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Analysis of Baryte Ore from Obubra Local Government Area in Cross River State, Nigeria

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Abstract
The physico-chemical properties of baryte from Obubra area of Cross River state was analysed to examine its suitability for use in drilling operations in Nigeria. Results obtained were compared with the American petroleum institute (API) specifications for drilling operations. From the analysis, the percentage composition of barium sulphate ($BaSO_4$) compound in the ore is 34.545wt%. This figure is well below the API's minimum value of 96wt%. The presence of the associated compounds, such as CaO (0.038wt %), MgO (0.00wt %), SiO_2 (59.297wt %), Fe_2O_3 (0.638wt %), Al_2O_3 (1.971wt %), K_2O (0.006wt %), were also observed. While the MgO content is within the API tolerable limit of 0.04wt%, the composition of SiO_2 is significantly beyond API's maximum limit of 0.01wt%. The Fe_2O_3 value of 0.638wt% is also above API 0.1wt% limit. Elemental results from the XRF analyses shows that apart from Fe, the composition of Ca, Mg, Zn, Cd, Cu and Cd were within API's acceptable threshold for drilling purposes. The physical and physico-chemical properties examined, puts the pH and moisture content within API specifications of 7-12 and 0.1% respectively. However, the specific gravity of the Obubra baryte sample, measured using a Le Chatelier flask is 4.14g/cm3. This value is slightly below the API requirement of 4.2g/cm^3, but above Nigeria's department of resources (DPR) mark of 4.0g/cm^3. Thus, in its raw form, the Obubra baryte sample can be used in producing good-quality papers, sodalime, glasses and as fillers for the production of rubber and paint. But requires more processing to satisfy API's limits for its associate compounds and specific gravity, when in use in drilling operations.

Keywords: *Baryte ore, API, Physico-chemical, Oil and gas, Drilling, Mineralogical Characterisation*

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1. Introduction

Baryte or barium sulphate (BaSO4) is an essential mineral used in making several products such as glass, paint, rubber, textile, paper, explosives, brake pads, as well as barium-based chemicals. In the medical world, baryte is utilised in the area of radiology, for the absorption of radiation for X-ray examination of the human digestive system (Ene et al., 2012; Siddiqui, 2016). According to Batouche et al. (2019), roughly 75% of its baryte's domestic output is used as a weighting agent for the formulation of drilling fluids, because of its high specific gravity. Afolayan et al. (2021) puts this value at over 80%. Summarily, the effective application of baryte is a function of the qualities and properties of the end-product comparable to globally acceptable standards and specifications.

Baryte mineralisation in Cross River state, Nigeria, occurs extensively in both the Cretaceous and Pre-Cambrian geologic terrain of central and northern Cross River State in the form of vein-filling materials (Akpeke et al., 2006; Oden, 2012). These veins are of variable thicknesses that usually range from less than 1 m to 2 m in some locations.

The occurrence of baryte has been reported at Obubra, Etung Yakur, Akamkpa, Ikom Biase, Okpoma, and Omoji areas of Cross River state Akpeke et al. (2006) and Ekwueme and Akpeke (2012). According to Oladapo and Adeoye (2011) areas in Cross River State such as; Alifokpa, Gabu, Obubra, Ogoja and Osina areas of Cross River State, Nigeria, holds an appreciable and commercial deposit of baryte reserves. In all, Obi et al. (2014) puts the estimated baryte reserves in Cross River State above nine million metric tons. This figure places Cross River as the State with the highest reserve of baryte in Nigeria. A recent research by Jimoh and Adeleke (2017), centring on the origin, distribution and economic evaluation of baryte reserves in Cross River State, gives credence to the significance of baryte deposits in Cross Rivers State, Nigeria.

Researches on the analyses of baryte, with the aim of improving its quality for drilling operations have centred on barytes located in Azara, Awe local government area of Nasarawa State and in Guma local government area of Benue, Nigeria. The analyses of baryte ore from Cross River State, Nigeria, have been unexplored. Therefore, the aim of the present study is to analyse baryte deposit from Obubra local Government Area of Cross River State, Nigeria.

