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Characterization and Beneficiation of Some Nigeria Bentonitic Clays for Formulation of Drilling Mud in Oil and Gas Industry

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Abstract

In the quest to develop sustainable materials locally for the drilling industry, the bentonitic potentials of two indigenous clays from Edikor and Ukukudung deposits were studied. The x-ray fluorescence (XRF), Fourier Transform Infrared (FTIR) and Scanning Electron Microscope (SEM) techniques were used to characterize the beneficiated bentonitic clay samples. This was done to ascertain the chemical composition and structural morphology of the samples. The minerals identified in FTIR results of the clay samples show that they are montmorillorite of the smectite group. The AlAlOH stretching band occurs at 2512 and 2504 cm^{-1} whereas the bending occurs at 1766 & 1796 cm^{-1} for Edikor and Ukukudung clays, respectively. The SEM microstructure images show that the bentonite samples are generally moderately dispersive with some large flows which were separated and dispersed from one another. For the drilling mud formulation, the two local clays were beneficiated with 1.5 g Na_2CO_3 / 350 ml of water.

Keywords: XRF, FTIR, SEM, Bentonite, Beneficiation, Characterization.

1.0 INTRODUCTION

Nigeria is among the top oil producers in sub-Saharan Africa, and her economy is largely based on this mineral. Hence, the continuous search for local materials in processing this mineral. Falode *et al.*, (2007), and Emofurieta (2010) reported at the international conference on “Modern Mining Processing” that Nigerian bentonite proven reserves have risen above four billion metric tons. However, research over the past years has shown that drilling activities in the petroleum and ground-water development industries in Nigeria are still consuming large amounts of clay for drilling mud formulations, of which a greater percentage is imported despite these large reserves of clay in Nigeria. (Omole *et al*., 1989). The bentonitic clays require some level of beneficiation to upgrade the quality before application in drilling fluid preparation making.

2.0. MATERIALS AND METHODS

2.1 Materials and Equipment

The Edikor and Ukukudung Bentonitic clays were sourced from UdungUko Local Government Area of Akwa Ibom State. All reagents used were of analytical grade.

All materials used are: Edikor bentonitic clay, Ukukudung bentonitic clay, Sodium carbonate (Analytical grade), Sulphuric acid (Analytical grade), pH paper, Distilled water, Tap water, and Masking tape

The equipment used for the work include: Fourier Transform Infrared (FTIR) spectrometer (8400S model)**,** Scanning Electron Microscope (Machine PHenom Pro – X model), Electric Oven (Haracus model), Weighing Balance (Adam 22 model), Viscometer (Rheometer) 6 speed (OFITE 900 model), Thermometer (Pyrex England model), Energy Dispersive X-ray Flourescence (EDXRF) spectrometer (X-Supreme 800 model), Mud balance 50 ml Graduated cylinder Pyrex (England model, 50 ml), Burette (Pyrex, England), 250 ml conical flask, Magnetic pill (Pyrex, England), Hot plate with magnetic stirrer (Thermo Fitcher), Ball mill machine (Kera Bv), Mud balance, Standard sieve mesh (US standard).

2.2 Methodology of Clay Preparation

The method of preparing the clay is highlighted in Figure 1.

