

Evaluation and Comparative Analysis of Wet and Thermal Beneficiation of Nteje Clay for Possible Drilling Fluid Formulation

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Abstract: This study evaluates the potential of Nteje clay, sourced from southeastern Nigeria, as a local alternative to bentonite in water-based drilling fluids. Raw clay (NC1) was treated using thermal (NC2) and wet beneficiation (NC3) methods to enhance its physicochemical and drilling performance properties. Characterization was conducted using X-ray diffraction, X-ray fluorescence, Scanning electron microscopy with energy dispersive X-ray spectroscopy, and standard American Petroleum Institute procedures (API standard test). Among the treated samples, NC3 demonstrated the most improvement, recording a pH of 10.21, apparent viscosity of 45 cp, yield point of 70 lb/100 ft², and gel strengths of 43 and 54 lb/100 ft² after 10 seconds and 10 minutes respectively (with 0.8 g Polyanionic Cellulose regular grade). Fluid loss was reduced to 11 ml with 0.8 g Carboxymethyl Cellulose and 9 ml with 0.8 g Polyanionic Cellulose regular grade, while filter cake thickness was minimized to 2.38 mm and 1.59 mm, respectively, both within acceptable API limits. XRF analysis showed NC3 contained 61.70 wt% SiO₂ and 17.80 wt% Al₂O₃, indicating favorable rheological potential, although Na₂O (0.25 wt%) remained below the ideal range. SEM analysis revealed improved particle dispersion in NC3, correlating with better hydration and additive compatibility. While NC3 does not fully meet API benchmarks for plastic and apparent viscosities without additives, its enhanced filtration control and gel strength with additives confirm its suitability for partial substitution of bentonite in drilling fluids. The findings highlight wet beneficiation as a cost-effective and sustainable route to improve local clay performance, offering a viable alternative for Nigeria's petroleum industry.

Keywords: Nteje Clay, Wet beneficiation, Dry beneficiation, Rheological properties, Filtration and Mud thickness.

1. INTRODUCTION

Drilling fluid, also known as drilling mud, is a critical component in oil and gas exploration, playing an essential role in facilitating the drilling process (Yang et al., 2023). Its primary functions include providing hydrostatic pressure to prevent formation fluids from entering the wellbore, transporting cuttings to the surface, cooling and lubricating the drill bit, and maintaining wellbore stability (Khodja et al., 2010). The efficiency and success of drilling operations are significantly influenced by the properties of the drilling fluid, which must exhibit optimal viscosity, yield stress, and filtration characteristics to ensure smooth drilling operations (Saboori et al., 2018). One of the key materials in drilling fluid formulation is bentonite, a type of clay primarily composed of montmorillonite, known for its excellent swelling and rheological properties (Zhang, et al., 2020). Bentonite is widely used as a viscosifier and filtration control agent in water-based drilling fluids (Ali et al., 2022). However, the growing demand for high-performance drilling fluids, coupled with the increasing cost of imported bentonite, has necessitated the exploration of alternative sources of clay that can be locally sourced, processed, and used in drilling fluid formulation. Developing a cost-effective and sustainable substitute for bentonite is crucial, particularly in countries like Nigeria, where the oil and gas industry is a major economic sector. Nigeria is endowed with vast clay mineral resources, many of which remain underutilized due to a lack of detailed studies on their beneficiation and suitability for industrial applications (Adeniyi, et al., 2023). Among these clay deposits, Nteje clay, found in southeastern

Nigeria, has attracted attention due to its abundance and possible potential for use in drilling applications. However, natural clays, including Nteje clay, often exhibit inferior rheological properties compared to commercial bentonite (Eyankware et al., 2021). This is due to impurities, poor swelling capacity, and suboptimal mineralogical composition, which hinder their direct application in drilling fluid formulations (Anthony et al., 2020). To overcome these limitations, beneficiation techniques must be employed to enhance the physicochemical properties of the clay formulations (Anthony et al., 2020). Two promising methods for improving the quality of natural clay for industrial applications are wet and thermal beneficiation. Wet beneficiation involves processes such as dispersion, sedimentation, and chemical treatment to remove non-clay impurities and improve hydration, swelling, and rheological properties (James et al., 2008). This method enhances the clay's ability to disperse in water, making it a more effective viscosifier in drilling fluids. Thermal beneficiation, on the other hand, involves heating the clay at controlled temperatures to alter its mineralogical structure, remove unwanted impurities, and modify its swelling and dispersion characteristics (Belghazdis & Hachem, 2022).. The application of thermal treatment can lead to improved rheological and filtration properties, making the treated clay more suitable for drilling fluid formulation. Several studies have demonstrated that both wet and thermal beneficiation methods can enhance the performance of natural clays in drilling applications (James et al., 2008). While wet beneficiation improves clay hydration and dispersion, thermal treatment modifies the crystalline structure, affecting plasticity and swelling behavior (Ogolo et al., 2023).

However, the comparative effectiveness of these two beneficiation techniques on Nteje clay, as well as their combined influence on its drilling fluid properties, remains largely unexplored. This study seeks to evaluate and compare the wet and thermal beneficiation of Nteje clay to determine its suitability for drilling fluid formulation. Specifically, the research will investigate the effects of these beneficiation techniques on the mineralogical composition, rheological behavior, and filtration properties of the clay. By processing the parameters for both wet and thermal beneficiation, this study aims to enhance the drilling fluid performance of Nteje clay, making it a viable alternative to bentonite. The findings of this research will provide valuable insights into the suitability of locally sourced Nigerian clays for oil and gas drilling operations, thereby reducing dependence on expensive imported bentonite and promoting the utilization of indigenous materials. Furthermore, the successful beneficiation of Nteje clay could have significant economic and environmental benefits. It could lead to cost savings for the petroleum industry, encourage local content development, and stimulate industrial applications of Nigerian clays. Additionally, utilizing locally available materials aligns with sustainability goals, as it reduces the carbon footprint associated with the importation of foreign drilling fluid additives (Al-Hameedi et al., 2019). Going forward, this study will bridge the knowledge gap regarding the potential of wet- and thermally-treated Nteje clay as a drilling fluid component. By systematically analyzing the changes in its physicochemical and rheological properties post-beneficiation, this research will contribute to the broader efforts of optimizing indigenous clay resources for enhanced industrial applications.

2. MATERIALS AND METHOD

2.1. Material Collection and Preparation

Local clay samples were randomly collected from Nteje, Oyi Local Government Area, Anambra State, Nigeria and all other chemicals and reagents including Na_2CO_3 , **polyanionic cellulose (PACR)** and **carboxymethyl cellulose (CMC)** used in this study are of analytical grade. The sample, labeled as **Natural Clay (NC1)**, were air-dried, then oven-dried for 6 hours at a temperature of 70°C to remove moisture before being pulverized and sieved for uniform particle size. The processed clay was stored in airtight containers to prevent contamination. All preparation steps, including drying, grinding, and sieving, were conducted at the **Petroleum Engineering Laboratory, Nnamdi Azikiwe University, Awka, Nigeria**.

2.2. Thermal Beneficiated Treatment

Following the method described by Ainsworth-Jienken (1994), the bulk local clay sample was crushed, and unwanted materials such as pebbles and dirt were removed. To further eliminate coarse particles, the crushed clay was sieved using a **2 mm sieve**. The refined material was then dried at **150°C – 200°C** to remove volatile organic compounds and excess moisture. After drying, the sample was milled and further screened using a **150 μm sieve**, ensuring a finer and more uniform particle size. The processed clay was designated as **NC2** and stored at room temperature for subsequent evaluation tests.

2.3. Wet Beneficiation Treatment

According to Na₂EDTA method of Isaac (1969) for sodium saturation which involved putting 25 g of screened 2mm sieved sample of local clay in 100 cm³ of a solution containing 0.01 M Na₂EDTA in 0.1 M NaCl. After stirring for 0.5 hr, the pH of the suspension was adjusted to 11.5 with 0.1 M NaOH solution. The resulting suspension was further shaken vigorously for 0.5 hr and allowed to settle for 48 hr. Repeated sedimentation and siphoning were conducted in order to extract 2mm sized clay samples. The sample was designated as NC3 then air dried for days to remove moisture content and kept at room condition for evaluation tests.

2.4. Evaluation Tests

2.4.1. Apparent Bulk Density

The apparent bulk density of the samples (**NC1, NC2, NC3, and bentonite**) was determined following the **ASTM D1895** standard method (**ASTM International, 2024**). This method is specifically designed for fine granular materials capable of flowing freely through a **V-shaped funnel**. During the test, each sample was allowed to flow into a cylindrical container with a known volume of **100 cm³**, and its apparent density was subsequently measured.

2.4.2. Surface pH Measurement

A **20 g** sample of **NC1** was dispersed in **100 ml** of deionized water and allowed to stand for **30 minutes** with intermittent stirring using a glass rod. A **pH meter (Model: PHS-25)** was then used to measure the pH of the supernatant solution. The same procedure was repeated for, **NC2, NC3 and bentonite** to determine their respective pH values.

2.5. Instrumental Analysis of Beneficiated and Non-Beneficiated Nteje Clay

Both beneficiated and **un-beneficiated** local clay (Nteje clay) and bentonite samples were prepared with a uniform particle size of **150 μm** and subjected to instrumental analysis. **X-Ray Diffraction (XRD)** and **X-Ray Fluorescence (XRF)** were utilized to determine the crystallographic structure, and chemical composition of the samples (Ali et al., 2022). Additionally, **Scanning Electron Microscopy with Energy Dispersive X-Ray (SEM-EDX)** was employed to analyze the surface morphology and elemental distribution of the clay samples (Akhtar et al., 2023). All instrumental analyses were conducted at **Rolab Research and Diagnostic Laboratory, Ibadan, Oyo State, Nigeria** to ensure precise characterization of the samples' physical and chemical properties.

