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Enhancing Barite Ore Processing in Nasarawa State Nigeria: An Integrated Approach of Desliming, Froth Flotation and Acidic Leaching

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ABSTRACT

Low-grade Barite ore with a specific gravity (SG) of 3.45, from the Azara area of Nasarawa State, Nigeria, was subjected to desliming by elutriation, froth flotation, and acidic leaching to improve the requisite specifications for use in oil and gas drilling purposes. Other physical properties, namely, moisture content and pH and were also investigated. The elemental composition was obtained by X-ray Fluorescence (XRF). The mineralogical analysis, which revealed the presence of garnet, gypsum, orthoclase, davyne, Illite, hematite, muscovite, and quartz in the sample, was determined using X-ray diffraction (XRD). The highest SG value of 3.80 after desliming was attained at a flow rate and agitation speed of 42.50ml/s and 20 minutes, respectively. The sample's SG increased to 4.21 when further subjected to froth flotation using 2g of sodium silicate acting as a depressant, and. 2g of sodium dodecyl sulphate as the collector. The SG further rose to 4.26 after leaching it with 0.2M of hydrochloric acid. The final SG of 4.26 was higher than the American Petroleum Institute (API) 4.20 specification for drilling operations. The pH (±7.4) and moisture content (±1%) were all within the API requirements of 7≥12 and 1% respectively. Drilling operation. The metallic content, Ca, Pb, Zn, Mg, Cu, Cd, and extractable carbonates were within the API specifications, too. Results from the XRD analysis showed a reduction in the gangue minerals at each processing step. After leaching, the quartz content experienced a reduction from 45.12% to 37.8%, while Orthoclase and Hematite were absent. The overall results show that the processed Barite is suitable for drilling operations in Nigeria.

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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 [1-2]. As a nation, Nigeria is greatly dependent on its oil and gas sector for its socio-economic development. Studies revealed that Nigeria's oil and gas sector generates 95% of the country's foreign exchange income (FOREX), and roughly 80% of its budget revenue [3].

Barite is an important industrial non-metallic mineral with several applications in the chemical and petroleum industries. It is used in the production of paints, paper, rubber, etc. However, its most important application is in the oil and gas sector, where its high specific gravity makes it suitable for use as a weighting agent in the formulation of drilling fluid. [9]. A geographical survey of Nigeria's solid mineral deposits by the Nigerian Geological Survey Agency revealed commercial quantities of Barite ore deposits in the Azara area of Nasarawa State [4-5]. The Nigerian Federal Ministry of Solid Minerals Development estimates a national Barite reserve of 730,000 tons [5]. Barite deposit in Azara is located in hydrothermal veins, within the Cretaceous Keana sandstone of the Middle Benue Trough in central Nigeria. Azara Development Area of Nasarawa State has been described as the leading area of Barite in Nasarawa and Nigeria at large [6]. Geochemical and Petrology studies further reveal that the Azara mineralization deposit potential has a relatively high quantity of Barite in its veins [7].

However, there have been very minimal activities in terms of Barite exploitation in Azara and Nigeria as a whole, thus leading to a loss of over ₦5 billion ₦5 billion ($3,127,854.17 USD) annually in foreign exchange, due to the importation of Barite by the International Oil Companies (IOCs) operating in the country [8]. The IOCs argue that the specific gravity of Nigerian barite falls below globally acceptable standards, as a result of impurities associated with it in the form of associated or gangue minerals. Recent research in Nigeria has focused on the physical-chemical and mineralogical analysis of raw Nigerian Barite ores for various industrial applications [9-14]. However, there is a dearth in research involving the processing of these ores using globally acceptable processing techniques to meet the required specifications for various industrial uses.

Froth flotation can be described as a physicochemical process, in which there is a reaction between the surfaces of mineral(s) and the reagents in solution to create hydrophobic complexes that impart the floatability to the mineral particles. It is a highly versatile method for the physical-chemical separation of minerals such as Barite from its associated (gangue) minerals, based on differences in the capability of air bubbles to selectively cling to a particular mineral surface in a mineral/water slurry, a separation technique based on surface selectivity that isolates hydrophobic materials from the hydrophilic part [15]. Thus, it is widely employed to liberate Barite from its associated minerals in highly fine size fractions and high-grade concentrate [16-17]. Normally, the feed used in a flotation cell is 40 mesh size or finer. Therefore, it is used when the liberation size is finer than what a recovery in depositional Barite/sulfide ore, as well as fluorite-associated hydrothermal Barite ore [18-19]. With a large development space, froth flotation is interactively applied in industries compared to other processing techniques. Froth flotation is the most inexpensive and widely applied metallurgical method that separates chemically similar minerals and concentrates ores [15].

However, the use of froth flotation is associated with the production of huge measures of fine coated gangue minerals, resulting from the fine grind or the clayey form of the ore [20-22]. These fine mineral impurities (gangue) are collectively referred to as "slimes". Slimes are capable of exerting several harmful effects on the efficiency of flotation. The presence of slimes is capable of increasing both the consumption of reagents and pulp viscosity, which are equally liable to entrain into the froth product [23]. Slimes equally provide a coating on the surfaces of the desired minerals, which in turn, considerably alter the flotation performance of the latter. Naturally, the slimes on the surface of the desired mineral develop a hydrophilic "armor", thus preventing the mineral from having a straight (or direct) contact with both the collectors and/or air bubbles, which in turn decreases the flotation recovery of the desired mineral [24-27]. The presence of slimes on the flotation is related to the increase in reagents consumption [28]. Therefore, for a successful flotation, a process to eliminate the slimes is a necessary prerequisite, as the removal of such

slimes decreases reagent consumption and improves the valuable mineral recovery during flotation [29]. The options for treatment of slimes are partially based on the notion that for a given mineral(s) of a particular particle size distribution (PSD), the ore contains certain minerals from which the desired mineral need to be liberated from. Among the various methods applied in the removal of slimes before froth flotation is a process referred to as desliming.

The use of desliming results in the removal of the slime coatings, which, as earlier stated, hinders the contact between the bubble-mineral and collector adsorption on the valuable minerals during froth flotation. This results in a better froth flotation response of the desired mineral. Furthermore, for desliming to be possible and effective, the slime fraction should be bereft of a large concentration of the desirable (or valuable) minerals. In the instance when the slime proportion has a large concentration of desired minerals, the use of individual flotation circuits for both coarse and slime fractions should be considered. Desliming can be carried out with a hydrocyclone, by manual screening with sieves or a digital sieve shaker, as well as by the process of elutriation [23].

