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**Research Article**

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# Study of the Physicochemical Properties of Nigerian Bentonitic Clay Samples for Drilling Fluid Formulation

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**ABSTRACT** The physicochemical properties (colour, pH, hardness, lump size, moisture content, loss on ignition (LOI) and specific gravity) of the Nigerian Bentonitic clay samples used for oil and gas drilling fluid formulation were investigated. The samples are generally light grey with brown stains on Garin Hamza, Bulabulin Maiduwa and Sabongarin Ngalda. Green and Yellow pigments were also observed on Pindiga Bentonite. Some samples such as Garin Hamza Futuk, Bulabulin Maiduwa and Sabongarin Ngalda are consolidated and slightly hard and brittle compared to Pindiga and Tongo Bentonite that are semi consolidated and soft. The moisture content of the fresh samples ranges from 9.49 to 59.77%.. While after drying the samples in an oven at 110°C for 48 hours, the moisture content drastically reduced with maximum of 12.30%.The LOI for all the Bentonitic clay samples ranges from 33.0 to 35.0 %.The pH for all the raw samples ranges from 6.18 to 6.40 contrary to standard 8.5-12.5 and the specific gravity of all the raw bentonites ranges from 2.25 to 2.56.

**Keywords** Physicochemical, Bentonitic clay, Consolidated, pH and Specific gravity

## Introduction

The term "bentonite" is ambiguous. As defined by geologists, it is a rock formed of highly colloidal and plastic clays composed mainly of montmorillonite, a clay mineral of the smectite group and is produced by *in situ* devitrification of volcanic ash [1].The transformation of ash to bentonite apparently takes place only in water (certainly seawater, probably alkaline lakes, and possibly other fresh water) during or after deposition [2-3]. Bentonite was named after Fort Benton (Wyoming, USA), the locality where it was first found.

Bentonite feels greasy and soap-like to the touch [4]. Freshly exposed bentonite is white to pale green or blue and, with exposure, darkens in time to yellow, red, or brown [1]. The special properties of bentonite are an ability to form thixotrophic gels with water, an ability to absorb large quantities of water with an accompanying increase in volume of as much as 12–15 times its dry bulk, and a high cation exchange capacity.

Substitutions of silicon by cations produce an excess of negative charges in the lattice, which is balanced by cations ($Na^+$, $K^+$, $Mg^{2+}$, $Ca^{2+}$) in the interlayer space. These cations are exchangeable due to their loose binding and, together with broken bonds (approximately 20% of exchange capacity), give montmorillonite a rather high (about 100 meq/100 g) cation exchange capacity, which is little affected by particle size. This cation exchange capacity allows the mineral to bind not only inorganic cations such as caesium but also organic cations such as the herbicides diquat, paraquat [5], and striazines [6], and even bio-organic particles such as rheoviruses [7] and proteins [8], which appear to act as cations.

Variation in exchangeable cations affects the maximum amount of water uptake and swelling. These are greatest with sodium and least with potassium and magnesium. Interstitial water held in the clay mineral lattice is an additional major factor controlling the plastic, bonding, compaction, suspension, and other properties of montmorillonite-group clay minerals. Within each crystal, the water layer appears to be an integral number of molecules in thickness.

The ability of montmorillonite to rapidly take up water and expand is lost after heating to a critical temperature, which ranges from 105 to 390 °C, depending on the composition of the exchangeable cations. The ability to take up water affects the utilization and commercial value of bentonite [1-2].

Montmorillonite clay minerals occur as minute particles, which, under electron microscopy, appear as aggregates of irregular or hexagonal flakes or, less commonly, of thin laths [2]. Differences in substitution affect and in some cases control morphology.

The USA is the major producer of bentonite Fuller's earth. Approximately 90% of world bentonite production is concentrated in 13 countries: the USA, Greece, the Commonwealth of Independent States, Turkey, Germany, Italy, Japan, Mexico, Ukraine, Bulgaria, Czech Republic, South Africa, and Australia.

Marine shale units that are highly enriched in montmorillonite are found in Nigerian sedimentary basins. Notable among these are the Awgu shale in Eastern Nigerian, the Imo shale that forms a belt across Southern Nigeria, the Fika shale in the North-eastern parts, and the Dukamaje and Kalambaina**.** Formations in the North West.