2.6. Drilling Fluid Formulation and Testing

The reference drilling fluid (RDF) was formulated in accordance with the standard stipulated in API RP 13B-1 (American Petroleum Institute, 2019). To prepare the RDF, **22.5 g** of bentonite was weighed using an **electronic Fann weighing balance**, while **350 ml** of distilled water was measured with a **500 ml measuring cylinder**. The measured distilled water and weighed bentonite were transferred into the **steel cup of the Fann multi-mixer equipment** and vigorously agitated for **20 minutes** until a homogeneous mixture was obtained. The mixture was then left undisturbed for **24 hours** to allow for proper hydration and aging. Thereafter **5 g of Na₂CO₃ (soda ash)** was added to enhance the clay content, swelling, and solubility (**Okon et al., 2014**). This procedure was repeated for **NC1, NC2 and NC3** to evaluate their performance compared to the bentonite-based fluid. Following the formulation, full-set measurements were conducted to assess and compare the properties of the drilling fluids. **Mud weight** was determined using a **mud balance**, while **rheological properties**, including **plastic viscosity, yield point, and apparent viscosity**, were evaluated based on **two data point measurements (300 and 600 rpm dial readings)** using the **expanded equations 1 and 2**. **Initial gel strength (after 10 seconds) and final gel strength (after 10 minutes)** were measured using the **Model 35 Fann rotary viscometer**. Additionally, **filtration characteristics**, such as **fluid loss (cc/30 min)** and **filter cake thickness**, were determined using the **API Filter Press at 100 psi (LTLP)** to assess the fluid's filtration behavior.

$$\text{Plastic Viscosity (PV)} = PV = \theta_{600} - \theta_{300} \quad (1)$$

The unit is centipoise (cp)

Yield Point (YP) can be determined by the following formula, (equation 2);

$$\text{Yield Point (YP)} = \theta_{300} - \text{PV} \quad (2)$$

The unit is lb. /100ft² or Pa.s

2.6.1. Apparent viscosity (AV) in drilling fluids

Apparent viscosity is the resistance of a fluid to flow under specific shear conditions (Galindo-Rosales et al., 2011). For drilling muds, it represents the effective viscosity measured at a high shear rate, typically at 600 rpm on a Fann viscometer (Agwu et al., 2021). Although drilling fluids are non-Newtonian, AV simplifies analysis by using a fixed shear rate to estimate flow resistance (Amaratunga et al., 2020)

$$\text{Apparent viscosity (AV)} = \frac{\theta_{600}}{2} \quad (3)$$

Where;

θ_{600} = Viscometer dial reading at 600 rpm

Units: Centipoise (cP)

2.6.2. pH Determination of Water-based Drilling Fluid

The pH of the formulated mud system comprising NC1, NC2, NC3 and foreign bentonite separately was measured using a pH meter (model PHS-25). The probe was placed into the mud sample, and the pH was recorded once a stable reading was achieved.

2.6.3. Mud Weight Determination

The API standard Fann Mud Balance was used to measure the mud weight or density. In this process, the cup was filled with a mud sample and covered to ensure no expulsion of mud through the hole in the cup. The balance arm was placed on the bottom, with the knife edge resting on the fulcrum. The rider was moved until the graduated arm was level, as indicated by the level gauge on the beam. The mud weight was read at the left-hand edge of the rider, and its value was recorded. The same procedure was repeated for other mud samples and their readings were recorded.

2.6.4. Determination of Gel Strength of Drilling Mud

This is an extension of mud's rheological properties. The 10seconds and 10 minutes gel strength of the drilling mud was determined by setting the speed of the rotary sleeve at 600rpm to stir the mud samples for 10 seconds, thereafter the viscometer was set at 3rpm with the help of the knob, then switched off to enable the mud sample to stand for 10seconds undisturbed. Thereafter the flip toggle was switched to a low position and the maximum dial reading was recorded as the 10 seconds or initial gel strength. For the 10 minutes gel strength the same procedure was repeated with the mud sample allowed to stand for 10 minutes undisturbed, then with the flip toggle switched to the rear position, the maximum deflection (dial reading) was recorded as the 10 minutes gel strength (Okon et al., 2014)

2.6.5. Determination of fluid loss and mud cake

The API standard low temperature low pressure (LT-LP) filtration (75⁰F and 100psi) experiment was conducted on the highest concentration of the additives on the mud samples. The API standard LT-LP filtration test was performed at room temperature and pressure of 100psi (700kpa) for 30 mins. The standard API filter press consisting of a cylindrical cell whose internal diameter is 3 inches and its height is 5 inches was used as a container for the drilling mud sample. Whatman No.50 filter paper was fitted at the bottom of the cell and filled with a bentonite mud sample to be measured. A pressure of 100psi from the air compressor pump was supplied to the top of the cell while a measuring cylinder was placed under the cells for the collection of the filtrates. The filtrate was collected for 30 minutes and recorded in milliliters (ml) as the API filtrate of the bentonite mud sample, while the residual (mud cake) thickness of the bentonite mud sample on the filter paper was measured using a digital caliper and recorded in millimeters (mm) as the mud cake thickness of the mud sample. The same procedure was repeated for NC1, NC2 and NC3.

2.7. Formulation and evaluation of the effect of Carboxymethyl Cellulose (CMC) and Polyanionic Cellulose – Regular grade (PACR) on bentonite, NC1, NC2 and NC3 based mud

To evaluate the impact of adding various concentrations of viscosifiers (CMC and PACR) on the rheological properties of the drilling mud, thirty two (32) mud samples were prepared in accordance

with the standard stipulated in API RP 13B-1 protocols (American Petroleum Institute, 2019), which specifies 22.5 g of clay per 350 ml of water, and then treated with 5 g of sodium carbonate (Na_2CO_3). These samples were categorized into eight groups: X1, X2, X3, X4, X5, X6, X7 and X8 as shown in table 2.1, with each group containing four samples. Group X1, X3, X5 and X7 were dosed with 0.2 g, 0.4 g, 0.6 g, and 0.8 g of carboxymethyl cellulose (CMC) and group X2, X4, X6 and X8 were dosed with 0.2 g, 0.4 g, 0.6 g, and 0.8 g polyanionic cellulose (PACR). After formulation, comprehensive measurements were performed to evaluate and compare the properties of the drilling fluids. Mud weight was measured using a mud balance, while rheological properties including plastic viscosity, yield point, and apparent viscosity were determined using two data point measurements (300 and 600 rpm dial readings) and calculated with the expanded equations 1 and 2. Initial (10-second) and final (10-minute) gel strengths were assessed with a Model 35 Fann rotary viscometer. Additionally, filtration characteristics, such as fluid loss (cc/30 min) and filter cake thickness, were analyzed with the standard stipulated in API RP 13B-1 protocols (American Petroleum Institute, 2019), using the API Filter Press at 100 psi (LTLP) to determine the fluid's filtration performance. The experiments for this study were carried out in the laboratories of the Petroleum Engineering Department at the Federal University of Technology Owerri, Imo State, and the Chemical Engineering Department at Nnamdi Azikiwe University, Awka, Anambra State, Nigeria.

Table 2.1. Mud formulation design table

Additives	CMC (g)X1	PACR (g) X2
	0.2	0.2
NC1	0.4	0.4
	0.6	0.6
	0.8	0.8
Additives	CMC (g) X3	PACR (g) X4
	0.2	0.2
NC2	0.4	0.4
	0.6	0.6
	0.8	0.8
Additives	CMC (g) X5	PACR (g) X6
	0.2	0.2
NC3	0.4	0.4
	0.6	0.6
	0.8	0.8
Additives	CMC (g) X7	PACR (g) X8
	0.2	0.2
Bentonite	0.4	0.4
	0.6	0.6
	0.8	0.8

3. RESULTS AND DISCUSSION

3.1. Physical and Rheological Properties of Local and Foreign Clay

Table 3.1 compares the physical and rheological properties of beneficiated and un-beneficiated Nteje clays (NC1, NC2, and NC3) with foreign bentonite, highlighting their suitability for drilling mud applications. From the physical and rheological results of various mud samples presented in table 3.1, NC3 shows the most improvement among the local clays, with a pH of 10.21 within API's acceptable range (9.5–12.5) suggesting better clay reactivity and compatibility with chemical additives (Eze & Chukwu, 2025). Its bulk density (1090 kg/m³) is lower than that of bentonite (1150 kg/m³), favoring reduced solids content in the mud. Mud weight for NC3 is 8.60 lb/gal, slightly below the API range (8.65–9.60 lb/gal), indicating it may require densification. Rheologically, NC3 performs better than NC1 and NC2, with viscometer readings of 13cp (600 rpm) and 5cp (3 rpm), compared to 4cp and 0cp respectively, but still far behind bentonite (54cp and 10cp for 600rpm and 300rpm respectively). This implies weaker flow properties and lower suspension capability (Agwu et al., 2021). Gel strengths of 6 lb/100ft² (10 sec) and 11 lb/100ft² (10 min) for NC3 reflect moderate structure-building capacity (Agbabi, 2025), though bentonite's 10 lb/100ft² and 21 lb/100ft² for 10 sec and 10 mins respectively will provide superior suspension during static periods. NC3 records a plastic viscosity of 4 cp and apparent viscosity of 6.5 cp, both below API standards (8cp–10 cp and 15 cp, respectively), indicating inadequate carrying and flow resistance (Agwu et al., 2021). Its yield point (5 lb/100ft²) and YP/PV

ratio (1.25) are also much lower than bentonite (32 lb/100ft² and 2.9), suggesting reduced hole-cleaning efficiency (Al-Rubaii et al., 2023). From the results, NC3 exhibits potential; however, further improvement is necessary for it to meet API standards required for effective drilling fluid performance.

Table 3.1. Physical and rheological properties of various mud samples sample

Parameter	NC1	NC2	NC3	Bentonite	API Standard
pH	6.21	5.83	10.21	8.82	9.5min–12.5max
Bulk density (kg/m ³)	1190	1140	1090	1150	
Swelling power					
Mud weight (lb/gal)	8.70	8.65	8.60	8.68	8.65 - 9.60
Viscometer dial reading at 3rpm	0	0	5	10	-
Viscometer dial reading at 300rpm	2	2	9	43	-
Viscometer dial reading at 600rpm	4	4	13	54	≥30cp
Gel strength @ 10 Sec	0	0	6	10	-
Gel strength @ 10 Min	1	1	11	21	-
Plastic Viscosity (cP)	2	2	4	11	8.0 - 10
Apparent Viscosity (cP)	2	2	6.5	27	≥15cp (usually)
Yield point (lb/100ft ²)	0	0	5	32	≥ 15 lb/100 ft ²
YP/PV ratio	0	0	1.25	2.9	3 (max)

American Petroleum Institute. (2019)

3.2. X-Ray Diffraction Analysis of Clay Samples

X-ray diffraction (XRD) analysis was conducted on NC1, NC2, NC3, and foreign bentonite using a Rigaku MiniFlex II diffractometer with CuK α radiation under standard conditions ($\lambda \approx 1.54\text{--}1.66$ nm, 40 kV, 20 mA). From table 3.2 and fig 3.1 – 3.4, the results, which are in consistence with ICDD data reveal distinct mineralogical compositions and crystallinity. In NC1, quartz was identified as the major mineral phase with sharp diffraction peaks observed at 2 θ values of 31°, 35°, 43°, 47.5°, 54°, 62°, and 67.5°, indicating a highly crystalline structure. Minor phases included kaolinite (30.44°, 40.62°), halloysite (37.86°, 60.86°, 68.8°), albite (25.4°, 36.36°, 45.32°), and allophane (49°). In NC2, which underwent thermal beneficiation, quartz also remained the dominant phase with peaks at 34°, 41.5°, 44°, and 54°. Impurity phases included calcite (45.5°), iron oxide (22°), cristobalite (27.5°, 64°), and zeolite (24°, 37.5°, 57.5°).

