



FORMULATION AND CHARACTERIZATION OF NIGERIAN FIKA BENTONITE FOR APPLICATION IN DRILLING



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Abstract:

The consumption of bentonite clay in Nigeria has continued to increase as drilling activities keep increasing. Huge amount is spent for the importation of this clay from overseas. In this work, water based muds were formulated from the local bentonite clay and characterized for application in drilling operations. The bentonite clay (calcium based) was beneficiated with soda ash (Na_2CO_3) for its conversion to sodium based bentonite through ion exchange. Five samples of bentonite muds (A-E) were prepared using 350 ml distilled water and additives comprising of 10g barite and polyanionic cellulose (PAC) of varying amount (1-2.5g). The rheological properties of clay were determined and compared with commercial bentonite. The results of the apparent viscosity were 3.5-24.5 cP, yield point 3.0-23.0 lbf/100ft², plastic viscosity were 2.0-12.0 cP and the gel strength 1.0-4.0 lbf/100ft². The yield point to plastic viscosity ratio and plastic viscosity of the samples without additives are within the specified limits of oil company material association (OCMA) standard. Fika bentonite clay exhibits good rheological properties that could compete favorably with that of commercial bentonite when beneficiated with soda ash, barite and polyanionic cellulose. Hence, drilling fluid could be formulated from Fika clay for application in drilling operations.

Keywords:

Bentonite, formulation, drilling fluids, rheology and characterization

Introduction

The most important industry in Nigeria is the oil and gas industry due to its enormous impact in foreign exchange. This has positioned the oil and gas industry in the forefront of the Nigerian economy. Drilling operation is one of the most vital cogs in the oil and gas industry. Drilling operation is important in determining the oil and gas as well as borehole water present beneath the earth and a vital ingredient for this operation is drilling fluid which is commonly called drilling mud. The importance of clay and clay minerals in drilling industry is from the fact that clays are added to drilling fluid to build viscosity, thixotropy, and contribute wall building properties. The most commonly used clay in drilling fluid formulation is the sodium based bentonite (Shuwa & Sabiu, 2019). Drilling is the process of creating pathway for the discovered hydrocarbon to be extracted at the surface, it involves the drilling of the earth crust thousands of feet to where the hydrocarbon are accumulated in reservoir by rotary drilling process (Dewu *et al.* 2012).

Drilling fluids are heterogeneous mixture of chemical, water or oil and clay materials that aid in drilling operation. They are important in successful well drilling as they have common properties that facilitate safe and satisfactory completion of the well such as bottom hole cleaning, controlling high pressure zones and removal of cutting surfaces. It cannot be overemphasized how important drilling fluids are as the knowledge of drilling fluid is a requisite in the rotary drilling operation in the petroleum industry. The cost of drilling operation is also influenced by the performance of the drilling fluid. This in turn makes the

design, formulation and maintenance of drilling fluids important (Afolabi *et al.* 2017).

Nigeria as a nation is blessed with abundant bentonite resources which if well harnessed will reduce the importation of drilling fluid ingredients and specialized drilling fluids. Every region in Nigeria has been reported to have a substantial deposit of bentonite clays. The proven reserve of bentonite in Nigeria has been modestly estimated to be above 700 million metric tons (Omole *et al.* 2013; Bilal *et al.* 2015).

Bentonite is primarily expandable montmorillonite clay. Montmorillonite is a 2:1 type of mineral, and its unit layer structure consists of one Al^{3+} octahedral sheets. The modification reactions alter the surface and structural characteristics of clay by replacing the interlayer cations (e.g., Na^+ , K^+ , Ca^{2+}) with specific species or sites. Montmorillonite clays have the smallest crystals and hence the largest internal and external surface areas for cation exchange. The current market price of montmorillonite clays is 20 times cheaper than that of commercial activated carbon, which brings about its prospective as adsorbent candidate for wastewater remediation (Jock, *et al.* 2018).

