



OPTIMIZATION OF RHEOLOGICAL AND FILTRATION PROPERTIES OF NIGERIA CLAY USING FACTORIAL DESIGN

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ABSTRACT

Nigeria is blessed with about 700 million tonnes of bentonitic clay which in its natural state does not meet the API standard for drilling fluid application. Hence, this research undertake a process optimization and model development for improving rheological properties of this clay using 2 by 2 full factorial experimental design techniques. The accuracy of the models developed for the beneficiation of the clay were confirmed by the adjusted R-squared values of 0.94, 0.924 and 0.995 for yield point, plastic viscosity and filtrate volume respectively, alongside with the comparison of the experimental and model prediction results. The best conditions for treating the clay was been deduced from the best beneficiated clay sample which was characterized for yield point, plastic viscosity and filtrate volume respectively and was found to meet the API conditions for its drilling mud applications. Thus, the model developed was confirmed to be safe and suitable for examining treatment variations. Investigating effective way of examining interaction effects of the treatments and predetermines the treatment of local clay properties for various compositions.

Keywords: Bentonite; Beneficiation; Model; Drilling; Factorial Design.

INTRODUCTION

Background of Study

Petroleum is one of the major sources of energy in which a lot of machines like automobiles, generators depend on and its importance cannot be over empathized. Petroleum is a naturally occurring complex oily mixture formed as a result of the decomposition of the plant and animal remains under high pressure and temperature which took place millions of years ago and are buried underground in tiny pockets in rocks. In order to access the oil reserves, we need to drill oil wells into the rocks to pump out the oil. Some wells are more than two miles deep and can stretch more than eight miles sideways [8].

Drilling mud is basically composed of water, bentonitic clay and other chemicals such as barite, calcium carbonate and exhibits the properties such as apparent viscosity, plastic viscosity and yield point which plays important role in designing efficient and optimized drilling operation. These properties serve the functions of cleaning the rock fragments from beneath the bit and carry them to the surface as well as cooling and lubricating the rotating drill-string and bit. Out of the constituents used for the formulation of drilling mud, bentonite has always been sourced from outside of this country ever since 1960 which had drastically led to the neglect of the local bentonite which is unhealthy for the economy of our country. This have also made the purchasing cost for drilling materials to be high [14], [2], [15]. Hence, this research devices an alternative and easier means of treating our local bentonitic clay so as to serve as substitute for the foreign grade bentonite.

Factorial Design Approach and Terminologies

Factorial design planning is simply applied to determine and represent the cause and effect relationship between true mean responses and input control variables influencing the responses. Three kinds of design of experiments [16] are possible between output and input variables.

- a. Screening designs are used in beginning of process where more than five factors are involved, to recognize the most critical factors.
- b. Characterization deigns narrow the numbers of factors to only a few and permit for some quantitative understanding of the interactions among factors.

c. Optimization designs focus on only one or two factors, but in much more depth to gain a precise understanding of relationships between factors

A full factorial design combines the levels for each factor with all the levels of every factor. It covers all combinations and provides best data. However it consumes more time and resources. While a fractional Factorial design, uses too many of resources, or if a slightly non orthogonal array is accepted a fractional design is used. To analyze the data from a design of experiment, evaluating the statistical significance by computing one way ANOVA, or for more than one factor N-way ANOVA is essential. The practical significance can be evaluated through sum of squares, line or column charts, and normal probability chart [16].

Review of Literature on Clay Beneficiation

Previous Approach

Previous researches have shown that Nigerian has a probable reserve of about 700 million tonnes of bentonite clay in North-Eastern Nigeria. Though the clay in its natural state has properties below the standard required for drilling fluid application [16], [17], [1] but in recent times, more researches has been conducted to device means of beneficiating the clays through the use of additives for a better performance. Some of the research works are one which beneficiated Nasarawa and Abia clay using starch. Another formulated and characterized a bentonite extended drilling mud from Nasarawa clay using biopolymer (Rel-Pac) additives and lots more [2, 7, 9]. All this previous research on beneficiation has been adopting the one factor at a time (OFAT) experiment which gave a satisfactory result when only one factor is changed but cannot be used to study the effect of two-factors at a time.

New Approach

In present work, Polypac and PacR were been used to improve the viscosity of the clay together with the addition of soda ash which helps to reduce the concentration of calcium ion and increase the sodium ion concentration present in the clay simultaneously.

This research also assesses the effectiveness of additives role in improving the rheological and filtration properties of the local clay by increasing the viscosity and reducing the filtrate.

Hence, factorial design techniques were been applied so as to provide great flexibility for exploring or enhancing the treatment of local clay for the formulation of water based mud in diverse ways [14], [9], [2], [15] with adoption of American Petroleum Institute (API) Specifications in testing and defining their properties.

MATERIALS AND METHODS

Locally sourced clay from Sangere in Yola, Adamawa State and Polypac, Soda Ash and PacR, sourced from Mi-Swaco Drilling Fluids, Port Harcourt, Rivers State in Nigeria were used.