Table 3.2. Summary of XRD Results for NC1, NC2, NC3, and Foreign Bentonite

Sample	Major Mineral Phase	2 θ Peaks of Major Phase (°)	Minor/Impurity Phases	2 θ Peaks of Minor Phases (°)	Crystallinity
NC1	Quartz	31, 35, 43, 47.5, 54, 62, 67.5	Kaolinite, Halloysite, Albite, Allophane	30.44, 40.62 (Kaolinite); 37.86, 60.86, 68.8 (Halloysite); 25.4, 36.36, 45.32 (Albite); 49 (Allophane)	Sharp peaks (highly crystalline)
NC2	Quartz	34, 41.5, 44, 54	Calcite, Iron Oxide, Cristobalite, Zeolite	22 (Fe ₂ O ₃), 24, 37.5, 57.5 (Zeolite); 27.5, 64 (Cristobalite); 45.5 (Calcite)	Sharp peaks (highly crystalline)
NC3	Quartz	11.5, 20, 25, 30	Calcite, Iron Oxide, Calcium Oxide, Cristobalite, Zeolite	16 (Zeolite), 17.5 (Cristobalite); 22.5 (Calcite), 31 (Fe ₂ O ₃), 36.5 (CaO)	Sharp peaks (highly crystalline)
Foreign Bentonite	Montmorillonite	6, 20, 27.5, 35, 55, 62	Quartz, Feldspar, Calcite	21, 27 (Quartz); 23 (Feldspar); 22 (Calcite)	Sharp peaks (highly crystalline)

Evaluation and Comparative Analysis of Wet and Thermal Beneficiation of Nteje Clay for Possible Drilling Fluid Formulation

Sample	: NC 2	File	: Sg2~1.ASC	Date	: June 06 9:28:40	Operator	:
Comment	: Qualitative	Memo					
Method	: 2nd differential	Typical width	: 0.065 deg.	Min. Height		400:00 c p s	

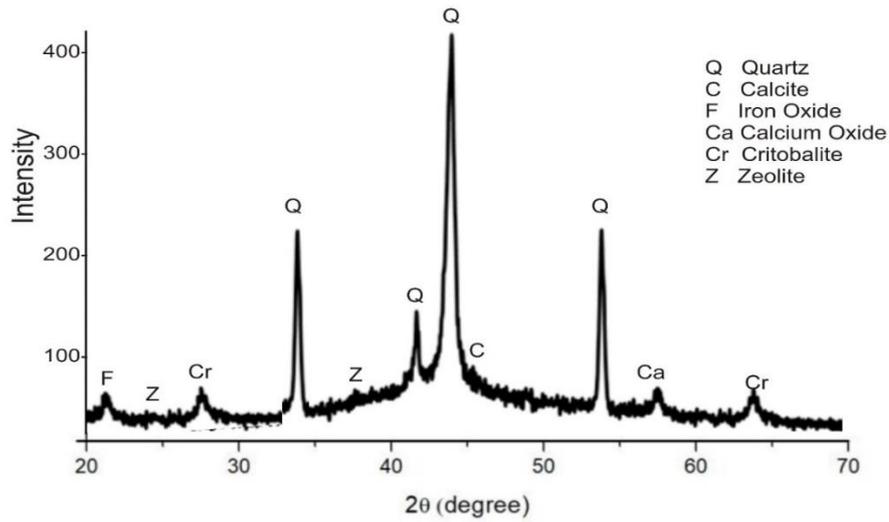


Figure 3.2. XRD patterns of NC2

Sample	: NC 3	File	: Sg2~1.ASC	Date	: June 06 10:28:20	Operator	:
Comment	: Qualitative	Memo					
Method	: 2nd differential	Typical width	: 0.065 deg.	Min. Height		280:00 c p s	

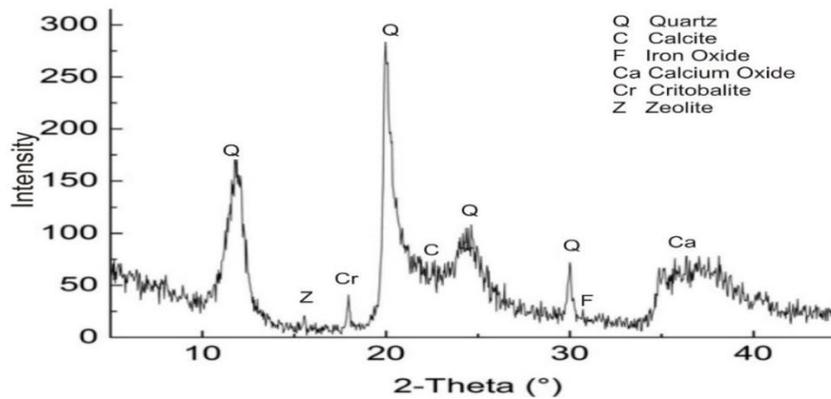


Figure 3.3. XRD patterns of NC3

Sample	: Foreign Bentonite	File	: Sg2~1.ASC	Date	: June 06 15:55:20	Operator	:
Comment	: Qualitative	Memo					
Method	: 2nd differential	Typical width	: 0.065 deg.	Min. Height		500:00 c p s	

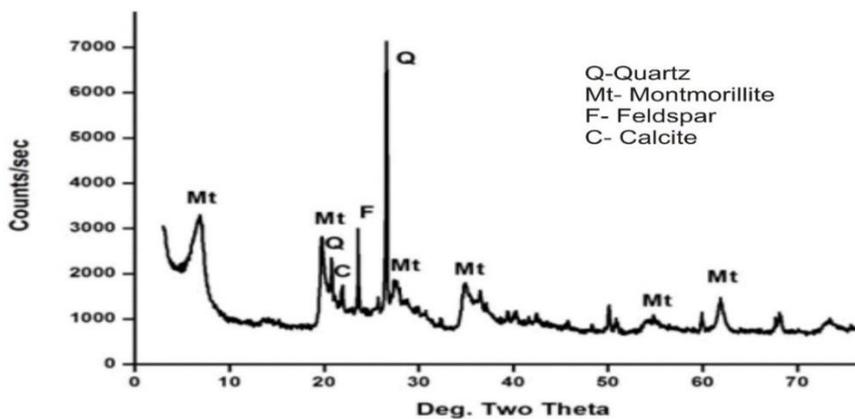


Figure 3.4. XRD patterns of foreign bentonite

The sharp peaks in the XRD pattern confirmed high crystallinity. NC3 similarly showed quartz as the primary phase, with major peaks at 11.5°, 20°, 25°, and 30°. Minor phases identified were zeolite (16°),

cristobalite (17.5°), calcite (22.5°), iron oxide (31°), and calcium oxide (36.5°), again with sharp peaks indicating a crystalline structure. In contrast, the foreign bentonite showed montmorillonite as the dominant mineral, with characteristic peaks at 6°, 20°, 27.5°, 35°, 55°, and 62°. Minor phases included quartz (21°, 27°), feldspar (23°), and calcite (22°). Like the Nteje samples, foreign bentonite also exhibited a highly crystalline microstructure. Generally, quartz was common to all samples, while montmorillonite was exclusive to the foreign bentonite, confirming its superior bentonitic quality.

3.3. Comparative SEM Analysis of NC1, NC2, NC3, and Foreign Bentonite

Scanning electron microscopy (SEM) was used to examine the morphological characteristics of NC1 (raw clay), NC2 (thermally beneficiated), NC3 (wet-beneficiated), and foreign bentonite as presented in figure 3.5 – 3.8. These analyses reveal how beneficiation methods and clay origin influence the microstructure, which directly affects their suitability for drilling fluid applications as compared in table 3.3. NC1 displayed a heterogeneous and porous microstructure with loosely packed, flaky particles and numerous voids. The surface appeared irregular and rough, with clear signs of impurities and poor crystallinity. These features indicate limited hydration, low gel strength, and poor suspension capability (Luckham & Rossi, 1999), rendering it unsuitable for direct use in drilling fluids without modification. Thermal treatment in NC2 resulted in a more compact and cohesive particle arrangement, with partially fused lamellar and reduced surface porosity. The improved microstructure reflects a higher degree of crystallinity, leading to enhanced rheological performance, including better gel strength and lower fluid loss (Pumchusak et al., 2021), making NC2 more compatible with drilling fluid formulations. NC3 showed moderate improvements in particle dispersion and surface uniformity compared to NC1. The wet beneficiation helped remove surface impurities and enhanced platelet separation, but the structure remained less compact than NC2. The presence of cleaner, thin flakes suggests improved hydration and dispersibility, making NC3 moderately suitable for drilling fluids when used with additives (Li et al., 2015). Foreign bentonite exhibited a highly compact, crystalline lamellar structure, with tightly stacked, smooth-edged platelets and minimal voids. This morphology is typical of sodium-based bentonite and indicates excellent swelling capacity, gel strength, and fluid loss control. The well-developed flake alignment and low porosity make foreign bentonite ideal for high-performance drilling mud systems (Wei et al., 2022), clearly outperforming the local samples.

Table 3.3. SEM-Based Comparison of Clay Samples

Sample	Treatment Type	Microstructure Summary	Implication for Drilling Fluids
NC1	Untreated (Raw Clay)	Loose, porous, flaky particles with surface impurities	Poor rheology; unsuitable without modification
NC2	Thermal Beneficiation	Densely packed, fused particles; high crystallinity	Strong rheology; improved yield point and fluid loss control
NC3	Wet Beneficiation	Moderately dispersed flakes; cleaner surface but less compact structure	Moderate rheology; better hydration and dispersion
Foreign Bentonite	Commercial (Imported)	Highly compact, layered lamellae with smooth, aligned flakes	Excellent rheology; ideal for drilling fluids