Bentonite has been used as a drilling fluid for many years. The mud of the drilling fluid must have high viscosity and thixotropic property, which prevents settling of fine materials and freezing the bits during oil well drilling (Harvey and Lagaly 2013). The common drilling fluids are mainly based on dispersed sodium bentonite. Bentonite is also used as a bleaching agent for oils by adsorption to remove some pigments. In some cases, calcium bentonite is activated with an acid (to increase surface area and bleaching activity) to produce bleaching earths as a refining

and clarifying agent to remove impurities such as fatty acids, gums, trace metals, and colors in the production of edible oils and fats (Murray 2007).

Beneficiation is simply a process of removing impurities or associated minerals that are not required thereby improving the quality of the bentonite clay. Beneficiation can be carried out using sodium salt such as sodium carbonate (Na_2CO_3) or sodium hydrogen carbonate (NaHCO_3) which permits for the conversion of the mostly calcium montmorillonite clays to sodium montmorillonite via an ion exchange mechanism (Igwilo et al. 2020).

Commercial bentonite clays imported for water borehole, oil and gas drilling are reported to be deficient in influential properties required for the drilling operation. Often, they fail to perform the basic functions of the drilling fluid during drilling operation. In spite of abundant deposit of bentonite clays, commercial drillers still import the clay from overseas. The Nigerian bentonitic clays are reported to be suitable for drilling application especially when activated with sodium salt to produce the high grade sodium bentonite. In this study, Nigerian Fika bentonite was activated with sodium carbonate and drilling formulated from the activated clay. Rheological properties of the drilling fluid were compared with commercially imported clay.

Materials and Methods

Sample collection and preparation

The bentonite clay was obtained from the department of chemical engineering, A.B.U, Zaria, originated from Fika, Yobe State, North-East, Nigeria. The clay in lumps form was sun dried for three weeks and crushed using a jaw crusher. The commercial bentonite was procured from a commercial driller.

Sample Beneficiation

Physical Beneficiation

The method employed for the beneficiation of the bentonite was adopted from Jock et al. 2016. Ten kilograms of the crushed bentonite was soaked in water for 24 h. The clay-water mixture was blunged (stirred) for 3 h at 25°C and was allowed to age for 4 days for the quartz impurities to sediment to the bottom leaving colloidal solution of clay and suspended particles at the top. At each day of sedimentation, decantation of the overflow was done and replaced with fresh tap water to the fourth day when the overflow has become less milky and free from suspended particles. The colloid clay sample was then collected and separated from the quartz sediments and sieved through a 230 mesh Tyler sieve (63 μm sieve opening) to further remove coarse impurities and organic particles present in the clay. The thick slurry clay was put in a filter cloth and pressed under heavy mass to squeeze out the water. The resulting cake was sun-dried and also oven-dried at 110°C to a constant weight. The dried clay was milled and sieved with a 125 μm mesh.

Chemical Beneficiation of Bentonite Clay

The procedure used for the chemical beneficiation was the modification of the methods prescribed by Musaab, 2014. The amount components used in the chemical beneficiation were soda ash, distilled water and bentonite clay. About 12g of soda ash was mixed with 350 mL of distilled water

and stirred vigorously for 5 min. 188g of the bentonite added to soda ash solution and agitated 20 min. The mixture was then allowed to age for 24 hr for the quartz impurities to sediment to the bottom leaving colloidal solution of clay and suspended particles at the top. The overflow water containing the suspended particles was decanted and the colloid clay sample was then collected and separated from the quartz sediments and sieved through a 230 mesh Tyler sieve (63 μm sieve opening) to further remove coarse impurities and organic particles present in the clay. The thick slurry clay was put in a filter cloth and pressed under heavy mass to squeeze out the water. The resulting cake was sun-dried and also oven-dried at 110°C to a constant weight. The dried clay was milled and sieved with a 125 μm mesh (Jock et al. 2016).

Bentonite Clay Characterization

The bentonite clay was characterized for elemental, microstructure and mineralogical composition using X-ray fluorescence (XRF), Scanning electron microscope (SEM) and X-ray diffractometer (XRD) respectively.

