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Morphological, Mineralogical and Chemical Characterizations of Azara Barite and its Yield Property as a Weighting Agent in Drilling Fluid

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ABSTRACT

Local Barite was mined from Azara Development Area in Awe Local Government Council of Nasarawa State, Nigeria. The mined mineral was characterized using X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM), and Electron Dispersion X-ray (EDX) techniques. Water-based drilling mud was prepared using commercial bentonite according to the API 13A Standard. Effect of gradual addition of Azara barite as weighting agent on the yield point of drilling mud was studied. XRD analysis showed that the dominant mineral phase in the material was barite. The SEM morphological analysis showed that Azara barite had lumpsome morphology having orthorhombic crystal shape. The average particle size of the microstructure was estimated as 100 µm. The specific gravity of Azara barite was determined as 3.9. Chemical composition analysis carried out showed that the material contained 83.61 wt% barite. Mud yield point analysis of the material showed that Azara barite was only suitable as weighting agent for drilling mud at composition below 60 wt%. Above 60 wt% the mud was completely unsuitable for drilling fluid applications. The optimum yield point of the formulated drilling mud using Azara barite as weighting agent was 2.0 lb/100 ft^2 corresponding to formulation with ≤ 40 wt% weighting agent composition.

1. INTRODUCTION

Drilling mud is a suspension fluid used to aid drilling of petroleum and gas exploitation wells in the oil and gas industry. Drilling mud is also used for exploration drill in the petroleum upstream operations. Drilling mud is either water-based, oil-based or gas based (Cheraghian, et al., 2018). The liquid-based drilling mud is the most popular in petroleum upstream operations. Drilling mud is generally prepared by forming a suspension of drilling powder in water making a homogeneous fluid. The drilling powder is basically made of bentonite and other appropriate clay additives.

Bentonite is a clay mineral belonging to the montmorillonite mineral family. Montmorillonite structural architecture consists of an octahedral alumina layer sandwiched between two tetrahedral silica sheets. They are regarded as 1:2 category of clay (Jianwei et al., 2018). Bentonite's chemical structure possess exchangeable interlayer cations which are responsible for their ion exchange capacity and swollen property. The exchangeable cation in bentonites is either monovalent-cation based or divalent-cation based. The monovalent exchangeable cation is mainly Na^+ while the divalent exchangeable cation is Ca^{2+}. Most naturally occurring bentonite minerals possess Ca^{2+} exchangeable cation (Sans et al., 2017). Bentonites with Ca^{2+} exchangeable cation have lower swelling and water absorption property, due to their low ion exchange capacity. On the contrary, bentonites with Na^+ exchangeable cation have higher swelling and water absorption property, due to their high ion exchange capacity. Therefore, the Na^+ based bentonites easily form a more viscous fluid (Eisenhour and Brown, 2009) which makes them excellent for drilling fluid application than their Ca^{2+} counterpart. Most of the commercial bentonites used for drilling fluid formation are made of Na^+ based bentonites, although, some are made of mixed exchangeable cations; Ca^{2+}/Na^+ (Jianwei et al., 2018). Other industrial applications of bentonite include cosmetics, pharmaceuticals, foods, ceramics, paints, paper and iron industries (Harben, 1995; Churchman et al., 2002; Allo and Murray, 2004; Murray, 2006; Güven, 2009; Maldonado et al., 2011; Bergaya and Lagaly, 2013; Antonio and Madelyn, 2017).

Additives incorporated in the production of drilling fluid powders are rheological property boosting polymers and weighting agents. The role of weighting agents in drilling fluid preparation is to ensure mud density enhancement, thereby ensuring stability of the drilled borehole and increasing the penetration rate of the borehole (Basfar et al., 2020). The presence of weighting agent in drilling fluid also results in hydrostatic pressure in the borehole thereby minimizing fluid loss as a result of deposition of thick filter cake on the wall of the well (Abdou et al., 2018).

