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Characterization and Beneficiation of Clays from Ewekoro for use as Drilling Mud

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ABSTRACT

The proven bentonite reserve in Nigeria has been modestly estimated at more than 700 million tonnes, however her bentonite does not meet the API regulation standard. Hence this work was done with the aim of characterizing and beneficiating Nigeria bentonite clay to meet the required standard. Bentonite clay was gotten from Ewekoro in Ogun state of Nigeria, it was characterized using X-Ray Diffraction (XRD) to identify the structural layers which indicates the arrangement of the atoms in clay samples and X-Ray Fluorescence Spectrophotometer (XRF) which was used in the quantitative analysis of the elements in the sample. The XRD confirmed the presence of montmorillonite meaning the sample contained bentonite, while the XRF confirmed that the sample is Ca-based rather than the standard Na-based, hence the need for beneficiation. Beneficiation of the bentonite was done by ion exchange with Na_2CO_3 followed by addition with different compositions of viscosifiers namely: carboxymethyl cellulose (CMC), drispac and guar gum. The rheological properties together with the swelling ratio and filter loss of the clay were determined. Although some of the bentonite compositions met the required standard, but the bentonite formulation with the combination of 20.125 g bentonite + 2.875 g guar gum + 0.6 g drispac gave the best result.

Keywords: Bentonite, Beneficiation, Montmorillonite, Mineralogical analysis, Chemical analysis

1. INTRODUCTION

Bentonite is a crystalline, plastic, and colloidal aluminium phyllosilicate formed from alteration of volcanic ash. It is made up of smectite minerals namely montmorillonite which constitutes about 80% and also biotite, feldspar, kaolinite, illite, pyroxene, cristobalite, crystalline quartz and zirconia [1-4]. There are three major types of bentonite namely; sodium bentonite, calcium bentonite and potassium bentonite. The world bentonite deposit is majorly calcium bentonite and it has calcium ion as the major exchangeable cations [3]. When compared to sodium montmorillonite, it has low swelling and liquid limit. It is one of the earliest materials in the world used as cleaner, and also as adsorbent of fats and oils in solutions [5]. In addition to this, it is also used as a leaching agent in cooking oil industries and lubricating oil recycling, as a catalyst, absorber and filler [5].

Sodium bentonite is high swelling clay with sodium ion as the predominant exchange cation. It also has high fluid levels, high thixotropic capabilities and high thermal durability thus making it to be used in oil and gas drilling mud formulation [6, 7]. Also as a results of its good cation exchange property, highly specific surface area, good swelling property, high level platelets and ease of modification, it has been a major material used in many scientific fields like cosmetics, pharmaceuticals, foods, ceramics, paints, paper, and iron industries [8]. Potassium bentonite is a non-swelling, potassium rich illicit clay formed from volcanic ash. It is also used as a leaching agent, cooking oil industries and lubricating oil recycling, as a catalyst, absorber and filler [9].

When drilling for petroleum, one of the components used is the drilling fluid and the major component of freshwater drilling fluids is sodium montmorillonite. Drilling mud is used to remove cutting, to lubricate and cool the bit, also to form an impervious filter cake on the wall of the drill hole to prevent the penetration of water from the drilling fluid into the formation and formation fluids from the drilling fluid. The drilling fluid which circulates and carries the drilling cuttings up the hole, removed them by screening [10, 11]. In addition to the property of high viscosity, it must be thixotropic.

This property occurs when the drilling process stops, the mud has to turn to gel which prevents the drill cutting from settling down into the hole and freezing the bit which may break the drill stem. Also when the drilling process starts again, the drilling mud must become fluid. This thixotropic property is what makes sodium bentonite to be used in drilling mud formulation [12].

Despite large abundance of bentonite resources in Nigeria, estimated to be more than 700 million tonnes deposited in various location of the country, large amount of the bentonite used for drilling purposes in the country are still be imported by the multinational and indigenous oil companies thus making the country to lose millions of dollars [2, 13-16]. The excuse given by them is that the local bentonites did not meet the required standards set by the American Petroleum Institute (API) for drilling purposes [16]. From the research work conducted it was shown that drilling fluids produced by Nigeria bentonites showed a high liquid loss as a result of poor rheological and fluid loss properties and are therefore needed to be beneficiated [17].

As stated earlier, there are more than 700 million tonnes of bentonites located in various part of the country. Majority of these are found in Afuze, Edo State, Bauchi State, Taraba State, North East region of the country which includes Borno, Gombe and Adamawa States [2, 13-16, 18, 19]. Most of these bentonites deposits had been estimated and characterized and found to be majorly low grade calcium bentonites, but there are still some deposits in some parts of

the country including Lagos, Ogun, Anambra and Abia States that have not been estimated and characterized [16, 18]. This is what this research work wants to achieve.

