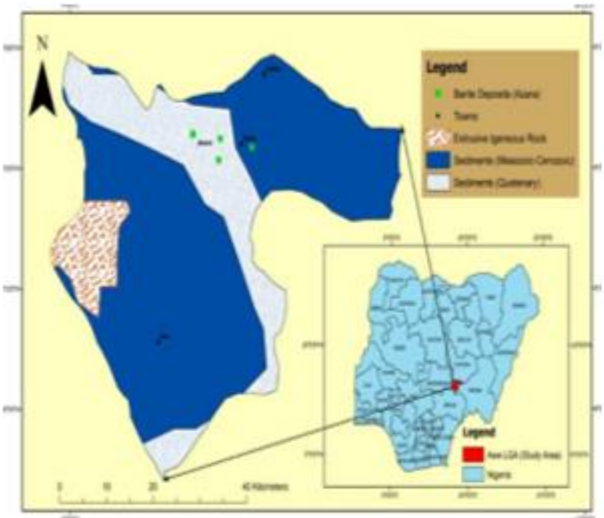

Fig. 1a: Map of barite deposit in Azara (Source: Kolawole, et al., 2021)

Fig. 1b: Map of Obubra in Cross River State (Ministry of Lands and Survey, 2008)

1. Azara Development Area in Awe local government council of Nasarawa State of Nigeria

Azara falls within 1:50,000 scale sheet 232 (Akiri NE) and is bounded by latitude $8^{0}19'00''$ to $8^{0}23'00'$and longitude $9^{0}16'00^{11}$ to $9^{0}20^{1}00''$. Azara development area in Awe local government council of Nasarawa State of Nigeria has an estimated barite ore reserve of about 731,000 tonnes with about 71,000 tonnes of good quality barites. The Azara barite deposit is located at about 98km south of Lafia the capital of Nasarawa State (Labe et al, 2018). The deposit which belongs to the Benue valley system can be described as a Vein and Cavity filling deposit with about 18 Veins. The deposit formed by precipitation from hot barium enriched fields in faults and fractures as a result of fluid mixing at reduced pressure and/or temperature. Sometimes the fluid dissolves the surrounding host rocks to form irregular replacement deposits, (Smith, 1978). Figure 1a shows the map of Azara development area in Awe local government council of Nasarawa State of Nigeria.

2. Obubra local government area of Cross River State, Nigeria

Fig. 1b shows the map of Cross River State indicating the second study area - Obubra local government area. Obubra Local Government Area lies on latitude 6° 05'N and longitude 8° 20' E. It is located in the central senatorial district of Cross River State, Nigeria, with a land mass of 1115 square kilometer. The 2022 local government news magazine puts the population of Obubra at 262,800 (Udama et al., 2024). 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 deposit and as mention earlier barite ore. Obi et al. (2014) puts the barite ore reserves in Cross River State at an estimate of roughly 9 million metric tons; thus, making Cross River State Nigeria's largest reserve of barite.

2.2 Sample collection

Roughly 2000g of Azara and Obubra barite ore samples of tested specific gravity 4.14 and 3.80 were collected from Qualchem global Nigeria Ltd. The samples were bagged and taken to the PTDF Bio-oil research laboratory, ETF Building, Abuja campus, located at the University of Port Harcourt, Rivers State for analysis.

2.3 Sample preparation

The samples, were cleaned, washed with water, and dried using a muffle furnace at a constant temperature of 50°C for a cumulatively period of 28 days. The ores were then reweighed until a constant weight of about 2000g was obtained for the Azara and Obubra respectively. The dry barite ores were then crushed, grinded and sieved to sizes of ≤ 75µm using a 75µm sieve (no 200). At the end of the sieving exercise, the quantity of barite was roughly 1500g. Figures 2a and 2b show the Pulverized samples of barite from Azara and Obubra respectively. Desliming was used to upgrade the ore sample at two factors; flowrate and agitation time. These factors were performed at two levels each. In the case of flowrate, the levels were 25.20ml/s and 42.50ml/s. While agitation agitation time was carried out at 20mins and 30mins. The factorial method (using the Designexpert version 13) software which was used to design various test conditions for the desliming experiments is shown in Table 1.

