



Beneficiation of Nigerian bentonite using local materials

Kevin C. Igwilo¹ · N. Uwaezuoke¹ · N. Okoli¹ · Franklin T. Obasi¹ · Emeka E. Okoro²

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Abstract

In previous studies, it has been found that the Nigerian bentonite is deficient in terms of its fluid loss and rheological properties which includes yield point. Also, due to its high calcium content and low sodium content as opposed to foreign bentonite, it does not meet the API standard of drilling. This research was carried out to beneficiate the Nigerian bentonite as regards its fluid loss properties, rheological properties and wellbore stability. Snail shell is seen as waste substance in Nigeria, and *Mucuna solan* on the other hand is a local major food supplement in Nigeria and is found in large quantities. The elemental and oxide compositions of the snail shell and *Mucuna solan* were determined through scanning electron microscope and X-ray diffraction measurements, respectively. The additives were used to beneficiate the Nigerian bentonite, and the test result proved that at considerable concentrations of the additives, the Nigerian bentonite was able to compete with foreign bentonite and also met API specifications. Both additives contain high concentrations of nanoparticles and inhibitive calcium and potassium for wellbore stability. Beneficiated Nigerian bentonite also proved to be viable economically when compared with foreign bentonite.

Keywords Beneficiation · Economic analysis · Fluid loss · Foreign bentonite · *Mucuna solan* · Nigeria bentonite · Rheological properties · Snail shell

Abbreviations

FB	Foreign bentonite
NB	Nigerian bentonite
SS	Snail shell
MS	<i>Mucuna solan</i>
YP/PV	Yield point-to-plastic viscosity ratio

Introduction

Nigeria as of the year 2016 had about thirty-seven (37) proven oil reserves according to Worldometers. These reserves can only be exploited if they are explored and drilled. For drilling operations to be successful, the use of drilling fluids must be employed which is formulated from combination of clay (mostly bentonite) and other materials to aid drilling. Though Nigeria has a substantial amount of clay deposits, it is not fully used during drilling operations

in the country due to its inability to meet API standards for drilling fluid.

Since the success of drilling operations depends on the proper selection of drilling fluid system (Bourgoyne et al. 1991), some of the bentonites used in mud formulation in Nigeria are imported to avoid catastrophe related to using substandard bentonite. According to previous research, beneficiation of Nigerian bentonite from its predominant calcium-based composition to sodium-based clay can go a long way in improving its properties. Bentonite is the main substance which provides fluid loss control in the drilling fluid as it aids filter cake formation. Other fluid loss control agents are additives that are added to drilling mud during formation to reduce the loss of fluid from the mud into the drilled formation. These additives help to minimize formation damage, maintain hole integrity, reduce log analysis problems, protect water sensitive shale, reduce fluid loss to protective formation and reduce hole washout to achieve better casing and cementing jobs (Chilingarian and Vorabutr 1983; Elward-Berry and Darby 1997).

Types of bentonites include calcium bentonite, sodium bentonite and potassium bentonite. Sodium bentonite expands when wet, absorbing as much as several times its dry mass in water. Because of its excellent colloidal

✉ N. Uwaezuoke
nnaemeka.uwaezuoke@futo.edu.ng

¹ Department of Petroleum Engineering, Federal University of Technology, P.M.B 1526, Owerri, Nigeria

² Covenant University, Ota, Nigeria

properties, it is often used in drilling mud for oil and gas wells and boreholes for geotechnical and environmental investigations. Calcium bentonite is a useful adsorbent of ions in solution, as well as fats and oils. It is the main active ingredient of fuller's earth, probably one of the earliest industrial cleaning agents. Calcium bentonite may be converted to sodium bentonite (termed sodium beneficiation or sodium activation) to exhibit many of sodium bentonites' properties by an ion exchange process. It is also known as potash bentonite or K-bentonite; potassium bentonite is potassium-rich illitic clay formed from alteration of volcanic ash.