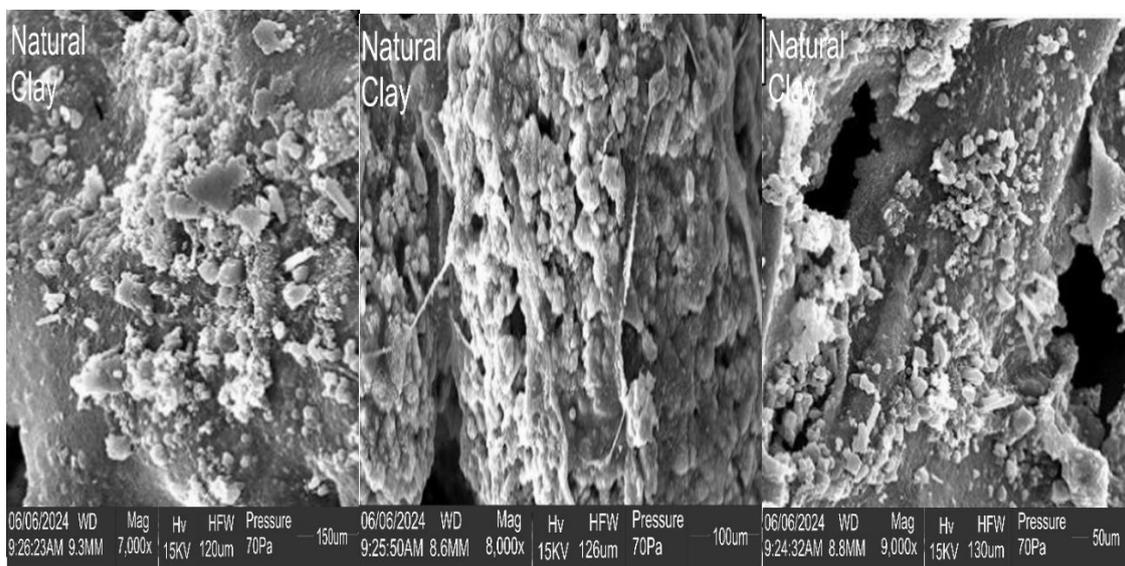


Figure 3.5. SEM images of NC1 at magnifications of (A); 7000x (B); 8000x (C); 9000x

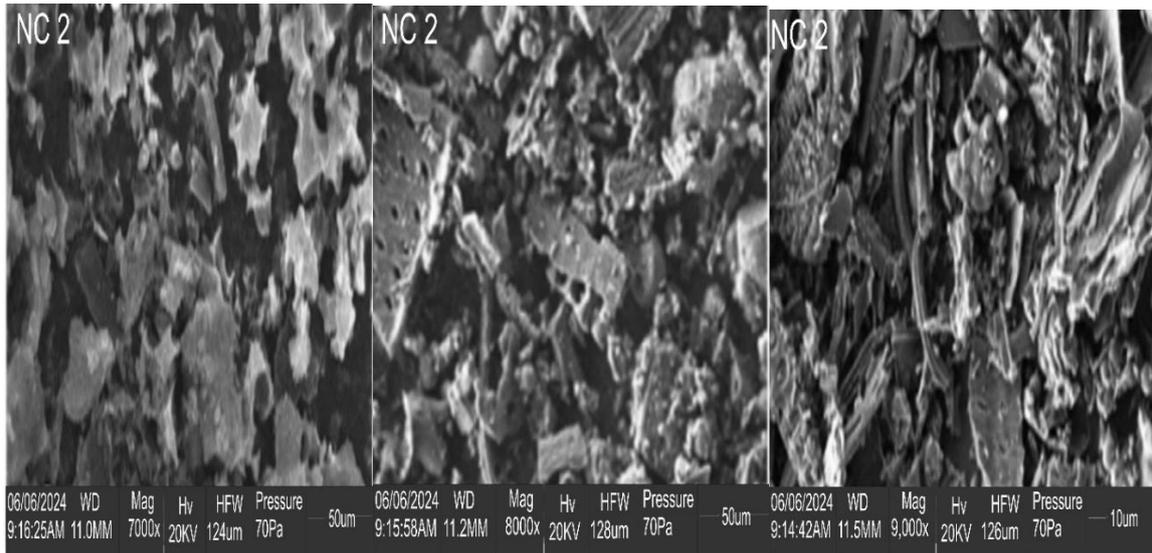


Figure 3.6. SEM images of NC2 at magnifications of (A); 7000x (B); 8000x (C); 9000x

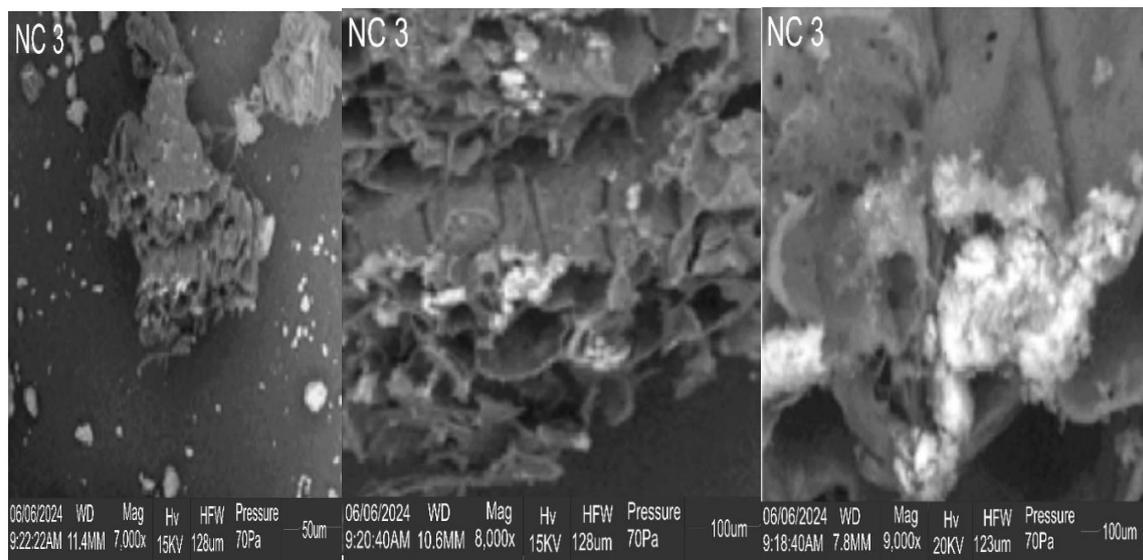


Figure 3.7. SEM images of NC3 at magnifications of (A); 7000x (B); 8000x (C); 9000x

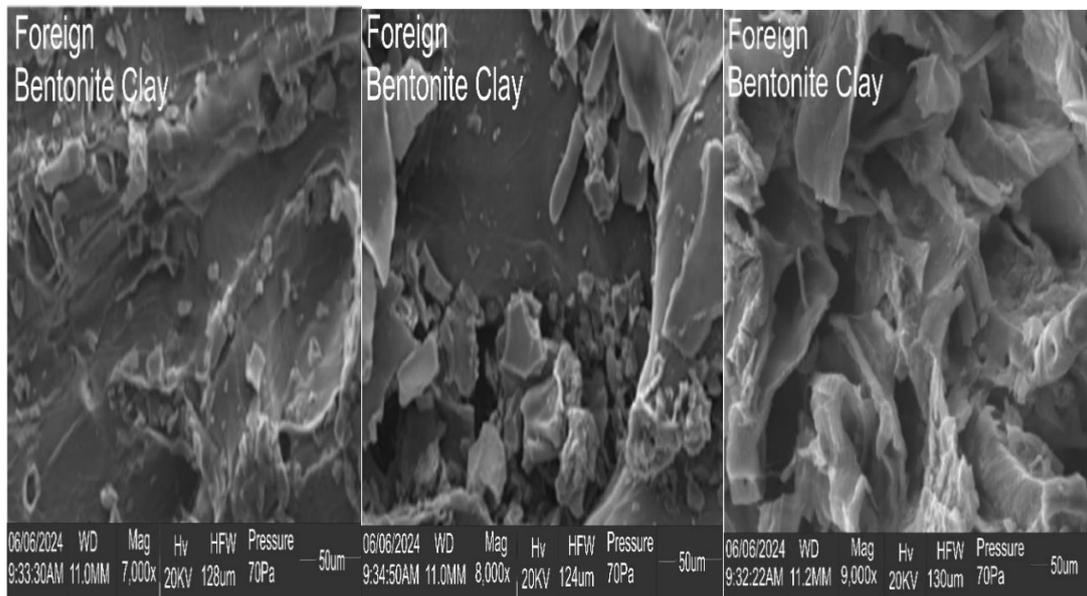


Figure 3.8. SEM images of foreign bentonite at magnifications of (A); 7000x (B); 8000x (C); 9000x

3.4. EDX Analysis of clay samples

Figure 3.9 – 3.12 presents the Energy Dispersive X-ray (EDX) elemental analysis of untreated and treated local clay used in the formulation of drilling mud. From fig 3.9 and fig 3.11 the chemical analysis has shown that the major chemical constituent of NC1 and NC3 were silicon as it contained 48.0 wt% and 46.0 wt% silicon respectively which were likely due to the associated quartz, critobalite and kaolinite phases identified in the XRD result (Monger & Kelly, 2002), while from fig. 3.6, the major chemical constituent of NC2 was carbon as it contained 45.0 wt% carbon which was likely due to the associated calcite phases identified in the XRD result (Santos et al., 2017). Other chemical composition of NC1 included 12.6 wt% aluminum and 20.1 wt% oxygen which was likely due to the associated albite, halloysite and kaolinite phases identified in the XRD result (Maj & Matus, 2023). Other chemical composition of NC2, were 20.0 wt% silicon and 20.2 wt% oxygen which was likely as a result of the presence of quartz phase identified in the XRD result (Jusnes, 2020). Also other chemical composition of NC3 included 7.4 wt% oxygen which are likely due to the presence of quartz phase identified in XRD analysis (Jusnes, 2020). Other compositions such as calcium, magnesium, iron, potassium, nitrogen and gold which are present in varying quantities in NC1, NC2 and NC3 collectively were likely due to impurity associated with Nteje clay. The dominant composition of the foreign bentonite was silica which was present up to 50.0 wt% followed by carbon which was 20.4 wt% and calcium which was 15.3 wt%. These show that the material is a calcium-silicate material. The silica-calcium ratio of the material was 3.27, this is typical of a montmorillonite clay group which bentonite belongs. (Naqi & Jang, 2019). These are likely contributed by associated mixed mineral phases in the bentonite or chemical additives added to the processed bentonite to improve its rheological characteristic so as to make it suitable for drilling fluid application.

