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Activation of Local Bentonitic Clays for Use as Viscosifiers in Water-based Drilling Fluids

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Authors' contributions

This work was carried out in collaboration between all authors. Author OEA designed the study, managed the literature searches and wrote the first draft of the manuscript. Author ANO wrote the protocol, analyzed the results and intellectual content in the manuscript. Author OIA reviewed the analyses of the results and important intellectual content in the manuscript. All authors read and approved the final manuscript.

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ABSTRACT

Activated locally sourced clay as replacement for imported commercial bentonite for use as viscosifiers was studied. Tests and analyses were performed on local clay samples obtained from five (5) locations: Ini, Ibiono, Ikono, Itu and Uyo Local Government Areas in Akwa Ibom State of Nigeria. The mineralogical composition of the unactivated samples determined using X-Ray Fluorescence (XRF), indicates montmorillonite contents less than the American Petroleum Institute (API) standard, so also are the rheological properties and fluid loss characteristics. The activation of the local clays with soda ash (Na_2CO_3) resulted in improved montmorillonite content. In addition, the activation process increased the clay yield and swelling power. Mud rheological properties and fluid loss potential improved more in the Ikono and Uyo than other clays. Thus, the activated clays can be classified as sub-bentonite that can be used as drilling mud for medium depth wells. The

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viscosifying potential of the locally sourced clays in water-based mud for oil well drilling can be exploited. This will reduce importation of foreign bentonite, lower total drilling cost as well as add value to the Nigerian economy.

Keywords: Local bentonitic clay; activation; viscosifiers, montmorillonite; Akwa Ibom State.

1. INTRODUCTION

Drilling fluids are used in the oil and gas industry for the drilling of boreholes and construction of oil wells [1]. The E&P operations can only begin to yield fruits when the hydrocarbon bearing formation is successfully drilled to the target depth. One component that has remained an integral part of the drilling process is the drilling fluid. The drilling fluid has always been a vital part of drilling operation for oil and gas deposits, dating back to the Spindletop; when a herd of cattle was turned loose in a partially-filled water pit to grind the saturated soil into mush with their hooves. After shooing the cattle out, more water was added to the pit, and presto, drilling "mud" was created [2]. Depending on the continuous phase, drilling fluid is classified either as water-based or oil-based. Although a new class is introduce - synthetic-based drilling fluid because of the environmental challenge of the oil-based drilling fluids. Therefore, it is heterogeneous mixture of clay, water/oil and other additives that aid the drilling operations; which is sometimes seen as the circulating blood of the drilling operation. Tailoring drilling fluid is an important key to the success of a drilling operation. During drilling, the deposit of solids in the form of a cake contributes to the borehole stability and limits the invasion of the permeable zone by the liquid phase and reduces the formation damage [3]. This characteristic of the drilling mud to perform deposition of solids to form cake is control by its viscosity which is enhanced by viscosifier - Bentonite. Additionally, filtration control in drilling fluids is usually based on the use of bentonite with the addition of other products to enhance fluid loss properties [4]. Bentonite is the name used for a range of clays that can swell and gel when dispersed in water [5]. It has been used extensively throughout the world in drilling, but the cost of transporting original "Wyoming" bentonite from the USA has led to the use of alternatives from other sources from other parts of the world. This has provided a window of opportunity for other parts of the world to look inwards to exploit their local clays which match bentonite quality. So far, companies involved in design and production of drilling fluid in Nigeria for the oil and gas sector have over the years imported the materials to produce mud or in some cases imported already designed and produced drilling mud [6]. Also, Odumugbo [7] added that the present consumption of bentonite clay in the drilling operations in Nigeria is put at over 50 thousand tons a year most of which is imported from USA; a trend that is expected to continue as drilling activity increase off the shores of the Niger Delta region. This has been a major challenge to the indigenous companies involved in the oil and gas as they have to import these materials at high costs. In this connection, the indigenous companies find it difficult to compete favourably with their foreign counterparts. The emergence of local content in the oil and gas industry by the federal government under the auspices of Nigerian National Petroleum Corporation (NNPC) to find local drilling fluid materials that can substitute the foreign ones has encourage the exploit of local clay deposit. Several studies on using local bentonite in Nigeria as drilling mud viscosifiers abound and most of those studies were reviewed in the work of Agwu et al. [8]. However, those of RMRDC [9], Abdullahi et al. [10], Udoh and Okon [11]; Onwuachi-Iheagwara [12]; Olugbenga et al. [13]; Imuentinyan and Adewole [14]; Nmegbu [15] and Wilfred and Akinade [16] are some of the many recent studies in this direction. This paper therefore evaluates and activates local clays from five different deposits in Akwa Ibom State for use as drilling fluid viscosifiers in water-based drilling mud.