2. Materials and methods

2.1 Study area

Fig. 1 gives a picture of the map Cross River State with the study area - Obubra local government area (LGA) of Cross River State. Obubra LGA is located in the central senatorial district of Cross River State, Nigeria and has a land mass of 1115 square kilometer. 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 deposit and baryte ore. Obi et al., (2014) puts the baryte ore reserves at Obubra at 9,660,306 metric tons.

Fig. 1: Map of Cross River State of Nigeria showing the study area

2.2 Physico-chemical analyses of the baryte sample

The physicochemical analyses carried out were physical appearance, specific gravity, grain size (fineness) and pH.

Appearance

The appearance of the un-pulvurised sample was ascertained by sensory evaluation using the eyes to ascertain the colour and hands for evaluating its texture.

Streak test

The streak test was conducted by scraping a portion of the un-pulverised baryte sample on a streak plate. A piece of ceramic material was used as the streak plate. The scraping action was performed with the ceramic plate firmly held with the left-hand. A little mass of powdery substance referred to as “streak” was produced on the ceramic plate from the streak action. Both the colour and texture of the streak were determined by sensory evaluation using the eyes and hands. As a confirmatory test, the streak test was repeated thrice.

Specific gravity determination

A Le Chatelier flask technique was used to determine the specific gravity of the Obubra sample. The procedure followed was in accordance with API Specification 13A (2019). Using this procedure, the bartye sample was first pulverized to 75mμ and dried in a muffle furnace for an initial period of 30mins. The sample was then weighed with digital weighing balance. This drying action was repeated 5 times until a constant weight was achieved. Kerosene was then introduced into the clean and dry Le Chatelier flask. The flask was then left for 30mins in a water bath after which it was taken out, thoroughly cleaned dry and reweighed. The gradated mark on the flask was read and recorded in millilitres as V_1. A funnel was then inserted into the top opening of the flask and precisely 80g of the dried baryte carefully introduced into the flask with the help of a spatula. 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. This procedure was repeated until no more air bubbles is seen rising from the Le Chatelier’s flask. The bulb was inserted and the flask returned into the water bath for 30mins. On removal from the bath, 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 millilitres as the final volume V_2. Fig. 2 is a diagram of a Le Chatelier flask, while Fig. 3 shows the insertion of the funnel into the flask.

Fig 2a. Diagram of the Le Chatelier

Fig 2b. Insertion of the funnel into the Le Chatelier flask

The baryte density, ρ, was calculated in grams per millilitre, 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 millilitres, V_2 is the final volume, expressed in millilitres, and ρ is the calculated specific density of the baryte sample.

Determination of sample grain size (fineness)

The grain size of the pulverised 75µm baryte sample was determined using a 75µm sieve No. 200. The baryte powder of size less than 75µm passed through the sieve after softly agitating it for approximately 15mins before it was collected onto a clean pan and weighed.

Fig. 3: A sample of Obubra baryte of size 75µm obtained after milling

The mass fraction of baryte powder greater than 75µm, w_1, was calculated 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, and W_1 is the calculated value of the grain size.

pH determination

Hanna instrument (HI98129 Model) was used to ascertain the pH value of the baryte sample. Having placed roughly 20g of the sample in a beaker, distilled water measuring 20ml was then poured into the beaker. The emerging slurry formed was left standing for 60minutes and then stirred for an estimated 10 minutes (Ibe et al., 2016). The probe of the Hanna instrument was afterwards dipped into the slurry and the pH reading read from the instrument.

2.3 Elemental and mineralogical analyses

The elemental analysis was carried out using X-ray fluorescent (XRF) Nitron 3000 while a Panalytical diffractometer was used for the XRD analysis of the sample.

XRF analysis

The chemical composition in the baryte sample was determined using XRF analysis at Spectral Laboratory Services, Kaduna State, Nigeria using an X-ray fluorescent (XRF) Nitron 3000. After placing the sample on the sample holder, and the ray point over the sample holder, the ray switch was activated and the data obtained automatically in triplicates. The percentage of chemical composition in oxide and the elemental form of the samples were obtained using this procedure.