Barite, having the chemical formula; $BaSO_4$, has been widely reported as a good weighting agent in drilling mud formation (Ezzat et al., 1999; Musaed and Ahmad, 2000; Güven, 2009; Abdou et al., 2018; Badr et al., 2019). Apart from application as additive in drilling mud, barite is also used in other industrial applications such as its use as raw material in paints, fertilizer, glass, match and pharmaceutical industries (Aladesanmi et al., 2018). Barite occurs in three forms, which include stratiform or bedded, residual and in veins. Geologist have described the barite veins in Piedmont belt, Virginia U.S.A being as a result of metamorphosed rocks (Kaiser et al., 1987). Residual deposits of barite are formed when disseminated barite or barite veins in pre-existing rocks accumulate on a basement as the previous host rock is eroded and weathered. The important deposits of Washington County occur in residual clay derived from Cambrian dolomite in which primary deposits occur (Mugel, 2017). Barite deposits in commercial quantity have been reported in many parts of the world including the huge veins widely occurring in the United States of America, Africa and Asia (Dominic et al., 2014).

Nigeria is one of the African countries hugely endowed with commercial deposits of barite. The Geological Survey of Nigeria has reported that huge commercial deposits of barite are found in Cross Rivers, Benue, Plateau and Nasarawa States (Dominic et al., 2014). The commercial deposit in Nasarawa State is located at Azara Development Area in Awe Local Government Council of Nasarawa State. Azara barites deposit is the best-known deposit of barites in Nigeria (Aladesanmi et al., 2018). The barite deposit in Azara occurs as hydrothermal veins within the Cretaceous Keana sandstone of the Middle Benue Trough (Aladesanmi et al., 2018). Based on report of the Federal Ministry of Solid Minerals Development, Azara has an estimated reserves of about 730, 000 tons of barite (Dominic et al., 2014). The global demand of barite for drilling operations has been reported as approximately 2,000,000 tons per year (Abdou et al., 2018).

As the major oil producing nation in Africa, Nigeria produces about 2 million barrel per day of crude oil, in all this, drilling fluid formulated with barite weighting agent is applied. The drilling fluid and barite weighting agent currently used in the Nigerian upstream operation are imported, causing the nation huge capital flight. Although, Nigeria is blessed with huge deposit of barite, sufficient research has not been carried out to ascertain properties of local barites so as to harness their qualities for industrial applications. Therefore, in order to change the current trend of capital flight resulting from importation of barite and determine suitability of Nigerian local barites for industrial applications, there is need for extensive study of the characteristics of local Nigerian barite. The aim of this work is to study the morphological, mineralogical and chemical characterization of Azara barite and investigate its impact on the yield point of drilling mud when it is applied as weighting agent.

2. MATERIALS AND METHODS

2.1. Materials

The materials used include raw barite mined from Azara development Area, Awe Local Government Council of Nasarawa State, Nigeria, commercial bentonite and distilled water. Apparatuses used include viscometer (Maker: Fann Model 35A), electronic compact scale (Maker: Kerro BL10001), Hamilton Beach mud mixer (CPM800), digital stop watch, beakers, measuring cylinders, spatula and mixing bowl.

2.2. Mud Preparation

The drilling mud was prepared according to the API 13A standard (Khan et al., 2017). For every preparation, 6% of drilling mud solid was applied in distilled water and was thoroughly mixed for about 30 min until a homogenous mixture was obtained. For 0 wt% weighting agent formulation, the drilling mud sample was prepared by dissolving 30 g of commercial bentonite in 500 ml of distilled water. The mixture was thoroughly mixed for about 30 min using Hamilton Beach commercial mixer until the drilling mud became completely homogeneous. For a 10 wt% weighting agent formulation, the above procedure was repeated but 10 wt% of the initial bentonite (30 g) was substituted with barite. Therefore, the formation was 27 g bentonite plus 3 g barite dissolved in 500 ml distilled. Similarly, for 20 – 100 wt% weighting agent formulations, the above procedure was repeated with barite introduced as weighting agent at various weight percent ranging from 20 – 100 wt% at intervals of 10 wt%.

