



Potential of Kaolin Clay on Formulation of Water Based Drilling Mud Reinforced with Biopolymer, Surfactant, and Limestone

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Abstract

Drilling the wellbore into subsurface formations is the earliest stage in the life of a well that requires a suitable drilling fluid with good rheological and filtration control characteristics to achieve a successful drilling operation. This paper reports on the formulation of water-based drilling mud using kaolin clay reinforced with biopolymer, surfactant, and limestone from Tanzania. The raw materials used for the formulation were characterized using XRD, XRF, FTIR, and TGA techniques. The rheological properties, filtration loss, pH, and density of the formulated mud were measured using a viscometer, filter press, pH meter, and mud balance, respectively. The XRD results revealed kaolinite and quartz as the main minerals in all kaolin clay samples which were in agreement with the FTIR findings. The mineralogical composition of the kaolin was observed to vary with the mining sites and influenced the rheological and filtration control characteristics of the mud. The beneficiated drilling mud showed better rheological and filtration control characteristics than the commercially available bentonite mud in Dar es Salaam market and was comparable with the API standards. These results, therefore, are not only new but rather important and novel information on the potential of kaolin clay for the formulation of water based drilling mud.

Keywords: Water-based drilling mud, kaolin clay, biopolymers, bentonite, surfactant.

Introduction

The exploitation of hydrocarbons from the subsurface requires drilling either a vertical or horizontal well in the ground that is referred to as a borehole (Chemwotei 2011, Yunita et al. 2016). The drilling process requires drilling mud to be circulated or pumped from the surface, down the drill string, through the bit, and back to the surface via annulus (Chemwotei 2011, Yunita et al. 2016). Drilling mud performs various functions including the removal of drilling cuttings from the drilled borehole, controlling borehole hydrostatic pressure, cooling and lubricating the drilling bits and string,

promoting borehole stability, and fluid loss control (Chemwotei 2011, Yunita et al. 2016). Depending on the composition and use in oil and gas drilling operations, drilling muds are classified as water-based, oil-based, or synthetic-based (Hossain and Wajheuddin 2016). In contrary to other drilling muds, water-based drilling mud has received special attention because it is cheap and environmentally friendly compared to the other drilling muds (Kania et al. 2015, Liu et al. 2016, Dankwa et al. 2018, Tiwari et al. 2020, Pinheiro et al. 2021). Furthermore, water-based drilling mud favourably demonstrates good rheological and filtration

control properties, adequate cooling and cutting removal ability, and simplicity to formulate and use in the oilfields (Horst et al. 2003, Li et al. 2015, 2016).

The formulation of water-based drilling mud from local, affordable, and available materials is a current growing research area that requires more exploration to find the best alternative to synthetic drilling mud (Udoh and Okon 2012, Behnamanhar et al. 2014, Omotioma et al. 2015). Local materials such as clay and rock minerals and agricultural wastes are marginally utilized in drilling mud formulation. Agricultural products and wastes such as corncobs, acacia gum, mango seeds, rice husks, sugarcane bagasse, soybeans, coconut and cashew nutshells, and cassava, potatoes, and bananas peels are rich in various chemical compounds such as biopolymers (Taiwo et al. 2011, Olatunde et al. 2012, Ghazali et al. 2015, Omotioma et al. 2015, Zarouri and Benmounah 2016, Talukdar et al. 2018, Al-hameedi et al. 2019, Li et al. 2019, Abro et al. 2020, Afolabi and Yusuf 2020, Al-Hameedi et al. 2020, Sulaimon et al. 2020, Zhou et al. 2021, Sarah and Ebidiseghabofa 2021). Both laboratory studies and field applications have ascertained the promising performances of biopolymers as drilling mud additives (Taiwo et al. 2011, Olatunde et al. 2012, Ghazali et al. 2015, Omotioma et al. 2015, Zarouri and Benmounah 2016, Talukdar et al. 2018, Al-hameedi et al. 2019, Li et al. 2019, Abro et al. 2020, Afolabi and Yusuf 2020, Al-Hameedi et al. 2020, Sulaimon et al. 2020, Zhou et al. 2021, Sarah and Ebidiseghabofa 2021). Biopolymers such as xanthan gum, guar gum, starch, cellulose, lignin, chitosan, gelatin, protein, and natural rubber have been reported as rheology enhancers and filtration loss controlling agents in water-based drilling muds (Coussot et al. 2004, Hamed and Belhadri 2009, Ghazali et al. 2015, Fink 2015, Zarouri and Benmounah 2016, Li et al. 2019, Oseh et al. 2019, 2020, Al-Hameedi et al. 2020, Zhou et al. 2021, Sarah and Ebidiseghabofa 2021). This paper, therefore, reports on the potential of Tanzania kaolin clay in the formulation of water-based drilling mud with reinforced additives from

locally available materials (limestone, mango seeds kernel, and soybean).