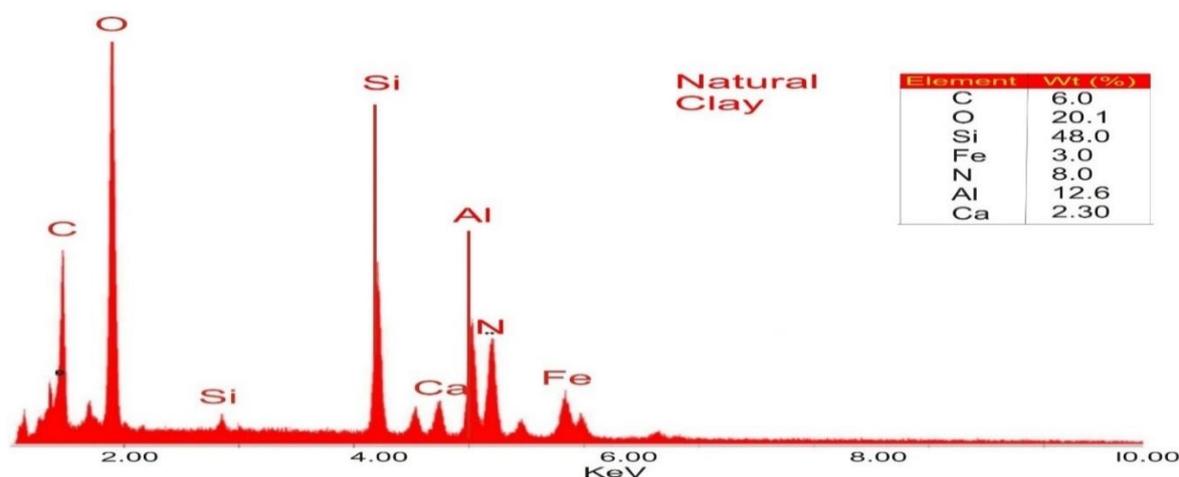


Figure 3.9. Energy dispersive spectroscopy of NC1



Figure 3.10. Energy dispersive spectroscopy of NC2

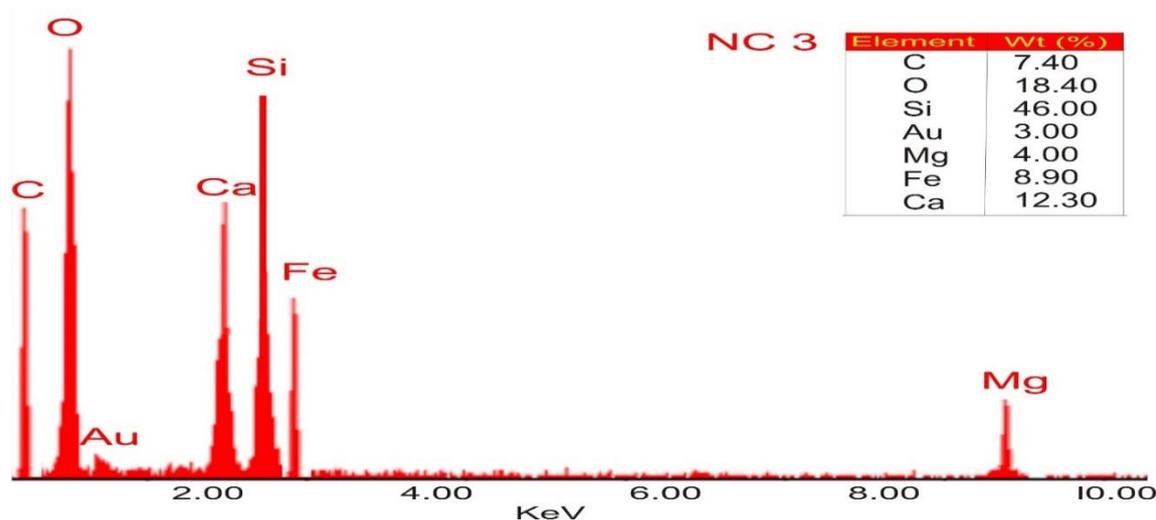


Figure 3.11. Energy dispersive spectroscopy of NC3

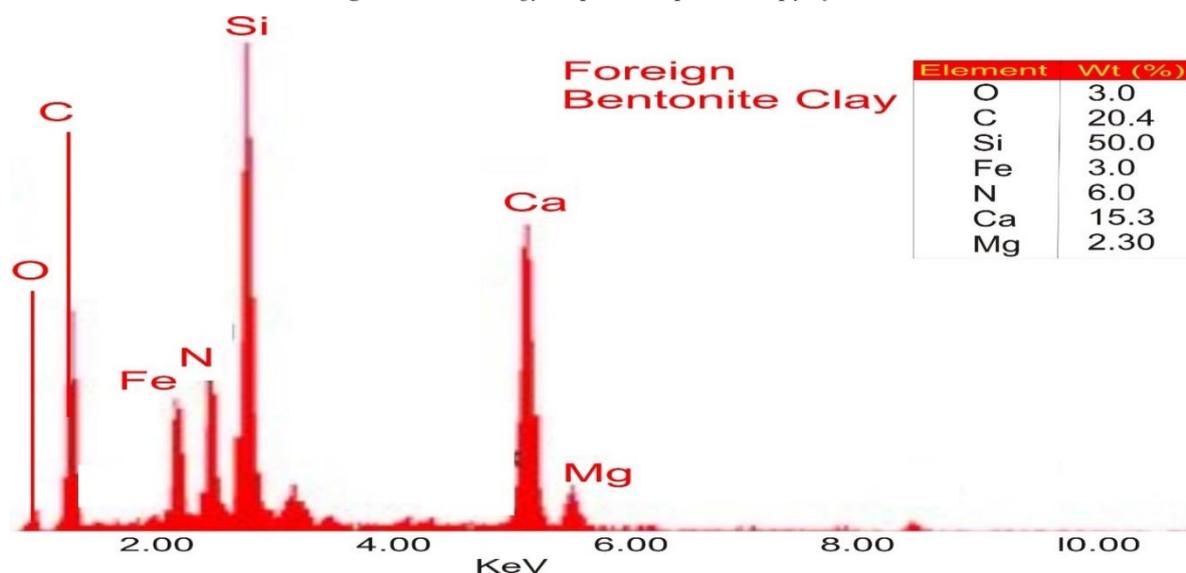


Figure 3.12. Energy dispersive spectroscopy of foreign bentonite

3.5. XRF Analysis of clay samples

Table 3.4 presents XRF results of both beneficiated and un-beneficiated Nteje clay (NC1, NC2, and NC3) and a foreign bentonite sample. From the table 3.4, NC3 shows the highest SiO₂ content (61.70%), aligning well with the API standard (58–64%), indicating good structural framework and potential for swelling (Murtaza, et al., 2020). NC1 and NC2 also fall within range but are slightly lower. However, NC1 and NC2 have Al₂O₃ contents of 27.50% and 30.27%, respectively, exceeding the API range (18–21%), which could affect the clay’s plasticity and dispersion in mud systems (Ali & Mohammed, 2024). NC3 with Al₂O₃ of 17.80% and Al₂O₃:SiO₂ ratio of 0.29 are close to ideal, supporting better rheological behavior. Fe₂O₃ levels in all local clays (NC1, NC2, and NC3) exceed the API limit (2.5–2.8%), with NC3 at 3.50% and foreign bentonite at 4.32%, potentially leading to undesirable effects like redox instability or higher abrasiveness (Mahamadou et al., 2024). CaO content in NC2 (1.45%) and NC3 (1.30%) also exceeds the API maximum (1.0%), suggesting possible flocculation issues that can affect mud stability (Biwott, et al., 2019). Furthermore, NC clays have deficient Na₂O levels (0.25–0.47%) compared to foreign bentonite (1.85%) and the API range (1.5–2.7%), indicating poor natural swelling and low exchange capacity, which are critical for viscosity development (Banganayi & Nyembwe, 2020). NC3 has a TiO₂ content of 1.23%, higher than foreign bentonite (0.70%), potentially affecting color and thermal stability (Napruszewska et al., 2024). Overall, while NC3 shows the most promise due to its balanced SiO₂ and Al₂O₃ levels, its low Na₂O and high Fe₂O₃ and CaO content suggest that further modification or enhancement through additives is needed for effective mud engineering applications.

Table 3.4. XRF results of clay samples

Element	NC1	NC2	NC3	Foreign bentonite	API Standard
SiO ₂	54.72	55.35	61.70	59.95	58 - 64
Al ₂ O ₃	27.50	30.27	17.80	19.20	18 - 21
Fe ₂ O ₃	2.82	3.60	3.50	4.32	2.5 - 2.8
TiO ₂	0.53	1.05	1.23	0.70	
CaO	0.20	1.45	1.30	2.02	0.1 - 1.0
P ₂ O ₅	0.03	0.01	-	0.30	
K ₂ O	0.80	0.98	0.90	0.34	0.2 - 0.4
MnO	0.01	0.01	0.01	0.04	
MgO	0.60	0.44	0.50	3.55	2.5 - 3.2
Na ₂ O	0.47	0.3	0.25	1.85	1.5 - 2.7
Al ₂ O ₃ :SiO ₂	0.50	0.55	0.29	0.32	0.31 - 0.38

3.6. Effect of additives (CMC) on viscosity (Dial reading @ 600rpm) of various mud samples

Figure 3.13 examines the impact of Carboxymethyl Cellulose (CMC) on the viscosity of various mud samples, including NC1, NC2, NC3, and foreign bentonite, using dial readings at 600 rpm. The API standard requires a minimum viscosity of 30 cp for effective drilling performance. At 0.2 g CMC, only NC3 and foreign bentonite meet this threshold. As CMC concentration increases, all samples show improved viscosity, but only NC3 and foreign bentonite consistently exceed the API standard. NC1 and NC2, despite showing gradual improvements, remain below the required 30 cp even at 0.8 g CMC. Foreign bentonite demonstrates the highest viscosities at all concentrations, peaking at 136 cp, indicating excellent compatibility with CMC. NC3 also shows strong compatibility, reaching 71 cp at the highest concentration. These results align with findings from Brito et al. (2018), Anthony et al. (2020), and Shuwa and Sabiu (2019), highlighting NC3 as the most effective local mud sample when combined with CMC. In contrast, NC1 and NC2 show limited performance enhancement, highlighting the variation in additive compatibility among mud formulations

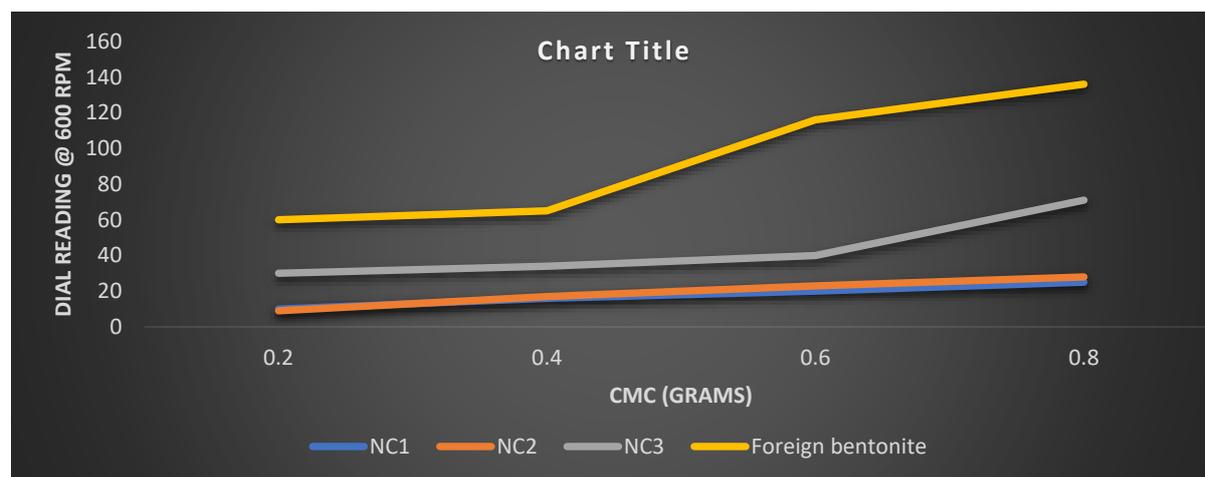


Figure 3.13. Effect of additives (CMC) on viscosity (Dial reading @ 600rpm) on various mud samples