Evaluation of the Local Clay for Mud Formulation

To test for the suitability of the prepared local clay from Sangere in Yola for mud formulation the following analysis were been carried-out;

Rheological Analysis

A suspension of the bentonite was prepared and 22.5 g of clay was been added to 350 ml of de-ionized water while stirring on the mixer. After been stirred for 5 minutes, the container was removed from the mixer and scraped on the sides with spatula to dislodge any bentonite adhering to the walls of the container having been sure that all bentonite clinged to the spatula was incorporated into suspension. The container was replaced on the mixer and the stirring continued. The container was removed from the mixer and scraped on the sides with spatula to dislodge any bentonite adhering to the walls of the container after another 5 minutes and after 10 minutes till a total stirring time of 20 minutes is completed. The bentonite suspension was then aged up to 16 h in sealed or covered container at room temperature. The storage temperature and duration was been recorded. After aging, the bentonite suspension was shaken well and then poured into the mixer container then stirred for 5 minutes on the mixer. The suspension was then poured into the viscometer cup provided with the viscometer. The dial reading of 600 rpm and 300 rpm rotor speed settings of the viscometer was recorded when at constant value for each of rpm at a room temperature of 25 °C.

$$PV = \Phi_{600} - \Phi_{300} \tag{1}$$

$$YP = \Phi_{600} - PV \tag{2}$$

Filtration Analysis

The prepared and tested suspension in 3.3.3 was recombined and stirred in a container for 1 minute on the mixer while the temperature was adjusted to 25 °C. The suspension was then been poured into the cell and before adding

the suspension it was ensured that part of filter cell was dried and that the gaskets are not distorted or worn, the filter paper and the O-ring groove was in place at the base cap of filtrate tube. The mud was poured to within about 13 mm from the base cap of the filter cell and the assembly was completed and a measuring cylinder was placed under the drain tube. One timer was set for 7.5 min and the second was set to 30 min. Both timers were started after adjusting the pressure to 100 psi (690 kPa) and the pressure was supplied by compressed carbon dioxide or nitrogen gas. The fluid loss was measured in milliliters. The cell was disassembled and the mud was discarded while extreme care was taken to save filter paper with a minimum disturbance of the cake.

Determination of Sand Content

The glass tube was fed to the “mud” line while sample and water is added to the sample in the tube until it attains the next scribed mark. Then, thumb is placed over the mouth of tube and shaken vigorously to ensure homogenous mixing. After that the mixture is poured onto the clean screen while more water is added to tube, shaken and poured onto the screen while the liquid that passes through the screen is been discarded. This was repeated until when the wash water is clean. The sand retained on screen is washed in order to free any adhering mud and finally the funnel is placed on top of screen assembly with the assembly slowly been inverted and the tip of funnel was inserted into glass tube while the sand is washed back into tube with a fine spray of water or a wash bottle. After the sand was allowed to settle, the volume percent of sand from graduations on the glass measuring tube was read and recorded in %.

Determination of Hydrogen ion Concentration

An indicator stick was placed in the mud and was allowed to remain in it until the color has stabilized, usually less than a minute. Rinse the stick off with de-ionized water but do not wipe. The colors of the stick were compared with the color standard provided and the pH of the mud was estimated and the pH of the mud was reported to the nearest 0.5 pH units.

Particle Size Analysis

10 g of dried bentonite was weighed and added to approximately 350 ml of water containing 0.2 g of sodium phosphate. The mixture was stirred on the mixer for 5min. The sample was then transferred into the 75 micron sieve. A water bottle was used to remove all material from the container to the sieve. Then, the material on the sieve with water controlled to 12 psi or 69 kPa from a spray nozzle was been washed for 2 min. While washing, the tip of the spray nozzle was held approximately in the plane of the top of sieve and moves the spray of water repeatedly over the sample. The residue from the sieve was washed into an evaporating dish and excess clear water was being decanted and dried in an oven to a constant mass. The mass of residue was been recorded.

$$P > 75 \mu m = \frac{M_R}{M_S} \times 100 \quad (3)$$

Determination of Moisture Content

An empty dish was weighed then a dish with 10 g of bentonite was weighed and their various weight readings as M0 and M1 respectively. The sample was been then kept in the oven for at least 3 hr and reweighed after drying. Its weight reading was recorded as M2.

$$\% M = \frac{(M_2 - M_0)}{(M_1 - M_0)} \times 100 \quad (4)$$

Determination of Calcium content (Ca⁺⁺)

5 - 10 ml of distilled water and 1 ml of the water or filtrate to be tested was added to titration vessel. Then, 1 ml or 20 drops of strong buffer solution (NH₄OH base) and 4 – 6 drops of calmagite were been added to the filtrate and mixed with the use of a stirring rod. A wine red color was developed to confirm the presence of calcium and/or magnesium.