Bentonite clays also exist in the North-east quadrant of Nigeria (Borno, Yobe, Taraba and Adamawa) where a probable reserve of more than 700 million tones has been indicated. Similarly, over 90 million tones have been reportedly found in Afuze, Ekpoma-Igunebon road, Ovibiokhuan and Okpebho areas of Edo State. Some occurrences have also been reported in Abia, Ebonyi and Anambra States.Clay mineral studies of the Tertiary to Recent subsurface in Niger Delta have revealed occurrences of bentonite as well as other non-swelling kaolinitic clays. Since the Niger Delta opens to the sea, there is likelihood that the subsurface Niger Delta bentonite may be enriched in sodium from the saline seawater. Exploration efforts aimed at commercial exploitation are going on in several localities now, while some companies have actually started test mining. Bentonite occurrence has been reported in Benue State, Abia State, Uturuumuahia, Anambra State (Awgu, Awka) [9], in Azara; Nasarawa State, Awa in Benue state, Akiri Hill in Plateau state, Wukari in Taraba State, Pindiga and Fika in Gombe and Yobe states respectively [10].

The availability of bentonite in Nigeria and the wide range of industrial application enhance the attractiveness of the bentonite processing ventures. The present level of consumption is about 200,000 MT (for oil well drilling only); much of this is sourced from the foreign market [11].

The importance of bentonite as a mud in drilling operations in water, oil and gas well construction cannot be overemphasized. The cost of drilling is reduced by about 15% when drilling mud is used and this is not small given the overall cost of drilling an oil or gas well.

The chemical composition of bentonite affects its usage [3,12& 13]. High swelling bentonite, in which sodium is usually the dominant exchangeable ion, is preferred for drilling muds, pelletizing iron ore, sealing and waterproofing, whereas low-swelling calcium bentonite is preferred for filtering, clarifying, absorbing and for serving as a filler, stabilizer, extender, carrier, bonding agent, or catalyst. Both types are used as a foundry sand bond. Sodium bentonites provide good dry strength in moulds, whereas calcium bentonites provide good "green" (condition prior to drying) strength.

The research is aimed at studying the physicochemical properties of the Nigerian Bentonitic clay samples collected from various locations of the sedimentary succession of Upper Benue Trough north eastern, Nigeria, for application in drilling fluid formulation.

## Materials and Methods

### Collection of Raw Samples

The raw Bentonitic clay samples to be used for the analysis were collected from the marine environment of upper Benue trough, north eastern Nigeria. The raw Bentonitic clays were collected from five different locations across the region by digging of Bentonite deposits to about 1.5m as shown in Figure 1. The fresh collected samples were kept in a polythene bags and then brought to the Department of Material Science laboratory, Centre for Energy Research and Training (CERT) in collaboration with Department of Chemical Engineering Ahmadu Bello University, Zaria, for experimental analysis.