Materials and Methods

Sample collection

The local materials (kaolin clay, limestone, biopolymers and biosurfactants from agriculture wastes) for the formulation of water-based drilling mud were collected from different parts of Tanzania. A total of 16 samples of kaolin clays were sampled from Ukemele, Kitalawasi, and Rungemba villages in Mufindi District in Iringa, whereas six samples of limestone were obtained from Kiomoni and Amboni in Tanga Region. The samples were placed in labelled plastic containers then packed into a cool box and transported to Chemistry Department University of Dar es Salaam for further processes. A total of 25 kg of Mango seeds were collected from Buguruni and Tandika markets in Dar es Salaam, and 30 kg of soybeans were purchased from Mafinga market in Iringa Region. All the collected samples were placed in labelled plastic bags and transported to Chemistry Department, University of Dar es Salaam for further processes.

Chemicals and reagents

Analytical grade chemicals and reagents; methanol, ethanol, n-hexane, dichloromethane, sodium bisulfate, sodium carbonate, and silica gel were bought from Scan Tanzania. Whereas the distilled water was obtained from the Chemistry Department, University of Dar es Salaam.

Preparation of kaolin clay cake

The collected kaolin clays were used to prepare the kaolin clay cake based on the method reported by Udoh and Okon (2012) with some modifications. The collected kaolin clays were sun-dried, crushed into a fine powder, and dissolved in distilled water to form a slurry. The formed slurry was then filtered using a 300 μm filter to remove impurities such as sand and organic matter. The filtrates were allowed to settle for 3 hours followed by water decanting to form a clay mud which was dried under sunlight for

2 days to form kaolin clay cake. The kaolin clay cake was ground and electrically sieved using 125 and 75 μm sieves to fine powder ready for characterization and formulation of drilling mud.

Preparation of limestone powder

The collected samples of limestone were crushed into small particles using a KHD Humboldt Wedag AG crusher which were then milled into limestone powder using planetary ball milling (PM 400). The obtained powder was electrically sieved using 250, 125, and 75 μm sieves to obtain the fine limestone powder for further use.

Extraction of starch from mango seed kernels

The starch required for the formulation of the drilling mud was extracted from mango seed kernels. The extraction process followed the method reported by Sonthalia and Sikdar (2015) with some modifications. The mango seed kernels were manually separated from seed coats using a knife and cut into small pieces. A total of 400 g of dried mango seed kernel pieces was soaked in 800 mL of 0.16 M of NaHSO_3 solution at 50 °C and left for 24 hours. The solution of NaHSO_3 was discarded and the seed kernels were blended in 500 mL of distilled water to form a slurry. The formed slurry was filtered using a 300 μm filter, washed thoroughly with distilled water, and left to settle for 1 hour. Thereafter, the supernatant was decanted and the settled layer was re-suspended in distilled water, centrifuged at 2800 rpm for 5 minutes followed by decantation and drying of the starch cake in an oven at 50 °C for 6 hours. The formed starch cake was ground and sieved using a 75 μm sieve, packed in a sealed plastic container, and stored at room temperature for further use.

Extraction and isolation of saponin surfactant from soya beans

Saponin surfactant was extracted from soya beans using the Soxhlet extraction technique (Fenwick and Oakenfull 1981, Khalil and EI-Adawy 1994). A total of 70 g of dry bean seed powder was initially

extracted by 500 mL of *n*-hexane to remove lipids and any interfering pigments (El-keiy et al. 2019). Thereafter, methanol was placed in the extractor for extraction of saponin from soybeans powder which was pretreated with *n*-hexane. The concentrated methanol extract was subjected to column chromatography packed with silica gel phase and eluted using dichloromethane, methanol, and water system at a ratio of 40:10:1. The collected fractions were assessed for the composition using TLC plates which were sprayed with a *p*-anisaldehyde reagent for the identification of the chemical composition in the ultraviolet lamp. The fractions that showed positive results with *p*-anisaldehyde were collected together and confirmed to contain saponin surfactants by shaking with distilled water.

