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BENEFICIATION AND EVALUATION OF THE POTENTIALS OF LOCAL (DIKWA) BENTONITIC CLAY FOR OIL WELL DRILLING FLUID FORMULATION

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ABSTRACT
Nigeria's economy depends on oil and one of the important activities in oil production is drilling in the presence of drilling fluid. A major component of drilling fluid that determines the success of a drilling operation is bentonitic clay. In this study, a local Bentonitic clay from Dikwa formation was procured and characterized by; x-ray diffraction, x-ray fluorescence, cation-exchange capacity and particle size distribution. The clay was beneficiated (wet and dry) using Na_2CO_3 (concentration ranges from 2-14%wt) as activating agent, and sample drilling muds (fluids) were formulated. Drilling fluids formulated from the beneficiated clay without additive addition did not possess the minimum rheological properties required for use in oil and gas drilling operations. However, an improvement in rheological and filtration properties was observed when treated with carboxy-methyl-cellulose (CMC) as an additive. The optimum values for plastic viscosity, gel strength and fluid loss (8.1cP, 9.4lb/100ft2 and 14mL, respectively) were obtained at 6%wt Na_2CO_3 concentration. The fluid loss of muds formulated from the local clay was improved by about 50% when treated with (CMC) and meets the API requirements. Results obtained after detailed experimental investigations showed that modified Dikwa (Kaza) bentonitic clay has great potentials to be used as a constituent of oil and gas drilling fluid.

Keywords: Drilling fluid, Bentonitic clay, Dikwa formation, plastic viscosity, fluid loss

INTRODUCTION
The importance of clay and clay minerals in drilling industry is evident from the fact that clays are added to drilling fluids to build viscosity, thixotropy, and contribute wall building properties. The most commonly used clay in drilling fluid formulation is Bentonite. Bentonite having sodium cation as either the dominant or as an abundant exchangeable ion typically has very high swelling capacities and forms a gel-like mass when added to water and is required in the formulation of drilling fluid. Bentonite in which exchangeable calcium cation is more abundant than other ions has much lower swelling capacities than sodium varieties (RMRDC, 2010). The calcium-based bentonitic clays are of inferior quality and need to be converted to the sodium-based to be used in drilling application. Most of the marine shale found in Nigeria are enriched in calcium and mixed bentonite (RMRDC, 2010).The purpose of this research is to improve the calcium based Kaza (Dikwa formation)bentonitic clay through chemical activation and investigate its rheological and filtration properties as a constituent of oil well drilling fluids.

Many researchers have investigated clays from various deposits across the nation in order to provide alternative to the imported Wyoming bentonite. Folade *et al*, 2007 evaluated Pindiga clay and improved its rheological properties obtaining a high plastic viscosity. (Okogbue, 2008) examined bentonitic clays from the South-eastern part of the nation and established their potential for use in drilling mud formulation. Yola bentonitic clay was also investigated. The results obtained revealed that the Yola clay was Ca-based montmorillonite (James *et al*. 2008). Treatment with a chemical additive improved its rheological properties but fell below the required standard for use in drilling mud formulation. Adeleye *et al.* (2009) treated locally sourced betonitic clay from the North-west of the country with indigenous gum Arabic and sodium carbonate and analysed its rheological properties using factorial design. The work gave an express method of determining the required treatment of the local clay. Ubakala clay from Abia State was evaluated for its potential as drilling fluid component. Its beneficiation with Na_2CO_3 and improvement with CMC enhanced its plastic viscosity by up to about 1207% (Apugo-Nwosu *et al.* 2011). Extensive works were carried out on clays from Pindiga and Fika formations in the North-eastern part of the country (Dewu *et al*. 2011a; Dewu *et al*. 2011b; Arabi *et al*. 2011; Dewu *et al.* 2012). Local raw materials were employed to modify the pH of the mud by Okorie (2009).Water and synthetic-based drilling muds were formulated using raw materials. The low value-pH muds were improved to the required standard of between 9.5 and 12.5 (Ajugwe *et al*. 2012; Udoh &Itah, 2012). Unprocessed Ota (Ogun State) Kaolin was employed as a weighting additive in drilling fluid formulation (Adebayo &Ajayi, 2011). Recently, Bilal used many analytical techniques to characterize and evaluate performance of some local bentonitic clays from Benue Trough (Bilal, 2015; Bilal *et al*. 2016; Bilal *et al*. 2017). No work in the open literature was conducted on clays from formations in Borno basin. Moreover, almost all the works on the use of the local clays in drilling application did not test their filtration properties. In this work, an inferior, locally available bentonitic clay (from Dikwa formation in Borno basin) was recovered, characterized, modified and tested in terms of rheological & filtration properties for use in oil and gas drilling fluid application.