Fig. 2a: Pulverized sample of barite from Azara

Fig. 2b: Pulverized sample of barite from Obubra

Table 1: Desliming condition by factorial experimental designs and representative responses

Runs	Factor 1 Flowrate (ml/s)	Factor 2 Agitation speed (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

1. Determination of the physical properties of the samples

Desliming was undertaken using two factors, flowrate and agitation speed, at levels of 25.20ml/s and 42.50ml/s, and agitation time of 20mins and 30mins respectively (see Table 1). The desliming experiment which consisted of 8 runs, for both samples were performed in duplicates, using a floccumetric machine. The experimental set-up is shown in Figure 3. The flowrates were selected based on the work done by Phiri et al. (2019).

Fig. 3: Experimental setup for the desliming of the barite samples

The experimental runs for the Azara sample was first performed, after which the Obubra runs was carried out. In each of the 8 runs, 110g test samples was placed in a plastic bucket with an opening at the bottom through which water was injected at flowrates of 25.2ml/s and 42.50ml/s. The upward flow of water which ran for 30 minutes, transported a measure of the barite into the overflow fraction. After 20 minutes and 30 minutes, the water flow was stopped, while the underflow fractions from were allowed to settle (to the bottom) for 20 minutes before decantation was carried out. The decanted barite from both buckets were added up to arrive at a single weight for each run. For each run, the deslimed barite were dried in a muffle furnace at 110^0C while weighing at a regular interval of 20 minutes until no further change in weight was observed. The dried barite, which at this point had caked, was then grinding and milled to obtain the barite in powder form. As before, the 75μm sieve (no.200) was utilised to obtain milled barite which were ≤ 75μm.

2. Determination of the specific gravity in accordance with API Specification 13A (2019)

a) A Le Chatelier flask was filled roughly 22 mm (0.8 in.) below the zero mark with kerosene.
b) The flask was placed upright in a constant-temperature waterbath. The flask was left for 30 minutes in the waterbath after which it was taken out, thoroughly cleaned, dried and reweighed. The level of water in the bath was higher than the 24 mL graduation of the flask but below the stopper level.
c) Kerosene was then introduced into the clean and dry Le Chatelier flask and the Le Chatelier's flask, reintroduced into the waterbath.
d) The flask and contents were allowed to equilibrate for an hour. Using the magnifying glass with care to keep eyes at meniscus level, the volume at the lowest portion of the curved interface was read, and the initial volume recorded in milliliters, to the nearest 0.05 mL without removing the flask from the waterbath and recorded as *V*1. When the kerosene level was outside the −0.2 mL to +1.2 mL volume range after equilibrating, a 10 mL pipette was used to add or remove kerosene to bring it within this range. The flask was allowed to equilibrate for at least one hour and the initial volume, recorded in milliliters, as V_1.
e) The Le Chatelier flask was then removed from the bath, wipe dry, and the stopper further removed. Several lengths of tissue paper were rolled diagonally along the length of the dowel and this assembly was used as a swab to dry the inside neck of the flask without allowing the swab to come into contact with the kerosene in the flask.
f) A funnel was then inserted into the top opening of the flask and precisely 80.00 g ±0.05 g of dried baryte with the help of a spatula and weighed using a weighing balance before carefully transferred into the Le Chatelier flask. Care was taken to avoid splashing the kerosene or plugging the flask with baryte at the bulb. The mass was recorded, in grams, as *m*.
g) The neck of the Le Chatelier flask, is cautiously tap with the hands and swirled, to dislodge potential baryte samples from clinging to the walls of the flask as well as eliminating entrained air from the sample, without allowing kerosene to come into contact with the ground glass stopper joint of the flask.)
h) The flask was returned to the bath and left standing for 30 min.
i) The flask was removed from the waterbath and Step (h) repeated to remove any remaining air from the baryte sample.
j) On the final removal from the waterbath, the flask was dried on the outside using a piece of dry cloth. The level of the kerosene in the flask was noted and recorded in milliliters as the final volume V_2.

Fig. 4 shows the insertion of the funnel into the Le Chatelier flask.

Fig. 4: Insertion of the funnel into the Le Chatelier flask

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

$$\rho = \frac{m}{v_{2-v_1}} \quad (1)$$

where, *m* is the sample mass, expressed in grams; V_1 is the initial volume, expressed in milliliters; V_2 is the final volume, expressed in milliliters. ρ is the calculated specific density of the baryte sample.

3. Calculation—Baryte Density by Le Chatelier Flask Method

The baryte density, ρ, was calculated in grams per milliliter, according to Equation (1).