Characterization of kaolin clay, limestone, starch, and saponin

The mineralogical composition of kaolin clay [Kitalawasi kaolin clay (KKC), Ukemele kaolin clay (UKC), Rungemba kaolin clay (RKC)] and limestone was determined using S8 Tiger-Bruker XRF-Spectrometer. During analysis, powdered kaolin clay and limestone were compressed under a pressure of 200 KN to form circular pellets with a diameter of 34 mm. Thereafter, the pellets were scanned to determine the elemental and metal oxides. The FTIR spectroscopic measurement of kaolin clay, limestone, starch, and saponin was performed using FT-IR spectrometer with an attached diamond attenuated total reflectance (ATR), Bruker optic GmbH 2011 USA model in Chemistry Department laboratories of the University of Dar es Salaam. A small amount of each finely powdered sample was placed and squeezed on the diamond crystal, and the IR spectra were recorded in the range of 4000 to 400 cm^{-1} in the transmittance mode with 4 scans per spectrum at a resolution of 4 cm^{-1} .

The determination of thermal stability of powdered samples of kaolin clay, limestone, and starch was performed using a thermal gravimetric analyzer (STA PT-1000 LINSEIS TGA). About 1.0 mg of each sample was measured in a platinum sample holder pan using a microbalance and heated

from room temperature to 800 °C at a rate of 10 °C min⁻¹ for 8 hours. Furthermore, the characterization of kaolin clay and limestone crystal phases was performed using a Bruker D2 Phaser XRD (Cu-K α at 1.54184 Å, 30 kV and 10 mA) with a LYNXEYE detector as well as corundum standard reference materials. The equipment was operated at 8 to 65° (degree 2 theta), with steps of 0.020° (0.05 sec) detector slit of 8 mm, a divergence slit of 0.6 mm, and a knife with 1 mm to obtain the diffractograms. The phase identification was performed with the DIFFRA.EVA software.

Formulation of water-based drilling mud

The water-based drilling mud were formulated according to the American Petroleum Institute (API 2017) standard (13B-1) (Edition 2017). About 25 g of each KKC, UKC, and RKC was dissolved into 350 mL of distilled water and agitated for 24 h.

Determination of the formulated water-based drilling mud properties

The pH, mud density, and filtration loss of the formulated water-based drilling mud were determined using a pH meter, mud balance (RCMB 140), and an OFITE filter press (14030), respectively. The rheological properties of the formulated water-based drilling mud were determined using an OFFITE viscometer (800) at 600 and 300 shear rates (rpm). The rheological properties investigated in this paper include yield point (YP), plastic viscosity (PV), gel strengths, and apparent viscosity (AV). The gel strength at 10 seconds (G10-s) and 10 minutes (G10-min) was recorded at 3 rpm. Whereas, the

AV, PV, and YP were calculated according to the following equations (Equation 1–3) (Zhou et al. 2021):

$$AV = \theta_{600}/2 \quad (\text{mPa.s}) \quad (1)$$

$$PV = \theta_{600} - \theta_{300} \quad (\text{mPa.s}) \quad (2)$$

$$YP = \theta_{300} - PV \quad (\text{lb}/100 \text{ ft}^2) \quad (3)$$

where θ_{600} and θ_{300} are dial readings at 600 and 300 shear rate (rpm), respectively, lb = lab barrel and ft² = feet square.

Beneficiation of the formulated water-based drilling mud

Beneficiation involves the addition of different chemical additives to improve the properties of the drilling mud. Thus, the formulated water-based drilling mud was beneficiated with starch, soda ash, limestone, and saponin surfactant.

Results and Discussion

Mineralogical composition of kaolin clay and limestone

In the analysis of kaolin clay for mineralogical composition, it was observed that KKC had the highest amount of SiO₂ (48.30%) and Al₂O₃ (34.96%) as compared to UKC and RKC (Table 1). Based on these results, KKC was considered to be suitable for the formulation of water-based drilling mud as it had a large amount of silica and alumina which are the major components of kaolinite (Diko et al. 2016). Moreover, the obtained values for the loss on ignition (LOI) indicated a low value on KKC (12.15 ± 0.74%) implying low content of organic matter as compared to the other two [UKC (13.75 ± 1.80%) and RKC (12.02 ± 1.41%)].