2.3. Yield Point Determination

The mud sample prepared was poured into a viscometer cup to the scribed mark. The viscometer cup was adjusted until the sleeve of the viscometer was into the mud unto the level indicated in the spindle. The viscometer knob was adjusted to obtain 300 and 600 rpm dial readings. The dial reading of the various mud formulations at 600 rpm was denoted as A. The dial reading of the various mud formulations at 300 rpm was denoted as B. Therefore, the yield point of the various mud formulations (in lb/100 ft^2) was determined using Equation (1) (Basfar et al., 2020).

$$\text{Yield point} = 2B - A \tag{1}$$

2.4. Specific Gravity

Specific gravity of the weighting agent was measured using density bottle. The specific gravity was determined using Equation (2).

$$\text{Specific gravity} = \frac{(W_2 - W_1)}{(W_4 - W_1) - (W_3 - W_2)} \tag{2}$$

Where:

W_1 = weight of empty bottle
W_2 = weight of bottle + barite
W_3 = weight of bottle + barite + water
W_4 = weight of bottle + full water

2.5. Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray (EDX)

SEM and EDX analyses were carried out using PhenomWorld Scanning Electron Microscope; Prox, manufactured by Eindhoven the Netherlands. The SEM machine was allowed to stabilize at about vacuum of 9 x 10^{-5} torr. The solid was pretreated by pulverization and spurting. Few samples of the solid, about 20 mg was mounted on the stub of a spurting machine to coat the solid surface with about 40 nm of gold. Then the spurted sample was placed in the SEM sample holder. Using the computer interface, magnification setting of 500x was made and images at different selected areas were collected. Similarly, at other magnification settings of 1000x and 1500x images at different selected areas were collected. Also using the computer interface EDX data of the sample was collected at about 1000 counts/s.

2.6. X-ray Diffraction

XRD patterns were recorded from Bragg's angle (2θ) of 5° to 70°; Rigaku Miniflex600, operated at continuous scanning, scanning speed of 5°/min, generator settings of 10 mA and 40 kV, and CuKα node material.

3. RESULTS AND DISCUSSION

Figure 1 shows XRD patterns of the weighting agent used in the drilling mud prepared. It could be observed that the material contained barite as the major mineral phase. The barite phases were identified at 2θ values of 23.1°, 25.1°, 26.1°, 28.9°, 31.7°, 41.0°, 43.1°, 51.3°, 55.0°, 60.5° and 65.8° (Chen, 1977; Al-Awad and Al-Qasabi, 2000). Other impurity phases present in the mineral were bauxite, gibbsite, hematite, quartz and vermiculite. Hematite and bauxite phases were identified at 2θ values of 36.46º and 44.30°, respectively. Gibbsite and vermiculite phases were identified at 2θ values of 27.03º and 33.02º, respectively. Quartz phases were identified at 2θ values of 20.62º and 38.93º. Sharp peaks observed in the XRD patterns of Azara barite show presence of highly crystalline microstructure.

