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Thermochemical Treatment of Nigerian Raw Clays for Oil and Gas Drilling Operations

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Abstract: Sodium-based bentonite is used for drilling operations because of its high swelling capacity. This type of bentonite clay is not sourced locally in many oil- and gas-producing nations. However, low-swelling clays (calcium- and potassium-based) are in abundant quantities in most of these countries. Hence, there is a need to convert low-swelling bentonite clays to sodium-based bentonite. The method used to convert low-swelling clays is more applicable to calcium-based bentonite. This research investigated a thermochemical treatment method that converted potassium-based bentonite to sodium-based bentonite. The raw clay materials were sourced from Pindinga (P) and Ubakala (U) clay deposits in Nigeria. An X-ray diffractometer (XRD), an energy dispersive X-ray (EDX), and a scanning electron microscope (SEM) were used to characterize the raw clay samples. Mud slurry was prepared by mixing 22 g of the local raw clays, 3 wt.% soda ash, and MgO at concentrations between 1 and 3 wt.% and heating at 90 °C. The result showed that the viscosities of samples P and U increased from 6 to 26 and 8 to 35.5 cP before and after thermochemical treatment, respectively. Also, due to the thermochemical treatment, the samples' yield point, consistency factor, consistency index, and thixotropy behavior were all significantly improved.

Keywords: thermochemical treatment; bentonite clay; beneficiation; drilling mud; viscosity; rheological properties

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1. Introduction

Drilling fluid is any fluid used to carry cuttings from the wellbore to the surface during drilling operation [1,2]. The successful completion of oil and gas wells is highly dependent on the properties of drilling fluids [3–5]. The rheological properties of drilling fluid are one of the major properties of the fluid and are responsible for the suspension and carrying of cuttings. Water-based mud (WBM) is one of the most common types of drilling fluid. It is prepared by mixing water, clay, and additives. The American Petroleum Institute (API) stipulates that any WBM used to drill wells should have a minimum viscosity of 30 cP at 600 rpm. Meeting this requirement depends largely on the type of bentonite clay used to prepare the drilling fluid. There are three types of bentonite clay, namely potassium-based, sodium-based, and calcium-based bentonite. Potassium-based and calcium-based bentonite have very low swelling capability [6–9]. When these types of clays are used to prepare WBM, the viscosity of the mud is usually poor. Sodium-based bentonite clay on the other hand has a high swelling capability [10]. Hence, they are the type of clay used during drilling operations.

The majority of bentonite clay in Nigeria is either calcium- or potassium-based [2]. Numerous studies have been conducted to improve the swelling capacities of clay sourced from Nigeria using three different techniques. The first technique involved beneficiating the raw clays with sodium carbonate [11]. This was performed by mixing the raw clays with sodium carbonate to convert the clays to sodium-based bentonite. However, this method could not improve the swelling capacity of the clays as the authors did not subject the raw clays to the conditions necessary for ion exchange to take place between the clay and sodium carbonate. The factors necessary for the thermochemical treatment of low-swelling clay include temperature (70–90 °C), the concentration of bentonite clay, and the concentration of sodium carbonate. The second method involved the addition of viscosifiers such as carboxyl methyl cellulose, poly-anionic cellulose, hydroxyl ethyl cellulose, and gum arabic to improve the rheological properties of the mud prepared using the local raw clays [12–14]. This method improved the rheological properties of the mud. This occurred because of the addition of viscosifier to the mud samples. The third method involved the combination of the first two methods [15–19]. Researchers resorted to using this technique as a result of the failure recorded using the first technique [20].

There is still a need to treat the local raw clays through an activation route that will not involve the addition of a viscosifier. Thermochemical treatment of low-swelling clays is an activation route that does not involve the use of a viscosifier. It can be used to convert calcium- or potassium-based bentonite to sodium-based bentonite. It involves the use of heat and chemical (soda ash) to beneficiate the clay. The application of heat during this process is used to decompose the sodium carbonate into sodium oxide and carbon dioxide [21–23]. Calcium ions in the low-swelling clay will then be exchanged for sodium ions in sodium oxide [21]. Magzoub et al. [10] looked into how heat, agitation, and sodium carbonate affected the rheological and swelling capabilities of calcium-based bentonite colloidal dispersions. The authors noted that the slurry's viscosity increased and satisfied the minimum level required by API after being heated and stirred simultaneously. The limitation of Magzoub et al. [10] was the length of time (6 h) needed to treat the clay. Also, this method of clay activation cannot be used to improve the swelling behavior of potassium-based bentonite [21]. This is because sodium (from sodium oxide) cannot displace the potassium present in potassium-based bentonite clay as a result of the positions of sodium and potassium in the electrochemical series.

Magzoub et al. [22] investigated the swelling kinetics of calcium-based bentonite clay using particle size analysis. The swelling of the clay was shown to be a kinetically controlled process that depended on time, temperature, sodium carbonate, and bentonite composition. It followed a second-order reaction. Mahmoud et al. [23] beneficiated calcium-based bentonite clay using sea water and soda ash. The bentonite was treated initially with sea water under simultaneous heating and stirring conditions. This process did not improve the swelling capacity of the clay. However, after the addition of soda ash to the mud, the swelling properties of the clay improved.

The method used by Magzoub et al. [22] and Mahmoud et al. [23] also had the same setbacks stated with the method used by Magzoub et al. [10]. There is still the need to utilize a thermochemical treatment method that can be used to beneficiate potassium-based bentonite. This treatment method will involve the use of reagents such as magnesium oxide. This will cause the substitutions of ions within the structure of the clay. Magnesium (from MgO) will substitute aluminum in the octahedral unit of the clay. This substitution process will create unbalanced charges that will attract sodium ions to the interlayer units of the clay [21,24,25]. When this occurs, the clay will be converted to sodium-based bentonite depending on the amount of sodium ions in the clay. This research, therefore, will employ this thermochemical treatment route for the improvement of the rheological properties of Nigerian raw clays (potassium-based).