MATERIALS AND METHODS

Sample Collection and Preparation

The raw clay sample was collected from a pit dug to a depth of about 3ft at Kaza village of Dikwa province of Borno State, Nigeria. About 15kg of the sample was crushed to finer particles and sun dried for three days to ease pulverising and sieving. The sample was then ground to powder with the aid of a jaw crusher and ball mill machine. It was then shaken with test sieve shaker to obtain 63µm particle size to suit API specifications for bentonite.

Sample Characterization

Determination of Chemical and Mineralogical Composition

Chemical and mineralogical compositions of the raw Warsale clay sample was determined using X-ray fluorescence, XRF (model Pan analytic B.V PW4030/45B) and X-ray Diffractometer (model Schmadzu 6000) respectively. Wyoming bentonitic clay was used as the standard (control) hence it was equally characterized for the chemical and mineralogical composition. The hydrometer method was used for *particle size distribution analysis* of the clay samples whereas, the Bentonite Laboratory Manual of the British Geological survey agency (Inglethorpe, 1993) was used for cation exchange capacity (*CEC)* determination.

Chemical Beneficiation (Activation)

Chemical activation was carried out on the 63µm fraction obtained above and the predominant calcium bentonite was converted to sodium bentonite through ion exchange using sodium carbonate as the activating agent (Arabi *et al*, 2011). Two modes (wet and dry) of beneficiation were applied for comparison purpose. In the wet technology, 2 to 14%wt. of Na_2CO_3 were added to the bentonite powder suspension in water at intervals of 2%. The suspension was stirred vigorously and allowed to stand for 2hours in order to ensure that proper ion exchange had taken place. The beneficiated samples were oven dried to reduce moisture content and ground to powder using ball mill. The ground sample was sieved again to obtain 63µm fraction. The dry technology was carried out by dry blending the sodium carbonate with the raw bentonitic clay sample powder using the above concentrations. It is usually considered that ion exchange will take place during clay hydration in water after formulation of the mud.

Drilling Mud Formulation

In order to formulate the sample mud, 24.5 g of the beneficiated bentonitic clay at the various concentration of the sodium carbonate was weighed with the aid of an electric weighing balance. The samples were poured into the mixer cups containing 350 ml of water, thoroughly mixed and then agitated vigorously with the aid of Hamilton Beach Mixer for 10 minutes to obtain a homogeneous mixture and improve hydration of the clay. In order to further improve the rheological and filtration properties of the formulated sample muds, 1g of carboxymethyl cellulose (CMC) was separately added to the mud samples with a view to ascertain their effectiveness on the mud samples. The mixture of clay, water and the additive was thoroughly mixed and the homogeneous mixture allowed to age for 24 hours. Filtration and rheological tests were carried out on the aged muds.

Drilling Mud Testing Procedures

Plastic Viscosity, Apparent Viscosity, Yield Point and Gel Strength Determination

Ofite 900 model viscometer was used for these measurements based on manufacturer's guidelines. The sample mud was poured in to the sample cell and the rotor sleeve of the viscometer was immersed in the mud exactly to the scribed line. The mud was allowed to stabilize for ten seconds before the dial reading at 600rpm (θ_{600}) was taken and recorded from the screen. After additional ten minutes and ten seconds, the instrument displayed plastic viscosity (PV), yield point (YP) and gel strength (GS). All the values were recorded. The plastic viscosity is in cP, the gel strength and yield point in pounds per hundred feet square (lb/100ft^2).