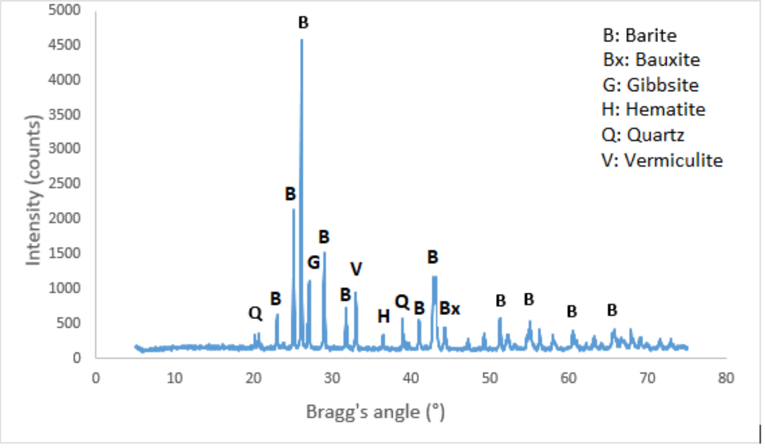

Figure 1: XRD patterns of Azara barite

Using Equation (2), the specific gravity of the commercial bentonite was determined as 1.03 while that of the weighting agent was 3.9. Azara barite has a slightly lower specific gravity compared to the API minimum specific gravity value of 4.2 for barite (Al-Awad and Al-Qasabi, 2000; Khan et al., 2017; Fadl et al., 2020).

Figures 2(A) – 2(C) show SEM images of Azara barite used as weighting agent in the prepared drilling mud, at various magnifications. Figures 2(A) and 2(B) are images at 500x and 1000x magnification, respectively.

It could be observed from Figure 2(A), that the material possesses a lumpsome morphology having fairly evenly distributed lumps of defined shape with irregular particle sizes. Figure 2(B) shows clearer particle distribution of the material than Figure 2(A), showing micrograph of fewer particles of the material at magnification of 1000x which makes the lumpsome morphology and defined shape more pronounced. Figure 2(C) shows the most detailed micrograph of a crystal. The crystal shape of the unit particle can be described as orthorhombic with some degree of crystal shape imperfection possibly due to high degree of crystal defects. The average particle size was estimated as 100 μm.

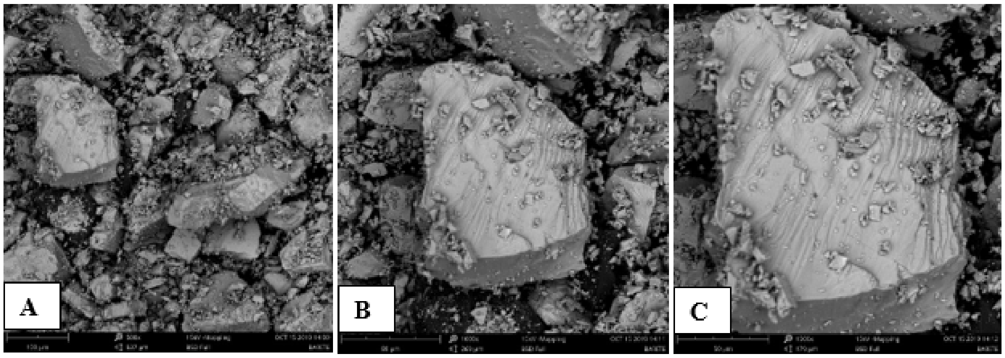

Figure 2: SEM images of Azara barite at magnifications of (A); 500x (B); 1000x (C); 1500x

Figures 3(A) – 3(C) show SEM images of commercial bentonite used for the water-based drilling mud, at various magnifications. Figures 3(A) and 3(B) are images at 500x and 1000x magnification, respectively. Although, Figure 3(B) shows clearer particle distribution of the bentonite than Figure 3(A), both images show relatively even distribution of micro-particles of the bentonite having irregular sizes and shape. Figure 3(C) shows a micrograph of the bentonite at magnification of 1500x. The micrograph is clearer as it reveals clearer details of the particles. It could be observed that the particles have clumpy morphology and irregular crystal shapes. The average particle size was estimated as 25 μm.