In the local scene, Odumugbo (2005) asserts that the present consumption of bentonite in Nigeria is about 50,000 tons per year and all of it is imported from USA. As reported by Terra mines in Edo State, the bentonite reserves in Nigeria have been estimated to be about 4 billion tons (Emofurieta 2001). Also, according to estimates from the Nigerian Mining Corporation and the Raw Materials Research Development Council (RMRDC), deposits of local bentonite clays in Nigeria have been modestly projected to be above 700 million metric tons (Aigbedion and Iyayi 2007; Raw Materials Research and Development Council 2007; Omole et al. 2013). Nweke et al. (2015) also carried out mineralogical characterization of clay samples from the Abakaliki formation in the Niger Delta part of Nigeria. The minerals found were majorly montmorillonite and illite with traces of kaolinite. Furthermore, elemental analysis carried out showed high calcium and potassium oxides content. Olugbenga et al. 2013 and Osadebe et al. 2011 experimented on the clay found in Niger Delta and Okada, respectively, and found that it was predominantly composed of SiO_2 . But Nwosu et al. (2013) found that the bentonite clay in Udi, Enugu state composed mainly of sodium-based montmorillonite. Nevertheless, that reserve is not enough to meet the demands of the Nigerian oil industry.

Bindei (1987) asserted that drilling operations began in the mid-fifties and local clay and additives were used until it subsided in early 1960s when imported commercial bentonite was introduced.

Substances that are rich with sodium have been found to serve as beneficiation materials for fluid loss control and hence the experimentation of snail shell and *Mucuna solan*. Also, some of these additives contain some percentage of inhibitive ions and nanoparticles that are very effective for wellbore stability. This is supported by Tawfik and Mukaila (2019), which states that nanomaterials with range of nano-sizes have been detected to greatly hinder shale swelling due to their ability to plug micro-openings in shale by forming compact filter cake to minimize fluid loss and prevent downhole pressure transmission. Onize (2003) discovered improved clay that meet API specifications.

Research by Uwaezuoke et al. (2017b) has found out that *Mucuna solan* has the capability of beneficiating the rheological properties of drilling muds. *Mucuna solan* is a readily available vegetable plant in Nigeria and can also be exploited in the oil industry if maximized. Several people have tried beneficiating local Nigerian bentonite via conducting tests and introducing additives for upgrade. They include but not limited to James et al. (2008) who conducted X-ray fluorescence (XRF), X-ray diffraction (XRD) and rheological analysis on local bentonite from Adamawa state using Na_2CO_3 for upgrade. Also, Dewu et al. (2011) conducted XRF and XRD on local bentonite from Gombe State using soda ash and viscosifier for upgrade. Nmegbu (2014) conducted rheological analysis on local bentonite from Rivers State using Na_2CO_3 , NaOH, quick trol and carboxymethyl cellulose (CMC) for upgrade. Research studies carried out by (Udoh and Okon 2012; Falode et al. 2007) on bentonite clay from Uyo and Pindiga, respectively, showed that the fluid loss property of Nigerian bentonite clay is fair and in line with API requirements though other rheological properties like plastic viscosity and yield point were below API specifications.

The need to compare viscometric readings taken at surface temperatures to downhole circulation temperatures has been identified over six decades ago. A study on the effect of temperature on the flow properties of some water-based muds has been conducted. However, the effects of temperature on mud prepared with some local materials have already been published. Understandable trends were observed. Fluid loss increased with temperature, and yield point showed scattering (Uwaezuoke et al. 2017a; Igwilo et al. 2017).

In petroleum economic analyses, the Net Present Value is used to evaluate one alternative or two and more alternatives. For single alternative cases, once the Net Present Value at the Minimum Acceptable Rate of Return in an investment is greater than or equal to zero, the Minimum Acceptable Rate of Return is met or exceeded and the alternative is viable. If two or more alternatives are considered, the Net Present Value at the Minimum Acceptable Rate of Return is calculated and compared. The alternative with the numerically largest Net Present Value is selected. For all negative NPV's, the least negative is selected, while for all positive NPV's, the most positive is selected. For both negative and positive NPV's, the more positive alternative is selected. The Net Present Value tool can also apply in incremental analyses cases (Leland and Anthony 2002).

Materials and method

Equipment and raw materials

The equipment used in carrying out this work includes graduated measuring cylinder (25 mL, 250 mL), beakers,

adventurer pro-weighing balance, Hamilton beach mixer, sieve, low-pressure low-temperature filter press, stop watch, spatula, and pH tester. The raw materials used in the formulation of drilling fluid required for the experiment are foreign bentonite, local bentonite, snail shell and *Mucuna solannie*.