Table 1: Mineralogical composition of KKC, UKC and RKC (n = 7 for KKC, 5 for UKC and 4 for RKC)

Composition	KKC (%)	UKC (%)	RKC (%)
SiO ₂	47.20 ± 2.25	44.10 ± 1.67	44.48 ± 2.35
Al ₂ O ₃	32.33 ± 3.33	31.49 ± 2.60	24.73 ± 0.66
CaO	0.22 ± 0.01	0.09 ± 0.01	0.24 ± 0.01
K ₂ O	4.37 ± 1.74	3.49 ± 1.46	9.01 ± 0.19
TiO ₂	0.70 ± 0.31	1.59 ± 0.28	2.96 ± 0.24
Fe ₂ O ₃	1.32 ± 0.97	1.73 ± 0.62	2.24 ± 0.16
LOI	12.15 ± 0.74	13.75 ± 1.80	12.02 ± 1.41

KKC = Kitalawasi kaolin clay, UKC = Ukemele kaolin clay, Rungemba kaolin clay = RKC.

For limestone, the determined mineralogical composition of both Amboni limestone (ALS) and Kiomoni limestone (KLS) showed a high percentage of calcium oxide (CaO) (Table 2) indicating the purity of the limestone (Hwidi et al. 2018). It was further noted that ALS had a higher percentage of CaO (59.54 ± 0.02 %) than that

of KLS (57.37 ± 0.01 %) which indicates the high purity of ALS, hence considered to be the best for beneficiation. Furthermore, it was noted that the organic matter composition was low in ALS compared to that of KLS as revealed by the values of LOI (i.e., 34.28 ± 0.08 % and 36.05 ± 0.06 %, respectively).

Table 2: Chemical composition (%) of Amboni and Kiomoni Tanga limestone

Composition	CaO	SiO ₂	Fe ₂ O ₃	Na ₂ O	MgO	LOI
ALS (%)	59.54 ± 0.02	>0.01	3.87 ± 0.04	0.24 ± 0.01	1.70 ± 0.03	34.28 ± 0.08
KLS (%)	57.37 ± 0.01	>0.01	2.55 ± 0.05	0.22 ± 0.01	1.64 ± 0.14	36.05 ± 0.06

The XRD analysis on kaolin clay showed the presence of kaolinite (Al₂Si₂O₅(OH)₄) and quartz in both KKC, UKC, and RKC, and that kaolinite was the major constituent

mineral as it showed prominent peaks in XRD diffractograms (Figure 1). These results agree with the XRF results presented in Table 1.

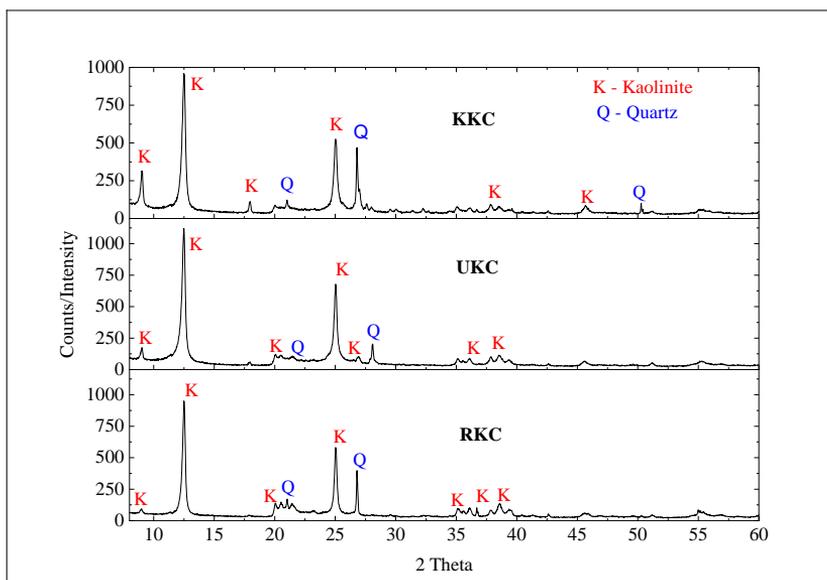


Figure 1: The diffractograms of kaolin clay; Kitalawasi kaolin clay (KKC), Ukemele kaolin clay (UKC), and Rungemba kaolin clay (RKC).

On the other hand, XRD results on the analysis of limestone indicated that both ALS and KLS had prominent peaks of calcite

revealing almost pure limestone (Figure 2). However, small peaks of magnesium oxide (MgO) were observed.