Fluid Loss Determination

Low temperature API filter press was used for fluid loss or water loss measurement as recommended by the manufacturer. The sample mud was poured in to the API filter press cell (not more than ¾ cup). The filter cell was then placed in the frame. The top cap was held tight with the screw and a graduated cylinder placed under the drain tube to collect the filtrate. Having ensured that the unit was tight, 100psi (690kpa) pressure from the mini carbon dioxide cartridge was applied to the filter cell through a regulator. A stop watch was set for 30 minutes. The test period begins at the time of pressure application. The volume of the filtrate or fluid loss was then read off from the graduated cylinder and recorded in millilitres (mL).

pH Determination

pH paper test strips was used for pH determination. A 1in. (25mm) of indicator paper was placed on the surface of the mud and allowed to remain until the liquid had wetted the surface of the paper and the colour stabilized (for about 30 seconds). The colour of the upper side of the paper (which had not been in contact with the mud solids) was compared with the colour standards provided with the test strips and the mud pH was estimated. The mud pH was reported to the nearest 0.5.

RESULTS AND DISCUSSION

Bentonitic Clay Samples Characterization

Chemical Analysis

Table 1 presents the chemical composition of the raw Kaza bentonitic clay and Wyoming bentonite as obtained using an XRF machine. It can be observed from the table that the

Al_2O_3/SiO_2 was approximately 1/3 in Wyoming bentonite as expected of smectites, which is the main component of bentonites. This ratio was higher in Kaza bentonitic clay. The obtained value is in agreement with the results reported by Falode *et al* (2007), Deer *et al* (1992) and Kirk-Othmer (1980). It can also be noticed that the Wyoming bentonite has higher percentage of Na_2O (3.22%) than the Warsale bentonitic clay (0.11%) which is an indication that the Wyoming bentonite consist of sodium montmorillonite. Kaza bentonitic clay however has higher percentage of CaO (2.05%) when compared with Na_2O present (0.11%) which indicates the presence of calcium montmorillonite as expected of Nigerian bentonitic clays (Falode *et al.* 2007). The Wyoming bentonite also shows high percentage of BaO, an indication of the presence of Barite ($BaSO_4$), a non-clay mineral.

Table 1: Chemical Composition of the Bentonitic Clay Samples

Chemical compound	**Wyoming (%)**	**Kaza (%)**
Al_2O_3	14.2	20.10
SiO_2	43.6	48.50
Na_2O	3.22	0.11
K_2O	0.93	1.75
CaO	7.05	2.05
TiO_2	1.30	2.14
MnO	0.11	0.10
Fe_2O_3	14.50	17.59
NiO	0.02	0.02
CuO	0.14	0.06
MgO	2.40	1.26
BaO	11.0	ND
PbO	0.06	ND
RuO	0.56	ND
ZnO	ND	0.03
SrO	ND	0.05
ZrO_2	ND	0.11
Cr_2O_3	ND	0.07
Others	ND	20.10
TOTAL	100.00	100.00

ND –Not Detected

Mineralogical Analysis

X-ray diffraction (XRD) analysis was carried out on the clay samples in order to ascertain their mineralogical composition. The Wyoming bentonite sample composed mainly of smectite (montmorillonite), sanderite, barite, morimotoinite and muscovite (Table 2). Other minerals detected in trace amounts are zinc arsenate, behierite and ammonium chlorate. The smectite dominates the Wyoming bentonite (about 50%) as expected. The smectite clay mineral is responsible for the swelling and high rheological and filtration properties of the drilling fluids. The Wyoming bentonite also shows highamount of barite (about 20%), a non-clay mineral used in drilling fluid as a weighting agent to increase the density of the drilling fluids. This could indicate that the Wyoming bentonite might have been treated with barite. It was also observed that the local bentonitic clay sample composed mainly of smectite, quartz, kaolinites, gismondine and serpentine

Table 2: Summary of Mineral Composition of the Clay Samples

*Wyoming*Bentonite		*Kaza* Clay	
Mineral	**Composition (%)**	**Mineral**	**Composition (%)**
Smectite	50	Smectite	26.67
Barite	19.4	Quartz	7.39
Sanderite	10	Kaolinite	4.04
Morimotoinite	7.9	Gismondine	41.50
Muscovite	6.9	Serpentine	5.40
Others	5.8	Others	15

Particle Size Distribution

Basic particle size distribution analysis was conducted in order to ascertain the percentage of particle size that constitute the clay sample (sand, silt and clay). Table 3 presents the particle size distribution of the Wyoming and Kaza bentonitic clay samples. From the table it can be observed that, the percentage composition of clay, silt and sand in the Wyoming and Kazasamples are 47, 33 and 20 and 63, 22 and 15 respectively.