The local and foreign bentonites were bought from a licensed dealer, in a local store in Port Harcourt, Nigeria. The snail shell and *Mucuna solannie* powder were sourced from a market in Nigeria and needed to be processed in order to be used. The snail shell was obtained when the organism was removed. This made processing easier as it just needed to be washed to remove extraneous particles. It was washed with fresh water at ambient temperature of 28 ± 1.5 °C for about 45 min. It was then sun-dried at 31 ± 1 °C for 5 days to remove moisture. Crushing was done in a mill to powder form with hammer mill (Model RLA 201 – 800014, UK). The powder was grinded further with a Hamilton Beach dry grinder (Model 80385) and sieved with 200 mesh sieve for quality assurance and quality check (QA/QC). Similarly, a sample of *Mucuna solannie* powder was obtained in the same manner associated with the method used by Uwaezuke et al. (2017a, b) from a Nigerian market. The processed samples were packed in high-density polyethylene (HDPE, 0.77 mm thickness) bags and heat-sealed with a sealing machine. A relative humidity of 77% was reported. 2 ½ kg each was stored in a refrigerator ready for use when required, in their preserved unrefined forms.

Methodology

Snail shell preparation

The giant African snail shell samples were collected from Nigerian local dish restaurants and local market at Ihiagwa, Imo state. The samples were washed and dried thoroughly. The samples were ground into fine particles, sieved and re-ground as explained.

Mucuna solannie preparation

Also, as explained, the *Mucuna solannie* seeds were collected from the local market, washed and dried thoroughly, and then ground to fine particles and prepared for the experiment.

Measurements of elemental and oxide compositions of additives with scanning electron microscope (SEM) and X-ray diffraction (XRD)

The elemental and oxide composition of the additives (snail shell and *Mucuna solannie*) required to beneficiate the Nigerian bentonite was evaluated using SEM and XRD, respectively. This is very imperative to ascertain and

know precisely the elemental and oxide composition of the additives.

Rheological and fluid loss measurements

Rheological measurement

Fifteen grams of foreign bentonite only was measured using the weighing balance. 250 mL of water was stirred for 5 s and the bentonite was gradually transferred into the cup, and the stirring process was allowed for a period of 30 min. The sheared mud was then transferred into the viscometer cup to the line of meniscus. The viscometer was put on, and the knob was set at 600 rpm and the dial reading was taken when the pointer was steady. The knob was then reduced to 300 rpm, 200 rpm, 100 rpm, 6 rpm and 3 rpm, and the dial readings were taken appropriately and accordingly when the pointer was steady at a particular point. This same process was repeated for 30 g of foreign bentonite only, 15 g of local bentonite only, 15 g of local bentonite plus 1 g of snail shell plus 1 g of *Mucuna solannie*, 15 g of local bentonite plus 2 g of snail shell plus 2 g of *Mucuna solannie*, 15 g of local bentonite plus 3 g of snail shell plus 3 g of *Mucuna solannie*, 15 g of local bentonite plus 4 g of snail shell plus 4 g of *Mucuna solannie*, 15 g of local bentonite plus 5 g of snail shell plus 5 g of *Mucuna solannie*, 30 g of local bentonite only, respectively.

Fluid loss measurement

The same quantity and mixing procedures carried out in the case of rheological properties determination were done for API fluid loss. After the mud has been prepared, it was poured into the cell of the API filter press. The filter press was pressurized to a pressure of 100psi, and the 25 mL measuring cylinder was positioned to receive the filtrate. The filtrate was received and reported for 30 min.

Economic analysis

A hypothetical example was used for the economic analysis for the materials. Assume 12 ¼" vertical hole drilled to a depth of 8000 ft with 3000 bbls of mud [the amount of mud will take care of both the hole content (including excess factor) and the total losses].

To make a comparison between the foreign bentonite and local beneficiated bentonite, an incremental analysis was performed using the Net Present Value as a decision tool for cash flow duration of 10 years. The choice of NPV over DCF-ROR is because they are service-based only (negative cash flow) series.

Results and discussion

Snail shell and *Mucuna solan* compositions

Figures 1 and 2 show the snail shell and *Mucuna solan* SEM image representations, while Figs. 3 and 4 show the additives elemental counts that gave rise to Tables 1 and 2 of the elemental compositions of the two additives obtained using SEM. In the SEM image of snail shell sample, phase separations with massive plates are observed as a heterogeneous surface morphology. Table 3 shows the oxide composition obtained using XRD method. Snail shell contains 90.30% calcium, 65.52% calcium oxide, 8.01% aluminum oxide and 10.21% silicon dioxide in both its elemental and oxide composition. *Mucuna solan*

contains 39.18% potassium, 4.82% silicon dioxide and 1.83% aluminum oxide. The high content of calcium and potassium portrays a high degree of shale inhibition during drilling operations. Also, the presence of aluminum oxide and silicon dioxide is the example of nanoparticles well known for their wellbore stability effectiveness.