James *et al.* [2] characterized and beneficiated bentonites from Yola and Adamawa which are in the North Eastern part of the country. The study showed that the bentonites are low-grade calcium montmorillonite which required beneficiation in order to be used for drilling purpose. They used both wet and dry methods for the treatment of the clay samples. From their results, it showed that the bentonite clay beneficiated by wet method has a better pH, cation exchange, swelling power and yield compared with the one by dry method. However, the dry method produced bentonite with better bulk density than the wet method. Abdulahi *et al.* [20] characterized bentonite clay from a Fika member of Pindiga formation in upper Benue trough in North Eastern Nigeria.

The mineralogy was carried out using XRD. The result showed that the bentonite clay is calcium montmorillonite and cannot be used for drilling purpose unless it is beneficiated to Na-montmorillonite. The elemental analysis using XRF showed that the Loss of Ignition (LOI), Al/Si ratio and moisture content of the local bentonite compared very well with standard bentonite used for drilling. After beneficiation with Na_2CO_3 , the clay was able to compare well with standard commercial bentonite.

Dewu *et al.* [21] investigated the use of bentonite clay of Pindiga formation from the Upper Benue trough in the North Eastern Nigeria for drilling purpose. The mineralogical and elemental analyses were done using XRD and XRF respectively. The results showed that the clay is Ca-montmorillonite and needed to be beneficiated to Na-montmorillonite. Also the elemental analysis showed that the Al/Si ratio ranges between 0.34-0.39 compared with 0.38 for the standard bentonite clay, the LOI was between 14.8-15.63 while for standard bentonite clay is 15.73. After beneficiation with Na_2CO_3 , the clays showed improvement to the extent that they can be used for drilling purpose. Apugo-Nwosu *et al.* [22] studied the tendency of Ubakala bentonite clay to be used for drilling mud formulation. This was compared with Wyoming bentonite as the standard clay.

The particle distribution showed that Ubakala clay is majorly clay (56% clay, 28% silt and 20% sand) which thus make it to be considered as pure clay sample. The cation exchange capacity showed that the clay is bentonitic and is predominantly calcium bentonite compared with Wyoming clay which is sodium dominant. The clay properties improved when beneficiated with calculated amount of carboxymethyl cellulose (CMC), polyanionic cellulose regular (PAC-R) and Na_2CO_3 . The results showed that clay at 24.5 g/350 ml of water beneficiated with 2 g CMC and 1 g of Na_2CO_3 has the best result.

Nweke *et al.* [19] investigated the use of bentonite from Abakaliki in South-Eastern Nigeria. He characterized the clay using XRD for the mineralogical composition and XRF for the elemental composition. The elemental composition showed that clay has low percentage of Na_2O compared with standard Wyoming clay and fairly high percentage of CaO and K_2O than that required for drilling purpose. The mineralogy showed that the clay is predominantly illite as well as montmorillonite with low amount of kaolinite. Abdulahi and Audu [23] did comparative analysis on chemical composition of bentonite clays from Ashaka and Tango deposits in Gombe State, Nigeria. The XRF analysis showed that the two abundant oxides in both samples are SiO_2 and Al_2O_3 .

The silica (SiO_2), alumina (Al_2O_3), iron (Fe_2O_3), calcium (CaO) and potassium (K_2O) contents of bentonite from Tango are more than that of Ashaka.

2. MATERIALS AND METHODS

2. 1. Sample Collection

The bentonite clay samples were sourced from Itori a town in Ewekoro Local Government Area of Ogun State which is in the South Western part of Nigeria. Six different samples of the clay were collected at a depth of about five to six feet as this are the depth in which samples of calcium, sodium and magnesium based elements are usually found. The samples were packed into plastic container, labeled to show the locations, date and time of collection. The samples were then subjected to different test to determine their nature and rheological properties.

2. 2. Sample Preparation

The clay samples were soaked in water for 72 hours and stirred every 24 hours to release organic matter. The collected samples were first crushed to smaller particles to increase their surface area and then left for 7 days in the sunlight to dry. The samples were then placed inside an oven at standard temperature for complete drying before grinding. Then the clay samples were grinded using mechanical grinder and passed through No 200 sieve (0.074 mm) to meet the standard API standard. The clay samples were subjected to both mineralogical and chemical analyses using X-ray powder diffraction (XRD) and X-Ray fluorescence technique (XRF) respectively.