2. Materials and Methods

2.1. Materials and Equipment

The materials used for the experiment include local raw clays, soda ash (Merck—99.9% purity), magnesium oxide (Merck—99.9% purity), distilled water, a spatula, a beaker (500 mL), and a measuring cylinder (1000 mL). The equipment used for the experiment include a scanning electron microscope (SEM) (Model-EVO®, Carl Zeiss Pvt. Ltd., Cambridge, UK), an energy dispersive X-ray (EDX) (Model-EVO® Carl Zeiss Pvt. Ltd., Cambridge, UK), an X-ray diffractometer (XRD) (Bruker-D8), a variable mud mixer (Ofite, Model 9B), a viscometer (Ofite, Model 800), a magnetic stirrer, an oven (U-Test GENO, DT104A), a jaw crusher (Retsch, BB 50), a weighing balance (OHAUS, Model AX124), and a mud balance (Ofite).

2.2. Material Collection and Preparation

Two locations in Nigeria were used to gather the regional raw clays. They were gathered in Ubakala (latitude 5°30′41″ N and longitude 7°28′32″ E), Abia State, and Pindinga (latitude 9°59′4″ N and longitude 10°57′8″ E), Gombe State. Prior research has demonstrated that the raw clay materials from these locations have subpar rheological properties [18,26–28]. The raw clay samples were taken at a depth of 1 m, and the moisture content was reduced by oven drying for 16 h at 70 °C. The dried clay samples were sieved using an API standard mesh size of 75 microns and crushed using a crusher [10].

2.3. Sample Characterization

The fine local raw clay materials were characterized using SEM, EDX, and XRD. A SEM was used to analyze the surface of the clay samples. The clay sample was placed inside the sample chamber of the SEM. The sample chamber was placed under vacuum conditions. This was performed to remove any gas particles inside the chamber. The electron beam was turned on and incident on the clay sample inside the sample chamber using an accelerating voltage of 25 kV. The interaction between the electrons and the clay sample generated signals that defined the surface morphology of the clay sample. The model of SEM used to analyze the clay sample also contained an EDX. When the clay sample was placed in the sample chamber in the SEM, the beam of electrons that was incident on the clay sample caused the clay sample to emit X-rays. The energy of the emitted X-ray was characteristic of the elements present in the clay material. The mineralogical composition of the clay samples was analyzed using a Bruker-D8 diffractometer. Cu Kα radiation was used for the analysis. The clay sample was placed in the sample holder of the diffractometer. A beam of electrons was incident on the clay sample with an accelerating voltage of 40 kV. The beam of electrons interacted with the clay sample and dislodged into the inner shell electrons to produce X-ray spectra that were characteristic of the clay sample.

2.4. Sample Preparation

The local raw clay samples were treated thermochemically using heat, soda ash (sodium carbonate), and magnesium oxide. Previous studies have shown that soda ash acts as a good source of sodium ions compared to other sodium compounds used to beneficiate low-swelling clays [10,22,23]. MgO was used to aid the substitution of ions within the structure of the clay [24]. A sample of mud was prepared by mixing 22 g of raw clay from Pindinga with 350 cc of distilled water. 3 wt.% of soda ash and 1 wt.% of MgO were added to the mud sample and mixed thoroughly. Additional two samples of mud were prepared by mixing 22 g of raw clay from Pindinga with 350 cc of distilled water. An additional two samples of mud were prepared by mixing 22 g of raw clay from Pindinga and 3 wt.% of soda ash with 350 cc of distilled water. 1 wt.% and 2 wt.% of MgO were added to the two samples respectively and mixed.

Another three samples of mud were prepared using 22 g of raw clay from Ubakala, 3 wt.% of soda ash, and varying concentrations of MgO (1–3 wt.%), as shown in Table 1. Table 1 shows the samples of mud prepared for thermochemical treatment. The mud samples prepared using Pindinga and Ubakala raw clay materials were tagged as samples P and U, respectively. The subscripts indicate the concentration of MgO and soda ash in each of the samples. The first item of the subscript (1–3) was used to indicate the level of concentration of MgO while the second item represents the fixed concentration of soda ash (3 wt.%). For example, sample $P_{1,3}$ was prepared using 22 g of Pindinga clay, 1 wt.% of MgO, and 3 wt.% of soda ash. Sample $P_{3,3}$ was prepared using 22 g of Pindinga clay, 3 wt.% of MgO, and 3 wt.% of soda ash. Each of the samples was heated at 90 °C for one hour using a magnetic stirrer.

Table 1. Mud samples prepared for thermochemical treatment.

Sample	Source of Clay	Mass of Clay (g)	Concentration of Additive	
			MgO (wt.%)	Na_2CO_3 (wt.%)
$P_{1,3}$	Pindinga	22	1	3
$P_{2,3}$		22	2	3
$P_{3,3}$		22	3	3
$U_{1,3}$	Ubakala	22	1	3
$U_{2,3}$		22	2	3
$U_{3,3}$		22	3	3

2.5. Testing Procedure

The effect of the thermochemical treatment of the local raw clays on the rheological behavior of the mud was determined using API standard procedures for mud testing [10,27,29,30]. The plastic viscosity, yield point, power law index, and consistency factor of the samples were calculated using Equation (1) to Equation (4), respectively. The shear stress to shear rate relationship of the samples before and after the thermochemical treatment was determined. Also, the thixotropy behavior of the samples before and after the thermochemical treatment was determined using the thixotropy loop test [31,32]. The elemental composition of the thermochemically treated clays was determined using EDX.

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

$$YP = \theta_{300} - PV \tag{2}$$

$$n = 3.32 \log (\theta_{600}/\theta_{300}) \tag{3}$$

$$K = \theta_{300}/(511)^{n} \tag{4}$$

where:

θ_{300} = Dial reading of the viscometer at 300 rpm;
θ_{600} = Dial reading of the viscometer at 600 rpm;
PV = Plastic viscosity;
YP = Yield point;
n = Power law index;
K = Consistency factor.

3. Discussion of Results

3.1. Characterization Results of the Local Clay Samples

The results obtained from the characterization of the clay samples are discussed in this section. Figures 1 and 2 show the surface morphologies of all the clay samples, as acquired from the SEM. The surface of all the clay samples look like a flake [33]. This is a unique characteristic of clay materials [24]. The raw clay materials contained small and large particles with irregular shapes. Table 2 shows the elemental composition of the clay samples. The clay sample from Pindinga contained the following elements: oxygen

(4.50%), magnesium (2.97%), silicon (53.26%), aluminum (33.17%), potassium (3.50%), and calcium (2.60%). Potassium was the dominant cation present in this clay. The raw clay material from Ubakala contained the following elements: oxygen (5.42%), magnesium (4.41%), silicon (39.21%), aluminum (44.50%), calcium (3.68%), and potassium (2.78%). Calcium is the dominant cation present in this clay sample. None of the local raw clay samples contained sodium.