2. MATERIALS AND METHODS

2.1 Samples Collection, Preparation and Clay Activation

The bentonitic clay samples were collected from five different deposits: Ini, Ibiono Ibom, Ikono, Itu and Uyo Local Government Areas in Akwa Ibom State. The locations of these deposits are indicated in Fig. 1. The coordinates of these locations are: Ini - $5^o 24' 0'' N$; $7^o 44' 0'' E$, Ibiono Ibom - $5^o 14' 0'' N$; $7^o 53' 0'' E$, Ikono - $5^o 12' 37.25'' N$; $7^o 47' 38.34'' E$, Itu - $5^o 10' 0'' N$; $7^o 59' 0'' E$ and Uyo - $5^o 1' 23.63'' N$; $7^o 55' 26'' E$ [17]. The choice of these locations are based on the work of RMRDC [9] about the existence of bentonite deposits in Itu and studies conducted by Udoh and Okon [11] to formulate water-based mud from clay in Uyo.

Thus, there is a need to expand the exploit for local bentonitic clay in Akwa Ibom State, Nigeria. The obtained clay lumps were sun dried for eight (8) days and pulverized into fine particles using mortar and pestle, after which they were dissolved in water to form slurries. The slurries were wet-sieved. The filtrates were allowed to settle for six (6) hours, decanted to obtain clay mud of the different bentonitic clay samples. These were sun dried for seven (7) hours to obtain clay cakes, which were disaggregated and sieved to 125 microns to obtain fine clay powder. A portion of each clay sample was activated with soda ash (Na_2CO_3), by kneading with 50 g of Na_2CO_3 in 350 mL deionized water and allowed for 24 hours to enable ion exchange to take place. The kneaded clay samples were sun dried for 5 hours. The activated clay samples were then pulverized and sieved into 125 microns; and analyzed for mineralogical content using with X-Ray Fluorescence (XRF) analysis. In addition, the composition of Wyoming bentonite - the clay of choice in drilling operation, was compared with the local clays' composition.

2.2 X-ray Fluorescence (XRF) Analysis

The mineralogical content of the non-activated and activated bentonitic clays from Akwa Ibom State was performed with the X-Ray Fluorescence (XRF) method. This approach (XRF) was used since it is a non-destructive multi elemental analysis technique with sensitivity in the range 10^{-8} [18]. The Bruker Tracer III-V analyzer was used. The analyzer was set with the filter composed of 1mil Titanium (Ti), 6 mil Copper (Cu) and 12 mil Aluminum (Al). This filter setting allows the analyzer to target potassium (K), calcium (Ca), iron (Fe), and titanium (Ti), among other montmorillonite content in the clay samples. In addition, the bentonitic clay was scanned at four locations with two reading per location for three minutes. This multi-location/multi-reading approach was adopted to allow for verification of the mineralogical composition reading consistency.

2.3 Mud Preparation and Beneficiation

Both the activated and non-activated bentonitic clays were used to formulate water-based drilling mud based on American Petroleum Institute (API) standard of 25 g of non-treated bentonite to 350 mL of water; Amoco Production Company [19], with addition of sodium hydroxide (NaOH) and Hydroxyethyl cellulose (HEC) of 10% concentration of the clay sample. The non-activated clays: Ini (Sample A), Ibiono (Sample B), Ikono (Sample C), Itu (Sample D) and Uyo (Sample E), and activated clays: Ini (Sample A-1), Ibiono (Sample B-1), Ikono (Sample C-1), Itu (Sample D-1) and Uyo (Sample E-1) were