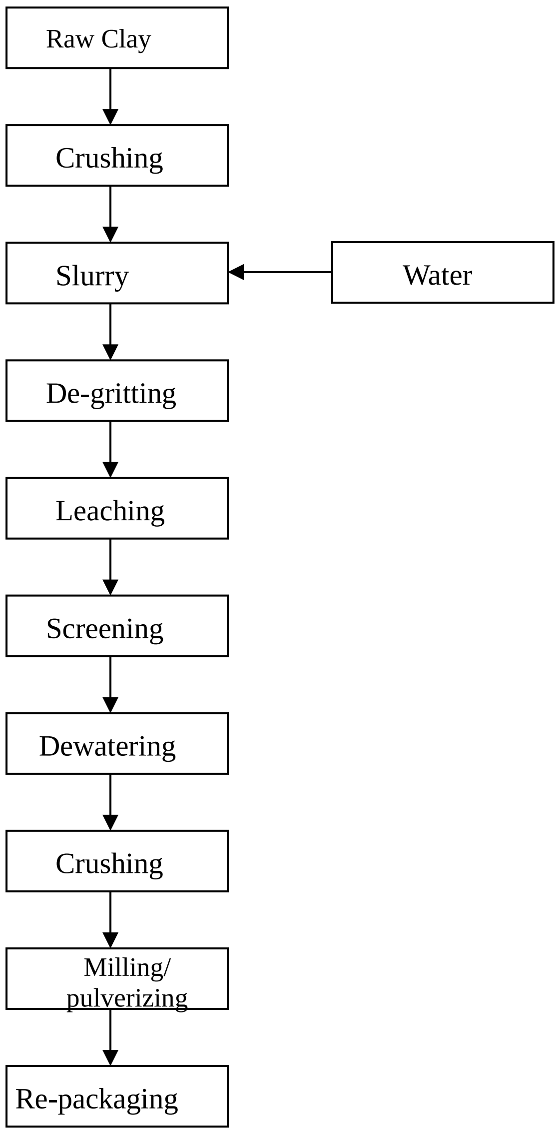

Fig 1: Flow Chart for Clay Processing

2.2.1 Sample Preparation

The clays were obtained from Ukukudung and Edikor all in Udung Uko Local Government Area of Akwa Ibom State. These samples were dried and crushed into a smaller sizes after which it was dissolved in water to form a slurry.The slurry was sieved to get fine particles of the clay which passed through as filtrate whereas sand and organic matter are allowed to settle as residues. The filtrate was allowed to settle for 4-5 hours and then decanted to obtain clear clay mud.

2.2.2 Drilling Mud Formulation

The mud samples were prepared by weighing out 25 grams of the clay samples. The measured clay samples were poured into a separate mixer cups containing 350 ml of fresh water. The clay samples and water were adequately stirred. The mud samples were allowed to age for 24 hours to hydrate properly, after which density, rheological properties, sand content, and pH were tested.

2.2.3 Clay Beneficiation

The clay samples were beneficiated with sodium carbonate 1.5g (Na_2CO_3) in 350ml of water, to convert calcium-based bentonite to sodium-based bentonite by cation exchange.

2.3 Experimental Procedure for Determination of Drilling Mud Properties

API RP-13B standard procedures were used in compliance with world best practices and acceptable laboratory means of determining rheological and fluid loss properties. All sample muds were based on the formulation of 350 ml of fluid that contains only fresh water.

pH value: The pH meter was calibrated using deionized water thereafter pH of the sample was measured.

Determination of the mud weight: The mud balance was standardized using distilled water. The balance cup was cleaned, dried, and filled with the sample to be measured.

Determination of rheological properties: The rotational viscometer gives a better measurement than the marsh funnel (Bourgoyne *et al.*, 1986).

The rheological properties are obtained from these equations

$$PV = \theta_{600} - \theta_{300} \tag{2.1}$$

$$AP = \frac{\theta_{600}}{2} \tag{2.2}$$

$$YP = \theta_{300} - PV \tag{2.3}$$

Swelling Power Test: The swelling capabilities of both beneficiated and non-beneficiated clays were carried out.

Fluid Loss: The API standard LT-LP filtration test was conducted at room temperature and 100 psi for 30 minutes (Akinde 2012).

3.0 RESULTS AND DISCUSSION

3.1 Chemical Composition Results

The results of the elemental analysis using X- ray fluorescence (XRF) as presented in Table 2.

The results indicates that the Al_2O_3 / SiO_2 ratio varies for non-beneficiated and Na_2CO_3 beneficiated samples, this is because of variation in the percentage of the two oxides (Al/Si) present in each sample. The presence of excess silica in all samples affected the Al/Si ratio, but after beneficiation, there was significant improvement of Edikor and Ukukudung clay samples.

