EFFECT OF BENEFICIATION OF GARIN-HAMZA FUTUK BENTONITE FOR OIL AND GAS DRILLING FLUID FORMULATION

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ABSTRACT
This research focused on beneficiation and characterization of raw Garin Hamza Futuk (GHF) Bentonite for oil and gas drilling fluid formulation. The FTIR results confirmed that GHF bentonitic clay is rich in montmorillonite mineral noticed at approximately 3620-3630 cm\(^{-1}\) stretching band in the higher frequency level. In the lower frequency region, montmorillonite had a strong band at 1024.24 and 1028.09 cm\(^{-1}\) for Si-O stretching vibration of layered silicates. The X-ray fluorescence (XRF) results showed slight reduction in free silica (Quartz) by about 1%. The SEM images of the beneficiated samples were more dispersed than the raw sample with some large flocs structure confirming their montmorillonitic nature. The optimum amount of poly anionic cellulose (PAC) used for instant drilling fluid formulation was found to be at 2.0 g, while the aged formulation was achieved at 0.8 g PAC when compared with the API grade. Hence, GHF bentonite can be used for drilling fluid formulation.

Key words: Bentonite, Beneficiation, Characterization, Drilling fluid and API Grade.

INTRODUCTION
Bentonite is a clay material rich in montmorillonite mineral used for formulation of drilling fluids in oil and gas industries. Since from the discovery oil in Nigeria, hundreds of oil wells have been drilled and no single well was drilled without using bentonite. Huge amount of money is being spent in procurement and importation of this bentonite while we have it in abundance in Nigeria, which needs little beneficiation (Bilal, 2016). It is therefore of interest to undertake beneficiation studies of locally sourced Garin Hamza Bentonite (GHB) located on latitude 09° 50’ 43.2” N and longitude 10° 54’ 10.3” E, Bauchi State-Nigeria. The GHB is available in commercial quantity and can be upgraded and compared with the API standard.

Bentonite is a montmorillonite and hygroscopic clay which is characterized by an octahedral sheet of aluminum atoms being infixed between two tetrahedral layers of silicon atoms (Lim et al., 2013). Bentonite has net negative electric charge due to the isomorphic substitution of Al\(^{3+}\) with Fe\(^{3+}\) and Mg\(^{2+}\) in the octahedral sites and Si\(^{4+}\) with Al\(^{3+}\) in the tetrahedral sites and is balanced by the cations such as Na\(^{+}\) and Ca\(^{2+}\) located between the layers and surrounding the edges. Natural Bentonite, when hydrated with water, is alkaline with pH of 8 to 10. It is hydrophilic in nature as it is strongly hydrated by water. This explains why bentonite has great water absorption capability. Water absorption of Bentonite occurs by means of diffusion and capillary suction (Lim et al., 2013). Bentonites are used as industrial raw materials in numerous applications such as palletizing iron ores, foundry bond clay, ceramic, drilling mud, sealant, animal feed bond, bleaching clay, agricultural carrier, cat box adsorbent, adhesive, catalyst and catalyst support, desiccant, emulsion stabilizers, cosmetic, paint, pharmaceutical, civil engineering, pillared clay organoclay and polymer-clay nanocomposites (Önal, 2006).

Commercially, there are two types of available bentonite; namely sodium bentonite and calcium bentonite. Sodium-Bentonite is characterized by its ability to absorb large amounts of water and form viscous, thixotropic suspensions. Calcium-Bentonite, in contrast, is characterized by its low water absorption and swelling capabilities and its inability to stay suspended in water (Bilal, 2016). Commercial deposits of Bentonites were reported in some areas in Nigeria with an estimated reserve of more than 4 billion metric tonnes; at the Benue Trough and around Jos plateau. Over 700 million tonnes deposits have been reported in the North-eastern part of the country in states like; Adamawa, Bauchi, Borno, Gombe, Yobe and Taraba. In the south-east deposits have been identified in Abia, Ebonyi and Anambra states (Arabi et al., 2011).
A drilling fluid, or mud, is any fluid that is used in a drilling operation which is circulated or pumped from the surface, down the drill string, through the bit, and back to the surface via the annulus. Drilling fluids serves many functions: suspending cuttings (drilled solids), removing them from the bottom of the hole and the well bore, and releasing them at the surface, controlling formation pressure and maintaining well-bore stability, sealing permeable formations, cooling, lubricating, and supporting the drilling assembly, transmitting hydraulic energy to tools and bit, minimizing reservoir damage, permitting adequate formation evaluation, controlling corrosion, facilitating cementing and completion, minimizing impact on the environment and inhibiting gas hydrate formation (Fred and Tim, 2005).