Beneficiation properties

In this section, the analysis was made with regard to API specifications for a good bentonite. The parameters analyzed as specified by API include the fluid loss, dial reading at 600 rpm and yield point-to-plastic viscosity ratio.

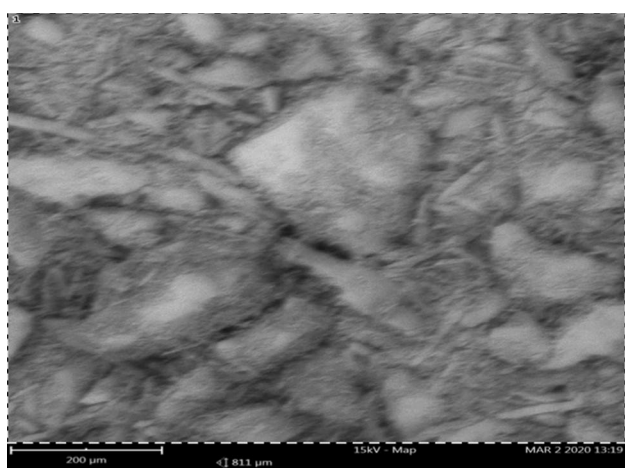


Fig. 1 Snail shell scanning electron microscope (SEM) image representation

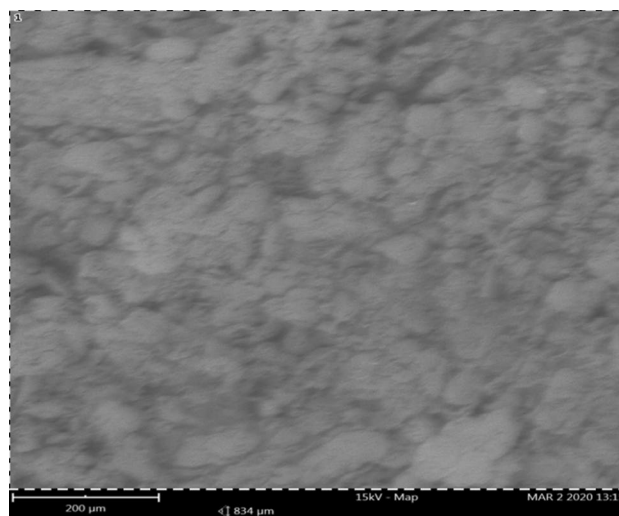


Fig. 2 *Mucuna solan* scanning electron microscope (SEM) image representation

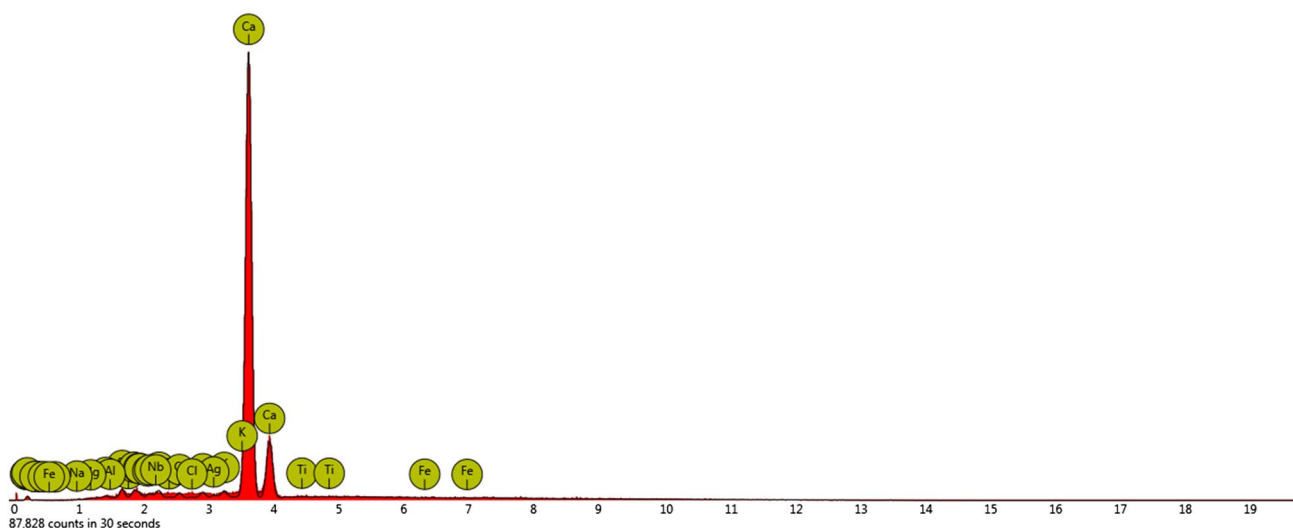


Fig. 3 Snail shell elemental count using scanning electron microscope (SEM)