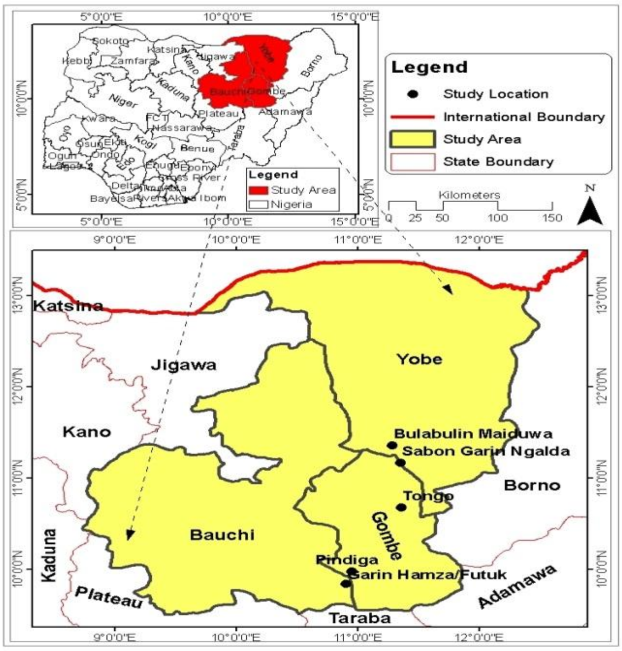

*Figure 1: Map of the study area showing sample locations*

### Sample Preparation

The raw samples collected from the field (having large particle size) were crushed and ground into smaller particle size with the aid of a jaw crusher and a vibratory disc mill machine (RS-200), in order to avoid cross contamination of the samples, acetone was used to clean the jaw crusher and the grinding machine before each sample was loaded into it. The samples were then sieved to API specification of 63 µ using 180 µ, 93 µ and 63 µ sieves mounted on a sieve shaker (Octagon 200) in that order to achieve a desired yield. The samples were dried in an oven at 110 $^{o}$C for 24 hours to remove the moisture that resists passage of the ground particles while sieving.

### Determination of Loss of Ignition of the Bentonitic Clays

Loss on ignition (LOI) was determined in order to measure the organic matter and carbonate content of the clay samples estimated by weight loss measurements in core sub-samples subjected to sequential heating [14,15 & 16).The crucible was initially washed, cleaned and dried at 105$^{o}$C for an hour and cooled in a desiccator for 30 minutes. The crucible was weight ($W_1$) and then filled to about one third with the sample ($W_2$) and heated to 550$^{o}$C for 4 hours. The crucible and the ash were reweighed ($W_3$) after cooling in a desiccator. The sample was then heated in a furnace for additional 2 hours after which the crucible and the ash were cooled in a desiccator and reweighed ($W_4$). Equations 1 and 2 were then used in calculating the LOI at 550 $^{o}$C and 1000 $^{o}$C respectively.

$$\%\ \text{Loss of ignition, LOI @ 550 o C} = \frac{W2-W3}{W3-W1} * 100\ \mathbf{(\%)} \qquad \text{Equation 1}$$

$$\%\ \text{Loss of ignition, LOI @ 1000 oC} = \frac{W2-W4}{W4-W1} * 100\ \mathbf{(\%)} \qquad \text{Equation 2}$$

### Determination of Moisture Content

The method is based on removing clay soil moisture by oven-drying a soil sample until the weight remains constant. The moisture content (%) was calculated from the sample weight before and after drying [17]. Moisture content was determined using similar procedure as explained in loss of Ignition determination procedure, the only difference between the two is the temperature difference. Moisture contents were determined at 110 $^{o}$C, while the Loss on ignition was determined at 550$^{o}$C for 4 hours to 1000$^{o}$C for 2 hours using equations 1 and 2.

## Determination of pH

The acidity and the alkalinity of the drilling fluid can be measured by the concentration of the ($H^+$) ion in the fluid. The pH of various formulated drilling fluids (Plate VII), were measured by direct measurement using a pH meter. The pH meter was calibrated using pH buffer 4, 7 and 9. The electrode was immersed into the clay and the pH meter reading was taken after 30 seconds.The pH of a mud seldom is below 7 and in most cases fall between 8 and 12.5 depending upon the type of mud. The pH is important because the pH affects the solubility of the organic thinners and the dispersion of clays presents in the mud.

## Results and Discussion

### Study of Physical Properties of Raw Clay Samples (Petrography)

This is hand specimen description of physical properties of raw samples such as colour, lustre, hardness and Lump size (as mined). The raw clay samples were carefully studied and their physical properties are:

### Colour

The colour of all the collected samples of bentonite is generally light grey with brown stains on Garin Hamza, BulabulinMaiduwa and SabongarinNgalda. Green and Yellow pigments were also observed on Pindiga Bentonite. The brown pigments could be iron impurities while green could be organic matter and the yellow pigments could be sulphur from the decomposed fossils deposited at the time of deposition of the sediments. It was observed that Tongo sample is the darkest of all the samples probably due to the presence of ferromagnesian minerals in the sediments as clearly shown in Plate I.

Plate I: Morphological features of raw bentonitic clays as collected from the field.

### Lustre and hardness

The samples are generally smooth due to relatively high content of fine grained clay forming minerals. Some samples such as Garin Hamza Futuk, BulabulinMaiduwa and SabongarinNgalda are consolidated and slightly hard and brittle compared to Pindiga and Tongo Bentonite that are semi consolidated and soft.