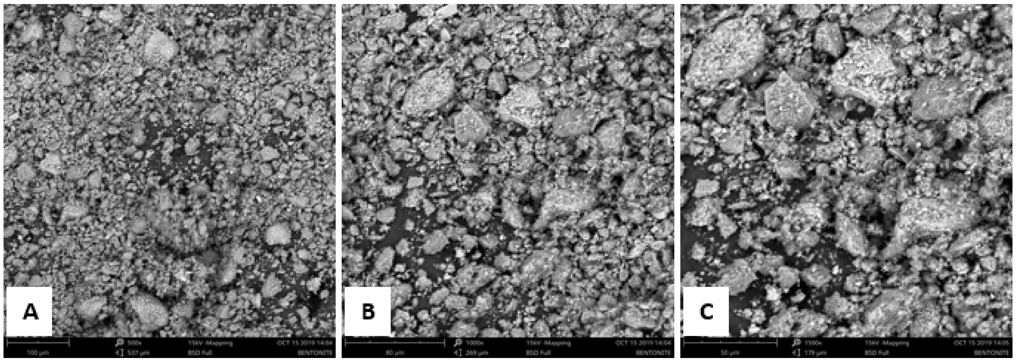

Figure 3: SEM images of the commercial bentonite used at magnifications of (A); 500x (B); 1000x (C); 1500x

Table 1 presents the Energy Dispersive X-ray (EDX) elemental analysis of Azara barite and the commercial bentonite used in the formation of drilling mud. The chemical analysis has shown that the major chemical constituent of the weighting agent was barite as it contained 83.62 wt% barite. Other chemical composition of the material included 2.47 wt% SiO_2 which was likely due to the associated quartz and vermiculite phases identified in the XRD result. The material had 0.56 wt% Al_2O_3 which was likely due to the associated gibbsite, bauxite and vermiculite phases identified in the XRD result. There was 0.95 wt% FeO which was likely due to the associated hematite phase identified in the XRD result. Other compositions such as calcium oxide, titanium oxide, silver oxide, manganese oxide, potassium oxide, vanadium oxide and chromium oxide, magnesium oxide and sodium oxide analyzed as 0.77, 5.01, 1.91, 0.96, 0.67, 1.96, 0.74, 0.17 and 0.21 wt%, respectively were likely due to impurity associated with the Azara barite.

The dominant composition of the commercial bentonite was silica which was present up to 39.37 wt% followed by alumina which was 24.87 wt%. These show that the material is an aluminosilicate material. The silica-alumina ratio of the material was 1.58, this is typical of a montmorillonite clay group which bentonite belongs.

Table 1: Energy dispersive X-ray (EDX) elemental analysis of Azara barite and the commercial bentonite used

Chemical composition	Composition (wt%)	
	Commercial bentonite	Barite
$BaSO_4$	4.24	83.62
SiO_2	39.38	2.47
Al_2O_3	24.87	0.56
FeO	7.39	0.95
CaO	4.32	0.77
TiO	3.77	5.01
Nb_2O	3.40	ND*
AgO	3.08	1.91
MnO	2.64	0.96
K_2O	1.92	0.67
VO_2	1.75	1.96
CrO_2	1.72	0.74
MgO	0.89	0.17
Na_2O	0.63	0.21
Total	100.00	100.00

*ND: Not determined

The elemental analysis indicated that the bentonite contains both monovalent and divalent cations. The calcium oxide content was 4.32 wt% while the sodium oxide was 0.63 wt%. The bentonite had a mixed interlayer cation which was largely calcium based, meaning that the Ca^{2+} is the exchangeable interlayer cation in the 1:2 structural architecture of the montmorillonite mineral constituting the bentonite. Other chemical compositions of the commercial bentonite were iron, barium, titanium, niobium, silver and manganese to the extent of 7.39, 4.24, 3.77, 3.40, 3.08 and 2.64 wt%, respectively. These are likely contributed by associated mixed mineral phases in the bentonite or chemical additives added to the processed bentonite to improve its rheological characteristic so as to make it suitable for drilling fluid application. In particular, the barium oxide of 4.24 wt% was likely due to presence of some barite additive. Vanadium and chromium were present at 1.75 and 1.72 wt%, respectively, they are likely due to presence of associated impurities. The potassium and magnesium compositions of 1.92 and 0.89 wt% were likely due to salt additives for enhancement of the rheology of the drilling mud.