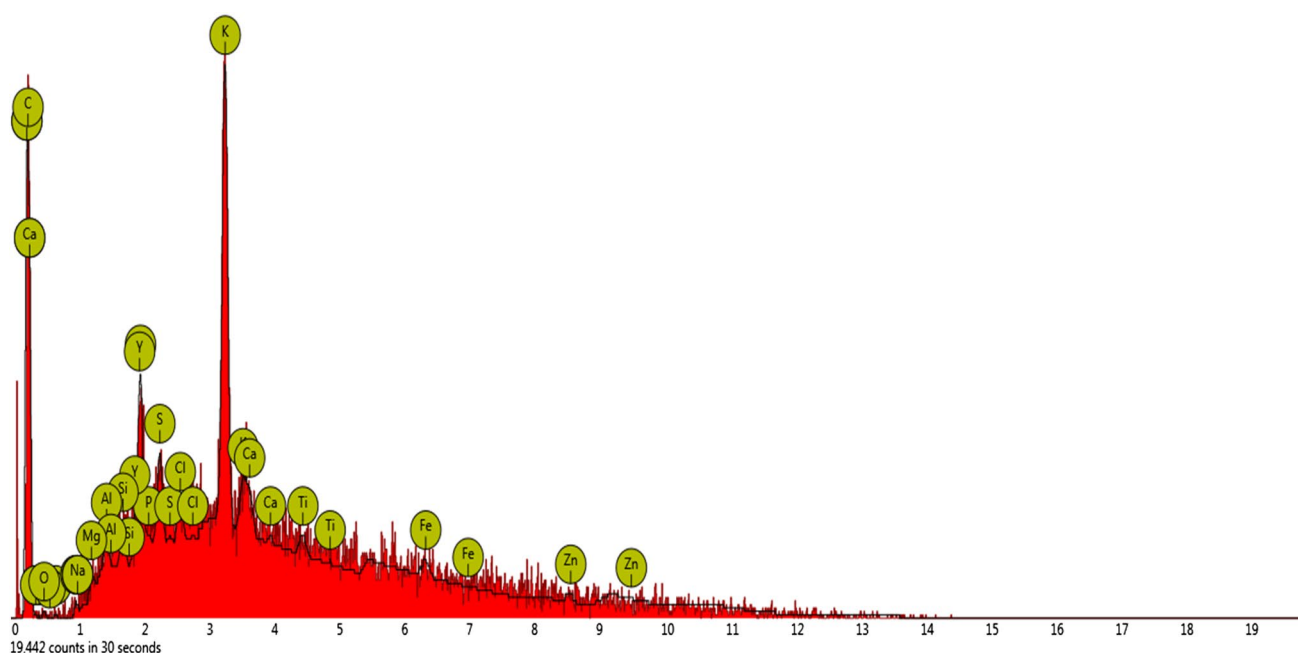


Fig. 4 *Mucuna solannia* elemental count using SEM

Table 1 Snail shell elemental composition

Element number	Element symbol	Element name	Atomic conc.	Weight conc.
20	Ca	Calcium	91.40	90.30
47	Ag	Silver	0.74	1.96
39	Y	Yttrium	0.83	1.82
41	Nb	Niobium	0.58	1.33
19	K	Potassium	1.09	1.05
14	Si	Silicon	1.20	0.83
16	S	Sulfur	0.95	0.75
17	Cl	Chlorine	0.77	0.67
8	O	Oxygen	0.73	0.29
15	P	Phosphorus	0.35	0.27
13	Al	Aluminum	0.35	0.23
6	C	Carbon	0.48	0.14
12	Mg	Magnesium	0.22	0.13
11	Na	Sodium	0.23	0.13
22	Ti	Titanium	0.08	0.10
26	Fe	Iron	0.00	0.00