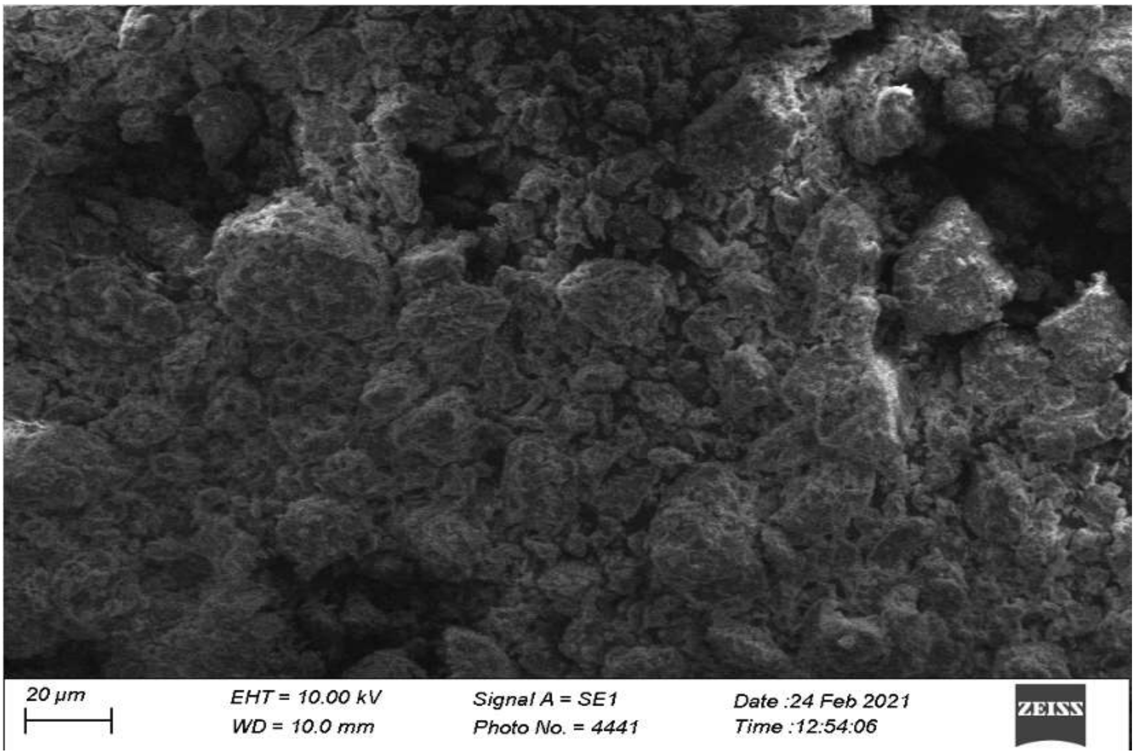

Figure 1. SEM Images of the raw clay material from Pindinga.

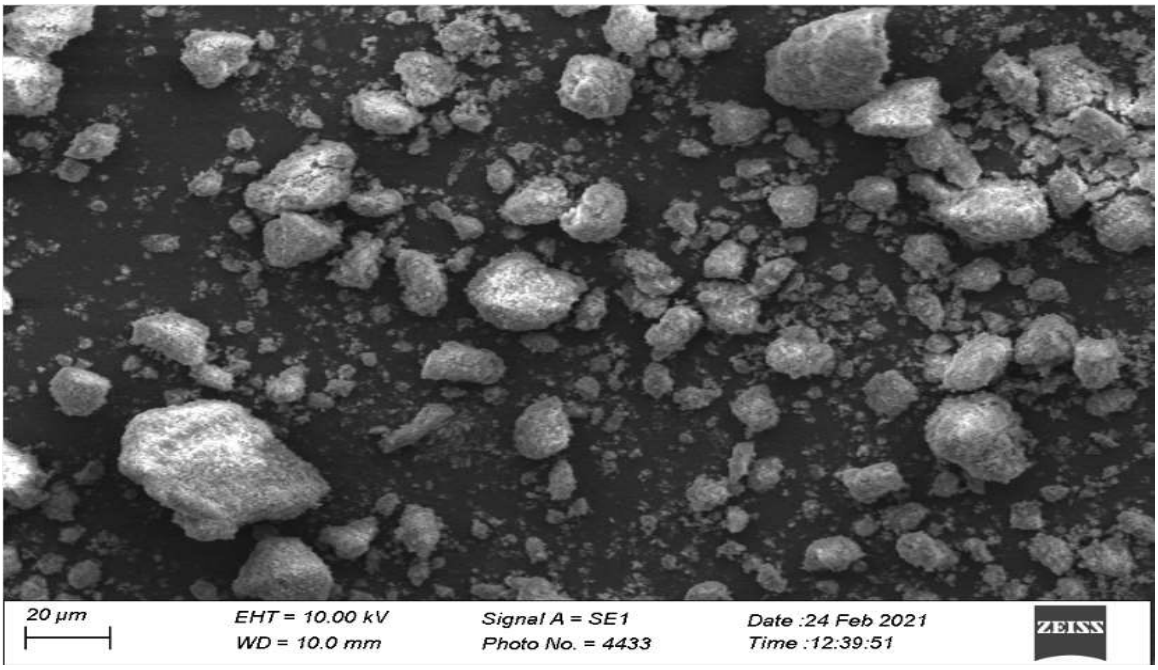

Figure 2. SEM Images of the raw clay material from Ubakala.

Table 2. Elemental composition of the clay samples before and after thermochemical treatment using EDX.