Elutriation is the process of separating mixtures of particles using an upward fluid stream [30]. The principle works by exploiting Stokes' Law, which states that a particle settling in a fluid medium will settle at a given velocity determined by the characteristics of the fluid and of the particle [31]. By suspending a heterogeneous mixture in an upward fluid stream, the upward velocity can be tuned to such a value that particles can be effectively separated from one another based on density, shape, and size.

After froth flotation, the concentrates obtained have to be further processed by leaching. Leaching is a liquid-solid process involving the use of a liquid solvent to bring about an extraction or a preferential dissolution of a desired component from solids [32]. In the leaching process, minerals obtained after undergoing froth flotation are enriched into a fully leached solution with the help of a reagent. Leaching in mineral processing finds its application in the recovery of various valuable minerals [33-35]. Considering that the occurrence of minerals such as Barite is mostly associated with a considerable quantity of unwanted or undesirable constituents, leaching is often further added in the processing of Barite ore.

Studies on jigging, froth flotation, and chemical leaching of −180 + 90μm size of Azara Barite, using pine oil, oleic acid, HCl, and HOCl showed a result of an increase in the specific gravity of Azara Barite ore from an initial value of 3.27 ± 0.03 to 4.38 [36]. A laboratory-based jigging and froth flotation of −350 + 180 μm size Azara Barite using NaOH and oleic acid test conducted reveals a result of an increased barite specific gravity from 3.72 to 4.23 at a pH of 7, while the pH at 3 gave the lowest specific gravity value of 3.78 [37]. A combination of jigging, froth flotation, and acidic leaching to improve Akpet 1 Barite specifications for use in oil drilling operations - for flotation, pine oil, oleic acid, and HCl were used as reagents, while 0.2 M of HCl was used in the acidic leaching. After acidic leaching, the specific gravity rose from an initial API value of 3.03 to 4.39, while the pH value was 6.8. These results show that Akpet 1 Barite can be processed to meet the physico-chemical specifications for use in the formulation of drilling fluid for oil and gas operations by using a combination of jigging, froth flotation, and acidic leaching [38].

The aim of this research, thus, is to show that the use of a combination of desliming (by desliming), froth flotation, and leaching can improve Azara Barite to meet globally acceptable specifications/standards for industrial use. This will ultimately reduce Nigeria’s overreliance on imported barite by oil companies operating within the country-thus resulting in a significant increase in its foreign exchange.

2. Features of the study areas

Fig. 1 gives the study areas of the research. A brief discussion of this study area follows:

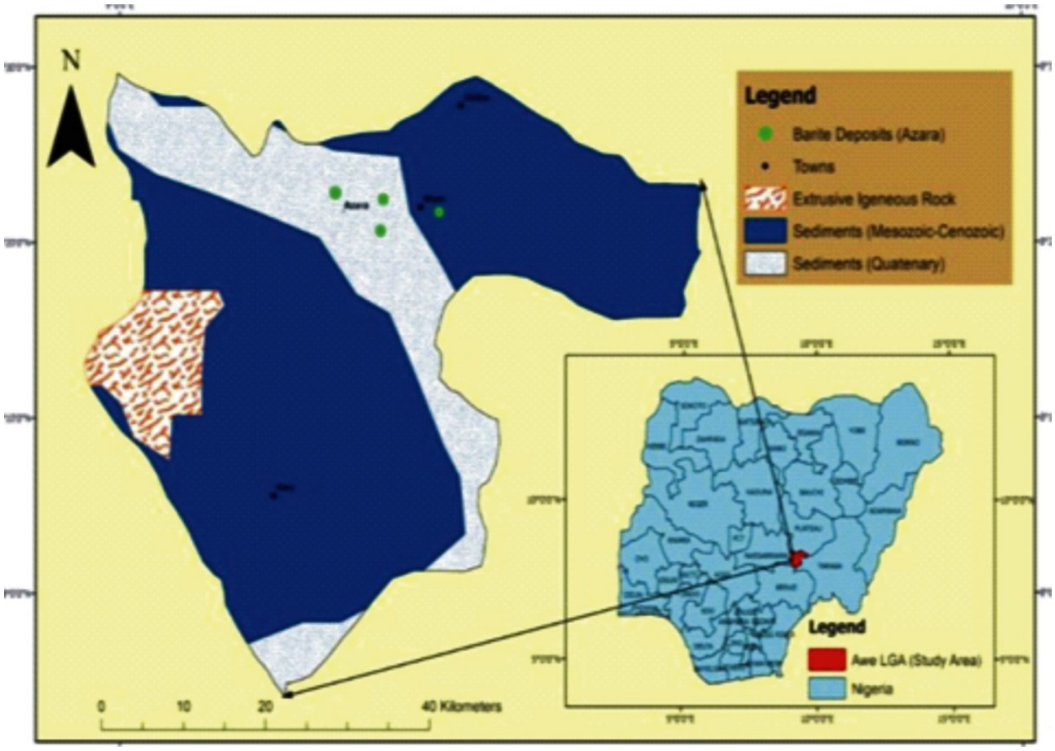

Fig. 1. Map of barite deposit in Azara [12].

3. 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 801910011 to 802310011and longitude 901610011 to 902010011. 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 Barite ores [39]. The Azara Barite deposit is located about 98km south of Lafia, the capital of Nasarawa State. 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 is 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 [40]. Figure 1a above shows the map of the Azara development area in Awe Local Government Council of Nasarawa State of Nigeria.

4. Experimental Procedure

4.1 Sample Preparation

The Barite ore samples from Azara were taken from Quelchem global L.T.D, and transported to the PTDF Bio-oil research laboratory in the Education Trust Fund (ETF) Building complex, Abuja campus, University of Port Harcourt, Rivers state, Nigeria. The ore sample was first crushed to several smaller pieces using a clean locally fabricated hammer crusher, before grinding, milling, and sieving, to obtain fine/coarse particles of sizes of ≥75 µm. The grinding and milling were done with the cheek of the fabricated hammer crusher, while sieving was carried out with a 75µm sieve (no 200). The specific gravity and pH values of the unprocessed Barite were ascertained using a le Chatelier flask and a digital pH meter, respectively. Fig. 2a and 2b give the samples of the crushed and milled Azara Barite, respectively.

Fig. 2a. Crushed sample of Azara Barite.

Fig. 2b. Milled sample of Azara Barite.

8000g of the previously milled and sieved Barite were used for the desliming of the sample. Desliming of the Barite samples was carried out by elutriation. Elutriation was carried out at water flow rates of 25.20ml/s and 42.50ml/s, and agitator speeds of 20rpm and 30rpm on each portion of the test sample using a floccumetric machine (J.P Selecta, Code, 3000833, serial no. 0463747, volt, 230, Amp, 0.1, W, 3.5, Hz, 50).