XRD analysis

The XRD analysis was carried out using a Panalytical diffractometer characterized by current and voltage equal to 40 mA, 45Kv, respectively, at temperatures of 21-23^{O}C. The computer system was switched on and the software of XRD switched on. The settings dialogue was clicked and all the required setting of power and temperature were checked to correspond to that of the XRD. After placing the sample on the sample holder and then placed in the sample chamber column. Then the door was shut and confirmed from the computer. The measurement setting was then set for scanned. The result of the scan was saved to a file and the results obtained were match with different library, such as the NIST and PubChem to obtain the name, chemical structure, and other physio-chemical characteristics of the sample.

3. Result and discussion

3.1 Physical Properties of Obubra baryte sample

Appearance

The colour of the un-pulverised Obubra baryte sample was ash with a fairly bright appearance. The higher the impurities especially the iron (Fe_2O_3) in baryte, the darker the baryte. Pure baryte is white to colourless in colour and bright in appearance. XRF analyses, showed that the Obubra baryte sample contains a relative low iron content (0.638%) when compared to a similar study on Azara baryte, by Edem et al. (2022) which recorded a darker colour with an iron content of 2.82%. The study also agrees with Afolayan et al. (2021) study, where the baryte sample with low impurities, (Fe_2O_3 = 1.39%) was white in colour.

Streak test

Baryte powder with a white colour an indication that the substance is possibly baryte (Bassey et al., 2021). Previous researches by Bassey et al. (2021) and Edem et al. (2022) on the characterization of baryte sample from Guma local government area of Benue State and Azara local government area of Nassarawa State respectively, equally produced a white streak.

Specific gravity

The use of the Le Chatelier flask method in determining the specific gravity of the sample recorded a value of 4.14g/cm^3. This value is slightly lower than the API specification of 4.2g/cm^3, meets Nigeria's department of petroleum resources 4.0 g/cm^3 mark. The specific gravity of Azara baryte by Abdullahi and Lawal (2010), recorded an even higher specific gravity value of 4.30g/cm^3 as did the specific gravity of baryte obtained from works by Bassey et al. (2021) both Torkula and Kaseyo of Guma local government area, Benue, Nigeria, which recorded a specific gravity value of 4.2g/cm^3 in both areas. The later three values all met the API specification of 4.2g/cm^3. However, in the benefication of Azara baryte by Mgbemere et al. (2018a) as well as a follow up research Mgbemere et al., (2018b) on the beneficiation of Azara baryte using froth flotation both recorded a specific gravity value of 3.027g/cm^3 and 3.72±0.06g/cm^3 and respectively. More recently, Edem et al. (2022) analyzed a sample of Azara baryte. The specific gravity was 3.45g/cm^3. The values from the last three researches all fell well below the API limit of 4.2g/cm^3.

Grain size (fineness)

Practically all the baryte sample passed through the 75μm sieve No. 200. Thus, signifying that all the particles were less than 75μm, and therefore, suitable for use in determining the specific gravity using a Le Chatelier flask

pH value

The recorded pH value which was 7.3, showed that sample was barely alkaline. This result puts the pH of the Obubra samples within API standard range of 7 ≤12.5 for drilling as a weighting agent (Christ, 2006; Osokogwu et al., 2014). This result is in line with similar researches carried out by Afolayan et al. (2021) on Torkula baryte samples from Guma local government area of Benue State, Nigeria. Here, the characterised baryte was essentially basic, with a pH value of 8.6. At 7.2, the pH research on Azara barytes by Edem at al. (2022) was equally basic.

Table 1: Summary of the physical properties of the Obubra baryte

S/N	Properties		Percentage (%) Azara and Torkula Baryte				
		Obubra Baryte	**API Specification (2010)**	**Abdullahi and Lawal (2010)**	**Tanko et.al (2015)**	**Afolanyan et.al (2021)**	**Edem et.al (2022)**
1.	Appearance	Grey	ND	ND	Pinkish	calabash	brownish
2.	Steak	White	white	ND	ND	ND	ND
3.	Specific gravity	4.0g/cm3	4.20g/cm3	4.30	ND	ND	ND
4.	Grain size	75μm	75μm	ND	ND	75μm	75μm
5.	pH	7.3	7 ≤12.5	ND	ND	8.6	7.5