2. 3. Mineralogical Analysis using XRD

The mineralogical composition of the bentonite clay samples was carried out using XRD. Each sample was run through the “Rigaku D/Max-IIIc X-Ray” diffractometer developed by the “Rigaku Int. Corp”. Tokyo, Japan and set to produce diffractions at scanning rate of $2^\circ/\text{min}$ in the 2 to 50° at room temperature with a Cu $K\alpha$ radiation set at 40 kV and 20 mA. The diffraction data (d value and relative intensity) obtained was compared to that of the standard data of minerals from the mineral powder diffraction file, ICDD which contained and includes the standard data of more than 3000 minerals. Similar diffraction data means the same minerals to standard minerals which exist in the soil sample.

2. 4. Chemical Analysis using XRF

The chemical composition of the bentonite clay samples was carried out using XRF. Each sample was crushed with an electric crusher and then pulverized for 60 seconds using “Herzog Gyro-mill (Simatic C7-621)”. Pellets were prepared from the pulverized sample, first by grinding 20 g of each sample with 0.4 g of stearic acid for 60 seconds. After each grinding, the” Gyro-mill” was cleansed to avoid contamination. 1 g of stearic acid was weighed into an aluminum cup to act as binding agent and the cup was subsequently filled with the sample to the level point. The cup was then taken to Herzong pelletizing equipment when it was passed at a pressure of 200 KN for 60 seconds. The 2 mm pellets were added into a sample holder of the x-ray equipment (Phillips PW-1800) for analysis.

2. 5. Purification and Activation of the Clay Samples

The mud samples were prepared by weighing out 50 g each of the clay samples in 16 beakers. Then added to each sample was 200 ml of deionized water followed by 25 g of Na_2CO_3 . They were left for 24 hours for ion exchange to take place.

The clay samples were further purified to remove impurities like carbonates, iron hydroxides and organic matters. This was done by dissolving the clay in distilled water and left for 12 hours to form a colloidal suspension. This purification was done by sedimentation. To the clay samples, 0.05M HCl was added to remove the carbonates and then followed by oxygenated water to remove the organic matters.

The clay samples were decanted and then dried. About 350 ml fresh water was measured in separate mixer cup and placed under the multimixer to shear. Then 0.1 g each of caustic potash and soda were added to the shearing water and allowed to shear for 2 mins. Then different clay samples were formulated as shown in Table 1 below and added to the sheared fresh water.

Table 1. Compositions of the bentonite clay formulated.

Sample no	Composition
1	23 g clay sample + 0.4 g CMC
2	23 g clay sample + 0.5 g CMC
3	23 g clay sample + 0.6 g CMC
4	20.125 g + 2.875 g guar gum + 0.4 g CMC
5	20.125 g + 2.875 g guar gum + 0.5 g CMC
6	20.125 g + 2.875 g guar gum + 0.6 g CMC
7	23 g clay sample + 0.4 g drispac
8	23 g clay sample + 0.5 g drispac
9	23 g clay sample + 0.6 g drispac
10	20.125 g + 2.875 g guar gum + 0.4 g drispac
11	20.125 g + 2.875 g guar gum + 0.4 g drispac
12	20.125 g + 2.875 g guar gum + 0.6 g drispac
13	Wyoming bentonite
14	Non-activated clay

2. 6. Rheological Test

Faan viscometer model 35 A was used to measure the rheological properties of the drilling activated clay. It is calibrated in revolutions per minute (RPM) and the results are in centipoises (cP). It is used to calculate the apparent viscosity, plastic viscosity, yield point and gel strength.

2. 7. Swelling Index Test

In this work, the test method ASTM D5890 is used to determine the swell index. A 2 g of each the non-activated and the activated clay was dispersed into a 100 ml graduated cylinder. This was done by adding 0.1 g of the clay every 10 mins so as to allow full hydration and settlement of the clay to the bottom of the flask. This was done until the whole 2 g of the clay was added. The sample was allowed to rest between 16 to 24 hours after which the level of settled and swollen clay was recorded to the nearest 0.5 ml.

Table 2. Mineralogical composition of the bentonites.

Constituents	Ewekoro bentonite	Wyoming bentonite
Major constituents	Quartz, Montmorillonite	Quartz, Montmorillonite, Feldspar
Major constituents	Quartz, Montmorillonite	Quartz, Montmorillonite, Feldspar

Table 3. Chemical composition of the bentonites.

Chemical oxides	Ewekoro (wt %)	Wyoming (wt %)
SiO ₂	66.80	58.60
Al ₂ O ₃	20.50	19.59
Fe ₂ O ₃	4.08	3.19
TiO ₂	0.15	1.68
CaO	1.69	0.37
P ₂ O ₅	0.08	-
K ₂ O	0.60	0.32
MnO	0.03	-
MgO	4.15	2.51
Na ₂ O	1.65	1.69
LOI at 110 °C	0.04	13.75

Table 4. Rheological properties, filter loss and swelling ratio of the bentonites (samples 1-7).