The Azara Barite desliming experiment consists of 8 runs (in duplicates), with 1000g per run. Each of the 1000g was performed in 4 batches, roughly 250g (to reduce potential losses of Barite during desliming). Each of the test samples was placed in a plastic bucket containing an opening at the bottom through which water was injected at a predetermined flow rate, and an opening at the top through which the overflow fraction flowed. The upward flow of water, which was allowed to run for 30 minutes, transported a portion of the Barite solids into the overflow. The overflow fraction was sent to a second bucket (with an opening at the top) through a pipe, to further collect any Barite from the overflow in the desliming bucket. The flow of water was shut off after 20 and 30 minutes (as shown in the experimental design), and the underflow fractions from both fractions were allowed to settle (to the bottom) for 20 minutes before decantation. The decanted Barite from both buckets was added up to arrive at a single weight for each run. Fig. 3 shows the experimental setup for desliming the Azara barite sample.

Fig. 3. Experimental setup for desliming the Azara Barite sample.

For each run, the deslimed Barite was dried in a muffle furnace at 110 °C, while weighing at an interval of 20 min until a constant weight was observed.

The caked Barite slurry formed after drying was milled to obtain the Barite in fine particles as it was after the initial milling. Finally, a 75µm sieve (no. 200) was employed to obtain Barite particles of less than

75µm in size. At the end of the desliming operation, the responses, the specific gravity, fineness (grain size), moisture content, and pH of each of the eight runs were determined.
After desliming, an average value of 640g of the sample was recovered from each run, out of which 80g was taken from each of the 8 runs (now consisting of deslimed samples) to obtain their respective specific gravities. The optimal deslimed run (i.e., the run that recorded the highest specific gravity during desliming) was then subjected to froth flotation. The optimal deslimed Barite measuring 640g was first divided into 4 portions of 160g each. For the first run, 100ml of water was first poured into a 250ml beaker. 2g of sodium silicate acting as the depressant was added to the beaker, and the mixture was well stirred until it was completely dissolved. 2g of sodium dodecyl sulphate acting as the collector was then added to the beaker and vigorously stirred until it was completely dissolved in the distilled water. 160g of the deslimed Barite was then added to the mixture and stirred for 10 minutes. During the stirring, white froth could be seen forming on the surface of the mixture. The mixture was then left for a conditioning time of 5 minutes, after which the froth was continuously skimmed off and collected in a separate beaker. The mixture was decanted, washed with more distilled water, and stirred again for another 10 minutes. Like before, the mixture was left for a conditioning time of 5 minutes, and the emerging froth was further skimmed off and collected as previously done, while the mixture was decanted and washed again. The ash-white Barite was also scooped and added to the froth collection. The entire process was repeated severally times until a thin layer of dark coloured slurry was left at the bottom of the beaker with no significant froth formation. The layer of dark coloured slurry was assumed to represent the gangue minerals depressed by the sodium silicate. The froth was allowed to settle, dewatered by filtration, washed, and dried in a muffle furnace at 110^0C while weighing intermittently, until a constant weight was achieved, indicating the near absence of moisture in the sample. The froth was completely dried after 8 hours. The entire process was repeated on the 2nd, 3rd, and 4th runs following the flotation experimental design as seen in Table 2. The optimal run (i.e., the run that recorded the highest specific gravity during froth flotation) was then subjected to acidic leaching. Here, the concentrate was poured into a conical flask and mixed with 300ml of 0.2M HCl. The mixture was gently washed severally times and weighed. The product was washed severally times before filtering it through a Whatman ‘41’ filter paper formed into a cone shape. The set-ups were left standing until no more of the HCl dropped into the beakers below. The leached Barite was dried in a muffle furnace while weighing intermittently at an interval of 20 minutes, until a constant weight was achieved. The leaching process was carried out to remove all traces of sodium dodecyl sulphate and sodium silicate from the Barite obtained after froth flotation, as well as to further wash the Barite sample of possible impurities. The pH value was recorded using a pH meter applied to the Barite substrate on the filter paper at the end of the leaching process.

4.2. Modeling of experiments for the desliming and froth flotation processes

Design-expert, as a statistical modeling tool, uses factorial, response surface, mixture, and custom designs for the design of experiments (DOE). The use of a design expert identifies the effective factors, thus minimizing the number of experimental runs. The optimal operational condition can then be calculated based on the DOE results obtained, and potential combinations of effective parameters, while also identifying the optimum condition. The use of design-expert software by modeling several tests generates different rules, which can streamline the DOE to achieve the minimum number of factor combinations that would be required for testing. The use of design-expert software has been used in several experimental studies, such as modeling and optimizing ultrasonic oxidation of arsenite with $H_2O_{2,}$ modeling of microwave-assisted extraction of natural dye from seeds of Bixa orellana (Annatto), in the use of response surface methodology (RSM) and artificial neural network (ANN) [41- 42]. In the pharmaceutical industry, it has also been of use for formulating development, optimizing floating Microballoons for oral delivery of domperidone, as an application tool for optimization in pharmaceutical preparations formulation, in the

design and optimization of the seed conveying system for belt-type high-speed corn seed guiding device [43- 45].

The experimental design for the desliming and froth flotation operation of the Azara Barite sample is shown in Table 1 and 2 below, and was prepared using the software Design-Expert (version 13). Desliming was done using the 2x2 factorial design, involving 2 factors, flow rate and agitation time at two levels (flow rate: 25.20ml/s, 42.50ml/s, agitation time: 20mins, 30mins). While froth flotation was done using 2 factors (quantity of the collector and quantity of the depressant) at two levels (quantity of the collector: 1g and 2g, quantity of the depressant: 1g and 2g).

Table 1. DOE for the Desliming Operation (Design-expert version 13).

Runs	Factor 1 Flowrate (ml/s)	Factor 2 Agitation speed (min)	Response 1 Specific Gravity	Response 2 Fineness	Response 3 pH
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			

Table 2. DOE for the froth flotation Operation (Design-expert version 13).