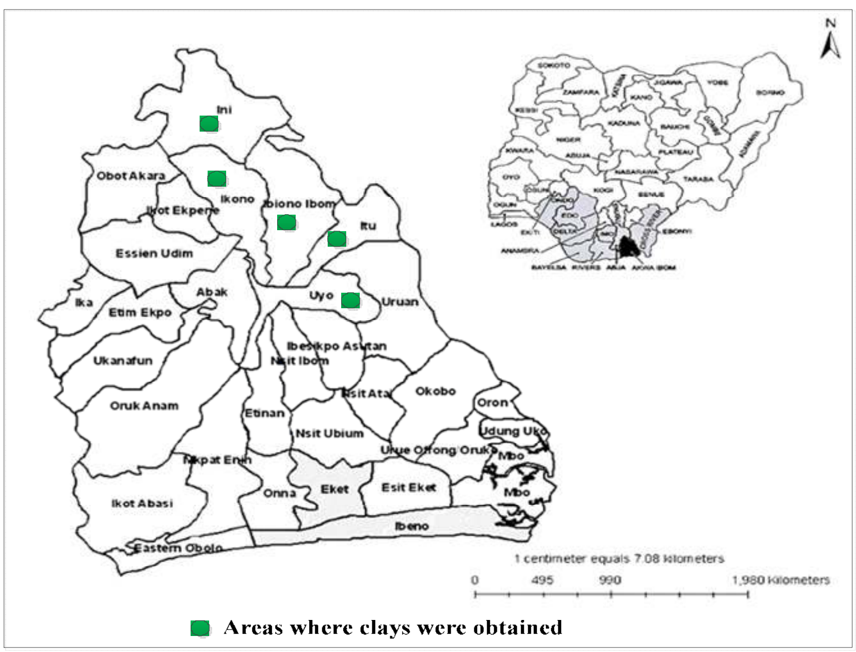

Fig. 1. Map of Nigeria (top right insert) and Akwa Ibom State (enlarged) showing areas where clays were obtained
[Source: Ite et al., [20]; modified by Authors]

Table 1. The API standard test and analysis values of mud parameters

Mud parameters	Mud density		Viscometer Reading @ 600 rpm	PV (cP)		YP (lb/ft^2)	pH	
	Min.	Max.		Min.	Max.		Min.	Max.
Value requirement	8.65	9.60	30cP (minimum)	8	10	3xPV	9.5	12.5

[Source: Amoco Production Company, [19]]

prepared and kept for 24 hours to age. Thereafter, the pH, fluid loss and rheological properties: apparent viscosity, plastic viscosity, yield point and gel strength of the muds were determined based on the API Specification 13B recommended practice for field testing water-based drilling fluids. The rheological parameters: apparent viscosity, plastic viscosity and yield point of the mud samples were evaluated from 300 and 600 rpm (revolutions per minute) reading using the expanded equations 1 through 3, from API Recommended practice of standard procedure for field testing drilling fluids [21]. Their results are presented in Table 3.

$$PV = \theta_{600} - \theta_{300} \tag{1}$$

$$YP = \theta_{300} - PV \tag{2}$$

$$AV = \left({}^{\theta_{600}}/_{2}\right) \tag{3}$$

where:

AV = *Apparent Viscosity*
PV = *Plastic Viscosity*
θ_{600} = *Dial reading at 600 revolutions per minute*
θ_{300} = *Dial reading at 300 revolutions per minute*
YP = *Yield Point*

2.4 Mud Properties Measurement Procedure

In drilling mud formulation, Wyoming bentonite - the clay of choice has been the yardstick for evaluating other local bentonitic clay as viscosifier in both water and oil based drilling mud. Therefore, the benchmark for the evaluation of the properties is provided in above Table 1.

The experimental procedures for pH value, rheological properties and fluid loss were adopted from the work of Okon et al. [22] which is in line with API Specification 13B for field testing of water-based drilling fluids' properties:

pH value: pH test is the measurement of the concentration of hydrogen ions in aqueous solution [23]. Therefore, this measurement establishes the acidity or alkalinity of the drilling mud. The pH meter was standardized using deionized water and the mud sample to be measured was poured into a glass beaker. The pH meter probe was immersed in the mud sample and at steady pH value indicated on the meter. This was recorded as the pH value of the bentonite mud sample.

Rheological properties: The rotational viscometer provides a more meaningful measurement of the rheological characteristics of the drilling mud compared to marsh funnel [24]. The non-activated clay mud sample (Sample A) was poured into the viscometer cup to the scribed mark and placed on the stand of the viscometer as it was lifted to immerse the rotating sleeve. With rotor speed at 300 and 600 revolutions per minute (i.e., two point data approach), their respective dial readings were recorded at steady values and used to evaluate the clay mud apparent and plastic viscosity as well as its yield point based on the expanded equations 1 through 3. Gel strength determination is an extension of mud rheological properties [22]. The 10 seconds and 10 minutes gel strength of the clay mud were determined. The rotary sleeve speed was set at 600 rpm position to stir the clay mud sample for 10 seconds, then, the knob was set to 3 rpm and the viscometer switched off to enable the mud sample to stand undisturbed for 10 seconds. Thereafter, the flip toggle was switched to low (rear) position and the maximum dial reading was recorded as the 10 seconds or initial gel strength. In addition, the same procedure was repeated as the clay mud sample was allowed to stand undisturbed for 10 minutes. With flip toggle switched to rear position, the maximum deflection (dial reading) was recorded as the 10 minutes gel strength. This procedure was repeated to determine the gel strength of clay mud samples B through E as well as samples A-1 through E-1.