Table 3: Particle Size Distribution of the Clay Samples

Sample	**40 seconds reading**	**2hours reading**	**% Clay** (<0.002mm)	**% Silt** (0.002-0.05mm)	**% Sand** (0.05-2mm)
Kaza	42	31	63	22	15
Wyoming	33	23	47	33	20

The percentage clay of the Wyoming sample is less when compared with the local clay samples, this may not be unconnected with the presence of high amounts of non-clay minerals like barite in the composition of the Wyoming clay as shown by the result of the mineralogical analysis.

Cation Exchange Capacity

Table 4 shows the CEC values of the investigated samples. The CEC of the Wyoming bentonite was found to be 86meq/100g which falls within the general range of (80-100) meq/100g (Table 4). This agrees with the values reported by Falode *et al* (2007) and, Irawan and Samsuri (2008). It can be seen from the table that, the untreated local bentonitic clay sample has low CEC, but when treated with 6% Na_2CO_3 about 50% increase in CECs was observed. This is still much less than the CEC value of Wyoming bentonite. The CEC of clay and the species of cations in the exchange positions are a good indication of the colloidal activity of the clay. Clay such as smectite that has high CEC swells greatly and forms viscous suspensions at low concentrations of clay particularly when sodium is in the exchange positions.

Table 4: Cation Exchange Capacities of the Clay Samples

Parameter	**Untreated Kaza clay**	**Treated Kaza with 6% Na_2CO_3**	**Wyoming bentonite**
CEC, meq/100g	30	46	86

Rheological Properties of the Sample Muds

Effect of Na_2CO_3Concentration on Plastic Viscosity

The commonly used direct indicating viscometer was specifically designed to facilitate the use of the Bingham plastic model. The PV is the slope of the Bingham plastic line. Plastic viscosity is used as an indicator of the size, shape, distribution and quantity of solids, and the viscosity of the liquid phase. The effect of Na_2CO_3 indicates an upward trend in plastic viscosity as the concentration of Na_2CO_3 present in the mud increased from 2% to 8%. The highest plastic viscosity was observed at 6% Na_2CO_3 for the sample mud treated with 1g CMC. Figures 1 & 3 show this trend. This shows that beneficiation improved the rheological properties of calcium based clays as demonstrated by Falode *et al* (2007) and Song *et al* (2005).The PV depends largely on the bulk volume of solids in the mud and on the viscosity of suspending liquid. The Na_2CO_3 converts the calcium bentonite to sodium bentonite and hence causes dispersion of clay. The dispersion is commonly used to describe subdivision of particle aggregates in a suspension and subdivision of clay platelet stacks as a result of electrochemical effect. The dispersion increased the PV of the sample muds until an equilibrium is reached where the PV is at its maximum value i.e. at optimum concentration of Na_2CO_3.