It can be seen that snail shell constituted primarily of calcium (about 91%), but it also contained a very little amount of Sodium (about 0.23%) which is the basic constituent of foreign bentonite. Hence, it can be seen that the basic elemental supplement of snail shell is calcium. Also it can be seen that *Mucuna solannia* constituted primarily of Carbon (about 14.71%) and potassium (about 39.18%). It also contains sodium (about 1%) as its least constituent element. Hence, it can be seen that the major elemental supplements of *Mucuna*

Table 2 *Mucuna solannia* elemental composition using Scanning Electron Microscope (SEM)

Element number	Element symbol	Element name	Atomic conc.	Weight conc.
19	K	Potassium	28.86	39.18
6	C	Carbon	35.27	14.71
15	P	Phosphorus	7.34	7.89
30	Zn	Zinc	2.81	6.37
26	Fe	Iron	2.63	5.11
16	S	Sulfur	4.54	5.05
20	Ca	Calcium	3.27	4.55
39	Y	Yttrium	1.15	3.54
22	Ti	Titanium	1.84	3.06
17	Cl	Chlorine	2.19	2.70
8	O	Oxygen	3.74	2.08
14	Si	Silicon	2.00	1.95
13	Al	Aluminum	1.89	1.77
12	Mg	Magnesium	1.39	1.17
11	Na	Sodium	1.08	0.86

solannia to the beneficiation process were carbon and potassium. Also, the major oxide composition of *Mucuna solannia* is silicon dioxide while that of snail shell is calcium oxide.

Fluid loss property

It can be noticed in Fig. 5 obtained from Table 4 (“Appendix”) that introduction of snail shell and *Mucuna solannia* as fluid loss control additive reduced the volume of filtrate

Table 3 Snail shell and *Mucuna solannia* oxide compositions using X-ray fluorescence (XRF) Cu–Zn method

Oxide	<i>Mucuna solannia</i>	Snail shell
CuO	0	0
NiO	0	0
Fe ₂ O ₃	0	0.103
MnO	0	0.019
Cr ₂ O ₃	0	0
TiO ₂	0.029	0.123
CaO	0.583	63.518
Al ₂ O ₃	1.828	8.008
MgO	0.243	0
ZnO	0.003	0.008
SiO ₂	4.816	10.21

loss during the experiment. It can also be noticed that an increase in the concentration of the additive in turn results in a decrease in volume of filtrate obtained which is actually the function of a fluid loss control additive. But according to (API 13A 1993) for a drilling mud to be accepted, the fluid loss additive used should give filtrate loss volume less than 15 mL over a period of 30 min. From Table 4, the filtrate volume was found to be 16 mL, a little above API standard and therefore can be concluded that snail shell and *Mucuna solannia* as fluid loss control additive for drilling fluids is not a viable prospect at low concentrations of 5 g and below.

Rheological properties

Also, according to API specification, the viscometer dial reading at 600 rpm should be greater than thirty (> 30),

but from Fig. 6, it can be found that the dial reading was 27 (for NB5 at 5 g conc.) which is lower than the standard API specification. Therefore, it can be concluded that snail shell and *Mucuna solannia* as drilling fluid additives to improve its rheological properties are not viable prospects at low concentrations of 5 g and below. It can be seen that local Nigerian bentonite does not satisfy API requirements of a 600-rpm dial reading greater than 30. Hence, that rheological factor needs improvement, as we can see that the effect of the beneficiating agents was an increase in the dial reading which if more were added, probably at 7–8 g each, the API requirement would definitely be reached.

The ratio of yield point to plastic viscosity (YP/PV) according to API specification for bentonite clay for drilling mud is a value below 3 (<3), and from the result in Fig. 7, this specification is met as opposed to foreign bentonite which has