3.7. Effect of additives (PACR) on viscosity (Dial reading @ 600rpm) of various mud samples

Figure 3.14 analyzes the impact of Polyanionic Cellulose Regular Grade (PACR) on the viscosity of NC1, NC2, NC3, and foreign bentonite mud samples using 600 rpm dial readings, compared to the API minimum of 30 cp. At 0.2 g PACR, only NC3 (38 cp) and foreign bentonite (63 cp) meet the standard, while NC1 and NC2 fall short. As PACR concentration increases, all samples show improved viscosities. At 0.4 g, NC3 and foreign bentonite maintain strong performance, while NC1 and NC2 remain below standard. At 0.6 g, NC2 and NC3 meet or exceed the threshold, and only NC1 stays slightly below. At 0.8 g, all local clays, including NC1, surpass 30 cp, with NC3 achieving 90 cp and foreign bentonite reaching 161 cp. These results confirm PACR's effectiveness in enhancing the rheological performance of local muds, particularly NC3, which consistently exceeds the API standard across all concentrations. The findings align with previous research by Udeagbara et al. (2019), Michael (2023), and Anabraba and Wami (2019), which all observed similar viscosity improvements in local clays with PACR. This highlights PACR's value as a cost-effective viscosifier for local drilling mud formulations.

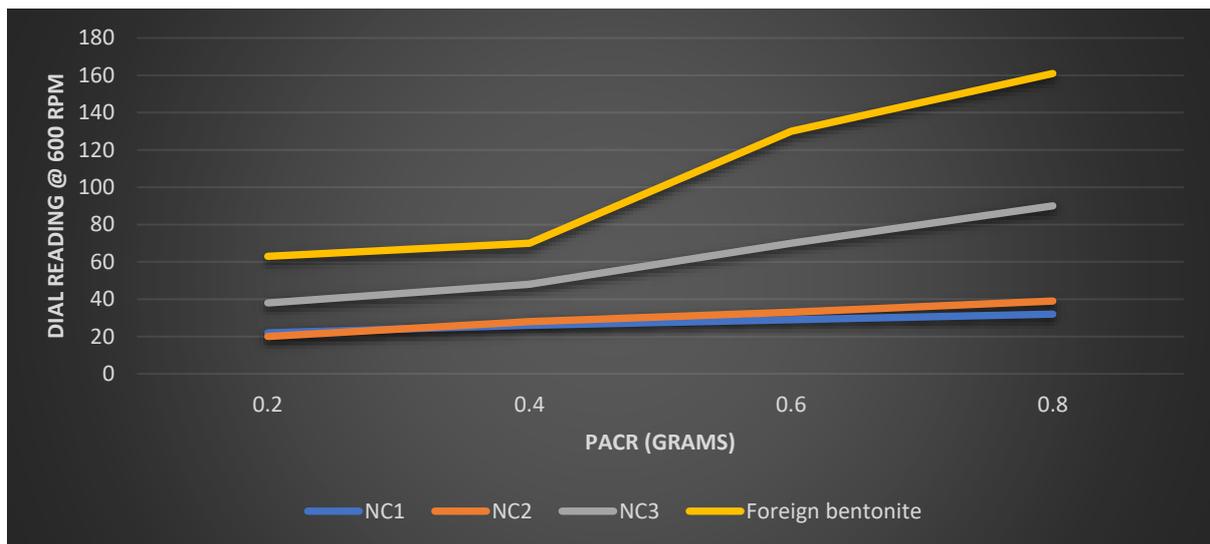


Figure 3.14. Effect of additives (PACR) on viscosity (Dial reading @ 600rpm) on various mud samples

3.8. Effect of additive (CMC) on gel strength @ 10 SEC and 10 mins on various mud samples

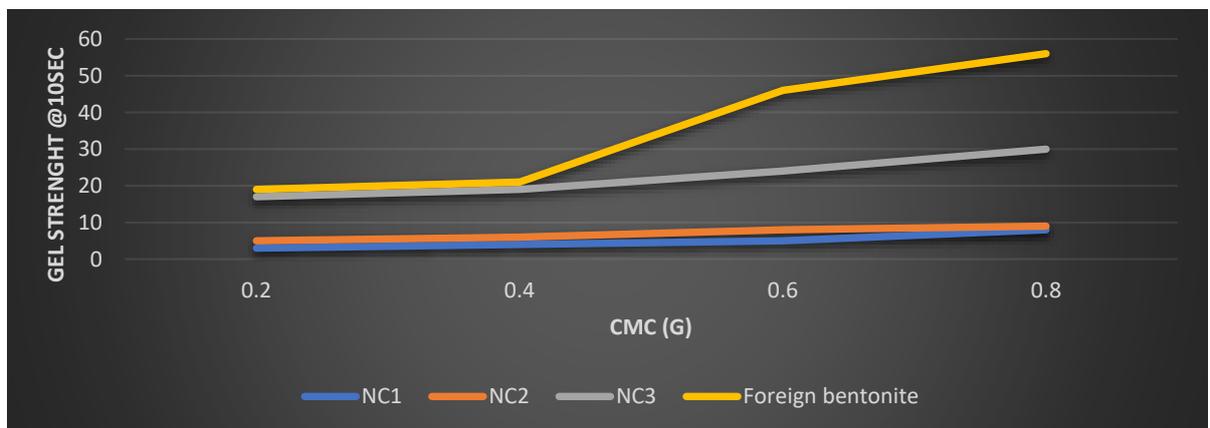


Figure 3.15. Effect of additives (CMC) on GEL STRENGTH @ 10 SEC on various mud samples

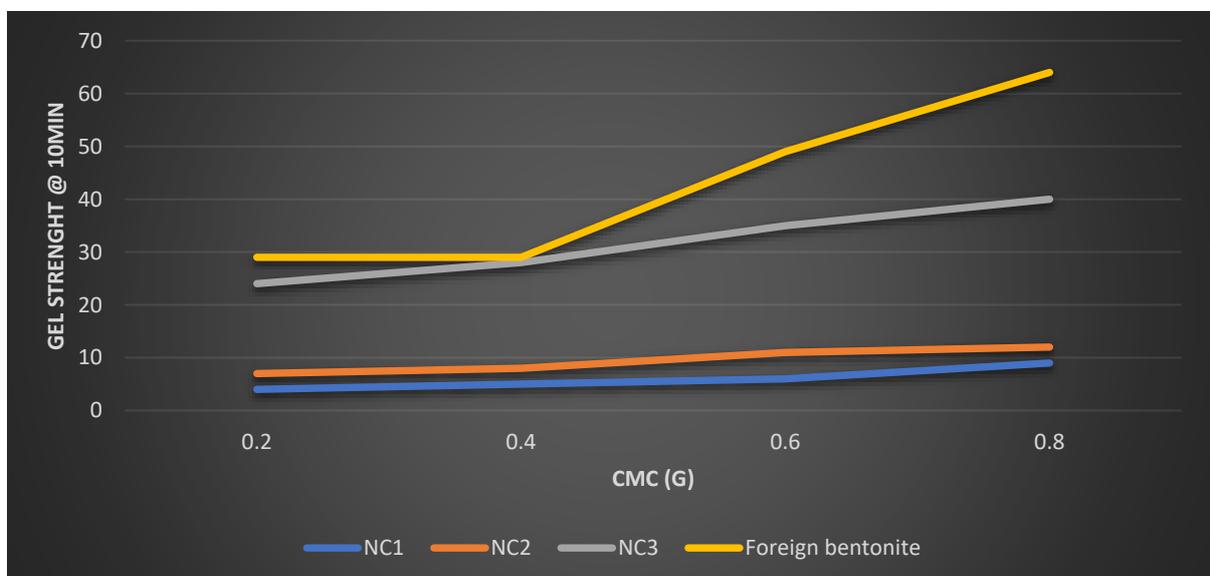


Figure 3.16. Effect of additives (CMC) on GEL STRENGTH @ 10 min on various mud samples

Figures 3.15 and 3.16 evaluate the effect of CMC on the gel strength of mud formulations of NC1, NC2, NC3, and foreign bentonite at concentrations of 0.2, 0.4, 0.6, and 0.8 g. Gel strengths at 10 seconds and 10 minutes increased with rising CMC concentrations. Foreign bentonite consistently exhibited the

highest values, with NC3 closely matching its performance, especially at higher CMC levels. NC2 showed moderate improvement, while NC1, though enhanced, remained the least effective. At 0.8 g CMC, 10-second gel strengths reached 8, 9, 30, and 56 lb/100ft² for NC1, NC2, NC3, and foreign bentonite, respectively; the 10-minute strengths were 9, 12, 40, and 64 lb/100ft² respectively. The findings align with studies by Shuwa & Sabiu (2019), Anthony et al. (2020), and Brito et al. (2018), confirming CMC's effectiveness. NC3 stands out as the most promising local mud, showing gel strength comparable to foreign bentonite.

3.9. Effect of additives (PACR) on gel strength @ 10 SEC and 10 mins on various mud samples

Figures 3.17 and 3.18 illustrate the effect of PACR on the gel strength (10 sec and 10 min) of NC1, NC2, NC3, and foreign bentonite muds at 0.2 to 0.8 g concentrations. Gel strength improved with increasing PACR, with foreign bentonite showing the highest values, followed closely by NC3. At 0.8 g PACR, 10-second gel strengths were 9, 15, 43, and 60 lb/100ft², and 10-minute strengths were 11, 19, 54, and 75 lb/100ft² for NC1, NC2, NC3, and foreign bentonite, respectively. NC3 consistently matched foreign bentonite's performance, especially at higher concentrations. NC2 showed steady improvements, while NC1, though improved, remained least effective. These results align with Udeagbara et al. (2019) and Anabraba & Wami (2019), who also reported enhanced gel strength in local clays using PACR. Overall, PACR proves effective in improving gel strength, with NC3 emerging as a strong, cost-effective local alternative to foreign bentonite for drilling operations.