Figure 4 shows the elemental distribution mapping of the energy dispersive X-ray (EDX) for (A); the the commercial bentonite used and (B); Azara barite. The distribution mapping for the commercial bentonite was collected at 1000 counts/s while that of the barite was collected at 1464 counts/s. It could be observed that the dominant peaks in the commercial bentonite were due to silicon and aluminum, this further shows that the material was aluminosilicate material and this agrees with the XRD and the chemical analysis results which have shown that the dominant phase of the material was montmorillonite. The EDX elemental distribution mapping image of the Azara barite showed that the dominant peaks in the material were due to barium and sulfure, this further confirm that the major mineral content of the material was barite as already identified by the XRD and chemical analysis results.

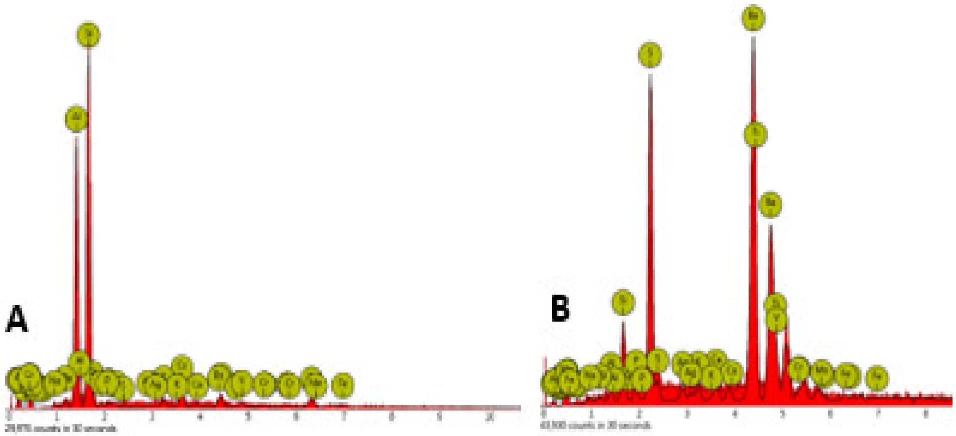

Figure 4: EDX elemental distribution mapping of (A) commercial bentonite (B) Azara barite

Figure 5 shows the viscometer dial readings at 300 and 600 rpm and the plastic viscosity against weight percent of the barite weighting agent. The plastic viscosity of a drilling fluid is given by Equation (3) (Fadl et al., 2020). It could be observed that the plastic viscosity of the drilling mud formulated reduced continuously from its initial value of 6.0 lb/100ft^2 at 0.0 wt% weighting agent to 3.0 lb/100ft^2 at 40 wt%.

$$\text{Plastic Viscosity} = \text{600 rpm reading} - \text{300 rpm reading} \tag{3}$$

The plastic viscosity value is a reflection of the fluidity and rheological behaviour of a drilling fluid. To analyze the observed trend of plastic viscosity properly, there is need to further analyze the rheological model of the mud. Using the power law model, it follows that:

$$\tau = k\gamma^n \tag{4}$$

Where τ = fluid shear stress (lb/100ft^2)

γ = shear rate (s^{-1})

n = power low index. When n < 1, the fluid is shear thinning and when n > 1, the fluid is shear thickening. n is defined by Equation (5) (Adewale et al., 2017).

k = consistency index and gives the level of the fluid thickness, the higher the k value, the thicker the fluid is. k is defined by Equation (6) (Adewale et al., 2017).