Fluid loss: The API standard LT-LP filtration test was carried out at surface (room) temperature and 700 kPa (100 psi) pressure for thirty (30) minutes. The LT-LP filter press consisting of cylindrical cell 3-inches in internal diameter (ID) and 5-inches high to contain the formulated drilling mud was used. The bottom of the cell was fitted with a sheet of Whatman No. 50 filter paper and filled with the clay mud sample to be measured. In this study, the filter press used consisted of six (6) filter cells mounted on a common frame. After the necessary connections, a pressure of 100 psi from air compressor pump was supplied to the top of the cells. With a measuring cylinder placed beneath the cell, the filtrate through the filter paper was collected over a period of 30 minutes and recorded in milliliters (mL) equivalent cubic centimeter (cm^3) as the API filtrate (fluid loss) of the bentonitic clay mud samples.

2.5 Swelling Power Test

The swelling powers of the non-activated and activated bentonitic clay samples were determined. Five (5) grams of each clay sample was divided into ten (10) portions, and each portion was suspended in 1% lauryl sulphonate solution in a 100 mL graduated cylinder of 3 cm diameter at 2 minutes interval in succession. Each portion was allowed to settle before adding the next. After the tenth portion, the cylinder was allowed to stand undisturbed for 2 hours. Then, the apparent volume of the sediment at the bottom of the cylinder was measured and presented in Table 3.

3. RESULTS AND DISCUSSION

3.1 Mineralogical Compositions

The result of the x-ray fluorescence (XRF) analyses presented in Table 2, shows the percentage of clay components in the samples and the proportion of montmorillonite minerals. The non-activated clay samples analysis indicate that the local clays contain appreciable montmorillonite: Aluminum Oxide (Al_2O_3) and Silicon Oxide (SiO_2) that is less than the American Petroleum Institute (API) bentonitic requirement. The results further depict the presence of other oxides: Magnesium (Mg), Iron (Fe), Calcium (Ca), Sodium (Na), Potassium (K) and Managanese (Mn) oxides in the obtained bentonitic clay in Akwa Ibom State. Frankly speaking, some of these oxides: SiO_2, Al_2O_3 and K_2O from Ini and Itu deposit are more than the stipulated API bentonite composition range. On the other hand, the activated clay samples' XRF results indicate that the montmorillonite content of the local clays were improved close to API standard range for bentonitic clay. Consequently, the same result is also observed with the oxides content of the activated bentonitic clay samples. Also, the Al_2O_3/SiO_2 ratio of the activated local bentonitic clays ranged between 0.35 - 0.41. However, Ini clay with 0.38, Ibiono 0.37 and Uyo 0.36 can be said to be within the API requirement of 0.38 for bentonite. This implies, technically speaking that the local bentonitic clays have met the required standard as drilling mud viscosifiers in water-based mud formulation for drilling purposes.

3.2 Mud Properties

The results obtained from the mud analyses for both the non-activated and activated clay samples are presented in Table 3. Figs. 2 through 5 present the rheological properties: apparent viscosity, plastic viscosity, yield point and gel strength, respectively of the bentonitic clays in non-activated and activated state. The apparent viscosity and plastic viscosity of the non-activated bentonitic clays was below the API required standard for drilling mud viscosifiers; as indicated in Table 3. This is probably due to the calcium-based nature of the bentonitic clays, which result in their low yield tendency; as further observed in their negligible swelling power in Table 3. In addition, there is a significant increase in the rheological properties of the activated bentonitic clay muds when compared to those of non-activated clay muds. This is an indication that the activation process increases the yield of the bentonitic clays as a result of the increased sodium content. Interestingly, some of the activated bentonitic clays' rheological properties: apparent and plastic viscosities were more or close to the API standard test for mud parameters as presented in Table 3. Following the criteria stipulated in Table 1, the plastic viscosity of activated bentonitic clays increased to between 8.0 – 9.6 cP, while the yield point (YP) and gel strength (10 sec./10 min.) were improve significantly to between 10.5 – 13.2 lb/100 ft^2 and 0.5/0.7 – 2.3/7.6 lb/100 ft^2. These indicate about 475% increase for the clays' yield point, and about 460% and 986% increase for the 10 seconds and 10 minutes gel strength, respectively. In addition, the muds' pH were increased to between 9.5 – 11.5 (about 396%); an indication implying that the activated bentonitic clays resulted in a favourable mud rheology that met the API test criterion for viscosifiers in drilling mud formulation.