Using a pipette, Standard Versenate Solution (EDTA) was been titrated against the mixture, while stirring the mixture continuously, until the sample first turns to blue with no undertint of red remaining. The number of ml of Standard Versenate solution used was recorded and the calcium ion and calcium carbonate content is calculated as:

$$Ca^{++} (mg/l) = \frac{ml \text{ of Standard Versenate}}{ml \text{ of sample}} \times 400 \quad (5)$$

Local Clay Treatment

To upgrade the local clay properties so as to meet the API Specification, the following were been carried-out;

Factors and Levels Selections for the Beneficiation of the Clay

Factors like the amount of water, mass of clay sample, mass of Polypac, mass of soda ash and mass of PacR that are involved in the clay beneficiation were considered to know which them influence the rheological and filtration properties of the clay. Review of literatures and previous work done shows that mass of soda ash and polypac have greater influence on the rheological and filtration properties of the clay [5]. The table 1 below shows the two major independent variables (factors) and the levels that they subdivided into.

Table 1. Level of Factors for Beneficiation of Clay

Level	Polypac (g)	Soda Ash (g)
Low	0.7	1.0
High	1.0	1.5

Design Matrix for the Beneficiation of the Clay

The design and operational matrix for this clay beneficiation was prepared using the above factors and levels where 1 represent the high while -1 represent the low level of the respective factors as shown in the Table 2 below

Table 2. 2 by 2 Factorial Design Technique for Beneficiation of Clay without Responses

Run	Design Matrix				Operational Matrix		Response		
	T	A	B	AB	P, g	S, g	YP, lb/100ft ²	PV, cP	FV, ml
1	1	1	1	1	1.00	1.50	21.50	18.50	10.40
2	1	1	-1	-1	1.00	1.00	21.00	18.75	10.60
3	1	-1	1	-1	0.70	1.50	16.50	16.00	14.20
4	1	-1	-1	1	0.70	1.00	15.25	16.00	18.20

Thus four beneficiated clay samples were prepared with various quantities of the materials as specified in Table 2 for every runs using dissolved 22.5 g clay in 0.0029 g /cm³ of concentrated PacR Solution. Then, each sample was thoroughly agitated until a homogenous mixture was obtained and aged to swell. The test for suitability in (a) – (g) above were carried out for raw local clay were repeated for this four beneficiated clay samples.

Model Design

The general model equation is given by equation 4 which shows 1 intercept coefficient, 2 main effects and 1 two-way interaction term (Davies O. L. and Hay W. A. 1950). Thus for the prediction of the local mud properties (plastic viscosity, yield point and filtrate volume) the fitted model equation is:

$$f = t + a + b + ab + e \tag{5}$$

The results obtained from experiments were recorded in Table 1. Full factorial designs were done with “Design Expert 8 Software” this software makes use of type III sums of squares and set the confidence intervals to 95% and significance value to 0.0500.

RESULTS AND DISCUSSION

Summary of effects of main and interactions effects between the factors for each model are given in Table 9 (a-c) for clay beneficiation while Figure 1 – 3 shows the effect of each factors on yield point, plastic viscosity and filtrate volume for the clay beneficiation.

Evaluation of the Local Clay for Mud Formulation

Rheological analysis

The rheological properties of the local clay are as follows:

(i) **Viscometer dial reading:** The viscometer dial reading at 600 rpm as specified by API for the physical requirement of bentonite clays is 30 cP (minimum). Table 4 shows that the result obtained for this raw clay sample does not meet the API requirement. Meanwhile, the result obtained is similar to what Igbafo [9] obtained as 4 cP (Abia clay) but different from the other clay (Nasarawa clay) which he obtained as 8 cP unlike Azeez [5] who obtained 9 cP (Afuze clay) and 19 cP (Maiduguri clay) and Aderinto [2] who obtained 12 cP (Nasarawa clay).

Table 4. Result of Rheological and Filtration Analysis of Raw Clay

Properties	API Spec	A _{Raw}
Viscometer dial reading at 600 rpm (cP)	≤ 30	4
Viscometer dial reading at 300 rpm (cP)	-	3
Viscometer dial reading at 6 rpm (cP)	-	1
Viscometer dial reading at 3 rpm (cP)	-	1
Plastic Viscosity (cP)	-	1
Yield Point (lbs/100ft ²)	-	2
Yield Point/Plastic Viscosity Ratio	≤ 3	2
Filtrate Volume or Fluid Loss (ml)	≤ 14	69

This result also confirms that the clay sample consists of low montmorillonitic content or clay minerals which account for low viscosity and yield. This signifies that the clay sample is composed largely of calcium ions in the view that clays with high calcium content gives low viscosity dial reading Azeez [5] when added to water which is shown in the result of raw clay sample analysis with 1001 mg/L calcium ions in Table 4. The clay sample can therefore be said to be a calcium bentonite.

ii. **Yield point/plastic viscosity ratio:** The Yield Point/Plastic Ratio specification by API is 3 maximum and from Table 4, the raw clay result was found to be 2 which satisfy the API specification and it is identical to the clay which Aderinto [2] and Igbafo [9] obtained as 2 (both for Nasarawa Clay) but is different from value obtained by Azeez [5] as 0.25 (Afuze clay) and 1.33 (Maiduguri clay), also has a wide distinction with the value obtained by Igbafo [9] as 0.67 (Abia clay). And the result obtained implies that the clay will exhibit good yield point and plastic viscosity when use for the formulation of drilling mud.