### Lump size

The sizes of various bentonites vary in sizes from sediment fragments, mineral chips to fine colloidal particles that range in size from 1cm to > 6 cm in Garin Hamza Futuk, Pindiga, and BulabulinMaiduwa Bentonites, while lump size in Tongo and SabongarinNgalda ranges from < 1 cm to 3 cm (see Plate I). The consolidated sediments are denser than the semi consolidated sediments due to compaction of the clay minerals within the consolidated bentonite as well as the mode of deposition and the environment of deposition of the bentonites.

### Moisture Content and Loss on Ignition.

The moisture content of the bentonitic clays indicates the amount of suspended water molecules within the sediment. The moisture contents of various fresh and dried bentonite samples are shown in Table 2. The result for the fresh sample indicates that Garin Hamza had the highest moisture content of 59.77% followed by SabongarinNgalda, Pindiga, Bulabulinmaiduwa and Tongo with the moisture content of 13.11%, 11.67%, 9.63% and 9.49% respectively. While after drying the samples in an oven at 110°C for 48 hours, the moisture content reduced with Tongo having the least moisture content of 9.6% followed by Garin Hamza, BulabulinMaiduwa, Pindiga and SabongarinNgalda having moisture content of 9.70%, 10.30%, 11.10% and 12.30% respectively. The high moisture content in some fresh bentonite samples is because they were collected close to the running stream channels where there is high moisture content, while those with low moisture content were collected from the dried exposures of the Bentonitic clay.

**Table 2:** Measured Physical properties of raw Bentonitic clay

| **S. No** | **Sample Name** | **pH** | **Moisture Content of Fresh Sample (%)** | **Moisture Content of Dried Sample (%)** | **Loss on Ignition** | **Specific Gravity** |
|---|---|---|---|---|---|---|
| 1. | API Standard literature Values | 8.5 | 10 – 27[a] | 10 – 13[a] | 10.64 | 2.83[b] |
| 2. | Garin Hamza/Futuk | 6.40 | 59.77 | 9.70 | 34.39 | 2.50 |
| 3. | Pindiga | 6.20 | 11.67 | 11.10 | 33.53 | 2.56 |
| 4. | Tongo | 6.29 | 9.49 | 9.60 | 34..47 | 2.47 |
| 5. | BulabulinMaiduwa | 6.18 | 9.63 | 10.30 | 34.62 | 2.39 |
| 6. | SabonGarinNgalda | 6.40 | 13.11 | 12.30 | 33.42 | 2.25 |

a- [18]$^{b-}$[19]

Table 2 also presents the loss on ignition (LOI) which indicates the percentage of carbonaceous materials or the organic matter content within the Bentonitic clay that oxidised to carbon dioxide and ash after burning in a furnace at 550°C for 4 hours and 1000°C for 2 hours. The LOI for all the Bentonitic clay samples ranges from 33 to 35 %. Some previous studies have shown that upper Benue clay had LOI of 24.2% [20], Fika clay 9.73% [21] and 6.6% for Ubakala clay [22] while [18] observed that the LOI for Wyoming, Baroid imported clay and Baroid Nigeria are 6.56 , 9.56 and 10.37% respectively, while Gambe clay has LOI 2.62% . When the result are compared with the standard commercial clay (Wyoming and Baroid imported clay) the lesser the LOI the better the rheology of the drilling fluid since the clay has less carbonaceous materials and vice versa.

The pH values that indicates the acidity or alkalinity of a sample indicates that the pH for all the raw samples range from 6.18 to 6.40 contrary to standard 8.5-12.5 [23]. While the specific gravity of all the raw bentonites ranges from 2.25 to 2.56. Pindiga bentonite is having the highest specific gravity of 2.56, followed by Garin Hamza with 2.50, Tongo 2.47, Bulabulinmaiduwa 2.39 and SabongarinNgalda with the least specific gravity of 2.25. The higher the specific gravity the better the ability of the formulated drilling fluid to exert a greater hydrostatic pressure that exists within formation pores during drilling.

## Conclusion

The studied physicochemical properties of the raw Nigerian Bentonitic clay samples indicates that the samples require treatment before they can be used for drilling fluid formulation, this can be achieved through impurity removal, increasing the pH from slightly acidic to basic (8.5 – 12.5) and specific gravity of the samples by addition of weighing agent such as barite to the formulated drilling fluid.

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