Therefore, the activated bentonitic clays as presented in Figs. 2 through 5 exhibited improved rheological properties. X-raying the apparent viscosity, plastic viscosity and yield point of the local clays, indicates that Ini clay resulted in about 480.77%, 258.33% and 1575.0% increase, respectively. While Ibiono clay was about 422.33%, 388.90% and 475.0% increase. Also, Ikono and Itu clays exhibited percentage increase of about 503.70%, 860.0% and 288.24%, and 582.26%, 411.11% and 950%, respectively. In addition, Uyo clay's rheological properties were activated to about 484.32%, 468.75% and 510.0% increase for apparent viscosity, plastic viscosity and yield point, respectively. Worth of mentioning is the flat gel potential of Ikono and Ini bentonitic clays; as observed in Fig. 5. This potential of the bentonitic clay is explorable during the waiting or non-circulating time in drilling operation.

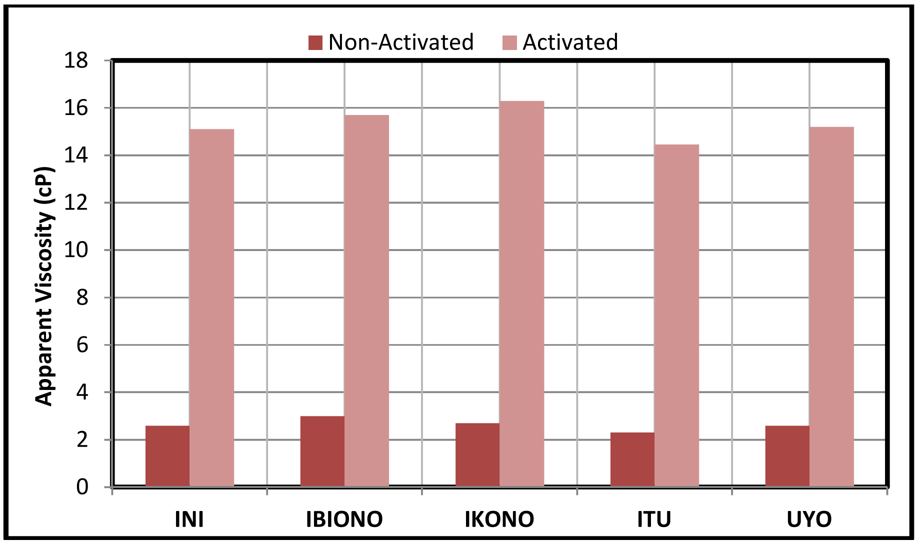

Fig. 2. Apparent viscosity of the bentonitic clays' water-based mud

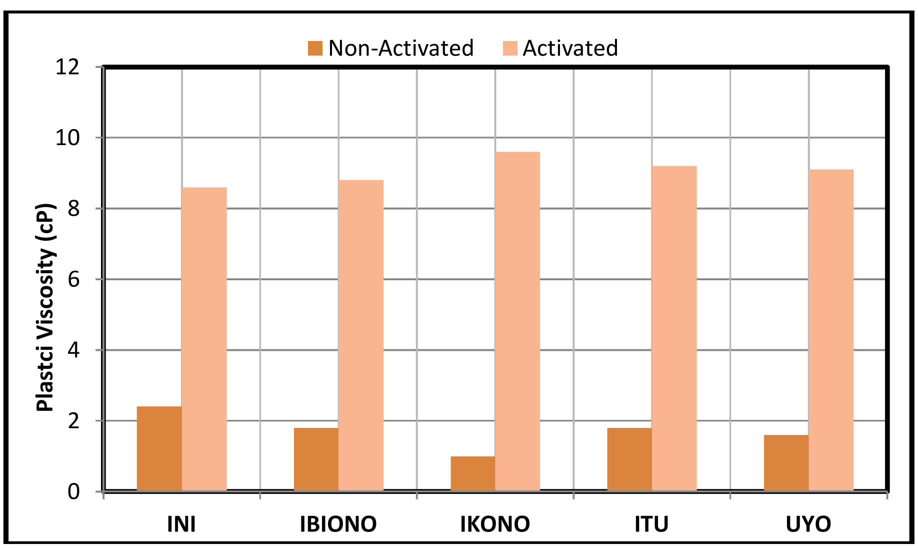

Fig. 3. Plastic viscosity of the bentonitic clays' water-based mud

Table 2. XRF results for non-activated and activated clay samples

Location	INI		IBIONO		IKONO		ITU		UYO		API Bentonite
Chemical oxide	Non-activated	Activated	Non-activated	Activated	Non-activated	Activated	Non-activated	Activated	Non-activated	Activated	
SiO_2	32.40	56.23	14.40	52.15	10.39	58.25	42.41	57.23	18.43	57.12	58-64
Al_2O_3	15.40	21.20	9.39	19.2	4.40	24.13	13.43	19.86	7.41	20.72	18-21
Fe_2O_3	4.56	7.05	5.53	7.42	6.63	8.51	7.12	8.43	5.63	7.13	2.5-2.8