Element	Pindinga		Ubakala	
	Before	**After**	**Before**	**After**
Oxygen	4.50%	4.12%	5.42%	4.52%
Sodium	-	10.85%	-	8.24%
Magnesium	2.97%	19.49%	4.41%	35.27%
Aluminum	33.17%	21.35%	39.21%	22.39%
Silicon	53.26%	37.45%	44.50%	22.85%
Potassium	3.50%	3.57%	3.68%	3.48%
Calcium	2.60%	3.17%	2.78%	3.25%

The elemental analysis of the thermochemically treated clay is also shown in Table 2. The same elements (oxygen, sodium, magnesium, aluminum, silicon, potassium, and calcium) were found in all the treated clay samples. The thermochemical treatment of the local raw clay materials had an impact on the chemical composition of the local clay materials. Initially, the raw clay material from Pindinga and Ubakala, respectively, did not contain sodium. However, after the thermochemical treatment of the raw clays, sodium was found in the clay samples. Also, the raw clays did not contain a lot of magnesium. However, after the thermochemical treatment, the composition of magnesium increased. This was due to the substitution of ions that occurred during the thermochemical treatment [21,25]. This process created some charges within the structure of the clay that attracted sodium ions (from soda ash).

Figures 3 and 4 show the XRD analysis of all the clay samples. The raw clay material from Pindinga contained a high composition of montmorillonite and quartz, a low composition of feldspar and illite, and traces of calcite. The raw clay sample from Ubakala also contained a high composition of montmorillonite, a low composition of feldspar and quartz, and traces of illite and calcite. The elemental composition and XRD analysis of the acquired local raw clay materials showed that all the local raw clay materials were bentonite clay.

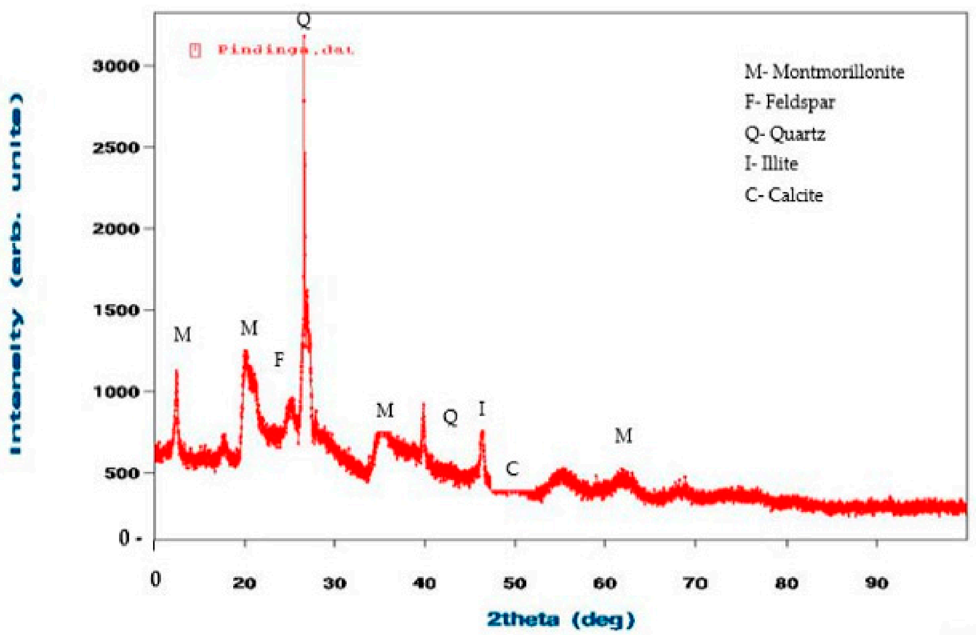

Figure 3. XRD Images of the raw clay material from Pindinga.

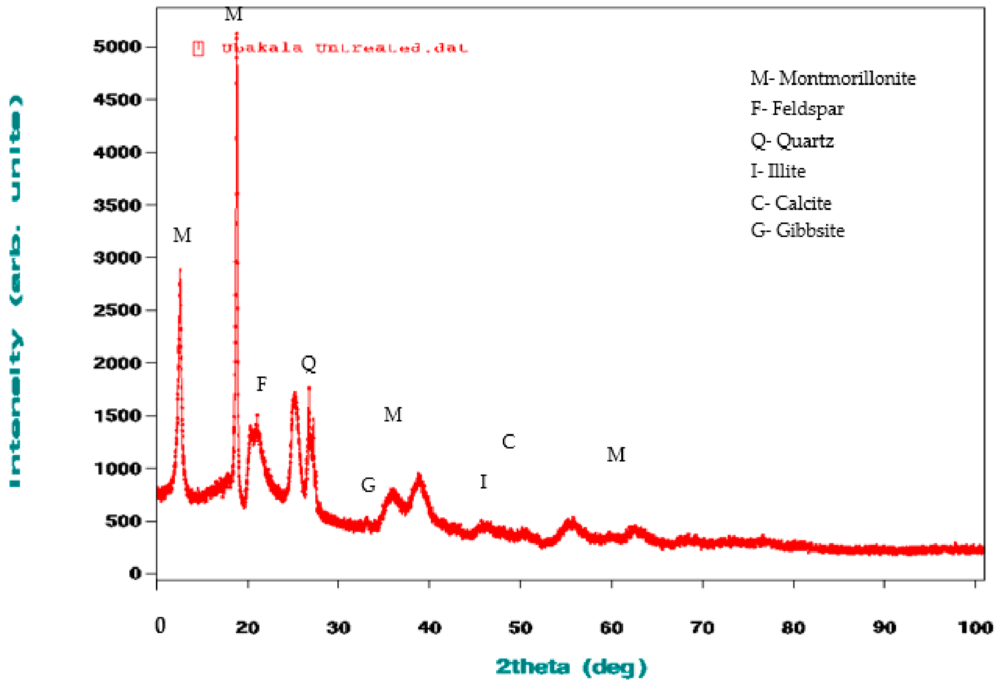

Figure 4. XRD Images of the raw clay material from Ubakala.

3.2. Effect of the Thermochemical Treatment of the Local Raw Clay Materials

This section discusses the effect of the thermochemical treatment of the local raw clay materials on the properties of water-based mud.