ND = Not determined

3.2 Chemical compound assessment of the Obubra baryte by X-ray Fluorescence (XRF)

Table 1 gives the chemical compound of Obubara baryte as compared with previous researches and API maximum limit. The chemical compound of the Obubra baryte by XRF shows the presence of BaO and SO_3 along with a host of associated minerals, namely; SiO_2, CaO, Fe_2O_3, Al_2O_3, and K_2. The results show the occurrence of silica and alumina based mineral compound (clay minerals), hematite, as the most foremost impurities. Summarily, the percentage of impurities in the Obubra baryte was ascertained as high with a very low concentration of $BaSO_4$ (34.545%). Thus, it was deemed unsuitable for use in the formulation of drilling mud. Research on the determination of the constituents and suitability of Azara barytes for industrial applications by Abdullahi and Lawal

(2010), and the geochemistry and petrology of Azara baryte by Tanko et al. (2015), revealed that like in the present research on Obubra baryte, BaO, SO_3 and Fe_2O_3 were the principal compounds of baryte in both researches. However, their respective $BaSO_4$ values of 83.28wt% and 86.39% placed both samples well below API's value of 96wt%. Again, the assessment of the chemical composition and properties of Torkula baryte deposit, located in Guma local government area in Benue State of Nigeria by Afolayan (2021), equally showed the presence of $BaSO_4$, silicate salts, CaO, and Fe_2O_3. In addition, MgO was found in trace percentages. The limited quantity of gangue minerals as well as the comparative value of its $BaSO_4$ (87.79%) as compared to the API standard, showed that Torkula baryte could be upgraded to meet global standards using identified processing techniques. Recent research by Edem et al. (2022) on Azara baryte followed a similar order of concentration; BaO, SO_3, $A1_2O_3$, and Fe_2SO_3. However, unlike the present research, the Azara sample recorded a higher percentage of $A1_2O_3$ when compared to Fe_2O_3. The percentage of $BaSO_4$ at 15.885, made it unsuitable for use in drilling operations.

Table 2: Chemical compound of Obubra as compared with past researches and API limit

S/N	Composition	Obubra Baryte	Azara and Torkula Baryte (%)				
			API Specification (2010)	Abdullahi and Lawal (2010)	Tanko et.al (2015)	Afolanyan et.al (2021)	Edem et.al (2022)
1.	$BaSO_4$	34.545	95.00	83.28	86.39	87.79	15.885
2.	SiO_2	59.297	0.010	6.23	3.70	6.66	70.576
3.	Fe_2O_3	0.638	0.030	3.46	0.12	1.39	2.820
4.	CaO	0.038	0.010	1.40	1.32	0.589	0.250
5.	MgO	0.000	0.040	ND	1.00	0.086	0.000
6.	$A1_2O_3$	1.971	0.035	ND	ND	0.750	5.655

ND = Not determined

3.3 Non-Metallic Elemental content of Obubra baryte

The XRF results as shown in In Table 2, further shows the percentage of the major non-metallic elements in the Obubra baryte sample. Sulphur (S), Si (Silicon), O (Oxygen), in Obubra baryte sample were the principal elements in the Obubra baryte. Among the elements, O was recorded as the principal element. The quantity of the major elements in the Obubra baryte sample in descending order are: O (43.933%), Si (27.718%) and Sulphur (5.372%). Si, and S were equally the major non-metallic elements recorded by; Mgbemere et al. (2018a), Mgbemere et.al. (2018b), while O, along with Si, and S were the major non-metallic elements recorded by Edem et.al. (2022).

Table 3: Non-metallic elemental content of Obubra baryte as compared with previous researches

S/N	Composition	Percentage (%)			
		Obubra Baryte	Mgbemere et.al (2018a)	Mgbemere et.al (2018b)	Edem et.al (2022)
1.	Si	27.718	1.41	1.555	32.990
2.	S	5.374	11.5	34.4265	2.920
3.	O	43.933	ND	ND	47.529
4.	Cl	0.000	0.10	ND	0.000
5.	P	0.000	ND	0.4034	0.000