Rheology	1	2	3	4	5	6	7
600 rpm (cP)	32	35	37.5	52	54	55	34
300 rpm (cP)	22	24	26	35	37	39	23
6 rpm (cP)	2.1	2.3	2.5	3	3.1	3.4	2.7
3 rpm (cP)	1.6	1.7	1.9	2.1	2.55	2.6	1.8
Gels 10 s	11	12	13	17	18	19	12
Gels 10 min	12	13	13.5	19	20	22	13.5
PV (cP)	10	11	11.5	17	17	16	11
AV (cP)	16	17.5	18.75	26	27	27.5	17
YP (lb/100ft ²)	12	13	14.5	18	20	23	12
YP/PV	1.2	1.18	1.26	1.06	1.18	1.44	1.09
Thixotropy	1	1	0.5	2	2	3	5
Filtration Filter loss (ml)	13.8	13.5	13.2	12.4	12.1	11.2	13.5
Swelling Swelling ratio	4.1	4.2	4.3	4.6	4.9	5.1	4.2

Table 5. Rheological properties, filter loss and swelling ratio of the bentonites (samples 8-14).

Rheology	8	9	10	11	12	13	14
600 rpm (cP)	37	40	56	59	62	61	5.5
300 rpm (cP)	26	28	38	40	42	43	3.5
6 rpm (cP)	3.1	3.3	3.8	4	4.1	3.95	1.5
3 rpm (cP)	1.9	2	2.7	2.8	3.1	3.3	0.9
Gels 10 s	12	12.5	20	21	22	19	4
Gels 10 min	14	15	22.5	23.5	25	21	5
PV (cP)	11	12	18	19	20	18	2

AV (cP)	18.5	20	28	29.5	31	30.5	2.75
YP (lb/100ft ²)	15	16	20	21	22	25	1.5
YP/PV	1.36	1.33	1.11	1.10	1.10	1.39	0.75
Thixotropy	1.5	2.5	2.5	2.5	3	2	1
Filtration Filter loss (ml)	13.1	12.3	11.3	10.7	10.2	12	21
Swelling Swelling ratio	4.3	4.5	5.2	5.3	5.5	5.1	2.1

3. RESULTS AND DISCUSSION

3. 1. Analysis of X-Ray Fluorescence (XRD)

Sample : A File : Sg2~1.ASC Date : Sept 28 15:35:20 Operator :
 Comment : Qualitative Memo
 Method : 2nd differential Typica width : 0.065 deg. Min. Height 450:00 c p s