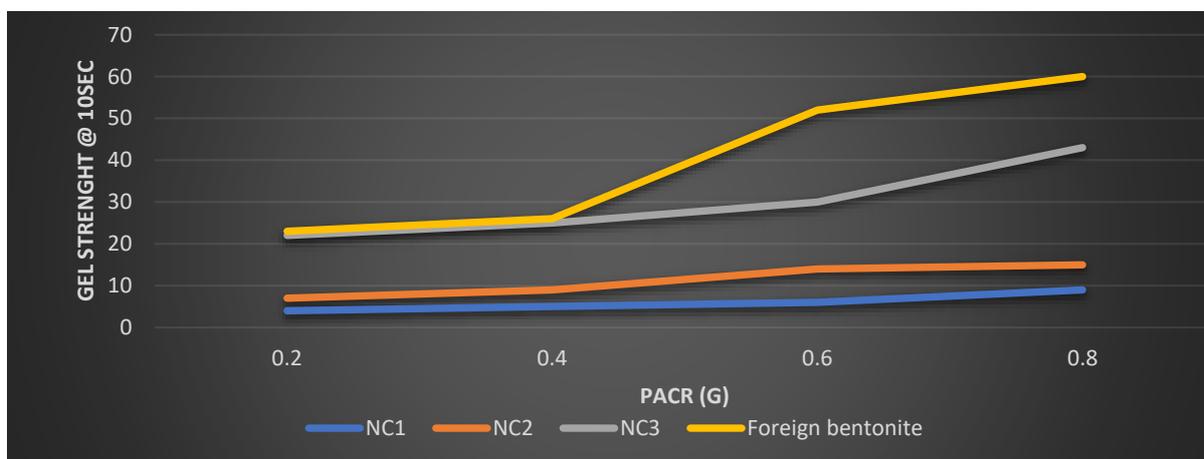


Figure 3.17. Effect of additives (PACR) on GEL STRENGTH@ 10 SEC on various mud samples

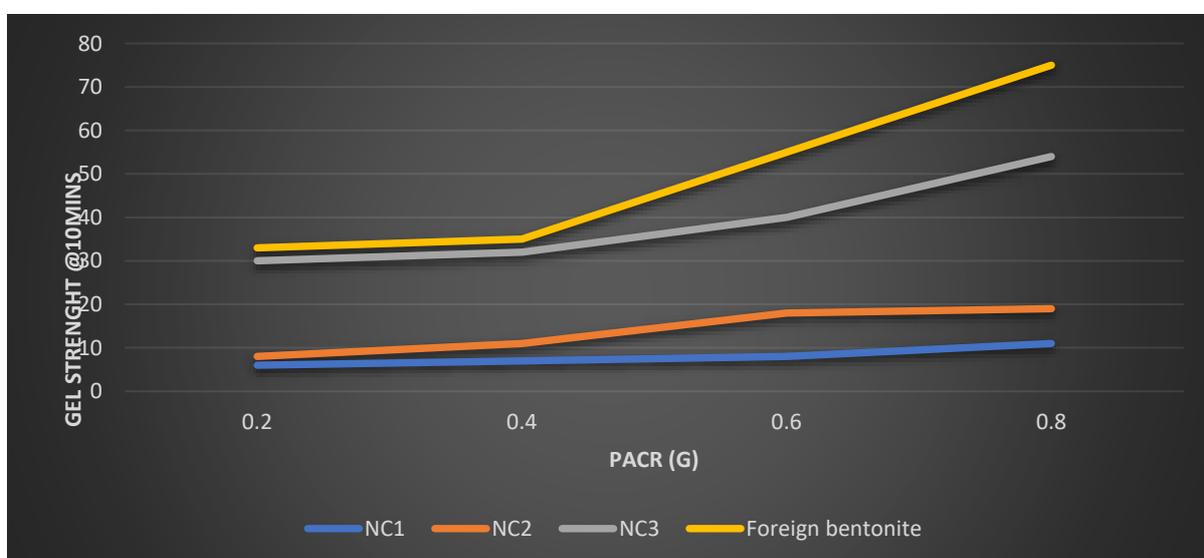


Figure 3.18. Effect of additives (PACR) on GEL STRENGTH@ 10 min on various mud samples

3.10. Effect of additives (CMC) on apparent viscosity of various mud samples

Figure 3.19 illustrates the impact of varying CMC concentrations on the apparent viscosity of different mud samples. An overall increase in viscosity is observed with higher CMC levels, though the response

varies by mud type. NC1 shows a steady rise from 5 to 12.5, indicating uniform polymer dispersion. NC2 increases from 4.5 to 14, with stronger effects at higher concentrations, suggesting enhanced particle-polymer interactions.

NC3 exhibits a sharp increase from 15 to 35.5, indicating strong CMC compatibility and possible flocculation effects. Foreign Bentonite shows the highest rise, from 30 to 68, reflecting a strong synergistic effect likely due to its high montmorillonite content. These findings align with Micheal et al. (2022) and Apugo-Nwosu et al. (2011), who reported similar improvements using CMC in local clays. Overall, the results highlight the importance of mud composition in determining the effectiveness of CMC in enhancing drilling fluid rheology.

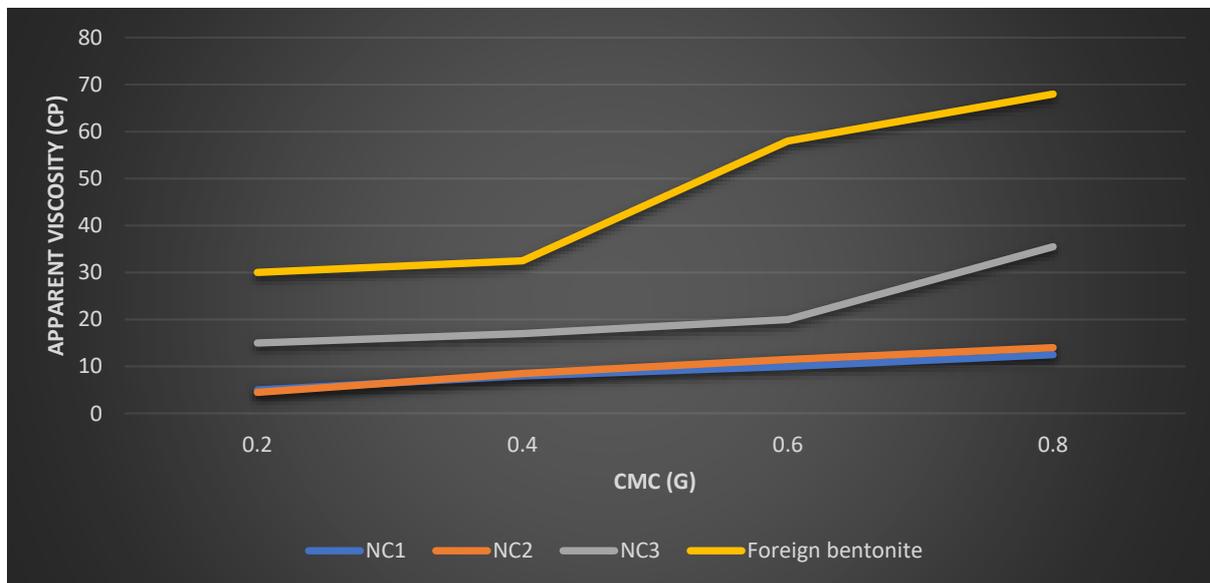


Figure 3.19. Effect of additives (CMC) on apparent viscosity of various mud samples

3.11. Effect of additives (PACR) on apparent viscosity of various mud samples

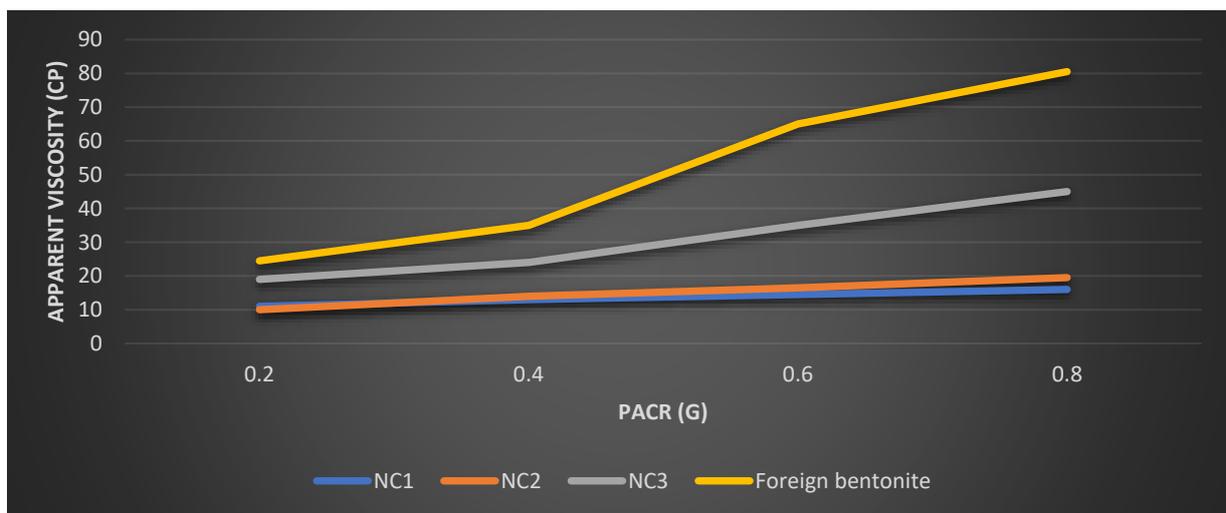


Figure 3.20. Effect of additives (PACR) on apparent viscosity of various mud samples

Figure 3.20 shows the effect of PACR on the apparent viscosity of NC1, NC2, NC3, and Foreign Bentonite mud samples. Apparent viscosity increased with PACR concentration across all samples, though the degree varied. NC1 and NC2 showed steady increases from 11cp to 16cp and 10cp to 19.5cp respectively, indicating consistent polymer interaction and predictable rheological enhancement. NC3 exhibited a sharp rise from 19cp to 45cp, suggesting strong polymer-particle interactions and effective gel structure formation. Foreign Bentonite showed the most significant increase, from 31.5cp to 80.5cp, likely due to its high montmorillonite content, which enhances PACR bonding and gel network development. These trends align with previous studies: Udeagbara et al. (2019) on Ebonyi clay, Olatunji

et al. (2012) on foreign bentonite, and Micheal et al. (2022) on Imo clay, all confirming PACR's efficacy in improving drilling fluid viscosity. The results highlight PACR's potential in tailoring mud systems for enhanced drilling performance.