Filtration analysis

The filtrate volume of the raw clay obtained is not in accordance with the API specification of 15 ml (maximum). The result obtained was found to be of large distinction and below the value obtained by Azeez [5] as 80 ml (Afuze clay) and 88 ml (Maiduguri clay), Igbafo [9] as 88 ml (Abia clay) and 80 ml (Nasarawa clay) but it is of low distinction and above the value obtained by Aderinto [2] as 38.5 ml. The result obtained in Table 4 implies that the raw clay has high water loss properties and shows no evidence of thixotropic properties.

Determination of Sand Content

The API specification states that the quantity of sand should not be greater than 1 % which this raw clay met the requirement with 1 % and this result was close to what was obtained by Azeez [5] as trace quantity (for Afuze and Maiduguri clay) and also Aderinto [2] as trace quantity (Nasarawa clay). The result (in Table 5) also proves that its formulated drilling mud will be of low solid content since the raw clay is of a very low sand content.

Table 5. Result of Chemical and Physical Analysis of Raw Clay

Properties	API Spec	A _{Raw}
Residue of diameter > 75µm (%)	≤ 4	3.00
Sand Content (%)	≤ 1	1
Ca ²⁺ (mg/l)	-	1001
pH	9-11	7

Hydrogen ion Concentration

The raw clay pH was obtained to be 7.0 which in agreement with what Igbafo [9] obtained as 7.0 (Abia clay) and 7.5 (Nasarawa clay); Aderinto [2] obtained as 7.5 (Nasarawa clay), but 14 % different from what was obtained by Azeez [5] as 8.0 (for Afuza and Maiduguri clay). Meanwhile, the obtained result in Table 4 was not in accordance with the API specification requirement of 9 – 11 and also shows that the clay is neutral (that is, 50 % basic and 50 % acidic).

Particle Size Analysis

The amount of residue with size greater 75 µm obtained in the raw clay was in agreement with the API specification of 4 % (maximum) for drilling mud formulation. The obtained result in Table 5 was found to be of large variation and low value when compared with what was obtained by Azeez [5] as 6.9 % (Afuze clay) and 7.5 % (Maiduguri clay). This result shows that its formulated drilling mud will be of low solid content.

Moisture Content

The amount of the water present obtained in the raw clay agrees quite well with what Azeez [5] as 3.95 % (Maiduguri clay) and slightly close to the other value he obtained as 3.23 % (Afuze clay). The obtained result in Table 5 was found to be in agreement with the API specification requirement of 10 % (maximum).

Calcium ion Concentration

Table 5 shows that the calcium concentration of the clay is high. This confirms that the clay is a calcium bentonite which normally exhibit poor filtration control and low viscosity.

Effect of Beneficiation on the Clay Properties

Rheological analysis

The effects of beneficiation on the rheological properties are as follows:

i. **Viscometer dial reading:** The result (Table 6) of clay beneficiation shows that sample A₁ to A₄ all meet up with the minimum requirement which was achieved with the addition of polypac and soda ash to a 1g/350 ml of pacR concentrated water which builds the lower-end rheology (6 rpm and 3 rpm) as shown in Table 6 when related to the result obtained for raw clay in Table 4.

The obtained results for sample A₃ (48.50 cP) and A₄ (47.25 cP) were in slight agreement with the value obtained by Azeez [5] as 38 cP (Afuze clay) and 37 cP (Maiduguri clay); Igbafo [9] as 37 cP (Abia clay) and 38 cP (Nasarawa clay) and Aderinto [2] as 23.4 cP (Nasarawa clay) while the other results obtained for A₁ and A₂ was of a large distinction from Azeez [5], Igbafo [9] and Aderinto [2].

The obtained results in Table 8 also show that the raw clay sample calcium content reduces for all clay samples during beneficiation process. It also confirms that the addition of additives (polypac, soda ash and pacR) resulted to an increase in the viscometer dial reading which by implication increases the sodium ion (Na⁺) and thereby increasing the viscosity [5], [6].

Table 6: Result of Rheological and Filtration Analysis of Beneficiated Clay Samples

Properties	API Spec	A ₁	A ₂	A ₃	A ₄
Viscometer dial reading at 600 rpm (cP)	≤ 30	58.5	58.5	48.5	47.25
Viscometer dial reading at 300 rpm (cP)	-	40	39.75	32.5	31.25
Viscometer dial reading at 6 rpm (cP)	-	4	3.75	3	2.75
Viscometer dial reading at 3 rpm (cP)	-	2.75	2.5	2	2
Plastic Viscosity (cP)	-	18.5	18.75	16	16
Yield Point (lbs/100ft ²)	-	21.5	21	16.5	15.25
Yield Point/Plastic Viscosity Ratio	≤ 3	1.16	1.12	1.03	0.95
Filtrate Volume or Fluid Loss (ml)	≤ 14	10.4	10.6	14.2	18.2

ii. **Yield point/plastic viscosity ratio:** The Yield Point/Plastic Ratio specification by API is 3 maximum. The results of samples A₁, A₂, A₃ and A₄ in Table 6 during beneficiation were found to be in accordance with the API standard. The obtained results was in quite agreement with what Aderinto [2] obtained as 1.13 but has a large distinction with what Azeez [5] obtained as 2.22 (Afuze clay) and 2.10 (Maiduguri clay) and Igbafo [9] obtained as 1.7 (Abia clay) and 1.9 (Nasarawa clay). The YP/PV ratio was found to increase with increase in mass of additive (polypac) been added.