3.2.1. Mud Viscosity

Figure 5 shows the effect of the thermochemical treatment of the local raw clays on the viscosity (600 rpm) of the samples. The thermochemical treatment of the raw clay materials has a strong effect on the viscosity of the samples prepared using Pindinga and Ubakala clays. The viscosity of samples $P_{1,3}$ and $P_{3,3}$ were improved from 6 to 14 cP and 8 to 26 cP, respectively, after the thermochemical treatment. The viscosity of samples $U_{1,3}$ and $U_{3,3}$ also improved from 6 to 10 cP and 8 to 35.5 cP, respectively. It was observed that the higher the concentration of MgO in the samples, the more the effect of the thermochemical treatment on the mud viscosity. The thermochemical treatment of Ubakala raw clay was able to improve the viscosity of the mud (sample $U_{3,3}$) to the standard specified by API. API stipulated that the minimum viscosity of a mud sample should be 30 cP.

The thermochemical treatment of Pindinga clay improved the viscosity of the mud (sample $P_{3,3}$) comparably with that of finished bentonite clay (26 cP) [34]. Hence, Pindinga raw clay can also be used for drilling operations after being treated thermochemically. The improvement in the mud viscosity after the thermochemical treatment of the local raw clay materials was a result of the substitution and exchange of ions that took place during the heating process [10,21].

3.2.2. Shear Stress to Shear Rate Relationship

Figure 6 show the effect of the thermochemical treatment of the local raw clay materials on the shear stress to shear rate relationship of the samples. All the mud samples exhibited a Bingham plastic fluid behavior. The shear stress to shear rate behavior of the samples before the thermochemical treatment was very poor. The higher the concentration of MgO in the samples, the more the impact of the thermochemical treatment. It improved the shear stress to shear rate behavior of the samples prepared using the raw clay from Pindinga and Ubakala. This was also observed in the research conducted by Magzoub et al. [35] where thermochemical treatment of calcium-based bentonite clay increased the shear stress behavior of the mud samples.

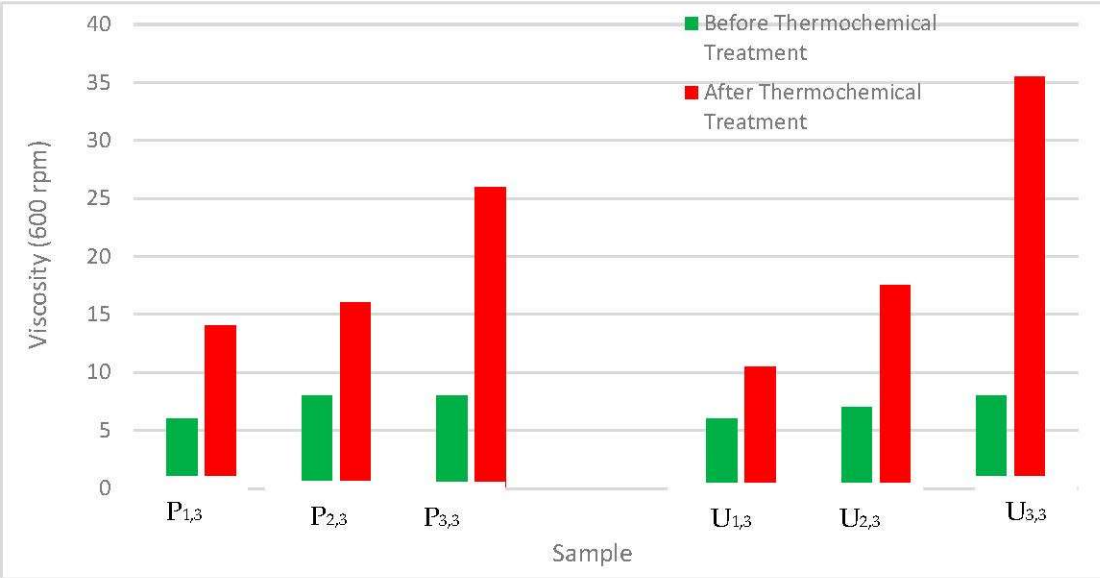

Figure 5. Effect of the thermochemical treatment of the local raw clay materials on the mud viscosity at 600 rpm.