MgO	0.06	1.45	1.87	1.87	0.14	1.42	0.09	1.22	0.08	1.43	2.5-3.2
CaO	0.30	1.01	0.60	1.09	0.72	1.03	0.43	1.41	0.43	1.08	0.1-1.0
Na_2O	0.14	2.89	0.13	1.29	0.27	3.92	0.14	3.16	0.15	8.14	1.5-2.7
K_2O	0.92	1.44	0.83	1.22	2.07	1.07	0.71	1.01	1.01	0.69	0.2-0.4
MnO	0.067	0.021	0.089	0.034	0.097	0.026	0.078	0.064	0.054	0.02	-
$Al_2O_3:SiO_2$	0.475	0.38	0.65	0.37	0.42	0.41	0.32	0.35	0.40	0.36	0.31-0.33

[API BENTONITE Source: http://www.lonestarbarite.com [25]]

Table 3. Properties from the non-activated and activated bentonitic clays' water-based mud

Parameter	INI		IBIONO		IKONO		ITU		UYO		API bentonite
	Non-activated	Activated	Non-activated	Activated	Non-activated	Activated	Non-activated	Activated	Non-activated	Activated	
Viscometer dial reading at 600rpm	5.2	30.2	6	31.4	5.4	32.6	4.6	28.9	5.2	30.4	>30[a]
Viscometer dial reading at 300rpm	2.8	21.6	4.2	22.6	4.4	22.8	2.8	19.7	3.6	21.3	-
Apparent Viscosity (cP)	2.6	15.1	3.0	15.7	2.7	16.3	2.3	14.45	2.6	15.2	15.0[b]
Plastic Viscosity (cP)	2.4	8.6	1.8	8.8	1.0	9.6	1.8	9.2	1.6	9.1	8.0[b]
Yield point ($lb/100ft^2$)	0.4	13	2.4	13.8	3.4	13.2	1.0	10.5	2.0	12.2	-
YP/PV ratio	0.167	1.51	1.33	1.57	3.4	1.375	0.56	1.14	1.25	1.34	3 (max)[a]
Gel Strength ($lb/100ft^2$)	0.1/0.2	0.5/0.7	0.1/0.2	0.6/3.5	0.3/0.4	2.8/3.2	0.2/0.2	0.8/1.6	0.1/0.3	2.3/7.6	-
Fluid loss (mL)	115	18.5	105	14.6	102	12.4	108	17.5	77	10	15 (max)[a]
Thixotropy	0.1	0.2	0.1	2.9	0.1	0.4	0	0.8	0.2	5.3	-
pH	3.49	11.47	3.88	10.2	3.73	9.78	2.26	11.21	5.34	9.5	8-10[a]
Swelling Power (cm^3)	Negligible	51	Negligible	48	Negligible	64	Negligible	40	Negligible	56	-

Source:
[a]Amoco Production Company [19]
[b]http://ww.lonestarbarite.com [25]