The presence of Na_2O in the samples indicates the sediments were deposited in an alkaline environment and its availability in clay materials enhances the viscosity and hydration ability of the clay as observed in some researches (Shuwa 2011 & Dewu et. al 2011) The Na_2O in Edikor and Ukukudung samples were 0.21% and 0.27% respectively but after activation with Na_2CO_3 the percentage of Na_2O improved to 1.18% and 1.71% respectively.

Table 1: XRF Results for Non-Beneficiated and Beneficiated Local Clays

Location	Edikor		Ukuku-Udung		API Bentonite
Elements	Non-Beneficiated	Beneficiated	Non-Beneficiated	Beneficiated	
SiO_2	57.80	62.82	66.90	67.30	58 – 64
Al_2O_3	35.10	22.62	26.30	23.90	18 – 21
TiO_2			2.18	2.17	
Fe_2O_3	2.15	8.92	1.65	2.08	2.5 - 2.8
MgO	0.08	1.02	0.14	2.01	2.5 - 3.2
CaO	0.42	2.05	0.72	1.95	1.1 - 1.0
Na_2O	0.21	1.18	0.27	1.71	1.5 - 2.7
K_2O	0.99	2.10	0.98	0.056	0.2 - 0.4
MnO	0.02	0.012	0.02	0.008	-
LOI	2.62	-	0.84	-	
Al_2O_3/SiO_2	0.61	0.36	0.39	0.30	0.31 - 0.33

LOI: Loss on ignition
API BENTONITE source: Agwu *et al.* (2016)

3.2. Rheological Properties

The rheological properties of a sample are plastic viscosity, apparent viscosity, yield point,and gel strength. The results obtained from mud analyses for both raw and beneficiated clay samples are presented in Table 2.

YP/PV is a significant indicator of drilling fluid condition. API stipulates 3 (max) for YP/PV. Both samples in raw nature are within API recommended range(Obaje 2013).

Mud Density: The mud was formulated and weighed in (g/cm^3) using mud balance and measuring cylinder directly.

pH Values: API recommended values ranged from 9.5 – 12.5(max). The PH of raw samples is 3.68 and 2.81 lb/g for Edikor and Ukukudung, respectively. Upon beneficiation with sodium carbonate (Na2CO3), the PH increased within standards (Okorie *et.al.*, 2016)

Sand Content: The lower sand content of a formulated mud, the better its performance.

Table 3: Mud Properties of Non-Beneficiated and Beneficiated Local Clays' Water-Based Drilling Fluid

Mud Properties	Edikor		Ukuku-Udung		API Bentonite
	Non-Beneficiated	Beneficiated	Non-Beneficiated	Beneficiated	
Mud Density (lb/gal)	8.74	9.20	8.48	8.90	-
Sand Content (%)	0.27	-	0.16	-	-
Apparent Viscosity (cP)	2.50	13.50	3.25	15.10	15.0
Plastic Viscosity (cP)	2.00	8.50	3.00	7.58	8.0
Yield Point ($lb/100ft^2$)	1.00	10.00	0.50	12.50	-
Gel Strength ($lb/100ft^2$)	0.5/1.0	1.0/2.6	0.5/1.0	1.0/3.0	-
YP/PV Ratio	0.50	1.18	0.17	1.65	3 (max)
Fluid Loss (mL)	98.0	17.50	120	22.50	15 (max)
Mud Cake Thickness (mm)	1.00	3.50	0.50	3.25	-
pH Value	3.68	11.48	2.81	12.72	8-10
Swelling Index (%)	42.0	73.0	49.0	81.0	-

API BENTONTE source: Agwu *et al.* (2016)

3.3 SEM Results of Clay at Different Magnifications

Edikor clay sample was viewed at different magnification of 500um, 1000um, and 1500um, respectively, as shown in Figures 3, 4, and 5. The morphology of Edikor samples was observed to be irregular with dispersed clay minerals which are suspected to be alumina. Ukukudung clay was viewed at 500um, 1000um, and 1200um as presented in Figures 5, 6, and 7. Clear cavities and particles which are poorly aggregated as flocs were observed, the smaller the flocs, the stronger and better the bonding in the sample.