Bentonitic clays were loaded in the plastic sample cup (sample holder) which has a thin layer leathering surface that allows the passage of the X-ray beam into the sample. The sample holder was positioned in the sample charger of a PAN analytic B.V (model PW 4030/45B) X-ray fluorescence equipment which has 12 compartments for samples to be analysed simultaneously. The measurements and its progress were monitored on the computer system together with the specific software for the analysis. To determine the mineralogical composition of the clay samples, X-ray diffraction (XRD) test was carried out on the samples using the X-ray Diffractometer (Schmadzu model 6000) as follows:

The clay samples were first pulverized and then sieved to obtain 1.5 μm particle size to meet the required equipment specifications. The samples were oven dried for one hour at 60°C before it was allowed to cool. The clay samples were then mounted on a specimen holder of the Diffractometer and the required parameters (voltage = 40kV, current = 30mA) were set and screened between the angles of 2.0 and 65degrees at a speed of 3.0 degrees per minute which was run for 21 minutes. The diffractograms were displayed automatically and processed using the accompanied Schmadzu diffractometry software. The analysis of the diffractograms obtained from the XRD screenings was based on the search and match techniques. The scanning electron microscopy was carried out at 10 kV using a field emission scanning electron microscope.

Drilling mud formulation

The drilling mud was formulated based on standard API specification for formulation of drilling fluid. This was performed by adding 350ml of distilled water into a beaker containing 24.5g of beneficiated bentonite and the mixture was stirred for 20 mins. 10g of barite and different amount of polyanionic cellulose were added to the mixture as presented in Table 1 and stirred continuously to attain homogeneity. The mud was aged for 24 hr to allow for complete hydration.

Table 1: Proportion of drilling mud formulation components

Sample	Bentonite (g)	Barite (g)	Distilled water (mL)	Polyanionic cellulose (g)
A	24.5	10	350	-
B	24.5	10	350	0.5
C	24.5	10	350	1.0
D	24.5	10	350	1.5
E	24.5	10	350	2.0
F	24.5	10	350	2.5

Rheological analysis of formulated drilling mud

The rheological properties determined on the bentonite mud include plastic viscosity, apparent viscosity, gel strength and yield point. Viscosity, yield point and gel strength were measured by using a fan viscometer (Direct-indicating viscometer FANN-35). It is a rotational

instrument powered by an electric motor operated at various speeds of 3, 6, 100, 200, 300 and 600 rpm. The formulated mud sample was shaken and properly poured into the cup until it reaches the marked point. The sample in the container was placed and immerse into rotor sleeve exactly to the marked line thereby positioning the sample in an annular space between the two concentric cylinders. The sample temperature was recorded, ran at 600 rpm and waited for the viscometer dial reading to stabilize at steady value. The speed was first reduced to 300 rpm and dial reading was taken. The speed was increased to 600 rpm and the sample was allowed to stir for 10 s before the rotor was stopped. The sample was run at 3 rpm and the maximum dial reading was recorded.

Results and Discussion

Chemical composition of bentonite clay

The chemical composition of the commercial and local bentonite determined is presented in Table 2. The chemical analysis (wt%) shows that the major components in both clay are SiO₂ (42.03-55.90 wt.%) Al₂O₃ (15.84-18.28 wt.%) and Fe₂O₃ (8.539-19.84 wt.%) as well as CaO with the composition 22.20 wt.% in local bentonite clay.

Table 2: Chemical composition of commercial and local bentonite mud

Components	Composition (wt.%)	
	Local bentonite	Commercial bentonite
SiO ₂	42.033	55.901
Al ₂ O ₃	18.284	15.854
MnO	0.368	0.321
Fe ₂ O ₃	8.539	19.839
CaO	22.201	1.612
NiO	0.005	0.012
CuO	0.035	0.058
Nb ₂ O ₃	0.010	0.019
Cr ₂ O ₃	0.022	0.057
P ₂ O ₅	0.000	0.191
SO ₃	0.942	0.085
MgO	4.390	0.000
K ₂ O	1.430	1.830
V ₂ O ₅	0.105	0.187
TiO ₂	1.024	2.816
ZnO	0.017	0.048
Ag ₂ O	0.023	0.059
Cl	0.497	0.859
ZrO ₂	0.034	0.161