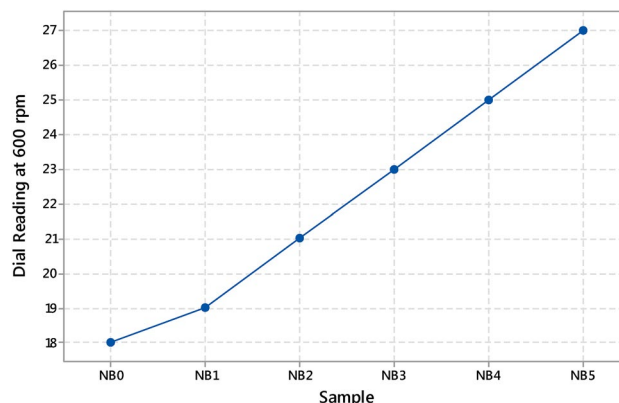
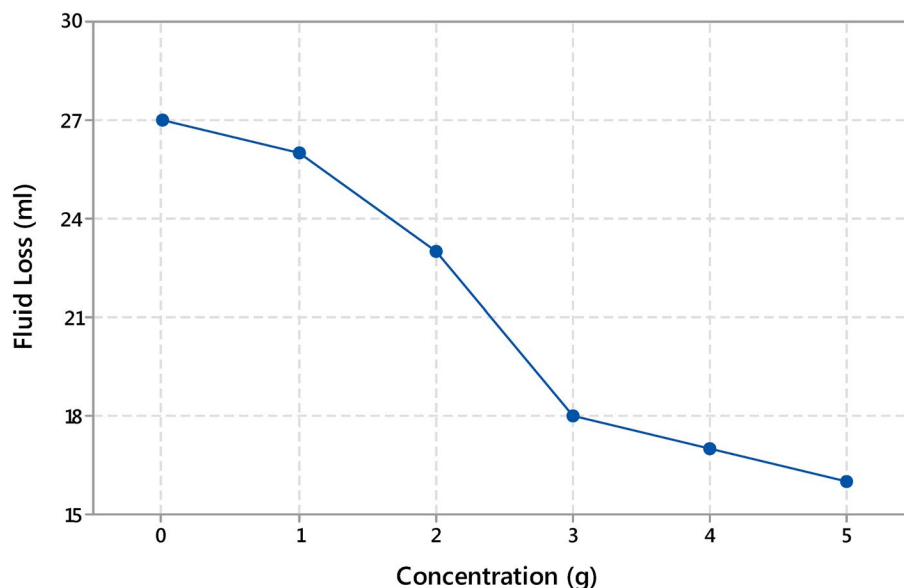
**Fig. 6** Dial readings at 600 rpm of unbeneficiated and beneficiated Nigerian (local) bentonite**Fig. 5** Fluid loss of drilling mud sample with snail shell and *Mucuna solannia* at different concentrations

Fig. 7 YP/PV of 15 g Nigerian bentonite beneficiated with different concentrations of snail shell and *Mucuna solan* at 0–5 g each

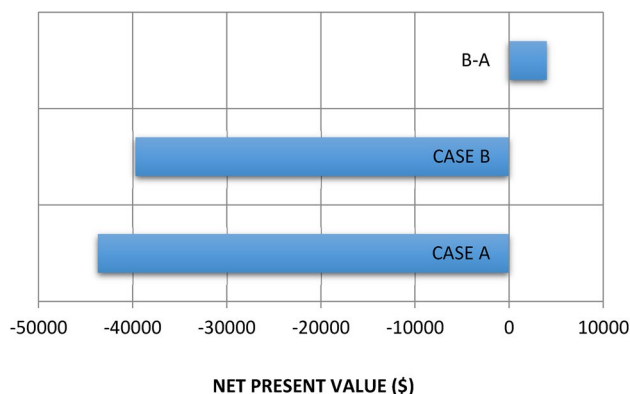
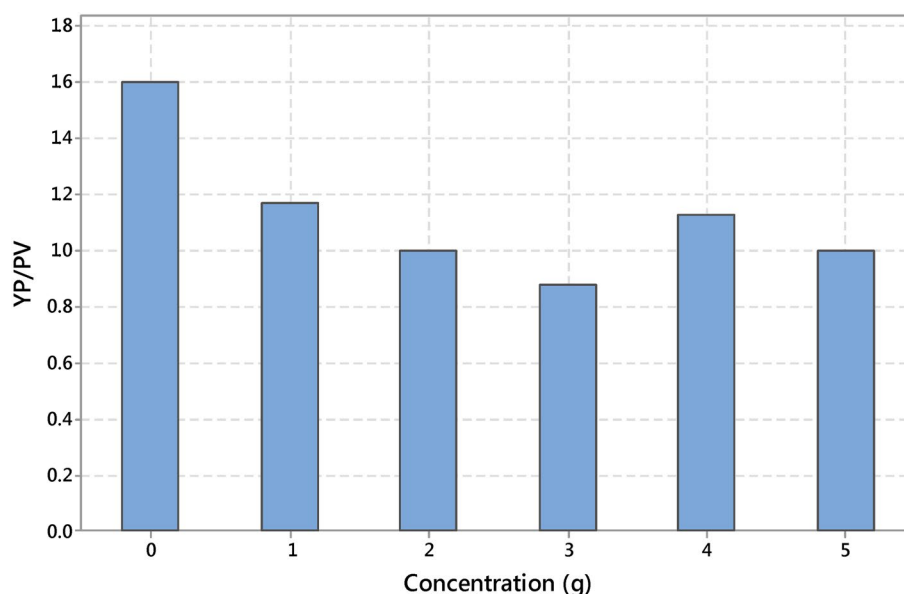