Runs	Factor 1 quantity of collector (g/t)	Factor 2 quantity of depressant (g/t)	Response 1 Specific Gravity	Response 2 Fineness	Response 3 pH
1	2	2			
2	2	1			
3	1	2			
4	1	1			

4.3 Sample Characterisation

4.3.1 Specific gravity determination

The specific gravity was ascertained by applying the API Specification 13A, Drilling Fluid standard, using a Le Chatelier flask, which is shown in fig. 4 below. Using this standard, the weight of 80g of dried Barite was determined with a weighing balance. The sample was then poured into a stainless steel pan and dried in a muffle furnace at 110 °C. The pan (housing the Barite) was removed at an interval of 30 minutes and weighed until a constant weight was achieved. The Barite sample was then poured into a Le Chatelier flask, which had been filled with kerosene up to 0.2 mL. It was left standing in a water bath for a time estimated at 30 minutes, after which it was taken out, and the 80g measured Barite was poured into it. By hand tapping and swirling actions, possible Barite samples clinging to the neck of the flask, as well as entrained air also in the flask (albeit in the form of bubbles), were both dislodged and removed, respectively.

The flask was reintroduced into the waterbath for 30min, after which it was taken out. The specific density, ρ, of the Barite was computed in grams per milliliter, as seen in equation (1) below:

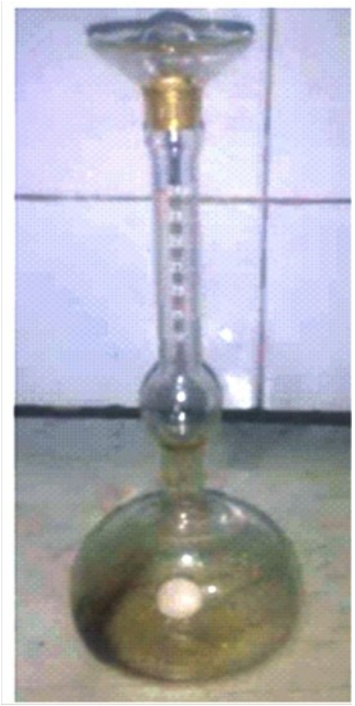

Fig. 4. Diagram of the Le Chatelier flask.

$$\rho = \frac{m}{V_2 - V_1} \quad (1)$$

Where:
m represents the Barite sample mass, in grams, while V1 and V2 represent the initial volume (read from the flask, just before the introduction of the sample) and the final volume (read from the flask at the end of the process), respectively. Both readings were recorded in milliliters (ml).

4.3.2 Particle size determination

Approximately 1100g of the powdered sample (obtained from the dried, crushed, and grinded Barite ores) was cooled to room temperature in a desiccator. The samples were transferred in batches to a 75µm sieve. A small brush was used to agitate the Barite particles to ensure that particles of size less than 75µm and below pass through the 75µm sieve into a clean pan positioned below the sieve. The total sieved Barite powder (less than 75µm) was added and weighed with a weighing balance. The mass fraction of Barite powder greater than 75µm (w_1, expressed in percentage (%), is computed using equation (2) below:

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

Where,

m_1 is the mass of the Barite sample measured in grams (g), while m_2 represents the powder mass retained by the 75μm sieve, also measured in grams (g)

4.3.3 Determination of sample pH

The Hanna instrument (HI98129 Model) was used to obtain the pH value. 20ml of water was added to 20g of the sieved sample. The mixture was placed in a beaker. The probe of the Hanna instrument was then inserted into the mixture, and the readings obtained.

4.3.4 Determination of moisture content

Ten grams of the weighed sample in a crucible was placed in a stainless clean metallic and placed in muffle furnace working at an operating temperature of 110 °C for 3 h. The cooled samples were measured as the dried weight of the samples. The differential weights of the samples were compared and computed using an analytical weighing balance. The entire process was repeated after 3 h until a constant weight was obtained [9].

The following formula was then used to obtain the moisture content calculated on the dry basis:

$$M_c = \frac{W - W_s}{W_s} \; x\; 100 \tag{3}$$

Where,

M_c = the moisture content (g water/g dry solids).

W = total product weight at each time (gram).

W_s = weight of the dry solids (gram).

4.3.5 Determination of the Barite mineralogical composition by X-ray diffraction (XRD)

X-ray diffraction (XRD), which was performed at the Spectral Laboratory Services, Kaduna State, Nigeria, determined the mineralogical composition and crystallographic structure of the sample using a Panalytical diffractometer characterized by current and voltage equal to 40 mA, 45Kv, respectively, at temperatures of 21-23^{O}C. To employ this technique, the finely ground, homogenized, and average bulk analyzed Azara samples composition was compressed in the flat sample holder to create a flat, smooth surface that was mounted on the sample stage in the XRD cabinet.

The computer system was then switched on, and the software for XRD was put on. The settings dialogue was clicked, and all the required settings of power and temperature were checked to correspond to those of the XRD. After placing the sample on the sample holder, it was put in the sample chamber column. The door of the chamber was shut, and confirmed on the computer. The measurement setting was then set for scanning. The result of the scan was saved to a file, and the results obtained were match with different library data, such as the NIST and PubChem to obtain the name, chemical structure, and other physio-chemical characteristics of the mineral components of the sample. The XRD was determined at each processing stage of the sample.

4.3.6 Testing the efficacy of the optimal desliming conditions on other low-grade Azara Barite ores

The particular run which gave the highest specific gravity value in Table 1, design of experiment for the desliming operations, was repeated for 4 low-grade Azara Barite ores samples (1, 2, 3 and 4) with specific gravity 3.43, 3.36, 3.50 and 3.60, to further test the efficacy of the optimal desliming conditions. The four samples were first washed, dried, crushed, grinded, milled, sieved and deslimed as previously done.

5. Findings and Argument

5.1 Desliming

Table 3 offers the measured physical responses to the experimental design after desliming. The interpretation of these results follows below:

Table 3. Results of the Design of Experiment (DOE) for Azara Barite desliming.

Runs	Factor 1 Flowrate (ml/s)	Factor 2 Agitation speed (min)	Response 1 Specific Gravity	Response 2 Fineness	Response 3 pH
1	25.20	30	3.73	<75mμ	7.5
2	42.50	30	3.74	<75mμ	7.4
3	42.50	30	3.80	<75mμ	7.5
4	42.50	20	3.55	<75mμ	7.5
5	25.20	30	3.58	<75mμ	7.6
6	25.20	20	3.60	<75mμ	7.5
7	25.20	20	3.64	<75mμ	7.4
8	42.50	20	3.45	<75mμ	7.5

5.1.1 Specific gravity

The Azara Barite sample obtained the highest specific gravity value rise of 3.80 at a flow rate of 42.50 ml/s and an agitation time of 30 minutes. This represents a differential of 0.35 in specific gravity from raw to deslimed (3.45-3.80). The increase in specific gravity is attributed to a decrease in the associated minerals or impurities responsible for lowering the specific gravity of the sample [36]. The optimal flow rate of 42.50ml/s agrees with similar works on the effect of desliming on the flotation response of Kansanshi mixed copper ore [46].