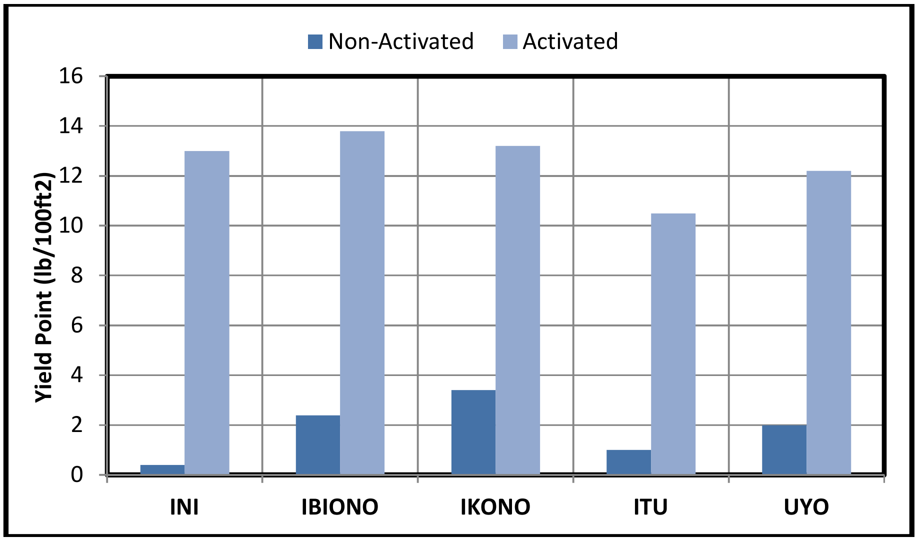

Fig. 4. Yield point of the bentonitic clays' water-based mud

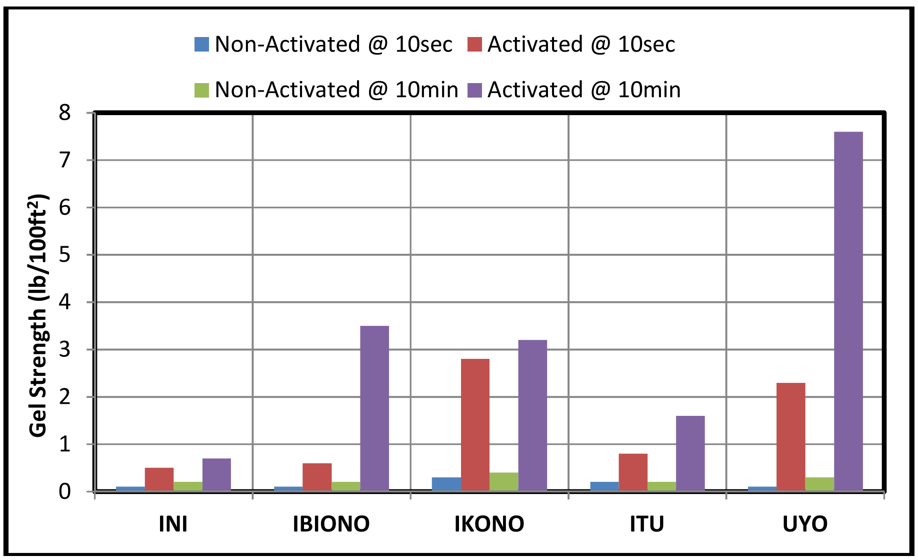

Fig. 5. Gel strength of the bentonitic clays' water-based mud

Fig. 6 depicts the fluid loss of the formulated bentonitic clay mud. Interestingly, the viscosifying nature of the clay materials in the drilling fluid is directly revealed in its fluid loss characteristics. The result indicates that there is a significant fluid loss reduction from the activated bentonitic clay muds compared to the obtained fluid loss result from non-activated bentonitic clay muds. This development is due to the fact that the high sodium ion (Na^+) content of activated bentonitic mud clay particles tends to form a better edge-to-edge bond (flocculation); preventing water penetration than the non-activated bentonitic clay muds with more calcium ion (Ca^{2+}). Therefore, the activation resulted in fluid loss reduction of 83.91%, 86.10%, 87.84%, 83.80% and 87.01% for Ini, Ibiono, Ikono, Itu and Uyo clay, respectively. Hence, this activation process gives Ibiono, Ikono and Uyo bentonitic clays a viable fluid loss potential as drilling mud viscosifiers. This assertion is based on the API Drilling-grade Bentonite requirement standard of 15 mL maximum, as the mentioned bentonitic clays had fluid loss of 14.5 mL, 12.4 mL and 10 mL, respectively.

Fig. 7 present the pH values for non-activated and activated clays. The acidic nature of the