$$n = 3.32\log\left(\frac{\theta_{600}}{\theta_{300}}\right) \tag{5}$$

$$k = 5.11\log\left(\frac{\theta_{300}}{511^n}\right) \tag{6}$$

Figure 6 shows the variation in the power law indices against weight percent of weighting agent. The power low index, n was less than 1.0 for drilling mud formation having weighting agent composition between 0 – 60 wt%. It could be observed that a gradual decrease of n value occurred from 0.807 at 0 wt% to 0.678 at 40 wt%. A reverse trend was observed above 40 wt% as n value rose from 0.678 at 40 wt% to 0.999 at 60 wt%. Above 60 wt%, value of n rose exponentially above 1.0 until a peak value of 2.0 was reached at 90 wt%. This implies that for weighting agent composition $\leq$ 60 wt%, the formulated mud characteristic was shear thinning while for the formulation > 60 wt% the mud characteristic was shear thickening (Adewale et al., 2017). It can also be inferred that the transition region between the shear thinning and shear thickening characteristics occurred between 40 – 60 wt%.

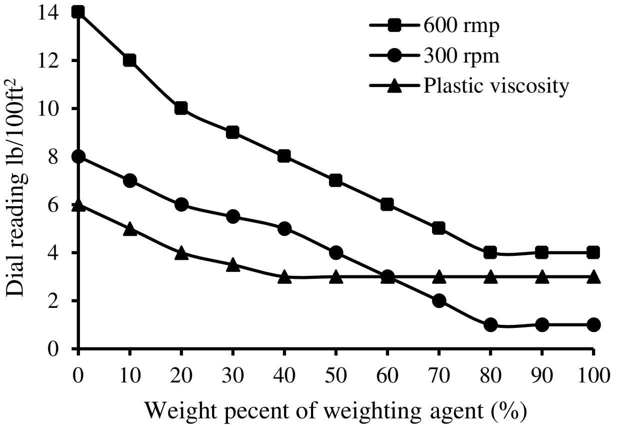

Figure 5: Dial readings and plastic viscosity against weight percent of weighting agent

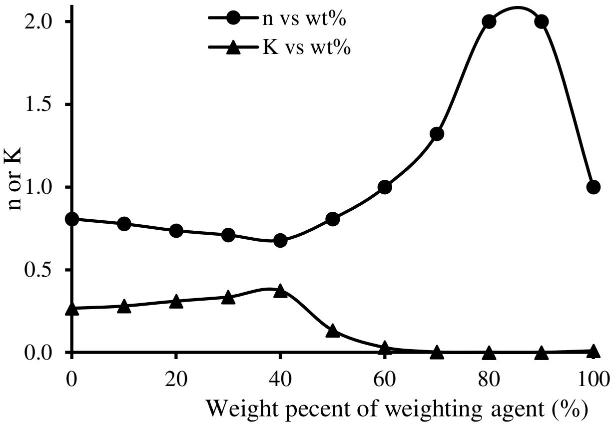

Figure 6: Power law model indices against weight percent of weighting agent

The fluid consistency index, k was 0.267 at 0 wt%. The value increased gradually until the peak value of 0.373 was attained at 40 wt%. Further from 40 wt%, k value decreased continuously until it became 0.0 at weighting agent composition of 60 wt% and above. It implies that the mud was thickening and having improved fluidity with addition of weighting agent between 0 – 40 wt% and its behaviour was non-Newtonian (Adewale et al., 2017). Above 40 wt%, the mud began to lose its non-Newtonian characteristics. At 60 wt% and above the mud became Newtonian (Saasen and Ytrehus, 2020).

Figure 7 shows the mud yield point against weight percent of barite weighting agent. It could be observed that the yield point of the drilling mud was constant at 2.0 lb/100 ft^2 for weighting agent composition of 0 – 40 wt%. Between 40 – 60 wt% of weighting agent the yield point of the mud decreased steadily until it was 0.0 lb/100 ft^2 at 60 wt% and above. It could be deduced that the rheological property of the mud was plastic for weighting agent composition of 0 – 40 wt%. Above 40 wt% the mud lost its plasticity and gradually became brittle. At weighting agent composition $\geq$ 60 wt% the mud was completely brittle. The region between 40 – 60 wt% could be seen as the transitional region between plastic and brittle behavior of the formulated mud. This further corroborate analyses of the power law indices which have indicated that the mud exhibited shear thinning and non-Newtonian characteristics for weighting agent composition $\leq$ 60 wt%. Whereas, for weighting agent composition > 60 wt% the mud the mud exhibited shear thickening and