iii. **Plastic viscosity:** From Table 6 above, it was deduced that with the addition of soda ash (B) in two levels at constant 0.7 g polypac (A), a constant clay plastic viscosity was been observed as 16 cP while the addition of soda

ash (B) in two levels at constant 1.0 g polypac (A) shows 1 % decrease in plastic viscosity of the clay from 18.75 to 18.50 cP. Unlike the average 14 % increment which was observed in the plastic viscosity when there was 30 % increment in polypac (A) from 0.7 g to 1.0 g at constant 1.0 g as illustrated in Figure 1. It can be concluded that polypac (A) made a larger contributions for the increment of a clay plastic viscosity as proved in Table 2.

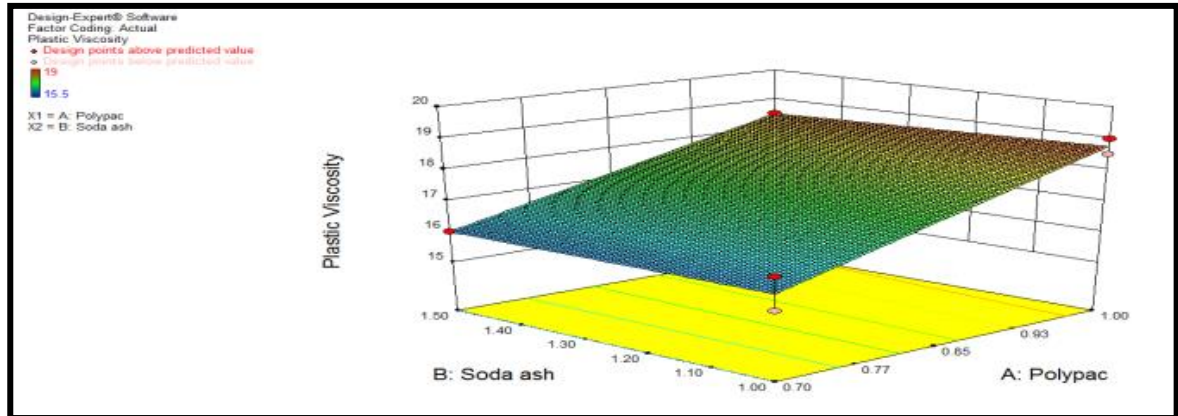


Figure 1. Effect of polypac (A) and soda ash (B) on plastic viscosity of local clay

iv. **Yield point:** It was deduced from Table 6 that the addition of soda ash (B) in two levels (as 1.0 g and 1.5 g) at constant 0.7 g polypac (A), 8 % increment in yield point from 15.25 to 16.50 lb/100ft² was observed while the addition of soda ash (B) in two levels (as 1.0 g and 1.5 g) at constant 1.0 g polypac (A), a 2 % decrement in the yield point of the clay from 21.0 to 21.5 lb/100ft² was observed. Unlike the average 38 % and 30 % increment in yield point from 15.25 to 21.0 lb/100ft² and 16.50 to 21.5 lb/100ft² that were observed as effect of the 30 % increment of polypac (A) from 0.7 g to 1.0 g at constant 1.0 g and 1.5 g respectively as illustrated in Figure 2. It can therefore be said that polypac (A) plays a major role in increasing the clay plastic viscosity as shown in Table 8.