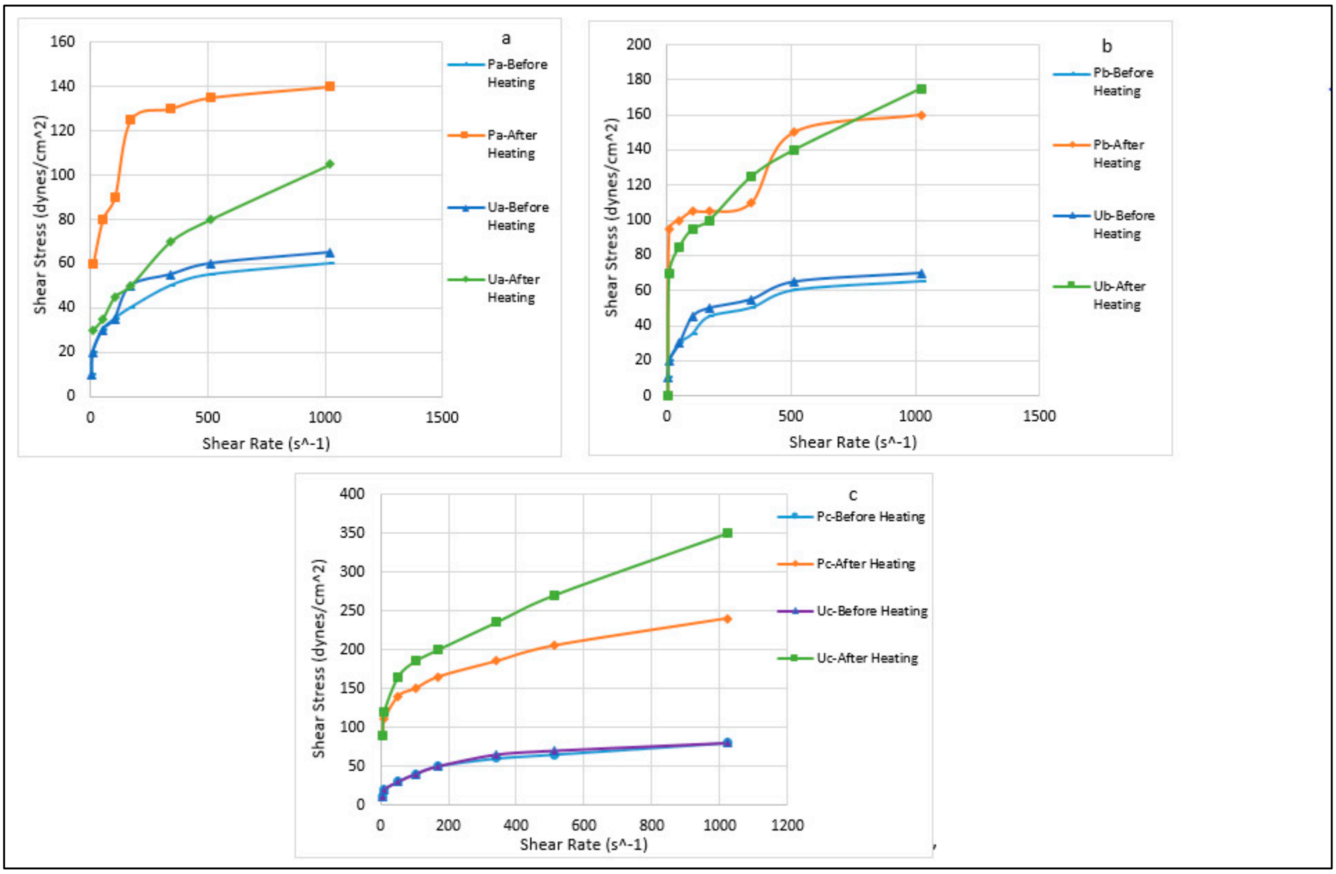

Figure 6. Effect of the thermochemical treatment on the shear stress to shear rate relationship of the mud samples: (**a**). Samples $P_{1,3}$ and $U_{1,3}$. (**b**). Samples $P_{2,3}$ and $U_{2,3}$. (**c**). Samples $P_{3,3}$ and $U_{3,3}$.

3.2.3. Plastic Viscosity, Yield Point, Flow Behavior Index, and Consistency Factor

Table 3 shows the effect of the thermochemical treatment of the local raw clay materials on the plastic viscosity, yield point, flow behavior index, and consistency factor of the mud samples. The thermochemical treatment of the local raw clay materials also had some significant effects on the plastic viscosity, yield point, flow behavior index (n), and consistency factor (k) of most of the samples. The thermochemical treatment of the samples improved the plastic viscosity of the samples prepared with Pindinga and Ubakala clay, respectively. The plastic viscosity of samples $P_{2,3}$ and $P_{3,3}$ increased from 0.5 to 1 cP and 1.5 to 3.5 cP, respectively. The plastic viscosity of samples $U_{2,3}$ and $U_{3,3}$ increased from 0.5 to 3.5 cP and 1.0 to 8.0 cP, respectively.

Table 3. Effect of the thermochemical treatment of the local raw clay materials on the plastic viscosity, yield point, flow behavior index, and consistency factor of the samples.

Property	$P_{1,3}$ (Before Heating)	$P_{1,3}$ (After Heating)	$U_{1,3}$ (Before Heating)	$U_{1,3}$ (After Heating)
PV (cP)	2.5	0.5	0.5	2.5
YP (lbf/100sq.ft)	1	13	5.5	5.5
n	0.777	0.052	0.115	0.392
k	0.027	9.734	2.921	0.694
Property	**$P_{2,3}$ (Before Heating)**	**$P_{2,3}$ (After Heating)**	**$U_{2,3}$ (Before Heating)**	**$U_{2,3}$ (After Heating)**
PV (cP)	0.5	1	0.5	3.5
YP (lbf/100sq.ft)	5	14	6	10.5
n	0.115	0.093	0.107	0.322
k	2.921	8.396	3.338	1.882
Property	**$P_{3,3}$ (Before Heating)**	**$P_{3,3}$ (After Heating)**	**$U_{3,3}$ (Before Heating)**	**$U_{3,3}$ (After Heating)**
PV (cP)	1.5	3.5	1	8
YP (lbf/100sq.ft)	5	17	6	19
n	0.299	0.227	0.193	0.374
k	1.005	4.968	2.107	2.618

The thermochemical treatment improved the yield point of the samples prepared with Pindinga and Ubakala clay. The yield point of samples $P_{1,3}$, $P_{2,3}$, and $P_{3,3}$ were increased from 1.0 to 13.0 lbf/100sq.ft, 5.0 to 14 lbf/100sq.ft, and 5.0 to 17 lbf/100sq.ft, respectively. The yield point of samples $U_{2,3}$ and $U_{3,3}$ were increased from 6.0 to 10.5 lbf/100sq.ft and 6.0 to 19.0 lbf/100sq.ft, respectively. The yield point of samples $P_{3,3}$ and $U_{3,3}$ after the thermochemical treatment met the API standard. API recommends that the yield point of any water-based drilling mud should be a minimum of three times its plastic viscosity. Magzoub et al. [35] also reported similar observations in their studies of thermochemical upgrading of calcium-based bentonite for drilling fluid applications. The thermochemical treatment improved the plastic viscosity and yield point of the mud samples prepared with calcium-based bentonite. The flow behavior index of the samples prepared with Pindinga and Ubakala clays was also affected after the thermochemical treatment. Despite the effect of the thermochemical treatment on the flow behavior index, the samples still behaved like a non-Newtonian fluid. The thermochemical treatment of the samples prepared using Pindinga and Ubakala clay also improved the consistency factor of the samples.

3.2.4. Thixotropy Behavior

Figures 7 and 8 show the thixotropy behavior of the mud prepared with Pindinga clay before and after thermochemical treatment, respectively. The mud samples prepared before the thermochemical treatment did not exhibit any thixotropy behavior. The up-sheared curve and the down-sheared curve of all the samples coincided with each other during the 10-s and 10-min thixotropy tests. This is undesirable during drilling operations. The thermochemical treatment affected the thixotropy behavior of this sample. The down-sheared curve lay above the up-sheared curve before a shear rate of 51 s^{-1} during the 10-min thixotropy test. This shows that at very low shear rate conditions, the fluid exhibited negative thixotropy [30]. The structure developed in the sample after the 10-min rest period did not break down completely at low shear rate conditions. The sample also exhibited negative thixotropy between a shear rate of 170 s^{-1} and 340 s^{-1}.