ND = Not determined

3.4 Metallic elemental content of Obubra baryte

The metallic content of Obubra baryte as compared with previous researches and API limit are shown in Table 3. Barium (Ba) has the highest concentration in Obubra baryte and is followed by strontium (Sr), titanium (Ti), aluminuim (Al) and iron (Fe). The zinc (Zn) and magnesium (Mg) contents were all within the limits set by the American Petroleum Institute (API). As an oxide, Obubra baryte contains, (barium sulfate) ($BaSO_4$), celestine ($SrSO_4$), rutile (TiO_2), kaolinite ($Al_2Si_2O_5(OH)_4$), hematite (Fe_2O_3), pyrite (FeS_2 (iron (II) disulfide)), magnetite (Fe_2+Fe_3+2O_4), sphalerite ((Zn,Fe)S), chalcopyrite ($CuFeS_2$) and chalcocite (Cu_2S), associated minerals, with celestine, rutile, kaolinite, and hematite as the principal gangue minerals.

Table 4: Metallic elemental content of Obubra baryte as compared with previous researches and API limit

Composition	Concentration (mg/l)/(mg/kg)						
	Obubra Baryte	**API specification (2010)**	**Ibe et al (2016)**	**Mgbemere et.al (2018a)**	**Mgbemere et.al (2018b)**	**Afolayan et.al (2021)**	**Edem et.al (2022)**
Ba	18.926	ND	ND	36.2004	36.2004	ND	7.698
Mn	0.007	ND	ND	0.2913	1.77	ND	0.069
Ti	0.713	ND	ND	14.710	14.710	ND	0.099
Sr	0.854	ND	ND	ND	ND	ND	ND
Au	0.009	ND	ND	ND	0.02	ND	0.016
Fe	0.446	Zero	62.20	0.1762	10.1	15.6094	1.973
Al	1.043	ND	ND	1.7642	ND	0.9	2.993
Sn	0.000	ND	ND	0.2779	ND	ND	0.000
K	0.005	ND	ND	ND	ND	ND	2.239
Ca	270	250	709.00	ND	ND	34.0135	0.179
Cu	37	36	160.00	ND	ND	0.3024	0.047
Zn	100	140	1.60	ND	ND	3.9051	0.001
Pb	ND	1000	6.00	ND	ND	113.8127	ND
Mg	0.000	250	64.20	ND	ND	8.5726	0.000
Cd	ND	5	1.60	ND	ND	0.0008	ND

ND = Not determined

3.5 Mineralogical analysis by x-ray diffraction (XRD)

Fig. 4 identified, kaolinite, muscovite ($KAl_2(AlSi_3O_{10})(F,OH)_2$, or $(KF)_2(Al_2O_3)_3(SiO_2)6(H_2O)$, hemaetite, ($Fe_2O_3$), lillite ($(K,H_3O)(Al,Mg,Fe)_2(Si,Al)_4O_{10}[(OH)_2\cdot(H_2O)]$, gypsum ($CaSO4\cdot2H_2O$) and garnet ($(X_3Z_2(TO_4)_3$ (X = Ca, Fe, etc., Z = Al, Cr, etc., T = Si, As, V, Fe, Al) as the major minerals in the Obubra baryte sample. This finding agrees with earlier works by Tanko et al. (2015); Aladesanmi et al. (2018); Mgbemere et al. (2018a,b) and Edem et al. (2022) as summarised in Table 3

The X-ray diffractogram in Fig. 5 shows the composition and crystallographic data of the Obubra baryte sample. Identifiable peaks in the diffractogram can be seen, with quartz having the most peaks. Other gangue minerals captured in the X-ray diffractogram include kaolinite, muscovite, hematite, lillte, gypsum and garnet. This is in line with reports given in the literature (Aladesanmi et al., 2018; Mgbemere et al., 2018a,b; Tanko et al., 2015).