The type and quantity of additives is based on the drilling method employed and the type of reservoir to be drilled. The drilling mud can be broadly classified as water-based mud (WBM), oil-based mud (OBM), synthetic based mud (SBM), emulsions, invert emulsions, air, foam fluids among others (Subhash et al., 2010). WBM is used for this research due to environmental and economic considerations.

An upgrade in the properties of the clay to that of the standard commercial bentonite for drilling fluid and other industrial uses will go a long way to save petroleum companies the huge capital spent on imports of a product that can be sourced locally, thereby creating more employment and sustainable development in the mining of Bentonite which can lead to boosting the solid mineral mining sector of the nation’s economy. This research is aimed at studying the effects of beneficiation of Garin Hamza Futuk Bentonites for oil and gas drilling application.

**METHOD AND MATERIALS**

**Collection, Preparation and Characterization of the Sample**

The sample collection, beneficiation and determination of physicochemical properties of the bentonite clay were fully explained by (Bilal et al., 2016a; 2016b and 2016c). The laboratory characterizations of Garin Hamza Bentonites were carried out using 8400s Fourier Transform infrared (FTIR) spectrophotometer, Phenom ProX Scanning Electron Microscope (SEM) machine and Oxford X-Ray Fluorescence (XRF) machine in order to determine the mineral, chemical constituents as well as the morphological features of the clay. A comparison of the generated result with the API grade (Wyoming) bentonite was also carried out, in order to see how well they correspond with each other, thus giving a hint about its suitability in oil and gas drilling application.

**Drilling Fluid Formulation**

Water based drilling fluid formulation was chosen because it is environmentally friendly, non toxic and easy to be disposed, cost effective, has high penetration rate during drilling due to low colloidal particles and can be used in hard Formations (Bilal, 2016). The drilling fluids were prepared from raw, sodium activated, treated sample and standard API grade Bentonite (Wyoming) as control. High concentration formulation described by Okorie, (2006) and Adamu, (2013) was adopted by dissolving 24.5g of the powdered bentonite into 350 mL of clean water, thoroughly mixed for about 10 minutes. Additives/Viscosifiers (PAC) at different concentrations (0.5 – 2.5 g) were then added to the clay-water suspension (mud), thoroughly mixed and allowed to stay for 24 hours in order to have homogeneous mixture and improve hydration of the clay. The rheological properties were determined using the following equations (King Fahd University of Petroleum & Minerals Resources, 2003).

1. **Apparent Viscosity (AV)** in centipoise- (cP): \[ AV = \frac{\text{Reading @ 600 rpm}}{2} \]  
2. **Plastic viscosity (PV)** in centipoise- (cP): \[ PV = \text{Reading @ 600 rpm} - \text{Reading @ 300 rpm} \]  
3. **Yield Point (YP)** in (lb/100 ft²): \[ YP = \text{Reading @ 300 rpm} - \text{Plastic Viscosity} \]