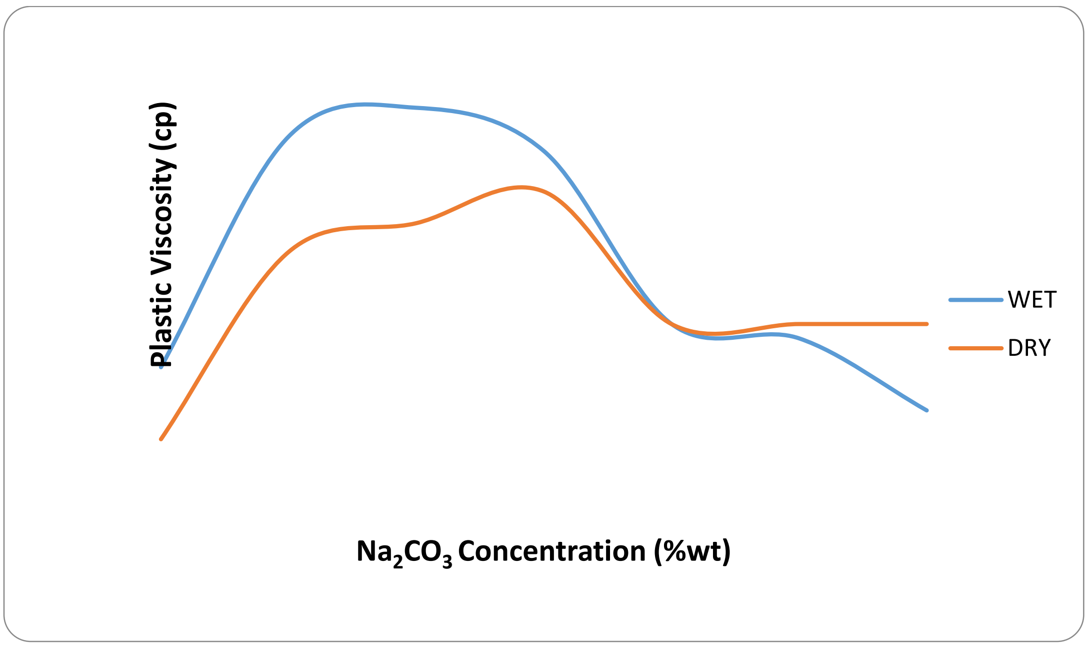

Fig. 1: Variation of Na_2CO_3 Concentration with Plastic Viscosity

for Kaza Muds without Treatment

The wet method also, proved more effective than the dry technology in terms of PV improvement.

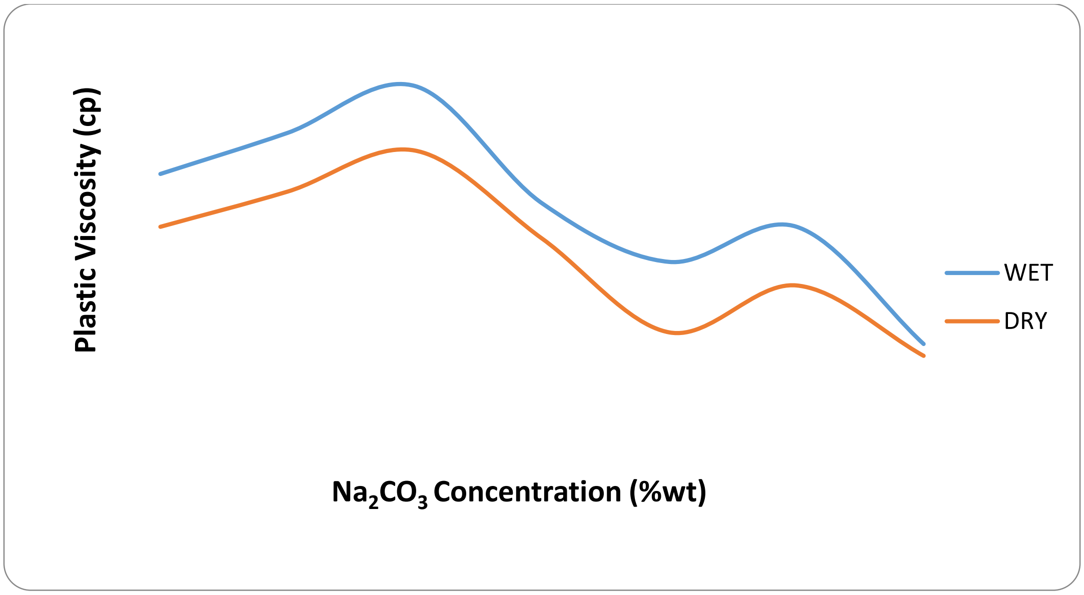

Fig. 2: Variation of Na_2CO_3 Concentration with Plastic Viscosity
for Kaza Muds Treated with 1g of CMC

Effect of Na_2CO_3 Concentration on Gel Strength

Gel strengths indicate the gelation or thixotropic properties of a drilling fluid and are the measurements of attractive forces under static conditions in relationship to time. Yield point, conversely, is a dynamic property (Darley and Gray, 1988). However, gel strength and yield point have a proportional relationship. Increase in one may results in increase in the other and vice versa. Figures 3 and 4 represent variation of Na_2CO_3 Concentration with gel strength of the muds treated with CMC and untreated muds for the sample clay. An increase in gel strength was observed when the Na_2CO_3 concentration in the mud increased from 4% to 8% for the sample muds without treatment. Further reduction was observed from the 8% to 14% Na_2CO_3 concentration. The same trend was observed for muds treated with 1g CMC. Gel strength and yield point depend on the presence of colloidal clays, and contamination by inorganic salts.