5.1.2 Fineness (particle size)

The particle size remained at (as in sieving) 75μm after desliming. Thus, indicating that desliming has no effect on the particle size after the milling and sieving processes conducted earlier.

5.1.3 pH value

As also seen in Table 3**,** the specific gravity of 3.80 corresponded to a pH of 7.5, up by a value of 0.1, from the pH of 7.4 recorded for the raw Barite. This is due to the use of water (in the desliming process), which reduced its acidity. This value was within the API specification for drilling weighting agent, which is between a value of 7 and 12.5 [47-49]. The pH results further show that agitation speed and the flow rate of the water barely have a slight effect on the pH of the Azara Barite sample.

5.1.4 Moisture content

The differential loss in weight of the deslimed Barite of each run, revolves within 0.9%-1%. These values were well within the API acceptable maximum of 1%. A moisture content over 1%, can bring about the collapse of the borehole as a result of variations in the mud viscosity [9]. Similar experimental observations were recorded in research where a moisture content of within the value of 1% was registered [10].

5.2 Application of the optimal desliming variables on 4 low-grade Azara Barite samples

The application of the optimal variables (flow rate: 42.50 ml/agitation time: 30 minutes) on the 4 other low-grade Azara Barite samples 1, 2, 3, 4 (in section 2.3.6) produced results that validated the use of the optimal

variables. The results, which are shown in Table 4 signify an increase in the specific gravity within the margins obtained in the experimental design.

Table 4. Application of the initial optimal desliming variables on 4 low-grade Azara Barite samples.

S/N	Specific gravity before desliming	Specific gravity after desliming
1	3.43	3.66
2.	3.36	3.43
3.	3.50	3.70
4.	3.60	3.77

5.3 Froth flotation

5.3.1 Specific gravity

The results, as given in Table 5, indicate that froth flotation, in general, improved the specific gravity flotation response of Azara Barite ore to a value of 4.21. This value met the API specification for the use of Barite in oil and gas drilling purposes, which is set at 4.20. The results further show that an increase in collector quantity results in an increase in the specific gravity. At run 1 (the collector-depressant ratio of 2:2), the highest specific gravity value of 4.21 was obtained. This value decreased to 4.15 during run 2, at a collector-depressant ratio of 2:1. At run 3, with a collector-depressant ratio of 1:2 (the first time the quantity of depressant was higher than that of the collector), the specific gravity recorded the lowest value of 3.98. At run 4, with a collector-depressant ratio of 1:1, the specific gravity value increased from 3.98 in run 3 to 4.04. An increase in collector dosage has a marginal increase in the specific gravity in froth flotation [50]. These findings also emphasize the efficacy of collectors in froth flotation, as seen in more recent research, which investigated the effect of pH on the specific gravity values of Barite processed using the froth flotation method. The results showed that by using a solution containing burnt empty palm (Eleais guineensis) bunch and analytical grade palmitic acid as the collector, a high specific gravity of 4.23±0.05 is obtained (at a pH of 7) from an initial value of 4.04 [36].

Table 5. DOE for the froth flotation Operation (Design-expert version 13).

Runs	Factor 1 Collector dosage (g)	Factor 2 Depressant dosage (g)	Response 1 Specific Gravity	Response 2 Fineness	Response 3 pH
1	2	2	4.21	<75mμ	7.3
2	2	1	4.15	<75mμ	7.4
3	1	2	3.98	<75mμ	7.1
4	1	1	4.04	<75mμ	7.2

5.3.2 Fineness (particle size)

Results of fineness followed the same pattern obtained during desliming. Regrinding the dried “caked” Barite obtained by drying after froth flotation, resulted in Barite Azara samples which were less than 75mμ, as obtained after sieving. Thus, indicating that the froth flotation does not affect the particle size of the sample.

5.3.3 pH Value

The pH value corresponding to the highest specific gravity of 4.21 is 7.3. Furthermore, the mean pH value of 7.35 was within the API specification of 7 to 12.5 for the use of Barite as a weighting agent in oil and gas drilling operations [51, 47, 48].

5.4 HCl Leaching of the Azara sample

The result of the HCl leaching on the Azara Barite ore sample is shown in Table 6 and further explained below:

Table 6: The responses after acidic leaching of Azara Barite samples.

	Factor 1	Factor 2	Factor 3
Leaching	Specific gravity	Fineness <75mμ	pH
	4.26		7.2

5.4.1 Specific gravity

The results presented in this study (as seen in Table 6 indicate that generally, HCl leaching further improved the specific gravity of the Azara Barite sample from 4.21 (after froth flotation) to 4.26.

5.4.2 Fineness (particle size)

Results (as seen in Table 6), of fineness followed the same pattern obtained during desliming and froth flotation, that is, the particle size remained at <75mμ after leaching. Thus, indicating that leaching has no effect on the particle size after the milling and sieving processes conducted earlier.

5.4.3 pH Value

As seen in Table 6, the pH value of the sample after leaching with HCl was 7.2, down by a value of 0.1, after froth flotation. This is due to the use of HCl (in the leaching process), which increased the acidity of the sample. Furthermore, the pH of 7.2 was (as in desliming and flotation), within the API specified operational range for use as a drilling weighting agent (7 or ≤12.5) [47, 48].

Results from other responses, namely, fineness (particle size) and moisture content, followed a similar pattern from the desliming and froth flotation processes.

6. Mineralogical analysis

6.1 Unprocessed Azara Barite sample

Fig. 5 gives the quantitative analysis result of the unprocessed Azara Barite sample using X-ray diffractogram. As seen in the table, the raw Barite sample contains the associated (gangue minerals, quartz, gypsum, orthoclase, davyne, and Illite. The presence of Barite in silicate carbonate rocks and siltstone-sandstone gives rise to the presence of these gangue minerals [51].

As seen in fig. 6, quartz is the dominant impurity found in the unprocessed Azara Barite sample. This is because quartz is the most common constituent of magmatic, metamorphic, and sedimentary rocks [52]. The presence of quartz in Barite ore decreases certain important physical properties of the Barite, such as specific gravity and brightness. It equally alters the colour, as Barite is usually alternates between colourless to white in its pure form. Therefore, it can be assumed that the light brown colour of the Barite sample, as

well as a reduction in its brightness, is because of the presence of gangue minerals (impurities) such as quartz, garnet, etc. The effects of the decolouration and brightness makes the Barite less valuable in industries where colour and brightness are of significant importance, such as the paint production and the medical industry. However, the separation of quartz from Barite can be achieved by gravity separation techniques and the use of the microbe, Bacillus licheniformis [27, 53]. Figure 5 shows the percentage of quartz and Barite in the unprocessed ore as 45% and 29% respectively. It further indicates the presence of Illite, davyne, garnet, Orthoclase, gypsum, and Muscovite.