**RESULTS AND DISCUSSION**

**FTIR Analysis**

The FTIR results for the raw, beneficiated/sodium activated and treated samples were compared with the API grade (Wyoming) sample as depicted in Figure 1. The results showed that the FTIR spectrum for the raw sample did not correspond with the API grade, which is basically due to presence of excess free silica that serves as impurities in the raw sample since it has not been beneficiated. However, the result for the beneficiated/\( \text{Na}_2\text{CO}_3 \) activated and treated samples showed significant similarity with the API grade bentonite indicating that there has been much improvement after beneficiating /Sodium activation and treatment with PAC respectively. The studied samples showed that \( \text{Al} \text{OH} \) stretching band occurred at 3622.44, 3619.54 and 3635.94 cm\(^{-1}\) for raw, beneficiated/\( \text{Na}_2\text{CO}_3 \) activated and treated Garin Hamza Bentonite (Shown in Figure 1(b), (c) and (d)) respectively. The bentonite samples that showed strong absorption bands approximately at 3630 cm\(^{-1}\) are said to be Al-rich smectites, mostly
montmorillonite. It was explained that \textit{AlAlOH} stretching band is typically at 3620-3630 cm\(^{-1}\) (Kumpulainen and Kiviranta, 2010). The band at 3413.15, 3436.30 and 3438.23 cm\(^{-1}\) gave a functional group with H-bonded and O-H stretch which is broad. This O-H stretching is the band of absorbed inter-water layer (Madejova and Komadel, 2001).

In the lower frequency region, montmorillonite had a strong band at 1024.24, 1017.48 and 1028.09 cm\(^{-1}\) for Si-O stretching vibration of layered silicates. The absorption peaks in the 1635.69, 1637.62 and 1640.51 cm\(^{-1}\) region was attributed to the O-H deformation mode of water. The band at 525.62 cm\(^{-1}\) for the raw sample corresponded to the deformation mode of Al-O-Si group. There were also some quartz or other polymorphs of SiO\(_2\) present as indicated by Si-O stretching band at \(~800\) cm\(^{-1}\) (Madejova and Komadel, 2001), which Garin-Hamza Bentonitic samples showed at 781.20 and 785.05 cm\(^{-1}\).

The FTIR analysis indicated that the sample is a montmorillonite of the smectite group, as the absorption bands depicted closely agree with that observed with montmorillonite clays.

Figure 1: FTIR Spectra for (a) API Grade bentonite (b) Raw (c) Beneficiated/Na\(_2\)CO\(_3\) activated (d) Treated Garin Hamza sample.

**SEM Images**

The SEM images displayed the microstructure of the clay samples at 1000x magnification for a detailed inspection of the morphological features of the clay samples. Montmorillonites tend to occur in thin equidimensional flakes that have a film-like appearance. In addition to flakes, some platy or needle particles may be seen (Velasco, 2013). Figure 2 depicted the SEM images of the API grade bentonite that revealed that the particles are flocculated but dispersed or loosely spaced together while the raw sample (Figure 3) is somewhat aggregated or clumped together and only moderately dispersed. The SEM images for the beneficiated/Na\(_2\)CO\(_3\) activated sample and treated samples were more dispersed than the raw sample. This anomaly is attributed to the impurities present in the raw sample. This has been confirmed by the explanation of Velasco (2013), that the SEM microstructure images usually indicate that the Bentonite samples are moderately dispersive to dispersive with some large flakes structure confirming their Montmorillonitic nature. This was observed in the beneficiated/Na activated and treated samples in Figures 4 and 5.
Figure 2: SEM image for API Grade (Wyoming) Bentonite.

Figure 3: SEM images for Raw Garin-Hamza Bentonite sample

Figure 4: SEM images for beneficiated/ Na$_2$CO$_3$ Activated Garin-Hamza Bentonite sample