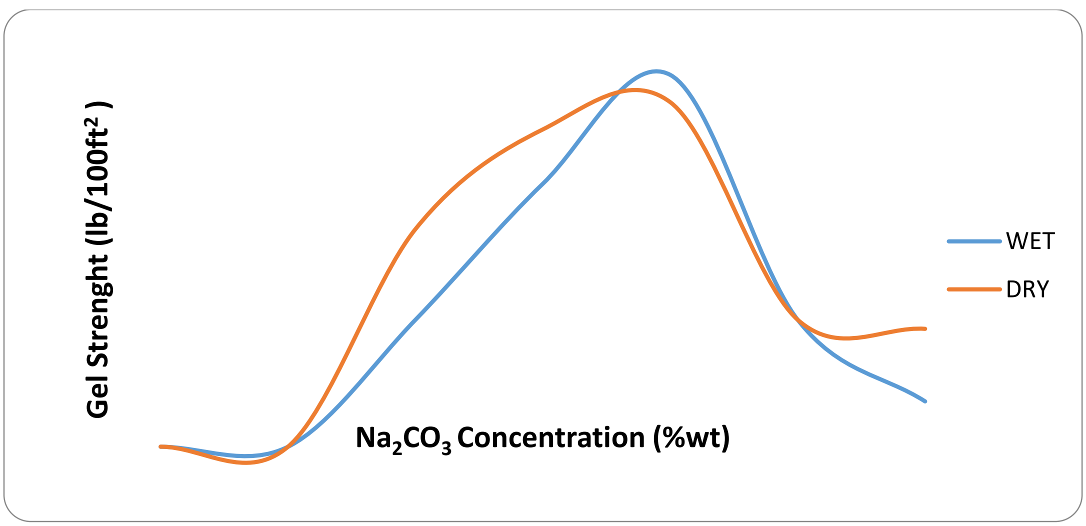

Fig. 3: Variation of Na_2CO_3 Concentration with Gel Strength for Kaza Muds without Treatment

Gel strength is due to flocculation and aggregation of clay platelets. Flocculation is limited to lose association of clay platelets which forms flocs or gel structures, while aggregation refers to the collapse of the diffuse double layers and the formation of aggregates of parallel platelets. Where flocculation causes an increase in gel strength, aggregation causes a decrease because it reduces the number of units available to build gel structures and the surface area available for particle interaction.

When the Na_2CO_3 concentration was increased from 2% to 10% gel strength continues to rise because of flocculation, but the particles reach equilibrium positions slowly (at 10%). Evidently, the attractive and repulsive forces are nearly in balance. Further increase in Na_2CO_3 concentration led to decrease in gel strength which can be attributed to the formation of aggregates of parallel platelets as explained by Darley and Gray (1988).