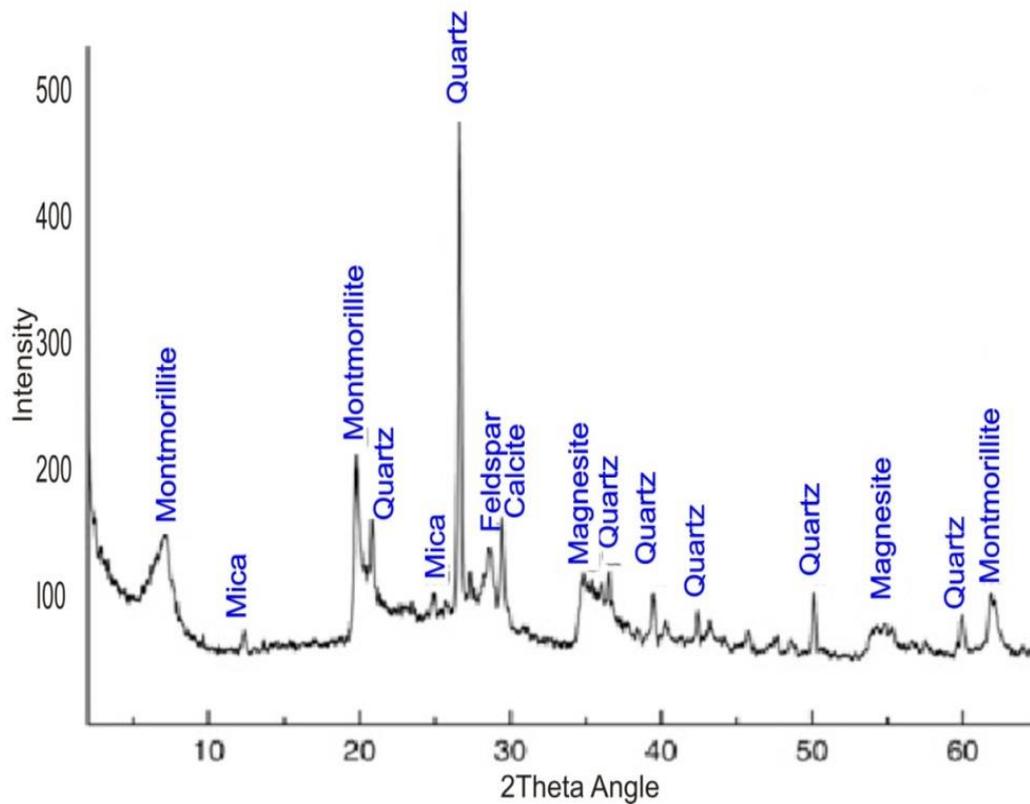


Figure 1. XRD diffractogram of clay sample

The mineralogical composition of the clay sample compared with the standard Wyoming bentonite clay is shown in Table 1. Also the diffractogram of the clay sample is shown in Figure 1. The XRD pattern shows that the main constituents of the clay samples are Quartz, Montmorillonite, Mica (Biotite), Feldspar (Albite), Calcite and Magnesite while the standard Wyoming clay sample is made up of Montmorillonite, Quartz, Kaolinite, Calcite, Biotite and Feldspar. It shows that the clay sample is significantly quartz and montmorillonite, while the other constituents being in minor quantities.

In Wyoming clay it is significantly montmorillonite follows by quartz and then the other constituents in small amount. This thus shows that the clay sample is montmorillonite.

3. 2. Analysis of X-Ray Fluorescence (XRF)

The XRF values of the clay samples from the XRF analysis and that of the standard bentonite clay are shown in Table 2. The values showed that the ratio of Al_2O_3 / SiO_2 for the sample clay and Wyoming clay are approximately 1/3 which showed that they are montmorillonite. Also the ratio of $[(Na_2O + K_2O) / (MgO + CaO)]$ of the sample clay is approximately 0.37 which confirmed that it is Ca-montmorillonite. Therefore the clay needs to be beneficiated by ion-exchange with Na_2CO_3 to Na-montmorillonite.

3. 3. Rheological Properties

3. 3. 1. Rheology and gel strength of clay activated with CMC

The rheological properties and the gel strength of the activated clay with CMC are shown in Table 4 and Figures 2 and 3. The apparent viscosity (AV) of the activated bentonite varies between 16 and 18.75 cP which is higher than 15 cp required by API. The plastic viscosity (PV) ranges from 10 to 11.5 cP which is within the API requirement. Yield point (YP) ranges from 12 to 14.5 lb/100ft². Gel strength after 10 s varies from 11 to 13 lb/100ft² while the gel strength after 10 mins varies from 12 to 13.5 lb/100ft² which is within the API requirement of 12 lb/100ft². The thixotropy changes from 0.5 to 1 while the ratio of YP/PV ranges from 1.18 to 1.26 which is within the API standard of maximum of 3.

3. 3. 2. Rheology and gel strength of clay activated with CMC and guar gum

These are shown in Table 4 and Figures 2 and 3. The plastic viscosity varies from 16 to 17 cP while the apparent viscosity ranges from 26 to 27.5 cP. Their values are more than the required API standard. Yield point ranges from 18 to 23 lb/100ft² while the gel strength at 10 s varies from 17 to 19 lb/100ft² and at 10 mins it varies between 19 and 22 lb/100ft². The gel strength is above the API standard. The thixotropy ranges from 2 to 3 lb/100ft² while the YP/PV ratio varies from 1.06 to 1.44. This shows that the addition of guar gum improved to a large extent the rheological and gel strength as compared to enhancing the clay with CMC alone.

3. 3. 3. Rheology and gel strength of clay activated with Drispac

These are presented in Tables 4 and 5 and Figures 2 and 3. Apparent viscosity increases from 17 to 20 cP and the plastic viscosity varies from 11 to 12 cP. The yield point ranges from 12 to 16 lb/100ft² while the gel strength for 10 s is between 12 to 12.5 lb/100ft² and that of 10 mins is between 13.5 to 15.0 lb/100ft². The thixotropy varies from 1.5 to 2.5 lb/100ft². The ratio of YP/PV ranges from 1.09 to 1.36 which is within the API requirement.

3. 3. 4. Rheology and gel strength of clay activated with drispac and guar gum

These are shown in Table 5 and Figures 2 and 3. Apparent viscosity ranges from 28 to 31 cP while the plastic viscosity increases from 18 to 20 cP. Their values are more than the required API standard. Yield point ranges from 20 to 22 lb/100ft² while ratio of YP/PV varies from 1.1 to 1.11. The gel strength at 10 s varies from 20 to 22 lb/100ft² and at 10 min it varies between 22.5 and 25 lb/100ft². The gel strength is above the API standard. The thixotropy ranges from 2.5 to 3 lb/100ft². This also shows that the addition of guar gum improved to a large extent the rheological and gel strength as compared to enhancing the clay with drispac alone.