They are spherical and slightly loose. Some large flocs indicating weak inter-particle bonding were observed especially in Figures 8 and 9. Beneficiation with sodium carbonate in Ukukundung samples shows more dispersion in the clay and formation of smaller flocs which aided Calcium-Sodium cation exchange compared to Edikor clay.

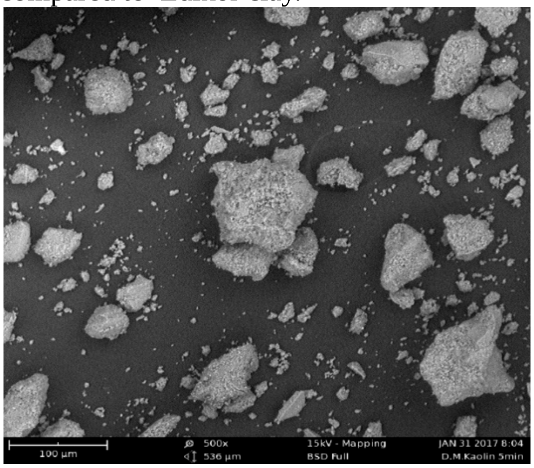

Fig 3: SEM result of Edikor sample @ 500um,

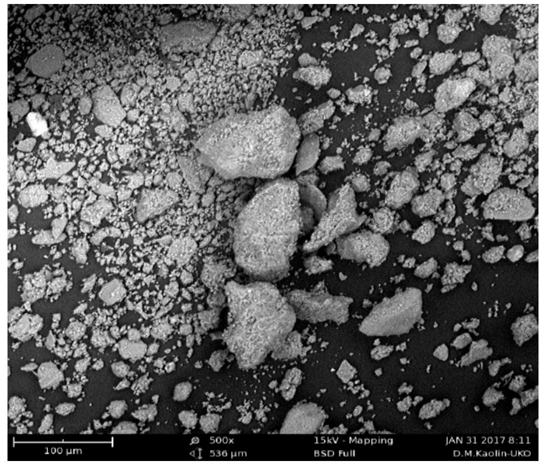

Fig 4: SEM results UkuUdung sample @ 500um

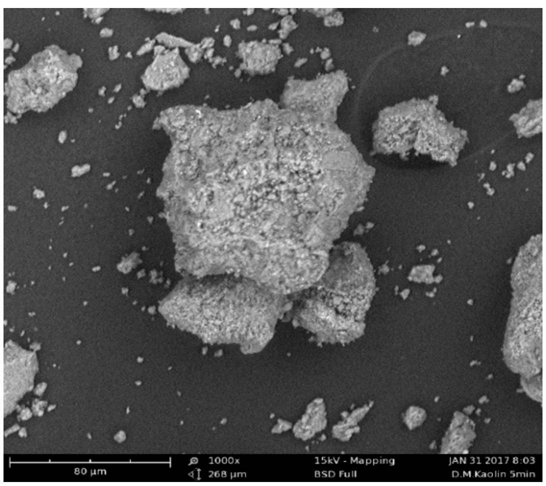

Fig 5: SEM result of Edikor sample @ 1000um,

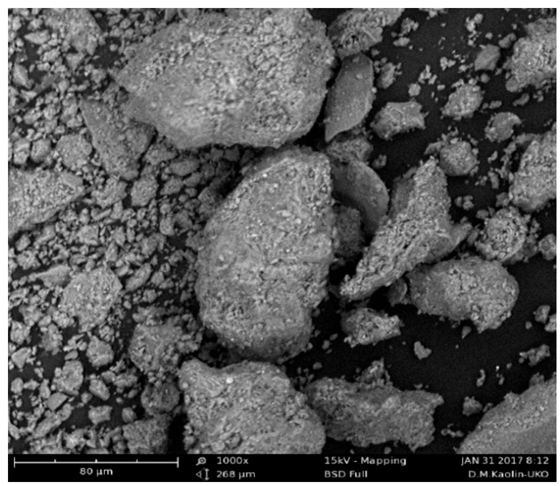

Fig 6: SEM results of Ukukudung sample @1000um

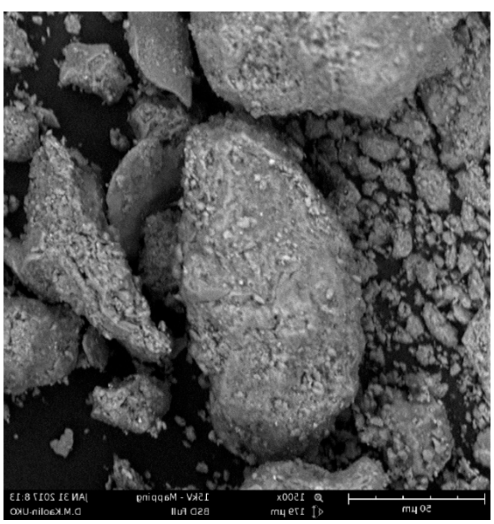

Fig 7: SEM result of Ukukudung sample, @1200um

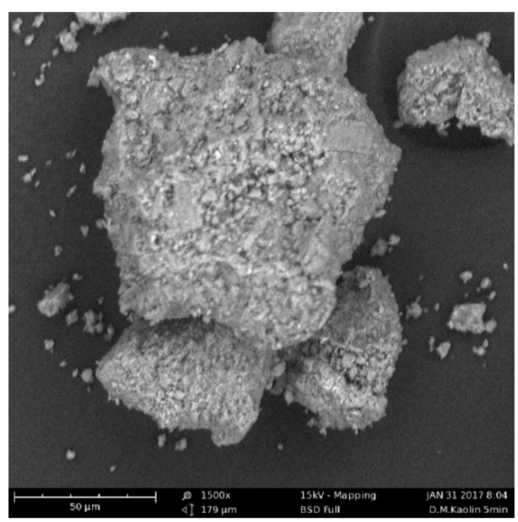

Fig 8: SEM result Edikor sample @ 1500um

3.4 FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR) ANALYSIS

A larger amount of kaolinite in the bentonite may result in less swelling and a lesser degree of sorption (Madejova *et al.*, 2001).Smectites generates inner –surface OH stretching bands at 3660 and 3630cm-1 mostly beidellite, 3632cm-1 for montmorillonite or 3564cm-1 for nontronite (Farmer and Russel 1967). In most cases, the band for smectites is broad and several unresolved spectral components can be attributed to it.