The amount of CaO in the commercial bentonite (1.61wt%) was less than in the local bentonite (22.20 wt%). This indicates that the local clay is a calcium based bentonite. The metallic oxides s MgO (4.39wt%) in local clay and others such as TiO₂, (1.02-2.82wt%), K₂O (1.43-1.83wt%), SO₃ (0.09-0.94wt%) and (0.11-0.19wt%) in both bentonite

are in minor quantities while oxides in traces amount include NiO, P₂O₅, Ag₂O, Nb₂O₃, CuO, Cr₂O₃ and ZnO. The high content of silica and alumina, defines the alumina-silicate type minerals of bentonite clay composed mainly of silica and alumina (Jock *et al.* 2020).

SEM analysis of bentonite clay

The SEM images depicting the clay micro structure are shown in Figure 1.

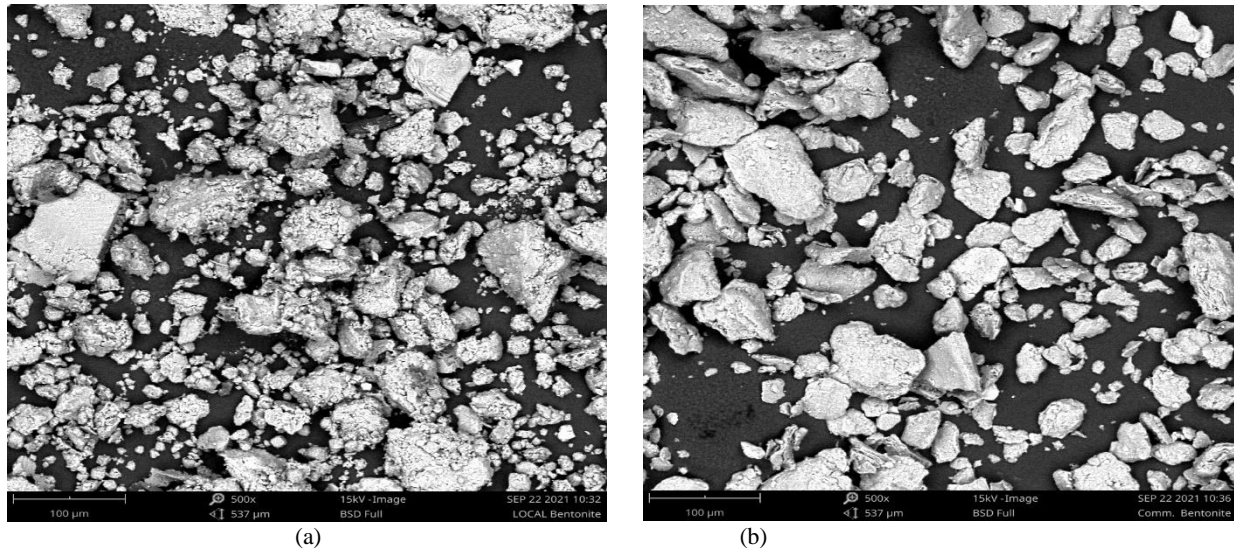


Figure 1: SEM image of bentonite clay (a) local bentonite and (b) commercial bentonite

The surface of the local bentonite appeared as random packing particles with a non-uniform structure while the commercial bentonite exhibited a more uniform structure and a cleared way layered pattern. The high degree of roughness and dull spots observed in the structure of the local bentonite could be due to the presence of impurities in the clay sample could be in form of high content of quartz, iron oxide, etc as shown the Table 2 and Figure 2.

Similarly, the local bentonite has no quoting surface while micrograph of the commercial bentonite appeared to be a covered with smooth quoting material. This is because commercial bentonite contained polymers and extenders.

XRD of bentonite clay

The XRD diffractogram of the local and commercial bentonite clay are shown in Figures 2 and 3 respectively.