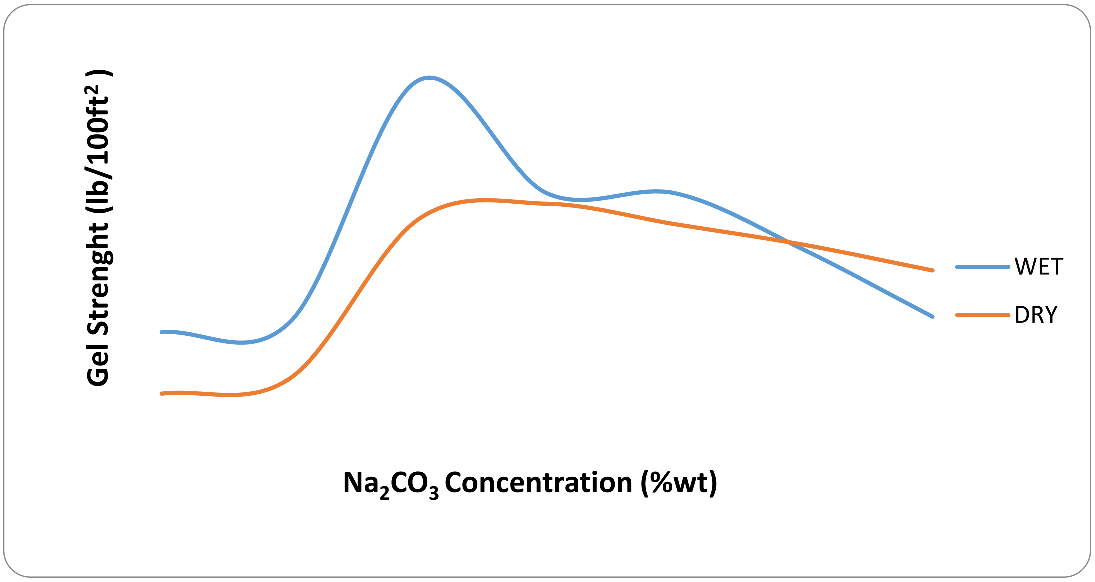

Fig. 4: Variation of Na_2CO_3 Concentration with Gel Strength for Kaza Muds Treated with 1g of CMC

Filtration Properties of the Sample Muds

The ability of the mud to seal permeable formations exposed by the bit with a thin, low permeability filter cake is another major requirement for successful completion of the hole. Because the pressure of the mud column must be greater than the formation pore pressure in order to prevent the inflow of formation fluids, the mud will continuously invade permeable formations if a filter cake is not formed.

Effect of Na_2CO_3Concentration on Fluid Loss

The fluid loss properties of a mud indicate how well the mud forms a seal against permeable formations. Figure 5 illustrates the fluid loss behaviour of the sample muds at 6%, 8% and 10% Na_2CO_3 concentration. The muds without treatment displayed poor filtration properties, but when treated with CMC a dramatic improvement in fluid loss was observed.50% reduction in fluid loss was observed when treated with 1g CMC for all the sample muds tested. The reduction was more pronounced at lower Na_2CO_3 concentration. The CMC being a synthetic polymer does the work by using its polymer chain to bind the flocculated particles together in the presence of salt concentration. This indicates that the CMC is a good filtration control agent.

The result obtained corroborates the work of Falode *et al* (2007) who improved on the filtration properties of Pindiga clay mud using starch. Filtration properties must be controlled in order to prevent thick filter cakes from excessively reducing the gauge of the borehole. Ideally the mud should build up a thin, tough, and impermeable cake fairly quickly.