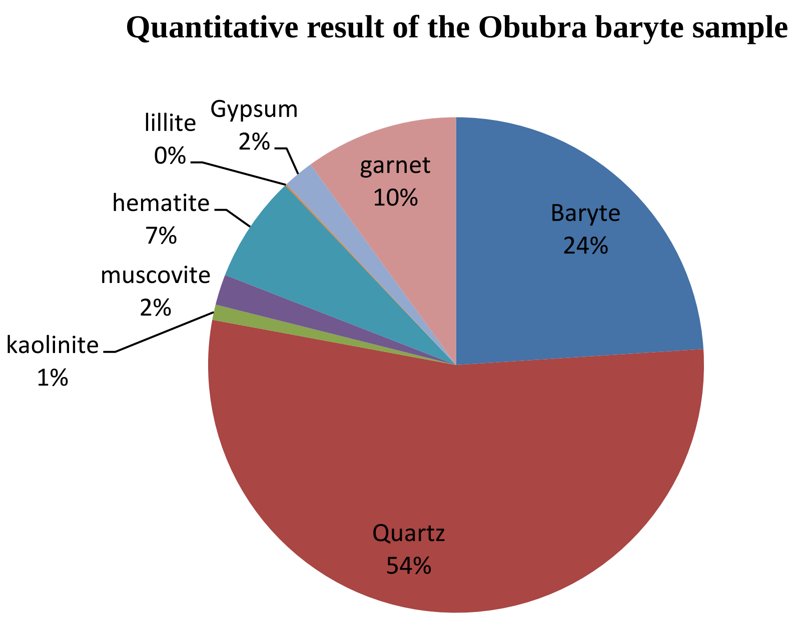

Fig. 4: Quantitative result of the Obubra baryte sample

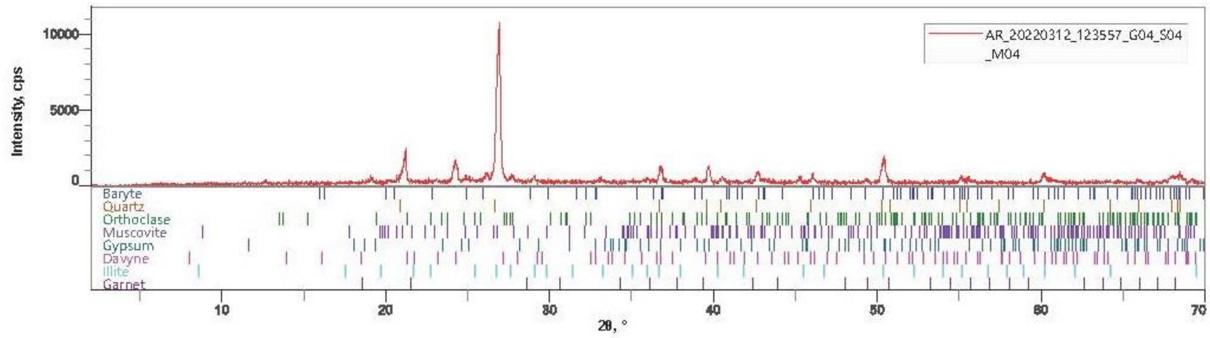

Fig. 5: Quantitative analysis report of the Obubra baryte sample

4. Conclusion

The experimental results in the presented study led to the following conclusions:

1. The baryte sample is ash in colour, possibly because of the low percentage of Fe_2O_3 in the sample
2. A white colour was obtained after conducting the streak test.
3. Its specific gravity of 4.14g/cm^3 fell short of the API's 4.2g/cm^3 for drilling operation. However, it was above the 4.0g/cm^3 limit set by Department of petroleum resources (DPR) drilling mud formulation in Nigeria.
4. The pH value (7.3) of the Obubra baryte is within the American Petroleum Institute (API) operational pH and the standard range for a weighting agent.
5. The Obubra baryte sample at a BaSO4 of 34.545%, is well below the API tolerable level of 96% for drilling operation
6. The Al_2O_3, SiO_2, CaO, and Fe_2O_3 were all above the API acceptable limit for use in drilling mud formulation. However, MgO 0.00% value met the API tolerable limit of 0.04%.
7. Quartz, kaolinite, Muscovite, Hematite, Illite, gypsum, and garnet were identified as the associated mineral present in the baryte ore.
8. The Obubra baryte can be applied in the production of paper, paint, and rubber.
9. If required for use in drilling operations, it must be processed, in order to bring the BaSO4, Al2O3, SiO2, CaO, and Fe2O3 percentages to API specifications.

10. The specific gravity, the ore can be subjected a fairly simple processing technique to bring it to API specification of 4.2g/cm^3.

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