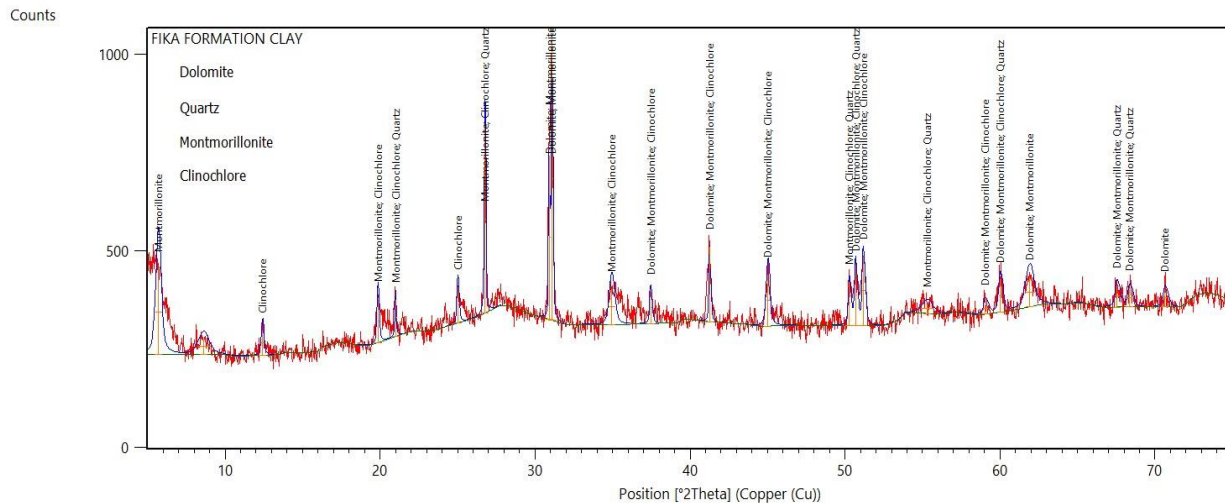


Figure 2: X-ray diffractogram of local bentonite clay

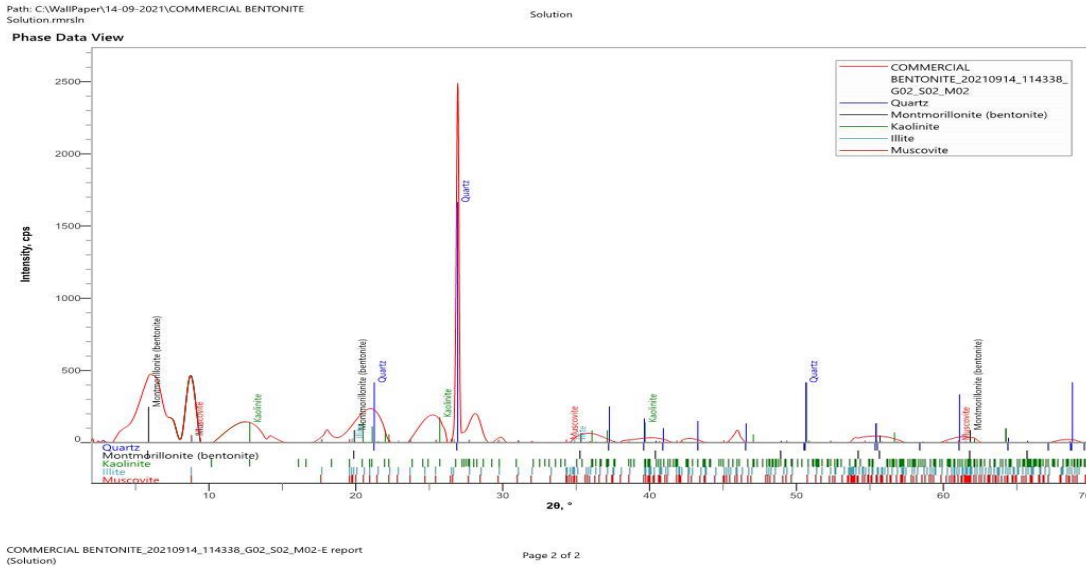


Figure 3: X-ray diffractogram of commercial bentonite clay

The presence of major minerals such as montmorillonite, quartz and dolomite were observed in the local (Fika) bentonite shown in Figure 2. This is in agreement with the chemical composition of the local clay containing high content of silica (42.03wt%) in the form of quartz and moderate amount of magnesite (4.39wt%) from dolomite as presented in Table 1. Similarly, high content of quartz (55.90wt%) and kaolinite were seen in the commercial bentonite. Other minerals found in Wyoming clay were muscovite and illite (Figure 3). The commercial bentonite