Spectra of clay minerals are rather complicated; the intensity and positions of IR absorption peaks depend on mineral formation and the presence of various impurity ions and elements including Mg, Ca. In the lattice of the minerals; the larger the ionic radius is, the more peaks shift to a longer wave region. Bands around 3695, 3666, 3650, and 3620 cm-1 and 3400cm-1 are the most diagnostically reliable for kaolinite and montmorillonite. (Vu Cong Khang 2004). The FTIR stretch bands and bendings for the Edikor sample are 872.2um, 1038.7um, 1390.3um, 2512.2um, and 872.2, 1028.7, 1397.8 and 2504um for Ukukudung samples. The API grade sample stretch between 870-890um confirms the presence of OH bending which is common in both samples. Again, the API grade that stretches between1032-10447 is most likely to be Si tetrahedral Si-O stretching as observed in Figure 10. The API bentonite CO3-Stretching is 1441um, but Edikor 1390um and Ukukudung 1390um is close to the result of standard bentonite.API Grade for H_2O stretching is 3485,3390,2516um. From Figures 9 and 10 both Ukukudung and Edikor possess H2O Stretching which is 2504.8um and 2512.2um, respectively. Although OH –stretching of the inner surface hydroxyl occurs around 3662um which depicts a typical montmorillonite, but Figures 10 and 11 did not clearly show montmorillonite stretch even though the swelling ability and other characteristics of both samples present montmorillonite potential.