4. Determination of the fineness

The grain size of the pulversized 75μm barite sample was determined using a 75μm sieve No. 200

The baryte grain size of diameter equal to or less than 75µm in the samples, was obtained using the following procedure.

a) Approximately 2000g and 13000g of the Obubra and Azara powdered baryte samples (obtained from the dried, crushed and grinded baryte ores) were separately cooled to room temperature in a desiccator in batches.
b) The samples were transferred in batches to a 75µm sieve No. 200. A small brush was used to agitate the baryte particles for approximately 15mins, to ensure that particles of size 75µm and below passes through the 75µm size unto a clean pan positioned below the sieve.
c) The baryte powder remaining on the sieve after agitating for roughly 15mins (until and insignificant quantity passed through the 75µm sieve) was transferred to a second clean pan
d) The process was repeated until all the baryte powder had been sieved.
e) The total sieved baryte powder (equal to or less than 75µm) was added and weighed with a weighing balance.

5. Calculation-baryte fineness

The mass fraction of baryte powder greater than 75µm, w_1, in percent, according to Equation (2):

$$w_1 = 100\left(\frac{m_2}{m_1}\right) \quad (2)$$

where, m_1 is the sample mass, expressed in grams; m_2 is the powder mass retained by 75µm sieve, expressed in grams. Record the calculated value.

6. Determination of sample pH

A Hanna instrument (HI98129 Model) was used to ascertain the pH value of the baryte sample using the following procedure:

a. A 20g sample of the sieved baryte was placed in a beaker, and 20ml of distilled water was added to it.
b. The emerging slurry formed was left standing for 60minutes and then stirred for an estimated 10mins
c. The probe of the Hanna instrument was afterwards dipped into the slurry until the meter stabilized and the pH reading then read from the instrument.
d. The pH was measured repeatedly after 24h and specifically at the temperature range of 26.3°C in line with the pH manufacturer's instruction manual (Washington State Department of Transportation (WSDT, 2009; Ibe, et. al., 2016).

7. Calculation-measuring baryte pH

The pH readings from the Hanna instrument (HI98129 Model) were recorded for all samples.

8. Determination of sample moisture content

a. the weighing and recording of an empty container
b. 10g barite sample was placed in the empty container and weighed.
c. The sample + container was dried in muffle furnace at 100^0C for 3 hours.
d. After 3 hours, the barite + container was removed, weighed and recorded.

Moisture content analysis was calculated using the formula in Equation (3)

$$\frac{w_2 - w_3}{w_2 - w_1} \quad (3)$$

where, w_1 - Weight of empty container; w_2 - Weight of container and sample before drying; and w_3- Weight of container and sample after drying.

3. Results and discussion

3.1 Percentage of each sample run recovered after desliming of both samples

Table 2 and Fig. 5a and 5b offer the percentage of each sample recovered after desliming of the Azara and Obubra samples. In the case of Azara, run 6 recorded the highest recovery of barite after desliming at 81.60%. While the least recovery was recorded at run 3 with 67.48%. Overall, the order of runs starting from the highest to the least is run 6 > run 8 > run 7 > run 5 > run 4 > run 1 > run 2 > run 3. Figure 5a clearly gives the highest recovery as 81.60% (run 6) and the lowest recovery as 67.48% (run 3). In the case of Obubra, run 5 recorded the highest recovery of barite after desliming at 80.12%., while the least recovery was recorded at run 1 with 69.25%. Overall, the order of runs starting from the highest to the least is run 5 > run 8 > run 7 > run 9 > run 2 > run 4 > run 6 > run 1. As follows, figure 5b clearly gives the highest recovery as 80.12% (run 5) and the lowest recovery as 69.17% (run 1).