Figure 5: SEM images for the Treated Garin-Hamza Bentonite sample

**XRF Analysis**

The XRF result gives an insight on the actual percentages of various compounds present in each sample and the results are compared with the chemical composition of the standard Bentonitic clay being used for drilling fluid formulations according to API specifications. The results in Table 1, indicate that the Al$_2$O$_3$/SiO$_2$ ratio varies in each sample for both the raw and Na$_2$CO$_3$ activated samples, this is due to variation in the percentage of the two oxides (Al/Si) in each sample. The presence of excess silica in all the samples affected the Al/Si ratio, but after beneficiation, there was slight increase in Al/Si ratio. The Al/Si ratio in raw Garin Hamza/Futuk is 0.31 while after silica reduction the ratios slightly increased to 0.32.
Presence of Na$_2$O in the samples indicates that the sediments were deposited in alkaline environment and its availability in clay materials enhances the viscosity and the swelling power of the clay as discovered by many researchers (Arabi et al., 2011, Dewu et al., 2011 and Shuwa, 2011). The percentage of Na$_2$O is slightly higher in the raw samples than in the pre-treated samples, this is because Na$_2$O that exists in form of Na$_2$CO$_3$ are generally soluble in water, that is why they were leached out and lost during beneficiation process but after activation with Na$_2$CO$_3$ some percentage were recovered and found out to be close to standard (2.56%) as shown in Table 1.

The magnesium content (MgO) is approximately close to standard 2.52%. Presence of excess MgO beyond the standard value may affect the rheological properties of the formulated drilling fluid.

### Table 1: XRF Analysis Results

<table>
<thead>
<tr>
<th>Elements</th>
<th>API grade sample</th>
<th>Raw GHF</th>
<th>Na$_2$CO$_3$ Activated GHF</th>
</tr>
</thead>
<tbody>
<tr>
<td>Na$_2$O</td>
<td>2.56</td>
<td>2.71</td>
<td>2.44</td>
</tr>
<tr>
<td>MgO</td>
<td>2.52</td>
<td>3.48</td>
<td>1.99</td>
</tr>
<tr>
<td>Al$_2$O$_3$</td>
<td>16.56</td>
<td>15.09</td>
<td>15.10</td>
</tr>
<tr>
<td>SiO$_2$</td>
<td>43.51</td>
<td>48.18</td>
<td>47.06</td>
</tr>
<tr>
<td>P$_2$O$_5$</td>
<td>0.96</td>
<td>1.20</td>
<td>1.11</td>
</tr>
<tr>
<td>K$_2$O</td>
<td>0.79</td>
<td>2.28</td>
<td>2.19</td>
</tr>
<tr>
<td>CaO</td>
<td>5.65</td>
<td>2.93</td>
<td>0.97</td>
</tr>
<tr>
<td>TiO$_2$</td>
<td>1.11</td>
<td>0.85</td>
<td>0.86</td>
</tr>
<tr>
<td>Fe$_2$O$_3$</td>
<td>5.71</td>
<td>4.53</td>
<td>4.45</td>
</tr>
<tr>
<td>Others</td>
<td>18.08</td>
<td>16.03</td>
<td>23.83</td>
</tr>
<tr>
<td>Al$_2$O$_3$/SiO$_2$</td>
<td>0.38</td>
<td>0.31</td>
<td>0.32</td>
</tr>
</tbody>
</table>

Presence of K$_2$O is an indication that the Bentonitic clay contains K-Feldspar which can also be associated with Kaolinite mineral. The API grade sample contains only 0.79 % K$_2$O, while in the local Bentonite clay the percentage is 2.28 % in the raw sample and 2.19 % in the beneficiated/activated samples respectively. Presence of much potassium may have effect on the viscosity as well as the rheological properties of the drilling fluid. CaO originated from CaCO$_3$. Its presence in the Bentonite affects the viscosity, swollen power as well as the rheological property of the drilling fluid. The percentage of CaO in the standard (Wyoming) clay is 5.64% in which raw GHF Bentonite is even better with less amount of calcium content 2.93% which changed to 0.97% after beneficiation. The percentage of Fe$_2$O$_3$ in the locally sourced Bentonite clay and activated bentonite are within the normal range (4.45 – 4.53 %) when compared with the standard bentonite having (5.71%) Fe$_2$O$_3$.

### Formulation Results

The drilling fluid formulations were carried out in three different ways as follows:

1. Formulation for raw bentonite samples.
2. Formulation after beneficiation and activation of Bentonitic clay with (12%) Na$_2$CO$_3$
3. Formulation with standard additive (PAC) for instant and aged (stayed for 24 hours) formulations.