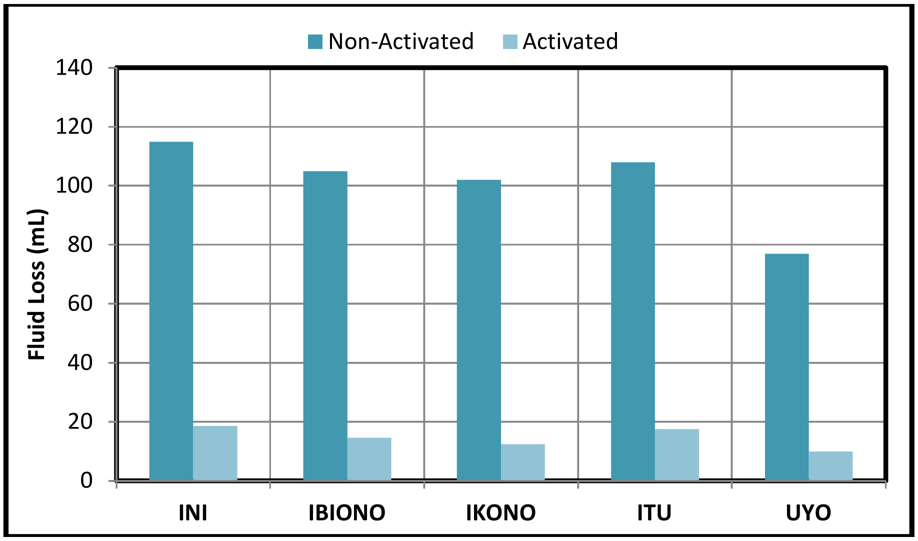

Fig. 6. Fluid loss of the bentonitic clays' water-based mud

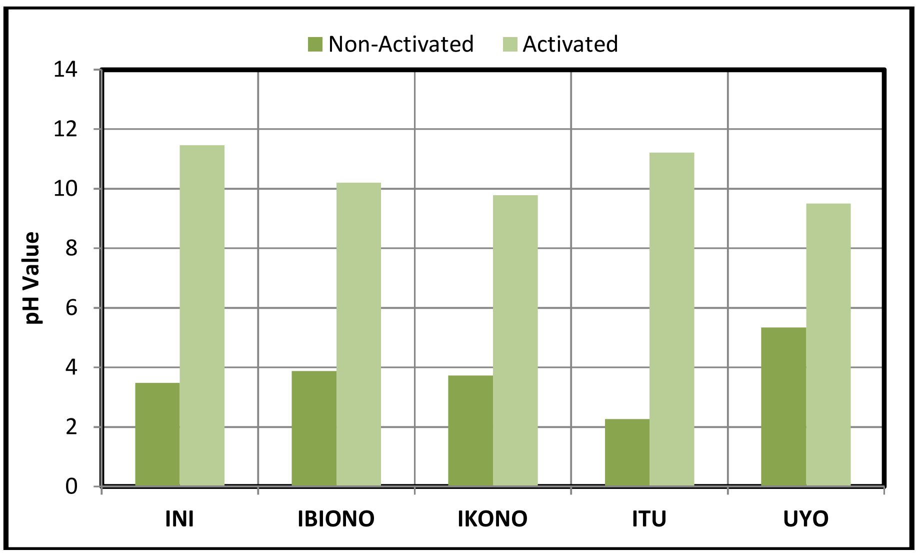

Fig. 7. pH value of the bentonitic clays' water-based mud

non-activated clay muds' pH of 2 – 5 were changed to alkaline clay muds (pH: 9 – 11), as the activation process introduces more sodium ion (Na^{2+}) into the clay structure. This development also accounted for the increase in the swelling power of the bentonitic clays. As the medium becomes alkaline, the solubility of the clay particles in the medium (water) is enhanced. Conversely, the acidic nature of the non-activated bentonitic clays hindered the solubility of the clay particles, thus, resulting in negligible swelling power as shown in Table 3. Fig. 7 further shows that the activation process resulted in muds' pH increase of 228.65%, 162.89%, 162.20%, 396.02% and 77.90% for Ini, Ibiono, Ikono, Itu and Uyo, respectively. A development which shows that the activated bentonitic clays' pH values were within the API bentonite pH value range as indicated in Table 1.

4. CONCLUSION

The use of clay as industrial product is not limited to its application in drilling fluid formulation. Its useful applications in ceramics, refractoriness and foundry industries cannot be overemphasized. Interestingly, Nigeria is endowed with this mineral resource – clays of varying composition. In the course of this, clays from local deposits in Akwa Ibom State were evaluated for its suitability as water-based mud viscosifiers. Thus, the following conclusions were drawn:

1. The compositions of the bentonitic clays are mixture of montmorillonite and kaolinite that can be improved to API specification by activation.
2. The rheological properties of the activated bentonitic clays were significantly improved to compare closely with the API specifications.
3. The activation of the bentonitic clays resulted in fluid loss reduction of 83.91%, 86.10%, 87.84%, 83.80% and 87.01% for Ini, Ibiono, Ikono, Itu and Uyo clay, respectively; with Ibiono, Ikono and Uyo bentonitic clays exhibiting a viable fluid loss potential as drilling mud viscosifiers.

Finally, tapping into these reserves of the bentonitic clay deposits in the state would be a value addition to the oil and gas industry; as cost of importation of foreign bentonite would be saved, especially now that the industry is cutting costs due to plummeting oil prices. Again, the mining of these clay reserves would add to the Nigerian economy through job provision. It is recommended that further research be carried out on the local bentonitic clay activation to determine the optimum Na_2CO_3 concentration for activation of local clays. Also, establish the effect of Na_2CO_3 on bentonitic clay's mineralogical compositions.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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