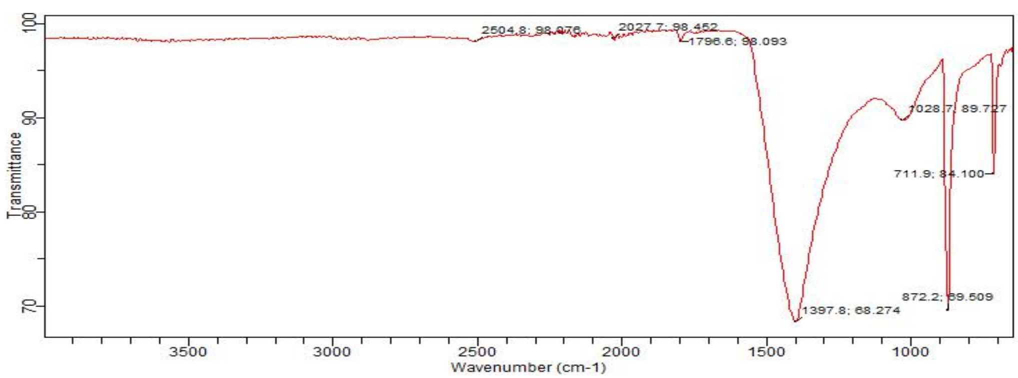

Fig 9: FTIR Analysis of Edikor Clay

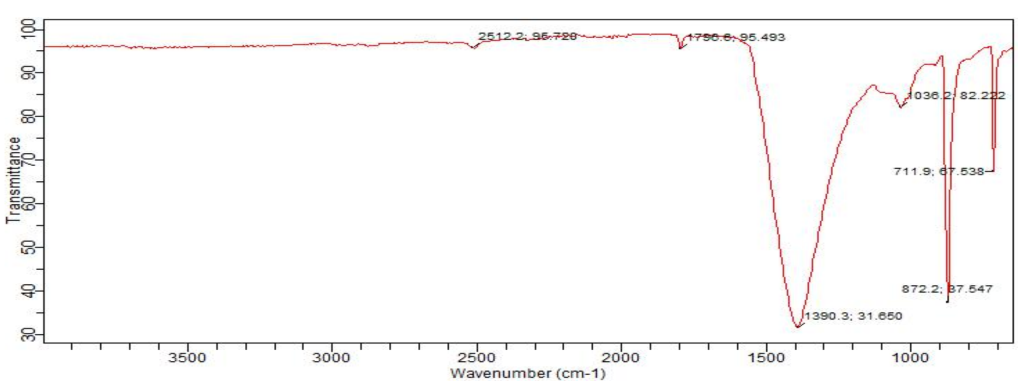

Fig 10: FTIR Analysis of Ukukudung Clay

4.0 CONCLUSION AND RECOMMENDATION

This study beneficiated and characterized some Nigerian bentonitic clay from Edikor and Ukukudung formation. The samples revealed low grade calcium montmorillonite making it unsuitable for utilization in drilling fluid formulation, but upon beneficiation the rheological properties improved significantly in comparism with imported standard Wyoming bentonite. The characterization was done with the application of XRF, SEM, and FTIR. FTIR shows Bentonitic characteristics but with a lower inner surface OH stretching band of 2504cm-1 and 2512 cm-1 for Edikor and Ukukudung, respectively the literature value 3627-1cm-1.However, the sample shows a bentonitic clay behavior rich in silica and Alumina and can be deduced to be montmorillonite of smectite. Scanning electron microscopy (SEM) carried out reveal that both samples possessed morphology which is a confirmation of its bentonitic clay nature.

The X-ray fluorescence conducted on the non-beneficiated and beneficiated samples again gives result within the API bentonite standard.

To reduce wasteful capital flight, it is imperative to develop a drastic policy intervention with the aim of developing the 'National strategy for competitiveness in raw materials (local clay) and other products in Nigeria'.

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