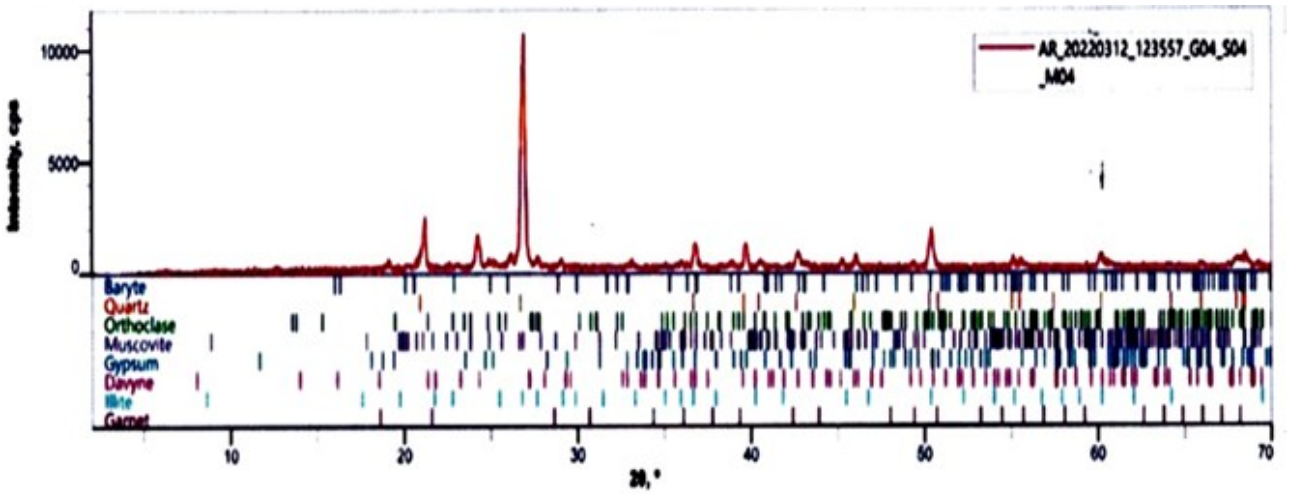

Fig. 5. XRD patterns of unprocessed Azara Barite sample (Quantitative analysis report).

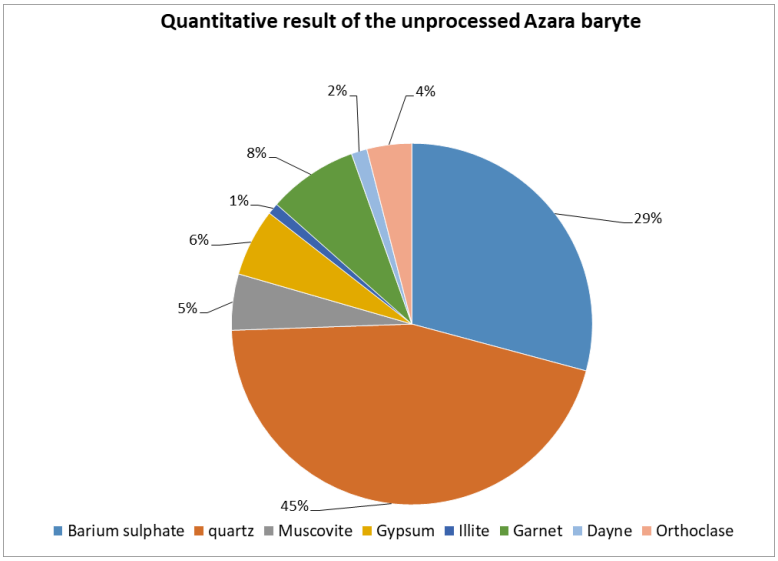

Fig. 6. Pie chart plot of the quantitative result of the unprocessed Azara Barite sample.

6.2 Mineralogical analysis of the deslimed Barite

The X-ray diffractogram information of the deslimed Barite is shown in the quantitative and qualitative analysis results in Fig. 7 and 8. The quantitative result of the deslimed Azara Barite sample, as shown in the figures, indicates that Barite and quartz are still the most dominant compounds at 24% and 54% respectively, after desliming. Therefore, this type of Barite is classified as quartz-Barite ore, with Barite as the useful mineral and the main quartz, the gangue mineral. Consequently, the qualified Barite products can be obtained generally by gravity separation, such as desliming or many times flotation methods [27]. When compared to the unprocessed Azara Barite analysis result, it is observed that the concentration of the gangue minerals in the unprocessed Azara Barite sample decreased after the desliming process. However, the gangue, associated or impure minerals were incompletely eliminated. There was a considerable reduction in the content of orthoclase from 4% to 2%, illite reduced from 1% to 0.04%, and garnet experienced a significant drop from an initial value of 8% to 0.7% after desliming. Davyne, which recorded a value of 1.4%, was completely removed from the sample after desliming. The specific gravity of the Barite increased after desliming from an initial value of 3.45 to 3.80.

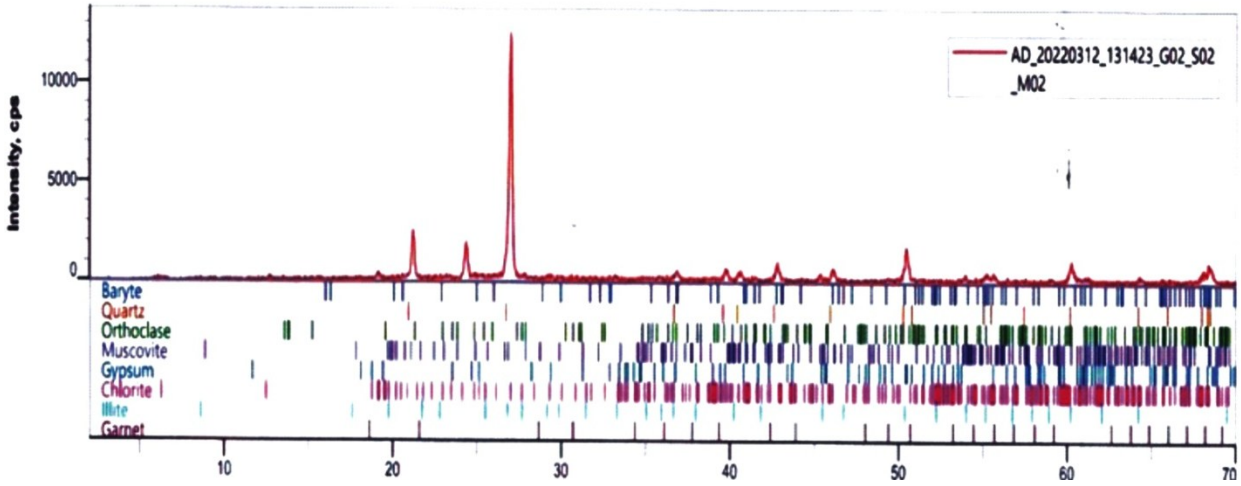

Fig. 7. XRD patterns of the deslimed Azara Barite sample.