3. 3. 5. Rheology and gel strength of Wyoming clay and unactivated clay

Table 5, Figures 2 and 3 showed the rheology and gel strength of both the Wyoming and unactivated clays. The apparent viscosity of Wyoming clay is 30.5 cP, plastic viscosity is 18 cP and yield point is 25 lb/100ft². The gel strength for 10 s is 19 lb/100ft² and that of 10 mins is 21 lb/100ft². The thixotropy is 2 lb/100ft² while the ratio of YP/PV is 1.39. Also for the unactivated clay, the apparent viscosity is 2.75 cP, plastic viscosity is 2 cP and yield point is 1.5 lb/100ft². The gel strength for 10 s is 4 lb/100ft² and that of 10 mins is 5 lb/100ft². The thixotropy is 1 lb/100ft² while the ratio of YP/PV is 0.75.

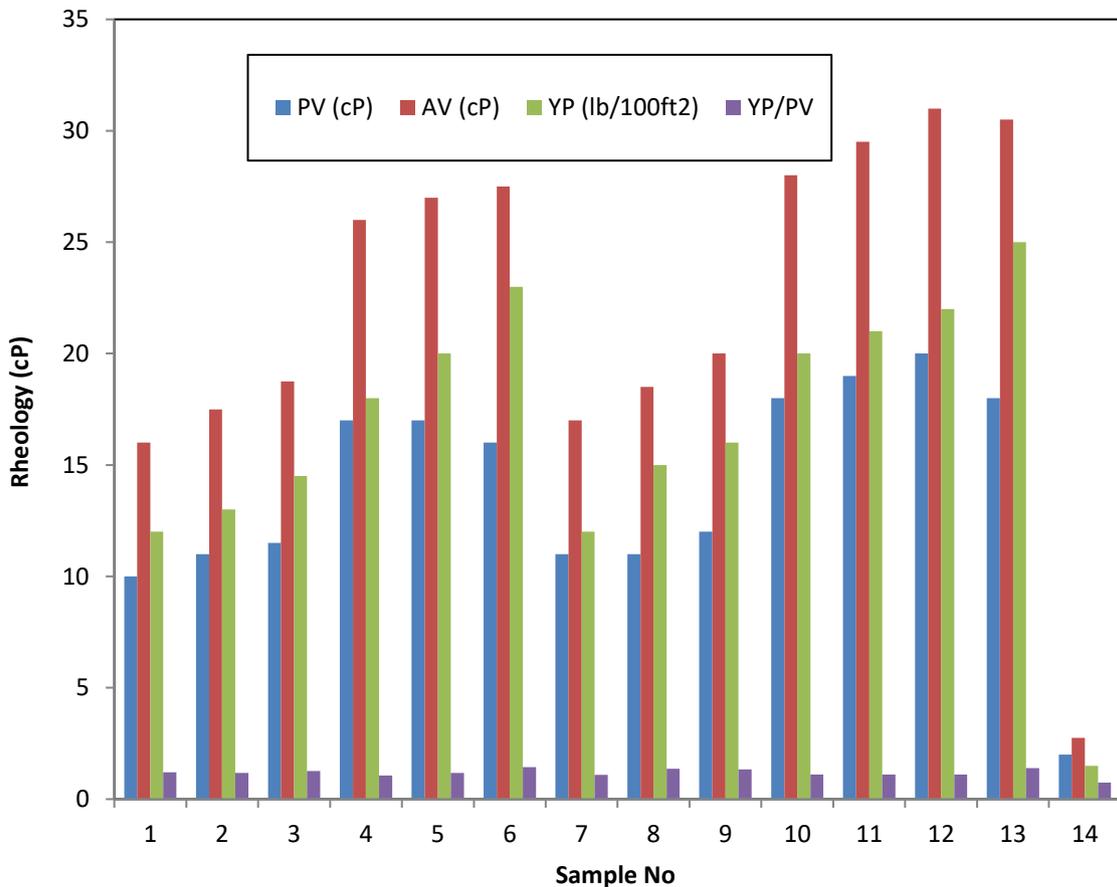


Figure 2. Rheology of clay samples

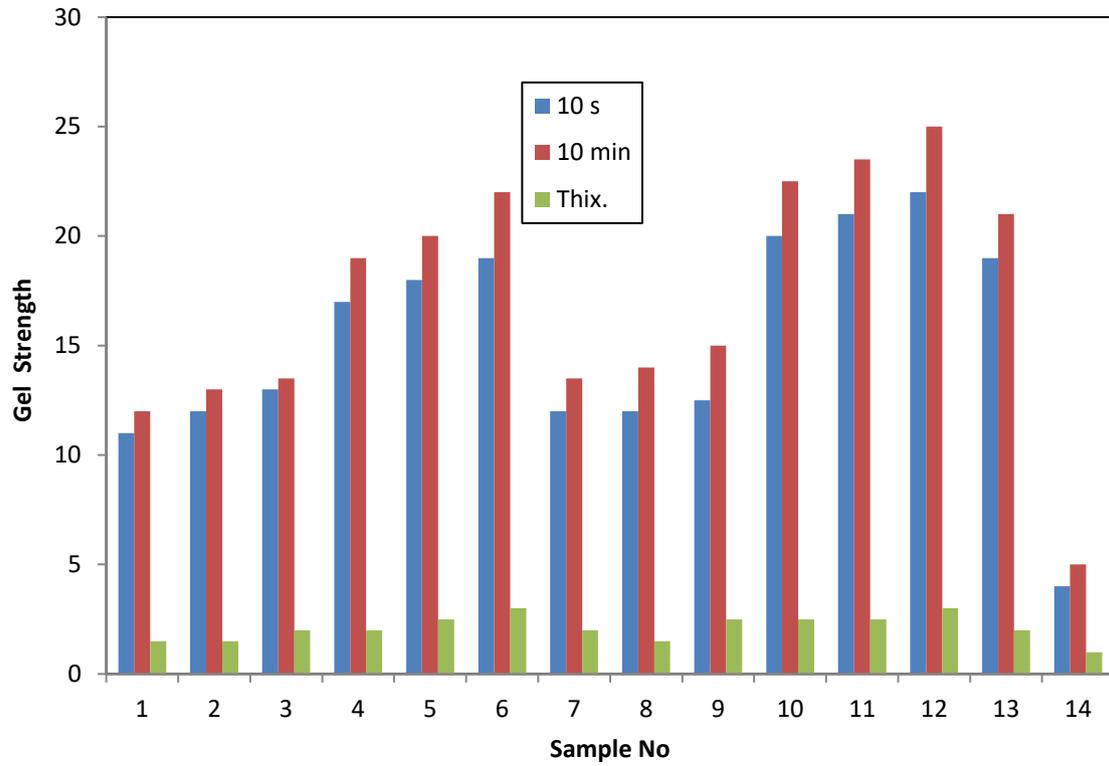


Figure 3. Gel strength of clay samples

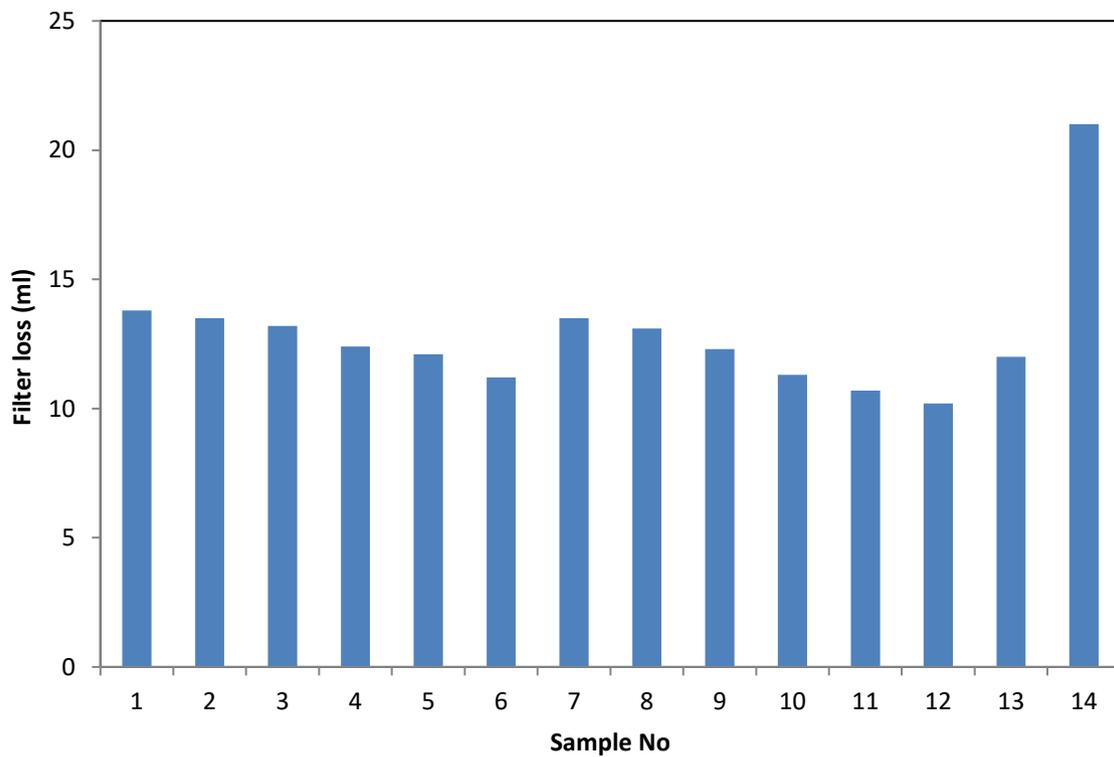


Figure 4. Filtration test of clay samples