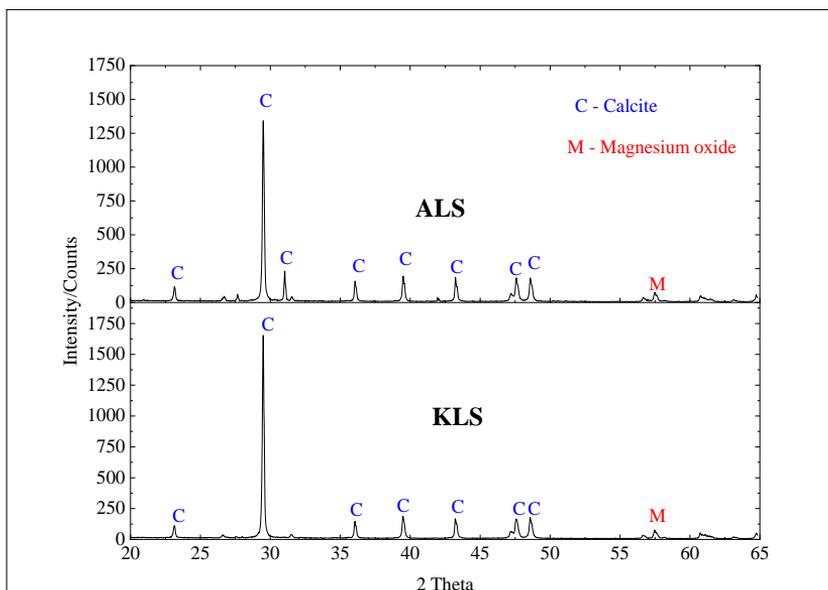


Figure 2: XRD diffractograms of limestone; Amboni limestone (ALS) and Kiomoni limestone (KLS).

FTIR spectroscopic measurement of kaolin clay, starch, and saponin

The obtained FT-IR spectra of KKC, UKC, and RKC had absorption peaks at 3685 cm^{-1} and 3888 cm^{-1} due to hydroxyls (O-H) groups which were ascribed to the out-of-plane stretching. Other peaks were observed at 3621 cm^{-1} and 3622 cm^{-1} which can be attributed to the inner plane vibration stretching of the O-H group of water found between the tetrahedral and octahedral sheet of kaolinite. This O-H vibrations stretching are in line with the kaolinite characteristic peaks (Panda et al. 2010, Saikia and Parthasarathy 2010, Diko et al. 2016). In the fingerprint region, many absorption peaks were observed which include; 1634 cm^{-1} for H-O-H bending vibration, $994\text{--}1005\text{ cm}^{-1}$ for

Si-O stretch, and $1110\text{--}1114\text{ cm}^{-1}$ for Si-O symmetrical stretching bending vibrations. The peak observed at $907\text{--}910\text{ cm}^{-1}$ is ascribed to vibration stretch of the O-H linked to the 2Al^{3+} ions of Al-Al-OH that signifies the presence of alumina (Murray 2006, Srinivasan 2011, Diko et al. 2016). More peaks were observed at $789\text{--}791\text{ cm}^{-1}$ assigned to vibration stretch of a hydroxyl group (O-H) attached to Al^{3+} and Mg^{2+} of (Al-Mg-OH) in the kaolin clay, $681\text{--}750\text{ cm}^{-1}$ assigned to the Si-O stretching due to quartz. A peak at $523\text{--}529\text{ cm}^{-1}$ was due to Si-O-Al asymmetrical vibration stretching, while that of $457\text{--}462\text{ cm}^{-1}$ was assigned to Si-O-Si asymmetrical bending vibration (Figure 3).