Newtonian characteristics. A drilling fluid formulation using weighting agent composition higher than 60 wt% will result into a poor petroleum production as a result of substantial pressure loss in the reservoir production line occasioned by presence of several local fracture fronts in the drilling mud as a result of deformation of the mud rheological characteristics from non-Newtonian for weighting agent composition ≤ 60 wt%. to Newtonian for weighting agent composition > 60 wt%.

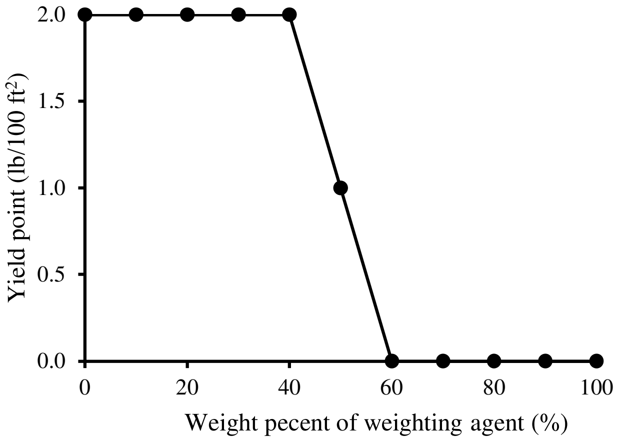

Figure 7: Mud yield point against weight percent of weighting agent

4. CONCLUSION

XRD analysis of the weighting agent showed that the material was a barite mineral having some mixed phases of bauxite, gibbsite, hematite, quartz and vermiculite. The chemical composition analysis carried out using EDX has further confirmed that the major chemical composition of the weighting agent was barite as it contained 83.61 wt% barite. SEM analysis of Azara barite showed that the material had lumpsome morphology having orthorhombic crystal shape. The average particle size was estimated as 100 μm. SEM analysis of the commercial bentonite revealed that the mineral possessed clumpy morphology with irregular crystal shapes and an estimated average particle size of 25 μm. Chemical compositional analysis of the commercial bentonite indicated that the material was an aluminosilicate mineral having a silica-alumina ratio of 1.58. The bentonite had a mixed interlayer exchangeable cation. Na^+ was the monovalent exchangeable interlayer cation and Ca^{2+} was the divalent exchangeable interlayer cation. The divalent cation was predominant as the calcium oxide content was 4.32 wt% while the sodium oxide content was 0.63 wt%. The specific gravity of Azara barite was 3.9. Although, this value is relatively low but relatively comparable with other reported values for barite. The yield point of the formulated drilling mud using Azara barite weighting agent was 2.0 lb/100 ft^2 for formulation having 0 – 40 wt% weighting agent composition. Above 40 wt% the yield point decreased steadily until it was 0.0 lb/100 ft^2 at 60 wt% weighting agent composition. The drilling mud formulated using Azara barite was good for drilling applications only for weighting agent composition less than 60 wt% as the mud losses its non-Newtonian rheological characteristics above 60 wt%. The Mud formulated was plastic and strongly non-Newtonian at weighting agent concentration (C) of $0 \leq C \leq 40$ wt%, this region corresponded to a yield point value of 2.0 lb/100 ft^2. At $40 \leq C < 60$ wt% the mud displayed necking property with continuous drop in the yield point value, this region also marked the gradual loss of non-Newtonian characteristics of the mud. At $C \geq 60$ wt% the mud became brittle and Newtonian having yield point value of 0.0 lb/100 ft^2. At weighting agent composition greater than 60 wt% the mud was completely unsuitable for drilling fluid applications.

5. ACKNOWLEDGMENT

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6. CONFLICT OF INTEREST

There is no conflict of interest associated with this work.

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