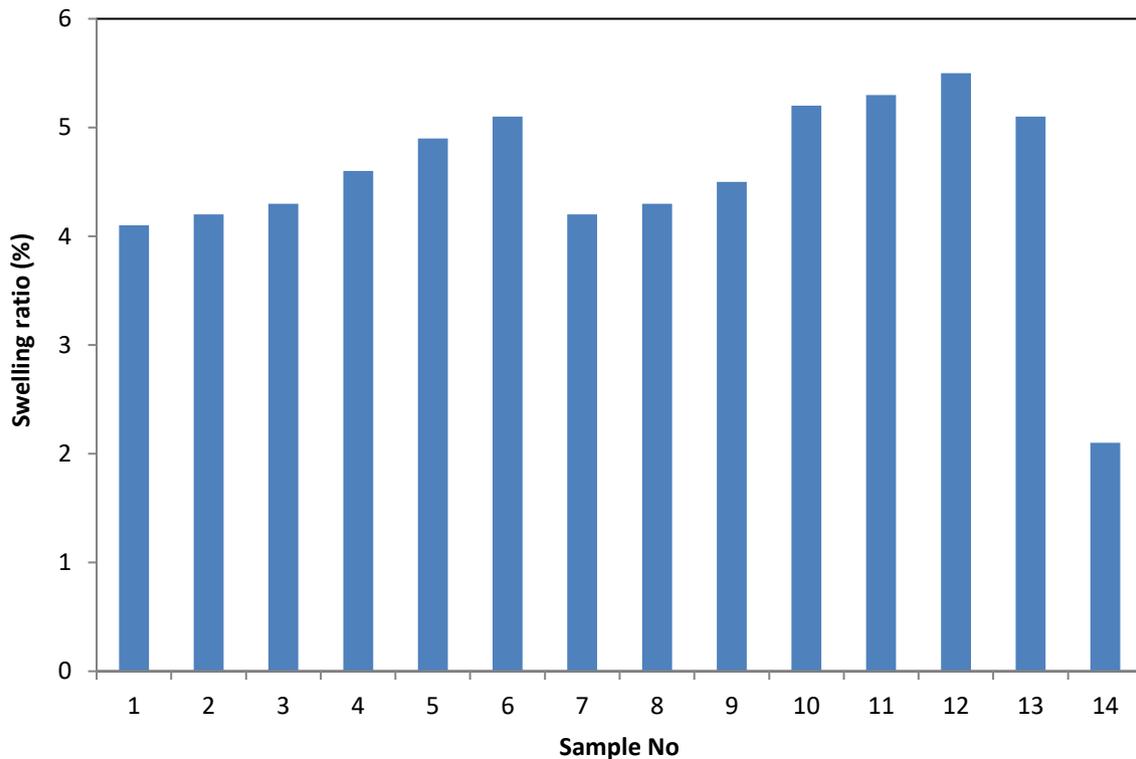


Figure 5. Swelling ratio of clay samples

3. 4. Filtration Abilities of Non-Activated and Activated Clay

As shown in Tables 4 and 5 and Figure 4, the filter loss for non activated clay is 21 ml and that of Wyoming clay is 12 ml. The filter loss for clay activated with CMC varies from 13.2 to 13.8 ml which is within the API standard of 15 ml maximum. The filter loss of the clay enhanced with both CMC and guar gum ranges between 11.2 and 12.4 ml while the filter loss for clay activated with drispac varies from 12.3 to 13.5 ml. Also the filter loss for the clay activated with drispac and guar gum ranges between 10.2 to 11.3 ml.

3. 5. Swelling Ratio

The result of the swelling ratio of the clay samples is shown in Figure 5. The value ranges between 4.1 and 5.5 for the first thirteen samples which fall with the API standard of 5 except samples 14 the untreated clay has a value of 2.1 which far below the API standard. The swelling ratio of the Wyoming clay is 5.1.

4. CONCLUSIONS

The local bentonite sample sourced from Ewekoro, possess no properties of its own for use as a viscosifier in drilling fluid. It is majorly Ca-montmorillonite not the standard Na-montmorillonite which is recommended for drilling purposes. Thus there is need to beneficiate to meet the required standard. The local bentonites was purified and activated with different

additives to improve its properties. After purification, different composition of CMC, PAC-R and guar gum were used to activate the bentonites. Although different combinations of additives used were able to improve its properties to almost the required standard, the clay activated with 20.125 g + 2.875 g guar gum + 0.6 g drispac gave the best result. It shows that addition of guar gum to each samples resulted in better bentonite clay.

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