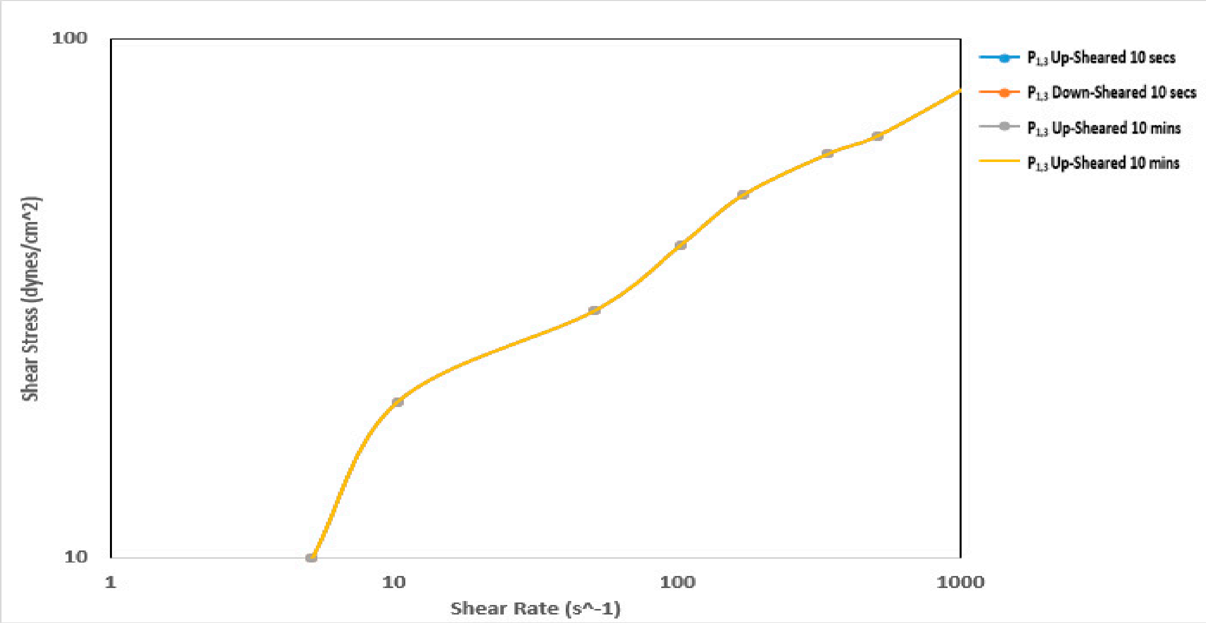

Figure 7. Thixotropy behavior of the mud prepared with Pindinga clay before thermochemical treatment.

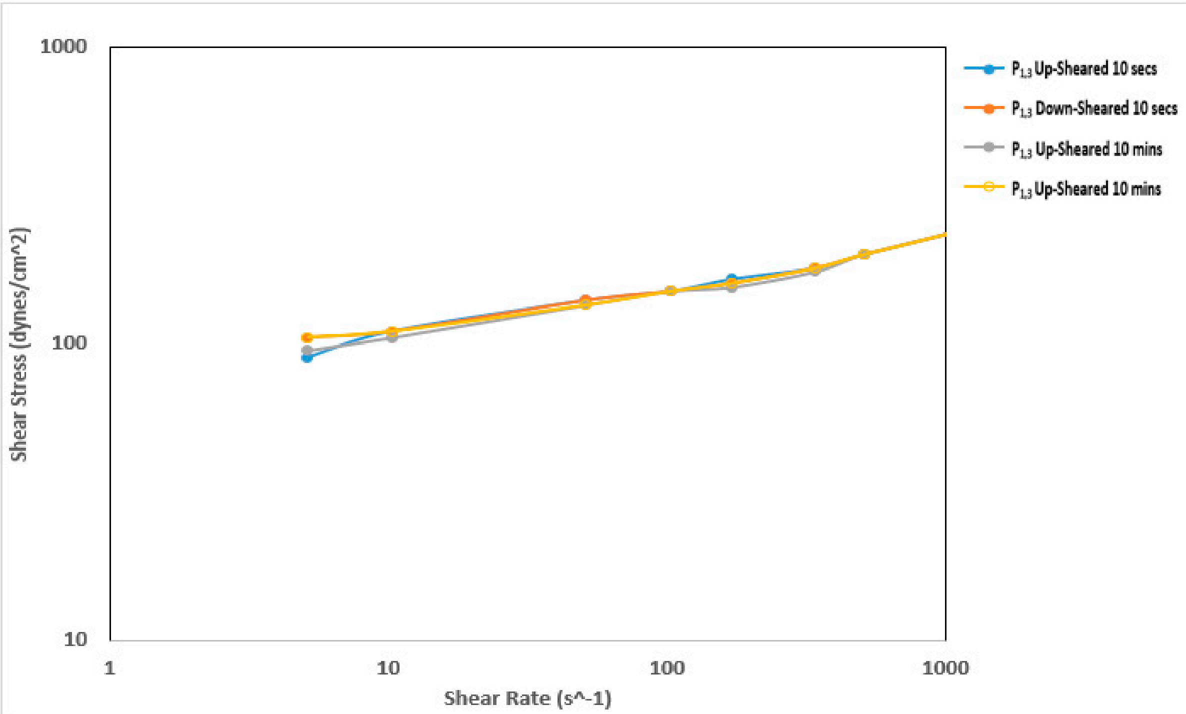

Figure 8. Thixotropy behavior of the mud prepared with Pindinga clay after thermochemical treatment.

Figures 9 and 10 show the thixotropy behavior of the mud prepared with Ubakala clay before and after thermochemical treatment, respectively. Initially, this sample did not exhibit any thixotropy behavior. However, after the thermochemical treatment, the sample exhibited prominent thixotropy behavior. The sample exhibited a negative thixotropy before a shear rate of 1022 s^{-1} during the 10-min thixotropy test. The down-sheared curve was lying above the up-sheared curve [30]. This implies that the structure developed during the 10-min rest period did not break down. The thermochemical treatment affected the thixotropy behavior of the samples prepared using Pindinga and Ubakala clay, respectively. It made the fluid samples exhibit negative thixotropy behavior. It also improved the shear stress required to shear the fluid. The structure generated during the rest period was a result of the improvement in the fluid viscosity during the treatment of the local raw clay materials.