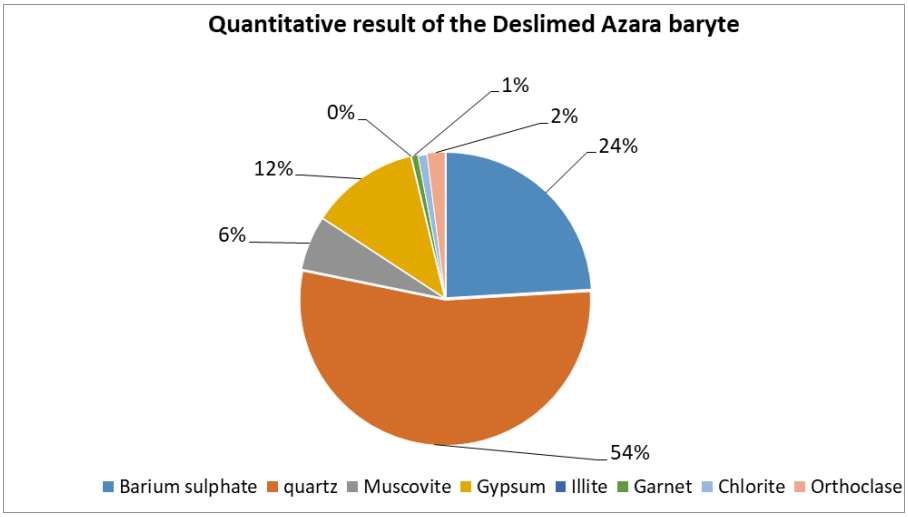

Fig. 8. Pie chart plot of the quantitative result of the deslimed Azara Barite sample.

6.3 Mineralogical analysis of the Azara Barite sample after froth flotation

The X-ray diffractogram of the deslimed Azara Barite sample, as shown in the quantitative analysis report in Fig. 9 and 10 reveals the composition and crystallographic information of the deslimed Azara 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 3700cps. The quantitative result of the deslimed Azara Barite sample, as shown in Figure 10, indicates that Barite and quartz are still the most dominant compounds after froth flotation. As earlier stated, this type of Barite is classified as quartz—barite ore, with Barite as the useful mineral and quartz, the gangue mineral. Consequently, the required Barite can be obtained generally by gravity separation, such as desliming or many times flotation methods [27]. When compared to the deslimed Azara Barite, the concentration of Barite increased from 24.4% to 38.3% after froth flotation, while the concentration of some gangue minerals in the sample decreased. In the case of quartz, there was a considerable reduction from 54.7% to 49.4%, Muscovite (KAl2 (Si3 Al) O10 (OH, F)2 reduced from 6.6% to 0.66., Gypsum (CaSO4·2H2O) reduced from 12.7% to 1.8%, while Chlorite was eliminated from the sample. The specific gravity of the Barite sample increased from 4.18 after desliming to 4.21 after froth flotation. This value (4.21) meets the API standard of a minimum of 4.2 required for oil and gas drilling operations.

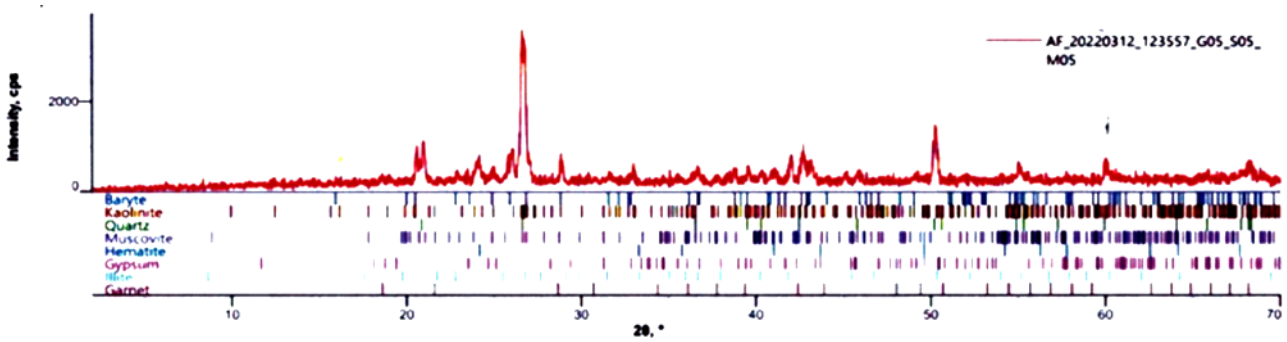

Fig. 9. XRD patterns of the Azara Barite sample after froth flotation.

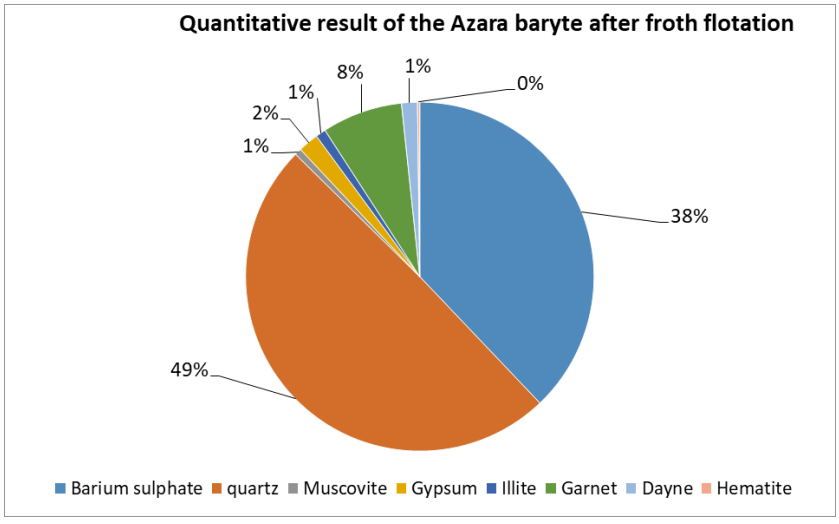

Fig. 10. A Pie chart plot of the quantitative result of the Azara Barite after froth flotation.