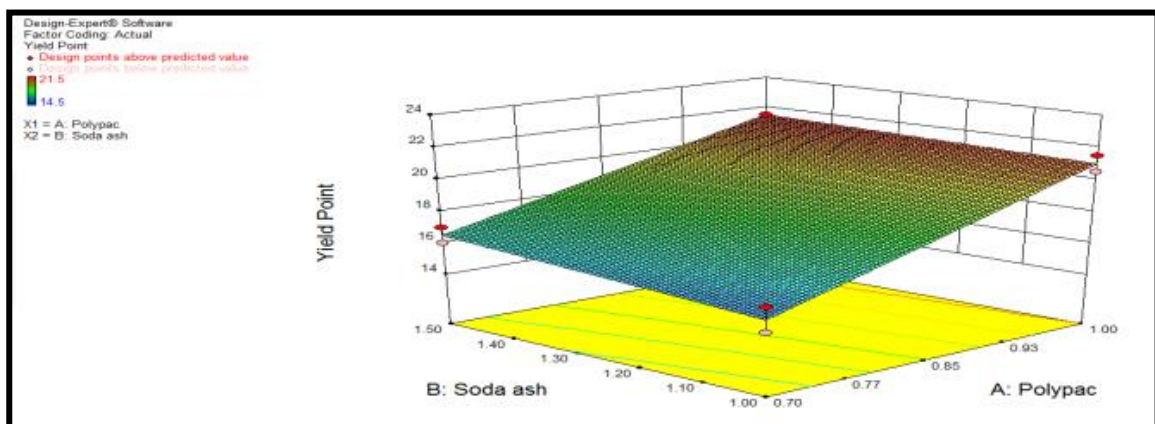


Figure 2. Effect of polypac (A) and soda ash (B) on yield point of local clay

Filtration Analysis

With the addition of various amount of polypac and soda concentration at constant pacR concentration using factorial design technique improves the fluid loss from 69 ml to the corresponding effect obtained as 10.40, 10.60, 14.20, and 18.20 ml respectively as shown in Table 6 and 2 in sample A₁, A₂, A₃ and A₄ due to their additions in two levels (as 1.0 and 0.7 g) for polypac with 1.5 and 1.0 g for soda ash as shown in Figure 3.

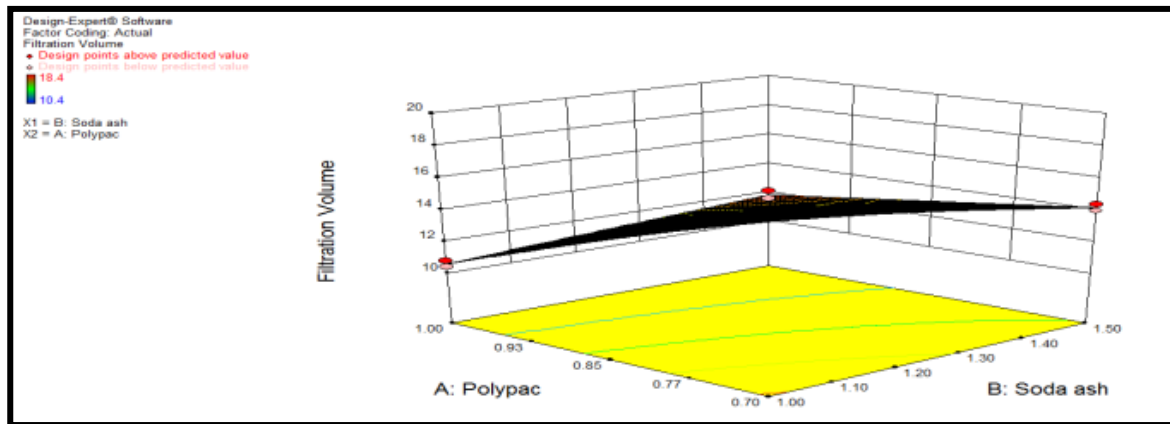


Figure 3. Effect of Polypac (A) and Soda Ash (B) on Filtrate Volume of Local Clay

From Table 6, it was deduced that an addition of soda ash (B) in two levels as 1.0 g and 1.5 g at constant 0.7 g polypac (A), 22 % decrement in clay filtrate volume from 18.20 to 14.20 ml was observed while the addition of soda ash (B) in two levels of 1.0 g and 1.5 g at constant 1.0 g polypac (A), 4 % decrement in filtrate volume of the clay from 10.60 to 10.40 ml was observed. Unlike the 42 % and 27 % decrement in clay filtrate volume from 18.20 to 10.60 ml and 14.20 to 10.40 ml which was observed as effect of the 30 % increment of polypac (A) from 0.7 g to 1.0 g at constant 1.0 g and 1.5 g respectively.

The results obtained for A₁ (10.40 ml) and A₂ (10.60 ml) was in accordance with API specification of 14 ml (maximum) while the other results obtained for A₃ (14.20 ml) and A₄ (18.20 ml) were not in accordance with the API specification. From Table 6, the fluid loss reduction is largely dependent on the quantity of polypac been added with slight addition of soda ash. These suggest that the basic function of pacR is filtration control which reduces the rate at which water is lost to the formation [11].

Sand Content

The API specification states that the quantity of sand should not be greater than 1 % in which the obtained results in Table 7 shows that the beneficiated clay samples A₁ to A₄ all met the requirement as trace quantity which is in accordance with the values obtained by Azeez [5] as trace quantity (for Afuze and Maiduguri clay) and also Aderinto [2] as trace quantity (Nasarawa clay). A low sand content implies that the clay will be of low flow resistance and yield point.

Table 7. Result of Chemical and Physical Analysis of Beneficiated Clay Samples

Properties	API Spec	A ₁	A ₂	A ₃	A ₄
Residue of diameter > 75µm (%)	≤ 4	3	3	3	3
Sand Content (%)	≤ 1	Trace	Trace	Trace	Trace
Ca ²⁺ (mg/l)	-	0	20	0	20
pH	9-11	9.5	9.5	9.5	9.5
Moisture Content (%)	≤ 10	0	0.02	0.05	0.05

Hydrogen ion Concentration

The results obtained in Table 5 and 7 was surveyed to rise from 7.0 to 9.5 for all beneficiated clay samples which shows that they all in accordance with the API specification requirement of 9 – 11 and which in agreement with what Aderinto [2] obtained as 9.5 (Nasarawa clay), but not in agreement with what Igbafo (2007) obtained as 9.0 (Abia clay) and 10.0 (Nasarawa clay) and Azeez [5] as 10.0 (for Afuza and Maiduguri clay). Meanwhile, the obtained result in Table 7 shows that the clay is basic.