The results were compared with the standard API grade (Wyoming) for each formulation until the desired formulation was achieved. The viscosity consistency curves and rheological properties for instant formulations are presented in Figures 6 and 7 respectively.
The viscosity consistency graph (Figure 6) showed that the raw GHF bentonite has less viscosity compared to the viscosity of API standard, but the beneficiated and activated sample showed slight increase in viscosity which is also not up to standard. This also affected the rheological properties of the bentonite as indicated in Figure 7.

![Viscosity consistency curve for instant drilling fluid formulations from GHF Bentonite](image)

**Figure 6:** Viscosity consistency curve for instant drilling fluid formulations from GHF Bentonite

The computed rheological properties (Figure 7) indicated that beneficiated and Na$_2$CO$_3$ activated GHF bentonite produced a better result compared with formulation from the raw sample. This can be attributed to the ability of the clay particles to undergo high cation exchange, in which Na ion displaces Ca ion to form Na activated bentonite with high swollen power.

![Rheological Properties for instant drilling fluid formulations from GHF Bentonite](image)

**Figure 7:** Rheological Properties for instant drilling fluid formulations from GHF Bentonite

The instant drilling fluid formulation using PAC as an additive was done with the aim of determining the optimum amount of viscosifier required to produce the best formulation for instant application during drilling of an oil/gas well. The amount of the additive used ranges from 0.5 to 2.5g of PAC. The viscosity consistency graph in Figures 6 has shown that at 2.0 g of PAC the viscosity of the GHF Bentonite is slightly lower than the standard. The API standard recommends that the maximum reading at 600rpm should be ≤ 30. Therefore, the optimum value was concluded to be at 2.0 g of PAC for the instant formulations. When the sample was allowed to age for 24 hours, outrageous (as high as 53.50 cP) value was observed, that means allowing the sample to stay overnight increases the hydration rate as well as the viscosity of the formulated fluid. Therefore, there was need to further investigate the effect of the viscosifier with aging and the result is depicted in Figure 8.

![Rheological Properties](image)
The rheological properties of instant formulated drilling fluids showed significant increment as the concentration of the viscosifier (PAC) increases from 0.5 to 2.5g. The optimum value for the sample was observed at 2.0 g of PAC. The YP, PV and AP were relatively equal to API standard as shown in Figure 7.

Figure 8: Viscosity consistency curve for aged drilling fluid formulations from GHF Bentonite

The rheological properties for the aged formulated drilling fluids (Figure 9) showed increment as the concentration of the PAC increases from 0.2 to 1.4g. The optimum value was found to be at 0.8g for GHF Bentonite. The YP at 0.8g of PAC was 6 (lb/100ft²) which correspond to the standard value (6 lb/100ft²) for API grade, as the concentration of the PAC was increased, the YP also increased. In practical terms, an increasing YP means a good and stable drilling mud which helps in removing cuttings from the hole, while a reducing YP means a bad and unstable mud which results in loosing viscosity, not removing cuttings, experience sagging. These problems may lead to stuck pipe and or loss circulation (Adeleye, 2012).

CONCLUSION

The FTIR results confirmed that GHF) bentonitic clay is a Montmorillonite of the smectite group. The SEM microstructure images for the Na₂CO₃ activated sample/treated samples were more dispersed than the raw sample with some large flocs structure confirming their Montmorillonitic nature. The XRF result indicated 1% reduction of
free silica (Quartz) after beneficiation. The optimum amount of PAC for instant formulated samples was found to be 2.0 g PAC when compared with API Grade, while for the aged formulated samples the optimum was achieved at 0.8 g PAC. This proved that the local GHF bentonite if properly beneficiated and activated and treated with Na₂CO₃ and PAC can be used for oil and gas as well as water borehole drilling application.

ACKNOWLEDGEMENT
We wish to thank members of Bentonite Research Team, Centre for Energy Research and Training, Ahmadu Bello University, Zaria, for providing the local GHF Bentonite samples for this research.

AUTHORS’ CONTRIBUTIONS
The first, third and fourth authors actively participated in conducting the research, while the second author supervised the research.

CONFLICT OF INTEREST
There is no any sort of conflict of interest among the authors.

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