has high content of montmorillonite (active bentonite clay mineral) and little impurities as compared to Fika bentonite as reported by Bila et. al. (2016).

Rheological characteristics of bentonite clay

The rheological properties of the bentonite clay determined include, plastic viscosity, yield point, apparent viscosity and gel strength as summarized in Table 3. These properties were compared with API and OCMA specifications in Table 4.

Table 3: Rheological properties of commercial and local bentonite samples

Bentonite Sample	Plastic viscosity (cp)	Yield point (lb/100ft ²)	Apparent viscosity (cp)	Gel strength (lb/100ft ²)	Yield point to plastic viscosity ratio
A	2.0	3.0	3.5	1.0	1.5
B	9.0	3.0	10.5	2.0	0.3
C	12.0	4.0	14.0	2.0	0.3
D	9.0	16.0	17.0	3.0	1.8
E	9.0	23.0	20.5	3.0	2.6
F	9.0	31.0	24.5	4.0	3.4

Table 4: API (13A) and OCMA specification for drilling grade bentonite clay

Parameter	Specifications	
	API	OCMA specifications
Plastic viscosity (cp)	-	Maximum 10.0
Yield Point to plastic viscosity ratio	Maximum 3.0	Maximum 1.5
Filtration volume at 30 min (cm ³)	Maximum 15.0	Maximum 12.5
Residue after sieved with 200 mesh (75 μm)	Maximum 4.0 % by weight > 75 μm	-
Moisture	Maximum 10.0 % by weight	-

Source: API, 1993.

The plastic viscosity evaluated was between 2cp and 12cp (Table 3). Sample A without PAC has the lowest plastic viscosity while Sample C has the highest, indicating that

sample A will flow more easily than C. Low plastic viscosity shows that the mud is capable of drilling rapidly

while high plastic viscosity indicates that the mud is capable of drilling slowly (Omole *et al.* 2013).

The yield point increases with increasing amount of additives (polyanionic cellulose) in the order B>C>D>E>F. the yield point of a drilling mud denotes the amount of tension required to initiate deformation in the fluid to cause its flow. Therefore, the lower the yield point, the better the ability of mud to flow. Similarly, the yield points to plastic viscosity ratio ranged from 0.3 to 3.4 and are within the API specification except for sample F which is slightly above API standard. The yield points to plastic viscosity ratio samples D, E and F are above OCMA specification (Maximum 1.5) as depicted in Table 4.

The apparent viscosity of bentonite clay determined was 3.5-24.5cp as presented in Table 3. Sample A (3.5cp) has the lowest viscosity while Sample F (24.5 cp) has the highest. Generally, the apparent viscosity increases with an increased in the amount PAC added in the mud formulation. The higher the apparent viscosity of the mud, the slower the drilling performance.

The gel strength of the bentonite samples shown in Table 3, also increases with an increased in the amount of polyanionic cellulose. Drilling mud with low gel strength will yield in a poor hole cleaning, inadequate cutting suspension ability etc, while a high gel strength drilling mud will create high pump pressure in order to break circulation after the mud is static for a long time.

Conclusion

Fika local bentonite clay was successfully activated using soda ash and formulated as a drilling mud for application in the drilling industry. Fika's clay characterized showed the presence of high content of montmorillonite as well dolomite. The clay characteristics on beneficiation and the rheological parameters evaluated on formulated mud were improved. Most of the drilling mud produced agreed with API and OCMA specifications. Therefore, Fika clay exhibited good rheological properties for drilling fluid formulation that could compete favorably with a commercial bentonite.

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