Particle Size Analysis

The amount of residue with size greater 75 µm in the raw clay is obtained to be 3 % which is in agreement with the API specification of 4 % (maximum) for drilling mud formulation. The obtained result in Table 7 was found to be

of large variation and low value when compared with what was obtained by Azeez [5] as 6.9 % (Afuze clay) and 7.5 % (Maiduguri clay). This result shows that its formulated drilling mud will be of low solid content.

Moisture Content

The amount of the water present in the raw clay was obtained to be 3.90 % which agrees quite well with what Azeez [5] as 3.95 % (Maiduguri clay) and slightly close to the other value he obtained as 3.23 % (Afuze clay). The obtained result in Table 7 was found to be in agreement with the API specification requirement of 10 % (maximum).

Calcium ion Concentration

Table 7 shows a low calcium concentration for different beneficiated clay samples. This confirms that the calcium ion present in the raw clay samples have largely been exchanged for sodium ion which also account for improvement in the filtration control and viscosity of the clay.

Statistical Analysis of the Designed Models

Shown in Table 9(a-c) are the summaries of values of the main and interaction effects between the factors of yield point, plastic viscosity and filtrate volume during the clay beneficiation.

Table 8. Summary of Effect and Contributions of the Factors and Their Interactions

Factors	Plastic Viscosity		Yield Point		Filtrate Volume	
	Effect	Contribution (%)	Effect	Contribution (%)	Effect	Contribution (%)
A	+2.62	95.25	+5.38	93.62	-5.70	79.97
B	-0.13	0.22	+0.67	2.48	-2.10	10.85
AB	-0.12	0.22	-0.37	0.46	1.90	8.89

a. **Plastic viscosity:** The mass of polypac (factor A) made a positive effects or contributions of (+2.62) 95.25 % to the plastic viscosity of the clay unlike soda ash (factor B) and interaction (AB) that made a negative contributions of (-0.13) 0.22 % and (-0.12) 0.22 % respectively as confirmed in the Table 8. With the level of significance obtained for different factors, it can therefore be said that the plastic viscosity of the clay is basically dependent on the amount of polypac (factor A) added which implies that for any significant change to take place in plastic viscosity of the local clay, the amount of polypac (factor A) added has to be changed or considered.

Table 9. (a) Analysis of Variance for Plastic Viscosity Model for Local Clay Beneficiation

Source	Sum of Square	Df	Mean Square	F-calculated	P-Value
Model	13.84	3	4.61	29.53	0.0034
A-Polypac	13.78	1	13.78	88.20	0.0007
B-Soda ash	0.031	1	0.031	0.20	0.6779
AB	0.031	1	0.031	0.20	0.6779
Error	0.63	4	0.16		
Total	14.47	7		R ² Adj = 0.924	

From the analysis of variance in Table 9(a), it was shown that the linear regression model of plastic viscosity with p-value of 0.0034 is significant in view of the fact that its p-value was less than 0.0500 and that it has (0.9966) 99.66 % chances of the model having significant relationship between the response and predictor variance and error of 0.34 %. The analysis of variance in Table 9(a) also show that model term A (Polypac) with p-value of 0.0007 is a significant model term in view of the fact that its p-value was less than 0.0500 while B (Soda ash) with p-value of 0.6779 and AB (Interaction of Polypac and Soda ash) with p-value of 0.6779 model terms are not significant which implies that no interaction took place. So, the developed linear regression model is therefore been reduced from $PV = 17.3125 + 1.3125A - 0.0625B - 0.0625AB$ to $PV^* = 17.3125 + 1.3125A$ which improves its

significance by neglecting the insignificant model terms B (Soda ash) and AB (Interaction of Polypac and Soda ash).

b. **Yield point:** Table 8 showed that the yield point of the clay is largely dependent on the amount of polypac (factor A) and slight dependent on soda ash (factor B) which necessitate that for any significant change to take place in yield of the local clay, the mass of polypac (factor A) has to be considered for either to be increased or decreased since it build a large positive effects or contributions of (+5.38) 93.62 % on the yield point of the clay contrasting soda ash (factor B) and interaction (AB) that build a low contributions of (+0.87) 2.48 % and (-0.37) 0.46 %.