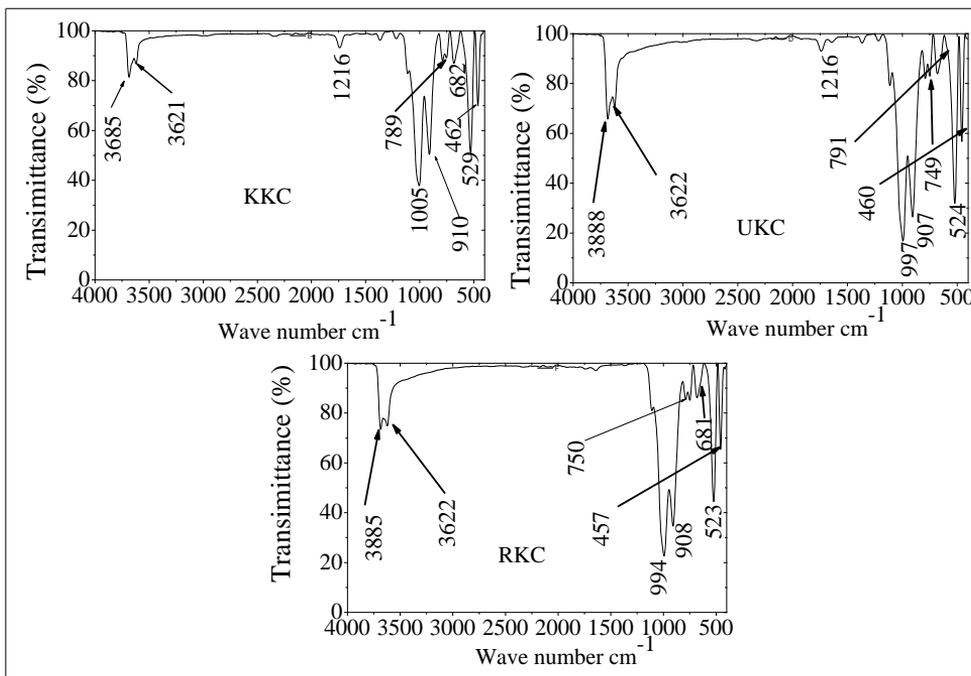


Figure 3: FTIR spectra of kaolin clay; Kitalawasi kaolin clay (KKC), Ukemele kaolin clay (UKC), and Rungemba kaolin clay (RKC).

The FT-IR spectrum of saponin (Figure 4a) showed an absorption peak at 3326 cm^{-1} that indicates the presence of OH stretching vibration bands. The sharp absorption peaks at 2928 and 2854 cm^{-1} were assigned to the C-H symmetric stretching band of CH_2 while that of 1613 cm^{-1} was assigned to the stretching vibration band of C=C. The oligosaccharide linkage to the saponin C-O-C exhibited an absorption peak at 1006 cm^{-1} . All the observed peaks on the FT-IR spectrum indicate the characteristics of saponin.

The FT-IR spectrum of starch (Figure 4b) showed a broad peak at 3286 cm^{-1} which is due to O-H stretching vibration, and peaks at 2921 and 2852 cm^{-1} which are attributed to

C-H stretching vibrations in starch. The peaks at 762 and 996 cm^{-1} are due to the C-C symmetric stretch and the C-O-C stretch of skeletal mode vibration of 1, 4 glycosidic linkage, respectively. The absorption bands at 1078 and 1150 cm^{-1} are due to C-C and C-O stretching vibrations, respectively. Furthermore, the peaks at 1343 , 1430 , and 1737 cm^{-1} are attributed to bending vibration of O-H, C-H, and aldehyde group stretching vibrations, respectively (Mohebbi 2008). Similar results were also reported by Silva et al. (2019) and Kizil et al. (2002) in their studies on the characterization of mango seed starch with FT-IR.

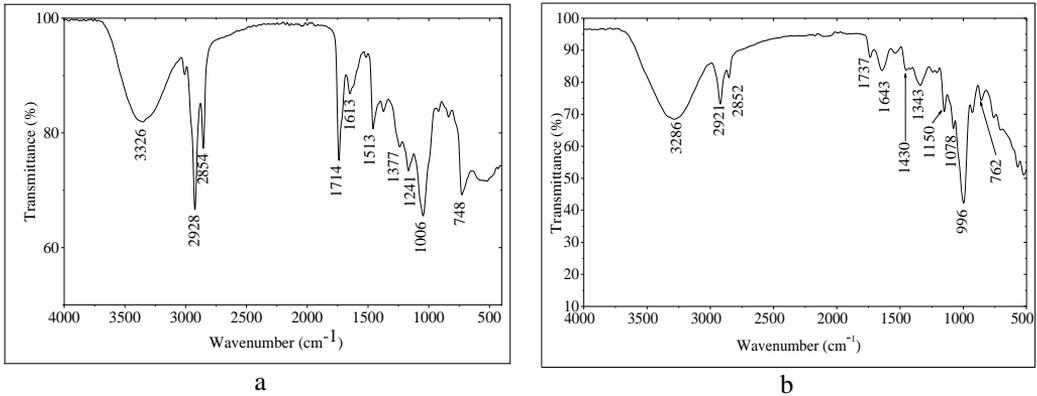


Figure 4: FTIR Spectrum for (a) saponin surfactant and (b) starch.