Table 2: Percentage of each sample run recovered after desliming

Runs	Quantity of recovered sample per run (g)		Percentage of recovered sample per run (%)	
	Azara	Obubra	Azara	Obubra
1	340.10g	83.00g	71.53	69.17
2	326.52g	89.90g	68.74	74.92
3	320.53g	90.10g	67.48	75.10
4	352.50g	87.00	74.21	72.50
5	358.00g	96.20	75.37	80.12
6	387.60g	83.10	81.60	69.25
7	375.00g	94.70	78.94	78.92
8	375.00g	94.70	80.86	78.93

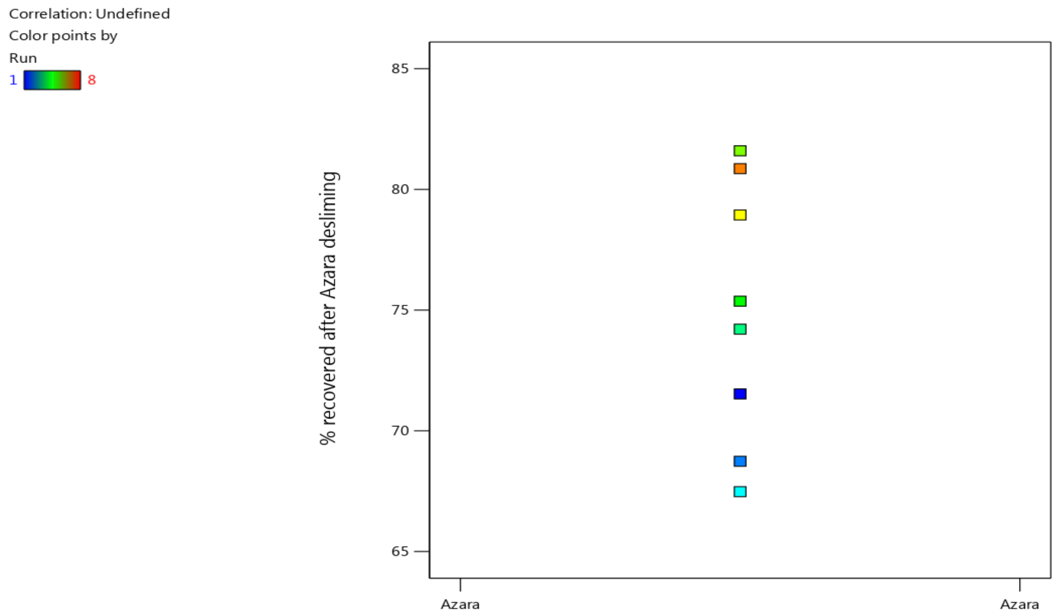

Fig. 5a: Percentage recovery from each desliming run drawn using (Designexpert version 13)

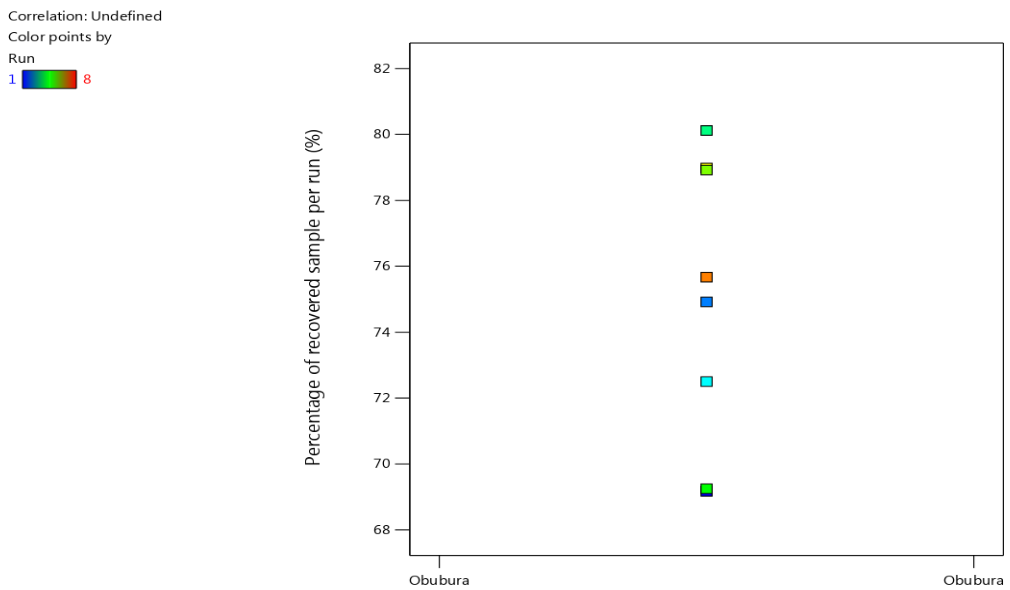

Fig. 5b: Percentage recovery from each desliming run drawn using (Designexpert version 13)

3.2. Physical properties of the both barite samples after desliming

Table 3 gives the physical properties of the barite ore samples analysed; specific gravity, grain size, and moisture content.