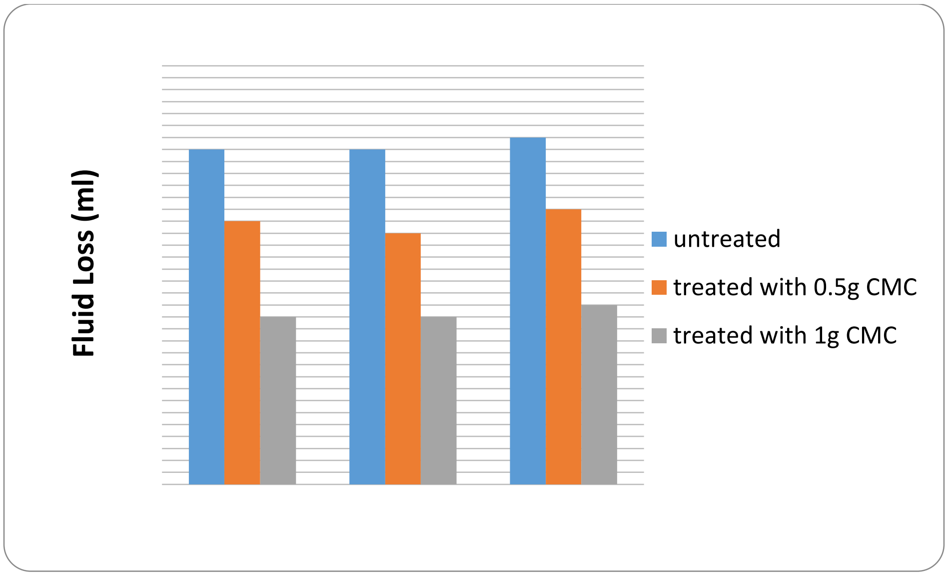

Fig. 5: Filtration Properties of Kaza Sample Muds treated with CMC

Effect of Na_2CO_3 Concentration on pH

Increase in Na_2CO_3 concentration led to increase in pH due to the presence of high concentration of sodium carbonate, Figure 6 illustrates this behaviour. The pH which can be defined as the negative logarithm of the hydrogen-ion concentration expresses the relative acidity or alkalinity of the suspension. The pH of the sample muds obtained fall within the API specification (8.5–12.5) as can be seen in the figure. See Table 5 for API specifications for drilling fluids.The pH is important because the optimum control of some mud systems is based on it, e.g. the detection and treatment of certain contaminants. Higher pH mud systems can be reduced by the use or treatment with chemicals like acidic polyphosphate. Conversely, the pH of some muds can be raised with NaOH.

Table 5: API Standards for Drilling Fluids

Parameter	**Specification**
Dial Reading at 600rpm	30cp minimum
Plastic viscosity	8cp-10cp
Yield Point	3* PV maximum
Fluid Loss	15ml/30 minutes
Sand Content	(1-2%) maximum
pH Level	8.5-12.5
Moisture Content	10% maximum
YP/PV	3

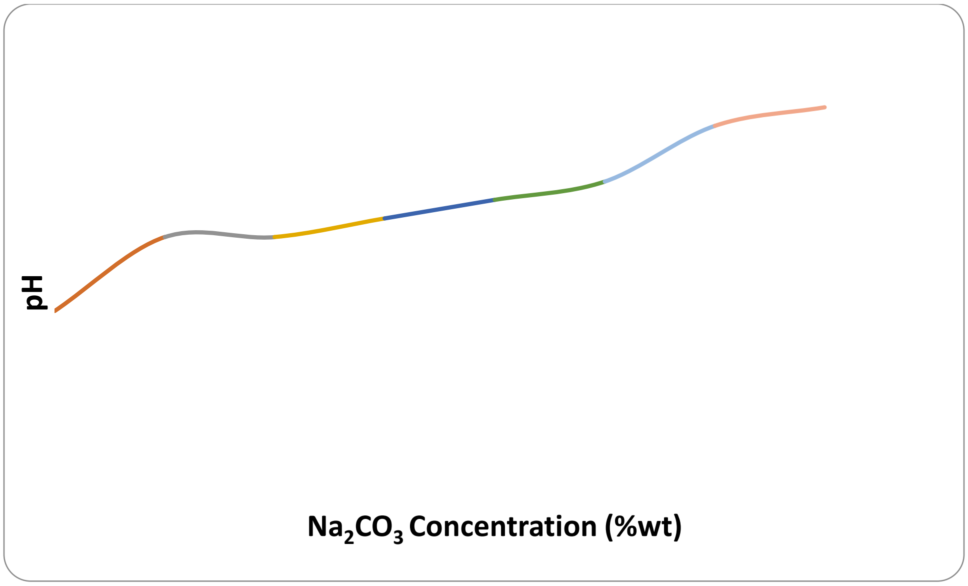

Fig. 6: Variation of Na_2CO_3 concentration with pH for Kaza Sample Muds without Treatment

CONCLUSIONS

After a detailed characterization and performance evaluation of the Kaza (Dikwa formation) local bentonitic clay, the following conclusions can be made from this work.

1. The sample muds formulated with the beneficiated local clay showed little improvement on flow properties and falls short of API standard. However, improvements in rheological and filtration properties were observed when treated with CMC.
2. The optimum values for gel strength (9.4 lb/100ft^2) and plastic viscosity (8.1 cP) were obtained at 6% Na_2CO_3 concentration. In all cases the wet method of beneficiation gave a better results than the dry technology method.
3. Treatment of the sample muds with sodium carboxymathyl cellulose (CMC) improved both rheological and filtration properties as the plastic viscosity and fluid loss mate API standard, and therefore confirmed that CMC is a good rheological and filtration control agent.

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