Fig. 8 Foreign and local beneficiated bentonites alternatives selection using incremental analyses

a value of 3.5, although this is not a decisive claim to conclude that it is a suitable substitute for foreign bentonite.

Tables 5 and 6 show the cost of materials and cashflow series, respectively. 7 g concentrations of additives for local beneficiated bentonite were considered. Figure 8 shows the incremental analyses and shows that local beneficiated bentonite is a good investment, with positive Net Present Value seen in the cashflow of incremental Case B over Case A.

In oil and gas exploration and production, drilling fluids are designed not only for chemical and thermal stability, but also to be biologically degradable. Hence, polymers and natural products are becoming relevant. *Mucuna solan* is a biomaterial and hence biodegradable. When a biocide is used in a mud formulated with it, it is expected that microbial actions would be reduced, just like it is applied in other mud types.

Conclusion

During the course of this research, it was observed that snail shell, which is considered as waste in Nigeria, plays a good role in supplementing the properties of the Nigerian bentonite to meet API standards. From the filtration and rheological measurements carried out on the drilling fluid samples that were beneficiated and the one which was not, it can be said that snail shell and *Mucuna solan* at substantial concentrations can be used to beneficiate the Nigerian bentonite and, therefore, can be a good substitute for foreign bentonite. Also, snail shell and *Mucuna solan* are good candidate additives for wellbore inhibition and stability. Beneficiation of Nigerian bentonite was shown to be economically viable.

Acknowledgements The SEM and XRD measurements were carried out as per API standard at Spectral Laboratory Services (SLS) in Kaduna, Nigeria.

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Appendix

See Tables 4, 5 and 6.

Table 4 Rheological properties and fluid loss of the additives

Additives concentrations	Fluid loss (mL)	Dial reading (rpm)					
		600	300	200	100	6	3
15 g foreign bentonite	13	27	21	15	11	8	6
15 g local bentonite	27	18	13	8	6	4	3
30 g foreign bentonite	5	52	43	32	25	17	14
30 g local bentonite	11	34	28	21	16	10	8
15 g local bentonite + 1 g snail shell + 1 g <i>Mucuna solannie</i>	26	19	13	9	6	4	3
15 g local bentonite + 2 g snail shell + 2 g <i>Mucuna solannie</i>	23	21	14	9	6	4	3
15 g local bentonite + 3 g snail shell + 3 g <i>Mucuna solannie</i>	18	23	15	9	7	4	3
15 g local bentonite + 4 g snail shell + 4 g <i>Mucuna solannie</i>	17	25	17	10	7	4	3
15 g local bentonite + 5 g snail shell + 5 g <i>Mucuna solannie</i>	16	27	18	12	8	5	3

Table 5 Cost estimate of materials

S/N	Item description	Size (metric tonne)	Unit price (\$)
1	Foreign bentonite (USA)	1	305.4
2	Local bentonite	1	150.6
3	Snail shell	1	5
4	<i>Mucuna solannie</i>	1	267

\$1 = N386

Table 6 Cashflow series

A Foreign bentonite			
B Local beneficiated bentonite			
Year	A	B	Incremental B-A
0	0	0	0
1	–8706	–7909.5	796.5
2	–8706	–7909.5	796.5
3	–8706	–7909.5	796.5
4	–8706	–7909.5	796.5
5	–8706	–7909.5	796.5
6	–8706	–7909.5	796.5
7	–8706	–7909.5	796.5
8	–8706	–7909.5	796.5
9	–8706	–7909.5	796.5
10	–8706	–7909.5	796.5
	(\$43,693.40)	(\$39,695.95)	
			\$3997.45
MARR	15%		Choose B

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