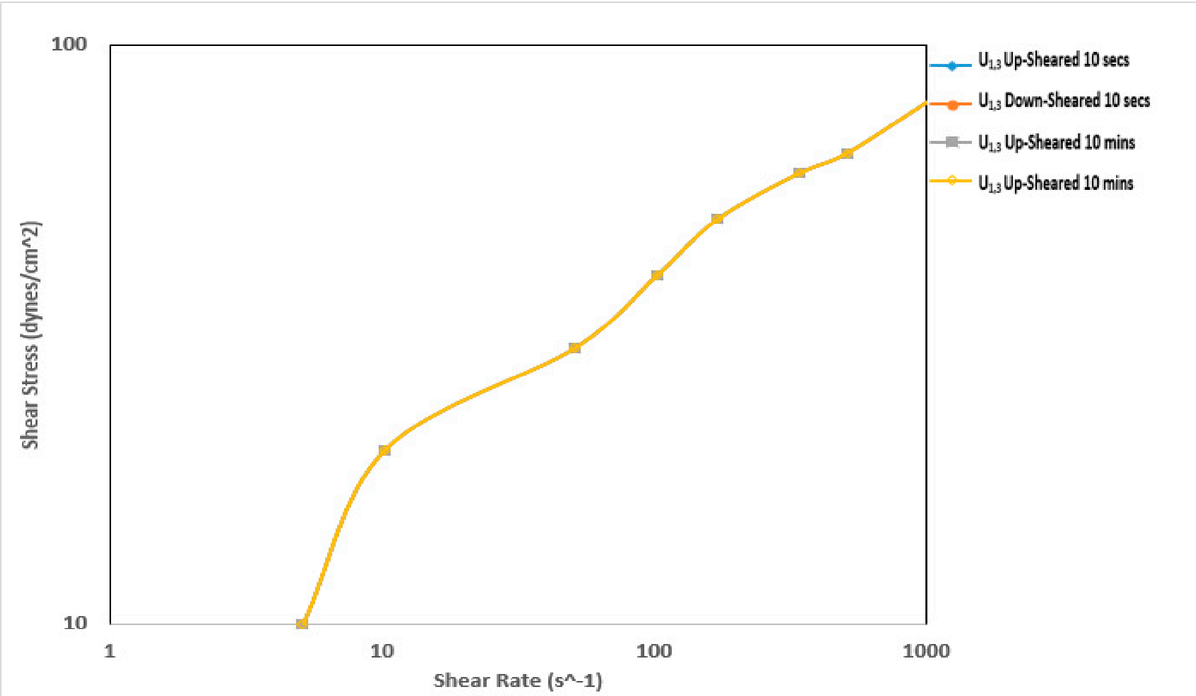

Figure 9. Thixotropy behavior of the mud prepared with Ubakala clay before thermochemical treatment.

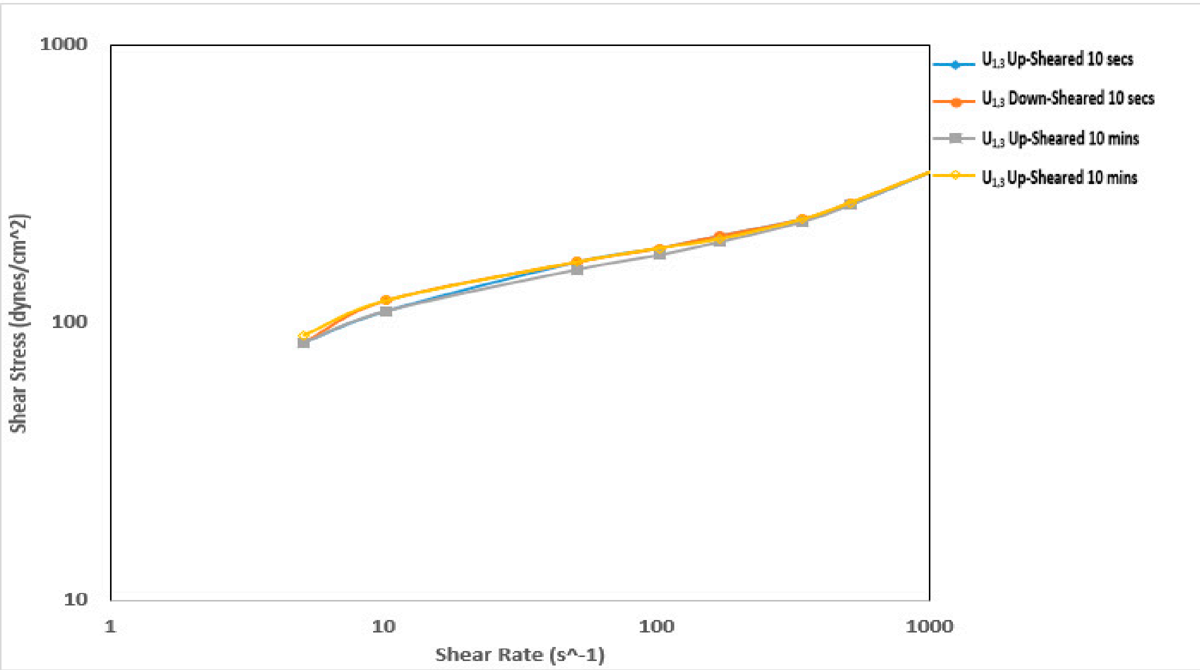

Figure 10. Thixotropy behavior of the mud prepared with Ubakala clay after thermochemical treatment.

4. Conclusions

The thermochemical treatment of the clay samples improved the rheological properties of the mud prepared with Pindinga and Ubakala clay, respectively. The viscosity (600 rpm) of the mud samples prepared with Pindinga and Ubakala clay was improved from 8 to 26 cP and 8 to 35.5 cP, respectively. It also improved the yield point, consistency factor, and consistency index of the mud samples to the standard recommended for drilling operations.

The thermochemical treatment also had an impact on the thixotropy behavior of the mud samples. It made the fluid samples exhibit negative thixotropy behavior. The time to treat the samples thermochemically to attain acceptable rheological properties was one hour. This is shorter than other treatment times observed in the literature.

It is recommended that further studies should be carried out to reduce the temperature requirement for thermochemical treatment of potassium-based bentonite clay. This will help to reduce the cost of treating potassium-based bentonite clay.

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