Table 9. (b) Analysis of Variance for Yield Point Model for Local Clay Beneficiation

Source	Sum of Square	Df	Mean Square	F-calculated	P-value
Model	59.59	3	19.86	37.39	0.0022
A-Polypac	57.78	1	57.78	108.76	0.0005
B-Soda ash	1.53	1	1.53	2.88	0.1648
AB	0.28	1	0.28	0.53	0.5072
Error	2.12	4	0.53		
Total	61.72	7		R ² Adj = 0.94	

The validity of the model from analysis of variance expressed in Table 9(b) confirms that the model with p-value of 0.0022 is significant in view of the fact that its p-value is less than 0.0500 and (0.9978) 99.78 % chances of the model having significant relationship between the response and predictor variance with error of 0.22 %. With the view that for any linear regression model term to be significant its p-value has to be less than 0.0500, the factor A (Polypac) with p-value of 0.0005 was confirmed to be significant while B (Soda ash) with p-value of 0.1648 and AB (Interaction of polypac and soda ash) with p-value of 0.5072 was confirmed not to be significant and with due consideration to their level of significance, the model is therefore been reduced from $YP = 18.5625 + 2.6875A + 0.4375B - 0.1875AB$ to $YP^* = 18.5625 + 2.6875A$ which improves its significance by neglecting the insignificant model terms B (Soda ash) and AB (Interaction of polypac and soda ash).

c. **Filtrate volume:** It was shown in Table 8 that the filtrate volume of the clay is largely dependent on the amount of polypac (factor A) with a large negative effects or contributions of (-5.70) 79.97 % and slight dependent on soda ash (factor B) with a small negative effect or contribution of (-2.10) 10.85 % to the filtrate volume of the clay which confirms that for any significant changes to be effected in filtrate volume of the local clay, the mass concentration of polypac (factor A) and soda ash (factor B) has to be considered whether to be increased or decreased because of the significance of their contributions. The analysis of variance also verifies that the interaction of polypac and soda ash (AB) is significant with (+1.90) 8.89 % positive effect or contribution to the filtrate volume of the local clay.

Table 9. (c) Analysis of Variance for Filtrate Volume Model for Local Clay Beneficiation

Source	Sum of Square	Df	Mean Square	F-calculated	P-Value
Model	81.02	3	27.01	450.11	< 0.0001
A-Polypac	64.98	1	64.98	1083.00	< 0.0001
B-Soda ash	8.82	1	8.82	147.00	0.0003
AB	7.22	1	7.22	120.33	0.0004
Error	0.24	4	0.060		
Total	81.26	7		R ² Adj = 0.995	

The result of the analysis of variance in Table 9(c) confirms that FV linear regression model with p-value of <0.0001 is highly significant in the view of the fact that the model p-value is less than 0.0500. The chances of the model having significant relationship between the response and predictor variance was obtained to be (0.9999) 99.99 %. Considering the p-values for the model terms derived from analysis of variance, the linear regression model term A (polypac) , B (soda ash) and AB (interaction of polypac and soda ash) with p-value of <0.0001, 0.0003 and 0.0004 respectively, were all confirmed to be significant with the view of the fact that they all have

their p-values less than 0.0500. Hence, the developed model, $FV^* = 13.35 - 2.85A - 1.05B + 0.95AB$ will required no removal of any model terms.

Hence, the clay properties improvement is highly dependent on the amount of polypac been added to it. The adjusted R-squared values yield point, plastic viscosity and filtrate volume models are 0.94, 0.924 and 0.995 respectively which implies that the model accounts for 94 %, 92.4 % and 99.5 % of variation in the experimental data respectively and have been explained by the factors in the model predictions which provided satisfactory results. Therefore, the developed model provides a means of determining the rheological properties (Yield Point, Plastic Viscosity and Filtrate Volume) with high degree of accuracy for the formulation of Water-Based Drilling Mud from Sangare clay.

CONCLUSION

This research confirms the fact that the Yola clay is a low grade which contains high calcium ion with a high filtrate volume and low viscosity which did not meet the API Standard Specifications. With the use of additives and employment of factorial design techniques, the beneficiated clay was found to meet all the API Standard Specifications with good filtrate volume and viscosity including a large drop in the calcium ion concentration at the end of the beneficiation process which justifies that the polypac are highly effectiveness.

Moreover, the analysis of variance confirms the fact that the mass concentration of polypac plays the major or largest role in the improvement of the fluid-loss control and viscosity including the yield point of the clay. It was also deduced that the factorial design aids the development of linear regression models that can predict yield point (YP), plastic viscosity (PV) and filtrate volume (FV) for the beneficiation of local clay from different mass concentration of polypac(A), and soda ash(B).

Result of ANOVA confirms that the models developed are safe and suitable for evaluating the best required treatment for a clay sample and optimizing the whole process of beneficiation. The developed model has proved to be a means of saving time, cost and rigorous procedures in the laboratory in the process of obtaining best clay flow properties.

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List of Nomenclatures

A _x	= Clay Sample X
T	= Intercept
A	= Polypac (P)
B	= Soda Ash (S)
P _{>75μm}	= Residue mass fraction (%) of diameter ≥75micron
M _R	= mass of residue, g
M _S	= mass of sample, g
M ₀	= Weight of dish
M ₁	= Weight of dish + sample before drying
M ₂	= Weight of dish + sample after drying
%M	= Moisture Content
Φ ₆₀₀	= 600 rpm reading
Φ ₃₀₀	= 300 rpm reading
PV	= Plastic viscosity in centipoises
YP	= Yield Point in lb/100 ft ²
AP	= Apparent viscosity in centipoises
cP	= Centipoises
lb	= Pounds
ft	= Feet

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