Thermal stability of kaolin clay, limestone, and starch

The TGA analysis of KKC, UKC, and RKC (Figure 5) indicated thermal decomposition at three stages. The first decomposition observed at 30 to 250 °C signifies the loss of water from the crystals. The second and third decomposition were observed at 250 to 500 °C, and 500 to 600 °C which signify the decomposition of organic matter and dehydroxylation of kaolinite to form the metakaolinite, respectively. The TGA analysis of ALS and KLS (Figure 6) indicated the first thermal decomposition at 30 to 180 °C due to the loss of water from the crystals. The second and third decomposition

were observed at 180 to 620 °C, and 620 to 800 °C which signify the decomposition of less volatile organic matter and calcium carbonate to form calcium oxide and carbon dioxide, respectively. The TGA analysis of starch indicated thermal decomposition at three stages. The first decomposition observed at 30 to 280 °C signifies the loss of water from the crystals and volatile matter. The second and third decomposition were observed at 280 to 340 °C and 340 to 510 °C which signify decomposition of less and non-volatile matter, respectively. In general, the TGA analysis revealed strong thermal stability of materials and therefore suitable for the formulation of drilling mud.

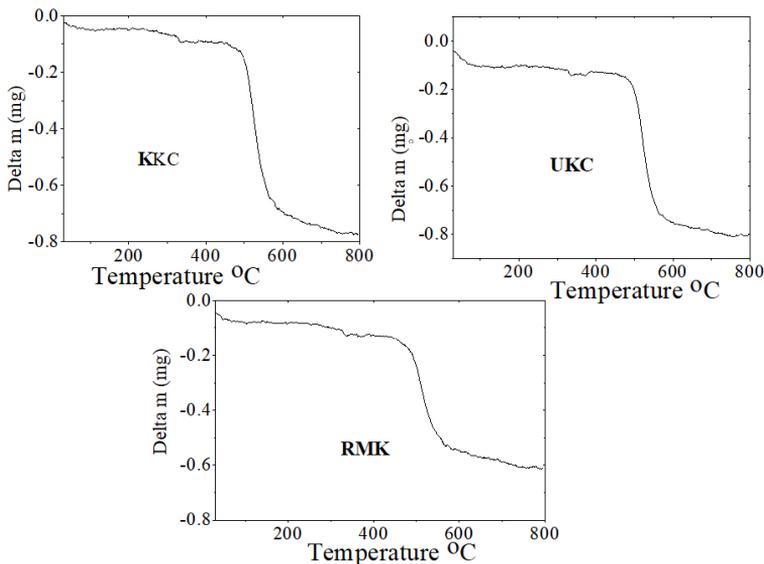


Figure 5: TGA thermogram of kaolin clays (KKC, UKC and RKC).