3.12. Effect of additives (CMC) on yield point of various mud samples

Figure 3.21 shows the effect of CMC on the yield point of NC1, NC2, NC3, and Foreign Bentonite muds. Yield point, a key indicator of a mud's suspension capacity, increased with CMC concentration but varied by sample. NC1 and NC2 showed low initial values, peaked at 0.6% CMC (10 and 11.5, respectively), then slightly dropped at 0.8%, indicating a threshold beyond which CMC becomes less effective. NC3 exhibited a steady and sharp increase from 10 to 53, reflecting strong CMC compatibility and superior gel strength. Foreign Bentonite showed the highest yield points (40–98), demonstrating its inherent gel-forming capacity, further enhanced by CMC. These findings align with Safi et al. (2016), Abdou & Abuseda (2014), and Shuwa & Sabiu (2019), confirming CMC's ability to boost suspension properties. While higher yield points improve cuttings transport and wellbore stability, excessively high values may increase energy demands, requiring careful formulation balance.

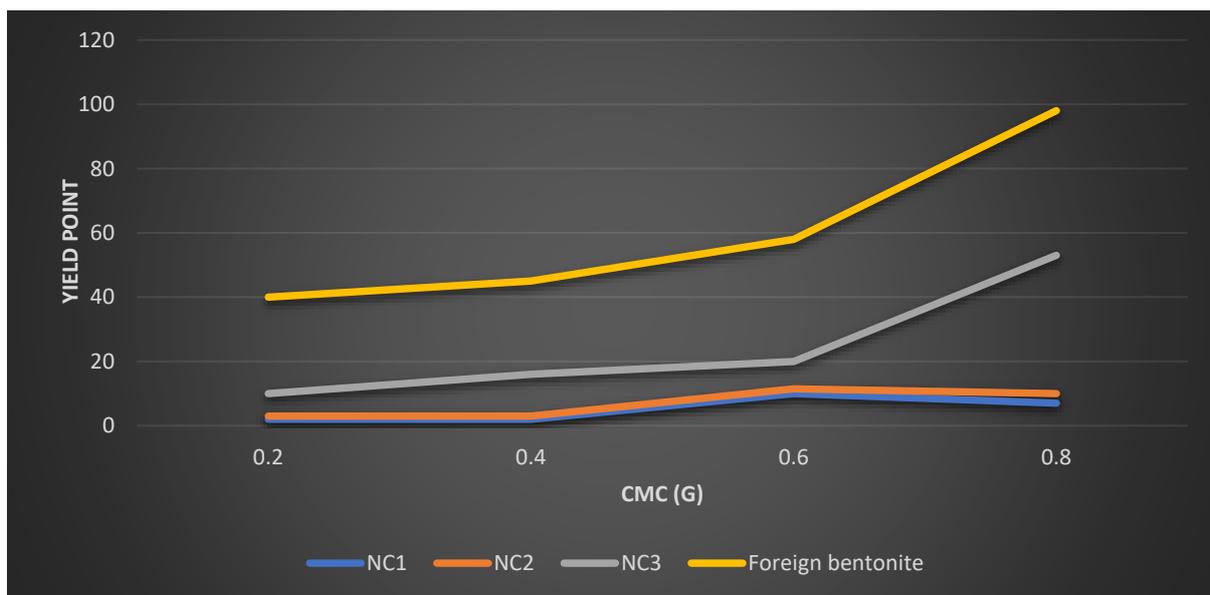


Figure 3.21. Effect of additives (CMC) on yield point of various mud samples

3.13. Effect of additives (PACR) on yield point of various mud samples

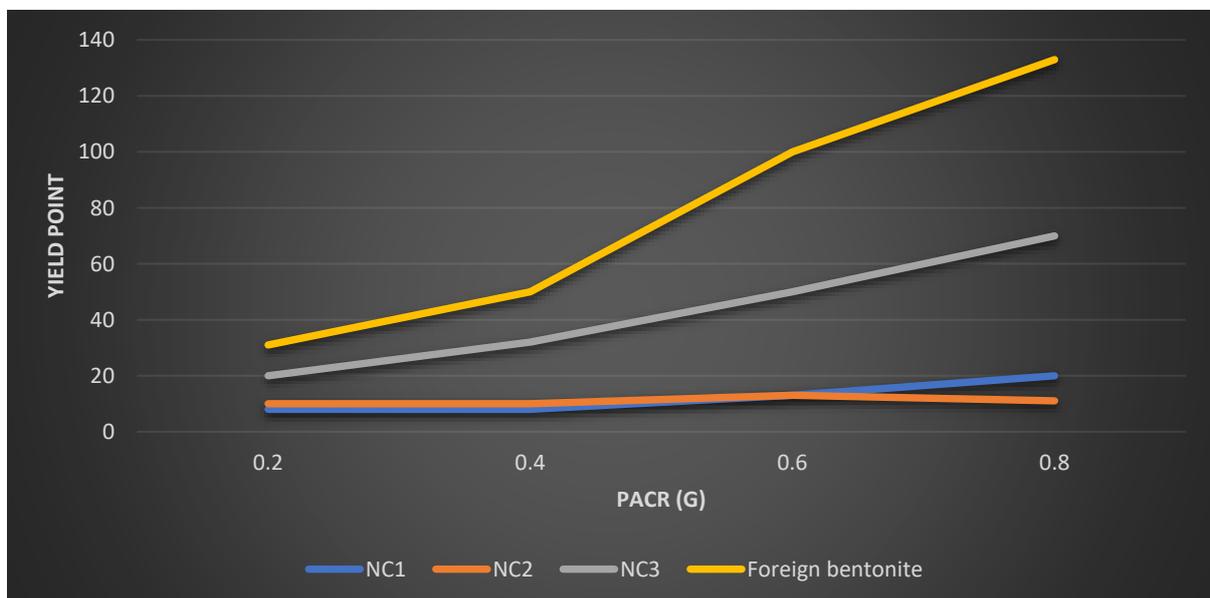


Figure 3.22. Effect of additives (PACR) on yield point of various mud samples

Figure 3.22 illustrates the influence of PACR on the yield point of NC1, NC2, NC3, and Foreign Bentonite muds. Yield point, a key indicator of suspension and gel strength, increased with PACR concentration in most samples. NC1 showed a steady rise from 8 to 20, indicating a threshold at 0.6% where PACR significantly enhances interparticle structure, consistent with Charles and Adetokunbo (2017). NC2 remained stable at lower concentrations, peaked at 13, then declined slightly at 0.8%, suggesting limited compatibility or oversaturation effects. NC3 showed a strong response, rising from 20 to 70, reflecting excellent interaction with PACR and superior suspension performance (Mahmoud, 2021). Foreign Bentonite displayed the highest yield points (45–133), due to its rich montmorillonite content (Deng, 2018), with PACR further strengthening its gel structure. These results, supported by Udeagbara et al. (2019), show PACR's effectiveness but also highlight the need to balance yield point to avoid excessive pump pressures (Yang et al., 2022).

3.14. Fluid loss and filter cake thickness of mud samples blended with 0.8g of CMC

Table 3.5 presents the filtration performance of water-based mud (WBM) systems formulated with 0.8 g of CMC using NC1, NC2, NC3, and foreign bentonite. **Fluid loss** is a critical parameter in drilling mud as it reflects the mud's ability to prevent filtrate invasion into the formation (Ali et al., 2022). NC1 and NC2 exhibit high fluid losses of 52 ml and 35 ml respectively, exceeding the **API maximum limit of 15 ml**, which indicates poor fluid loss control (Jia et al., 2017). In contrast, **NC3 (11 ml)** and **foreign bentonite (8.5 ml)** fall within acceptable limits, showing improved performance. Similarly, **filter cake thickness**, which affects wellbore stability and risk of pipe sticking (Cook et al., 2016), is excessive in NC1 (11.11 mm) and NC2 (7.94 mm), but significantly reduced in NC3 (2.38 mm) and foreign bentonite (1.59 mm), the latter meeting the **API standard (<2 mm)**. These results imply that **wet beneficiation (NC3)** greatly enhances the filtration control of local clay, making it a viable, eco-friendly alternative to imported bentonite in drilling fluid formulations.

Table 3.5. Filtration loss and filter cake properties of WBM formulated with 0.8g of CMC in various NC1, NC2, NC3 and foreign bentonite

Parameter	NC1	NC2	NC3	Foreign bentonite	API Standard
Fluid loss (ml)	52	35	11	8.5	15.0 ml Max
Filter cake Thickness (mm)	11.11	7.94	2.38	1.59	< 2mm

3.15. Fluid loss and filter cake thickness of mud samples blended with 0.8g of PACR

Table 3.6 shows the performance of water-based mud (WBM) systems formulated with 0.8 g of **Polyanionic Cellulose Regular (PACR)** using NC1, NC2, NC3, and foreign bentonite. In drilling operations, **fluid loss control** and **filter cake quality** are critical for formation protection and borehole stability (Siddig et al., 2020). NC1 and NC2 exhibited high fluid loss values (47 ml and 25 ml), exceeding the **API limit of 15 ml**, indicating poor filtration control and higher risk of formation damage (Krueger, 1986). However, **NC3 (9 ml)** and **foreign bentonite (8 ml)** complied with API standards, demonstrating effective fluid retention (Caenn et al., 2011). Filter cake thickness followed a similar trend: NC1 (9.53 mm) and NC2 (5.56 mm) formed thick cakes, which can lead to **differential sticking** and reduced wellbore quality (Cook et al., 2016). In contrast, NC3 (1.59 mm) and foreign bentonite (0.79 mm) produced thin cakes, meeting the **API standard (<2 mm)**. These results indicate that **wet beneficiation (NC3)** significantly improves the suitability of local clay for drilling mud, offering a cost-effective and environmentally friendly alternative to imported bentonite when enhanced with PACR.

Table 3.6. Filtration loss and filter cake properties of WBM formulated with 0.8g of PACR in various NC1, NC2, NC3 and foreign bentonite

Parameter	NC1	NC2	NC3	Foreign bentonite	API Standard
Fluid loss (ml)	47	25	9	8	15.0 ml Max
Filter cake Thickness	9.53	5.56	1.59	0.79	< 2mm

4. CONCLUSION

This study comprehensively evaluated the effect of wet and thermal beneficiation on Nteje clay for drilling fluid applications. The results indicate that untreated Nteje clay (NC1) exhibited poor rheological and filtration properties, rendering it unsuitable for drilling mud without modification. Thermal beneficiation (NC2) improved the structural compactness and crystallinity, enhancing gel

strength and reducing fluid loss moderately. However, wet beneficiation (NC3) demonstrated the most promising improvements, yielding enhanced hydration capacity, reduced fluid loss (11 ml with CMC; 9 ml with PACR), and thinner filter cakes (<2 mm), which complied with API standards. SEM and XRD analyses confirmed mineralogical improvements, while XRF data revealed that NC3 had the most favorable SiO₂ and Al₂O₃ balance. Though NC3 still underperforms compared to commercial bentonite in some parameters (e.g., viscosity, yield point), its performance was significantly boosted when combined with CMC and PACR. Therefore, wet-beneficiated Nteje clay presents a sustainable and cost-effective local alternative for partial replacement of imported bentonite in water-based drilling fluid systems, particularly when supported by suitable additives.

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