6.4 Mineralogical analysis of the Azara Barite sample after HCl leaching

The further extraction of gangue minerals from the Azara sample (after froth flotation) was carried out using 2M of hydrochloric acid (HCl). The X-ray diffractogram of the HCl leached Azara Barite sample, as shown in the qualitative and quantitative analysis report in Fig. 11 and 12 gives both the composition and crystallographic information of the leached Azara 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 3700cps. The quantitative result of the leached Azara Barite sample, as shown in Fig. 12 shows that Barite and quartz are still the most dominant compounds after HCl leaching.

The concentration of some gangue minerals in the sample decreased after HCl leaching. In the case of quartz, there was a sizable reduction from 49.4% to 37.8%, while Orthoclase and Hematite were completely absent in the leached sample. The specific gravity of the Barite sample increased from 4.21 after desliming to 4.26 after HCl leaching. As in the immediate froth flotation process, this value (4.26) represents a further increase in the API standard minimum of 4.2 required for drilling operations.

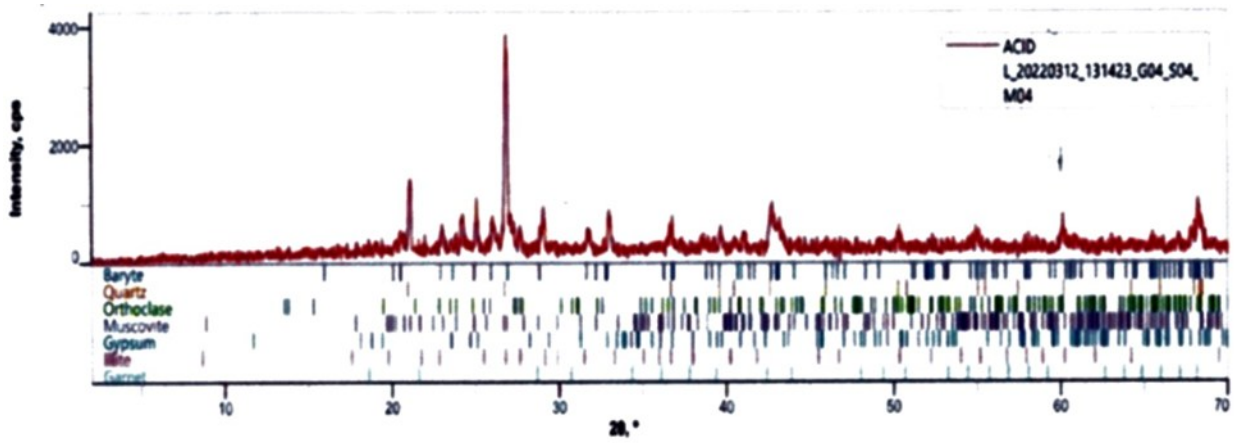

Fig. 11. XRD patterns of the Azara Barite sample after HCl leaching.

6.5 Environmental concerns

Since desliming by elutriation involves the use of only water (in the form of reagents), harmful effects on the environment from the use of chemicals is absent. Thus, desliming by elutriation can be described as an environmentally friendly technique for Barite processing. Unlike elutriation, which involves few factors, froth flotation involves the application of several reagents, and is dependent on several factors such as pulp pH, pulp concentration, pulp temperature, flotation time, flotation reagent, flotation time, etc. These factors raise some environmental concerns [54]. Similar concerns also apply to acidic leaching, which involves the application of acids in various concentrations. However, the use of water in place of acid in leaching can significantly minimize these concerns.

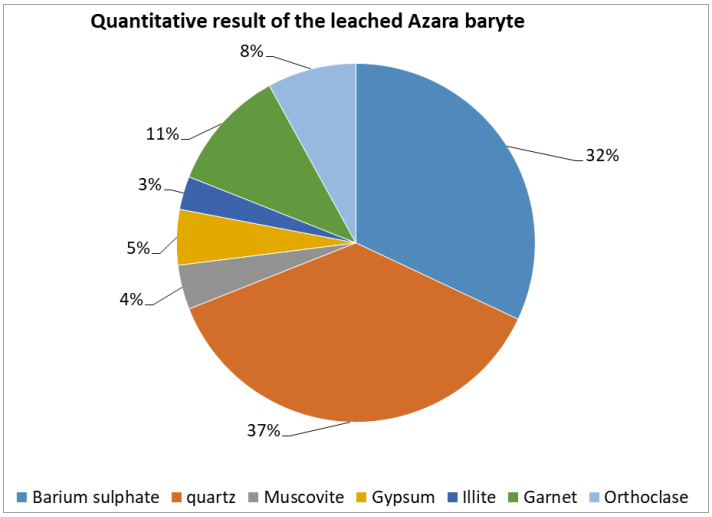

Fig. 12. XRD patterns of the Azara Barite sample after HCl leaching.

7. Conclusion

At the end of the processing, the specific gravity of the processed Azara Barite at 4.26 met the API standard of 4.2 for use in the formulation of drilling fluid. The particle size of the Azara Barite sample at each processing step met the API standard for oil drilling operations. The pH value of the Azara Barite sample at its raw state and during all the processing steps is within the operational pH and the standard range for a weighting agent, as recommended by the API. Desliming (through elutriation) improved the specific gravity of the Azara Barite sample from the raw value of 3.45 to an optimal value of 3.80. The change in the specific gravity was due to the removal of some unwanted minerals in the Barite ore. These unwanted minerals, often referred to as associate or gangue minerals, were mainly galena, calcite, hematite, pyrite, magnetite, sphalerite, brucite, chalcopyrite, and chalcocite. The use of sodium dodecyl sulphate (collector) and sodium silicate (depressant) improved the specific gravity to the optimal value of the deslimed Azara Barite. The use of 2g of sodium dodecyl sulphate (collector) and 2g of sodium silicate (depressant) offered the maximum specific gravity estimate of 4.21. Azara Barite leached with 2M of HCl (after froth flotation) increased the specific gravity values to 4.26, thus making it suitable for oil and gas applications. The X-ray diffraction at each processing step revealed a highly crystalline peak with quartz (SiO2), the dominant gangue mineral at each processing step.

Ethical Considerations

The authors avoided data fabrication, falsification, and plagiarism, and any form of misconduct.

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Conflict of Interest

The authors declare no conflict of interest.

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