Table 3: Physical properties of the barite ore samples

Runs	Factor 1 Flowrate (ml/s)	Factor 2 Agitation speed (min)	Response 1 Specific Gravity		Response 2 Fineness		Response 3 Moisture content		Response 4 pH	
S/N	Azara/ Obubra	Azara/ Obubra	Azara	Obubra	Azara	Obubra	Azara	Obubra	Azara	Obubra
1	25.20	30	3.73	4.14	<75mµ	<75mµ	0.01	0.00	7.5	6.96
2	25.20	30	3.74	4.14	<75mµ	<75mµ	0.01	0.02	7.4	6.89
3	25.20	20	3.80	4.14	<75mµ	<75mµ	0.00	0.01	7.5	6.97
4	25.20	20	3.55	4.14	<75mµ	<75mµ	0.01	0.01	7.5	6.98
5	42.50	30	3.58	4.18	<75mµ	<75mµ	0.01	0.00	7.6	7.06
6	42.50	20	3.60	4.16	<75mµ	<75mµ	0.00	0.02	7.5	6.81
7	42.50	30	3.64	4.14	<75mµ	<75mµ	0.01	0.02	7.4	7.02
8	42.50	20	3.46	4.18	<75mµ	<75mµ	0.01	0.01	7.5	7.02

a. Specific gravity

Table 5 shows the measured responses to the experimental design after desliming. From the Table, the optimal response value in specific gravity for the deslimed Azara and Obubra barite samples, as indicated by the highest specific gravity value of 4.18 occurs at a flowrate of 42.50 ml/s and an agitation time of 30mins. This indicates a rise in specific gravity of 0.3 and 0.04 from the initial specific gravity of the unprocessed ore, which was measured as 3.80 and 4.14. Respectively. Thus desliming had more effect on the Azara than the Obubra samples. This is possibly due to the effect that the Obubra sample was already much closer to the 4.5 specific gravity of barite in its pure state Ekwueme (2013). Unlike the Azara sample, which at 3.80, was much further.

b. Fineness (grain size)

Results of fineness as shown in table 5, showed that the grain size remained at 75µm after the desliming processes for both samples. This shows that the action of milling and sieving does not affect the grain size of both samples.

c. Moisture content

Drying both samples after desliming, showed that their moisture content for each run, revolves within 0.00-0.01g. These values were well within the API acceptable limit of 0.01g. A moisture content over 0.01g, can bring about the collapse of the borehole as a result of variations in the mud viscosity Afolayan et al. (2021). Similar experimental observations were recorded in researches carried out by Edem et al. (2022) registered a moisture content of 0.01g in its analysis.

d. pH value

The results in Table 5 showed that the pH of both samples after desliming were roughly alkaline ranging from 7.4 to 7.6 in the case of Azara,, and 7.0 to 7.5 in the case of Obubra. The pH of the values corresponding to the highest specific gravity were 7.50 and 7.02 for Azara and Obubra respectively. All pH values at each run of both experiments fell within API range for drilling drilling (7 to ≤12.5) pH=7─≤12.5 (Osokogwu et al. 2014; Ádewale and Salihu 2014; Ibe et al. 2016; Afolayan, et al. 2021). These results reveals that both the flowrate and agitation speed (of the flocummetric spindle) had a negligible effect on the pH value of both samples at each run.

4. Conclusion

The results of the desliming of Azara and Obubra barite for oil drilling operations, shows that at a final value of 3.80, desliming was unable to bring the raw specific gravity value of (3.45) the API required value of 4.2. In the case of Obubra, a specific gravity of 4.18 obtained (after desliming) was marginally below the API standard of 4.2, but is above Nigeria's Department of Resources (DPR) standard of 4.0. In terms of; moisture content, pH value and hardness tests carried out, both barite samples met the API physical properties for drilling. Thus, the deslimed Obubra baryte it suitable as a drilling mud additive in Nigeria's oil and gas industry. However, the processed Azara barite, failed to meet the API specific gravity specification for oil drilling use. Hence, it is primarily suitable in paint, ceramics, asbestos, glass, as well as a component for several other industries where barium compounds is required. For Azara barite to meet the API specification for drilling, it may require further processing using tested processing techniques, such as gravity separation, froth flotation etc.

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