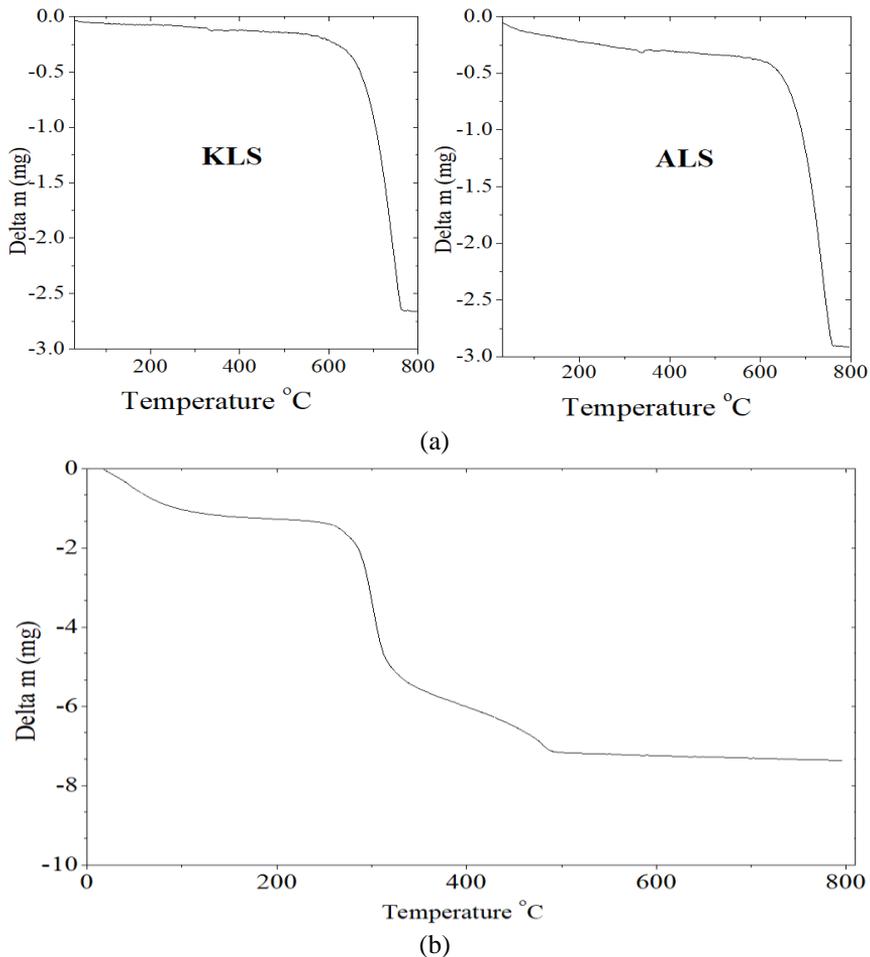


Figure 6: TGA thermogram of (a) limestone (ALS and KLS) and (b) starch.

Formulation of water-based drilling mud

Water-based drilling mud was formulated from the three collected clay samples (KKC, UKC, and RKC). However, among the three clay samples, KKC showed the best dispersion in distilled water, thus it was the only one considered for further beneficiation. This can be explained by the high amount of SiO_2 (48.30%) and Al_2O_3 (34.96%) (Table 1) as compared to the other two. Furthermore, this was also supported by a low value of a loss on ignition (LOI) (12.13%) indicating low content of organic matter as compared to the other two [UKC (14.50%) and RKC (12.14%)].

The beneficiation of the water-based drilling mud was achieved by the addition of various chemical additives such as starch,

soda ash, limestone, and saponin at different weights (Table 3). During the formulation, several attempts of different ratios were performed of which the best formulation was selected as the final water-based drilling mud based on the comparability (i.e., when the property was similar or better than that of CBM) of the rheological properties of commercially available bentonite mud in Dar es Salaam market. The comparison of commercial bentonite mud (CBM) and kaolin clay mud (KCM) before and after beneficiation is shown in Table 4.

The beneficiated kaolin clay mud showed improved rheological properties, filtration loss, pH and mud density compared to that of commercial bentonite and un-beneficiated kaolin clay mud (UKCM).

Table 3: Formulation and beneficiation of water-based kaolin clay mud (KCM)

Additives	Unbeneficiated KCM	Beneficiated KCM
Distilled water (mL)	350.00	350.00
Kaolin clay (i.e., KKC) (g)	25.00	25.00
Starch (g)	-	50.00
Limestone (g)	-	15.00
Saponin surfactant (g)	-	5.00
Soda ash (g)	-	2.50

Table 4: Comparison of commercial bentonite mud (CBM) and kaolin clay mud (KCM) before and after beneficiation

Properties	CBM	Unbeneficiated KCM	Beneficiated KCM
Density (lb/gal)	9.10	7.60	10.40
pH value	10.80	7.80	10.60
Dial reading @600 rpm	28.00	3.00	40.00
Dial reading @300 rpm	20.00	2.00	28.00
Plastic viscosity (cp)	8.00	1.00	12.00
Yield point (lb/100 ft ²)	12.00	1.00	16.00
Apparent viscosity (cp)	14.00	1.50	20.00
Gel strength 10 sec. (lb/100 ft ²)	5.00	0.50	7.00
Gel strength 10 min. (lb/100 ft ²)	10.00	1.50	10.00
Filtration loss (mL)	6.00	28.00	2.50

In most cases, the properties of the completely beneficiated KCM were better than those of CBM with exception of pH (< than that of CBM; 10.60 vs 10.80) and gel strength at 10 min (which was equal to that of CBM; 10.0 lb/100ft²). It was further noted that the yield point (YP), plastic viscosity (PV), gel strengths (G10-s), apparent viscosity (AV), and filtration loss were significantly improved as compared to those of CBM. Thus, YP, PV, G10-s, AV, and filtration loss were improved by 33, 50, 40, 42.9, and 58%, respectively.

Conclusion

This study has successfully formulated and characterized water-based drilling mud from kaolin clay reinforced with limestone, surfactant and biopolymer from Tanzania. The rheological and other properties of the formulated water-based drilling mud were determined and compared to the commercial bentonite drilling mud. Based on the experimental findings, it is concluded that kaolin collected from Kitalawasi was the most suitable for the formulation of water-based drilling mud being characterized by a high content of silica and alumina. The

complete beneficiation of the formulated drilling mud with starch, saponin surfactant, limestone, and soda ash resulted in rheological properties better than that of commercial bentonite but comparable to the API required water-based drilling mud properties. Therefore, the findings suggest kaolin clay can be used to produce water-based drilling mud to replace the costly and non-environmentally friendly drilling mud.

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