



Surface Active Characteristics of Potash ($K_2CO_3 \cdot H_2O$) Extracts of *Vernonia amygdalina* and *Terminalia mantaly* in Formulating Water-Based Drilling Mud

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Abstract Surface active characteristics of Potash ($K_2CO_3 \cdot H_2O$) extracts of *Terminalia mantaly* (Almond) and *Vernonia amygdalina* (bitter-leaf) were evaluated, when used in the formulation of water based drilling mud. Plastic viscosity, yield point, gel strength, filtration loss volume, specific gravity and pH of the formulated muds were studied with respect to the effect of surface active agent present in the mud. The pH of the various formulated drilling fluid are in the order: ALEM (9.2) > BLEM (9.1) > C1 (8.3) > C2 (8.2), with ALEM and BLEM being more alkaline than C1 and C2. The highest PV value was 17 cP which was exhibited by BLEM at 30 °C and ALEM at 49 °C. The YP values obtained from the formulated muds were relatively desirable with the exception of C2 which gave very high YP values (70, 64 and 60 at 30, 49 and 60 °C respectively), and this could be due to the absence of surface active agent during its formulation. Furthermore, the gel strength obtained was observed to be flat gel, as there was not much difference between gel at 10 seconds and 10 minutes, while the filtration loss volume are in the order: C1(2 ml) < ALEM (6 ml) < BLEM (7 ml) < C2 (15ml), indicating that the plant extracts enhanced reduced filtration loss.

Keywords Surface active, Yield point, Rheological properties, Potash ($K_2CO_3 \cdot H_2O$), Water-based mud

1. Introduction

Surfactants or surface active agents are a special class of versatile amphiphilic compounds that possess spatially distinct polar (hydrophilic head) and non-polar (hydrophobic tail) group [1]. They show interesting phenomena in solution by modifying the interfacial and bulk-solvent properties. The unusual characteristic properties of surfactants in solution especially at the interface owe it to the presence of distinct hydrophilic, as well as hydrophobic domains in the same molecule [2, 3]. In view of its amphiphilic nature and distinctive capability of lowering the interfacial tension; surfactant finds applications in almost every aspects of our daily life directly or indirectly in household detergents, personal care products and industrial processes such as pharmaceuticals, food processing, oil recovery, nanotechnologies, etc. [4].

Generally, based on the nature and type of surface active moiety present in the molecule, surfactants are classified as anionic, cationic, non-ionic surfactants and zwitterionic (amphoteric) surfactants [5].

Anionic surfactants, which are relatively less expensive, are employed in extremely wide variety of surfactant based applications [6]. While anionic surfactants mostly contain carboxylates, sulfonates, sulfates or phosphates moiety as hydrophilic head group, it is often an amine or ammonium groups in case of cationic surfactants [7]. On the other hand, non-ionic surfactants generally have ethylene oxide chains or hydroxyl groups as the polar center and are less reactive compare to the ionic ones. Zwitterionic surfactants contain both cationic and anionic centers, the ionic behaviour of which is altered according to pH of the solvent [8, 9]. These surfactants are



effectively used in personal care and household cleaning products because of the excellent dermatological properties of the surfactants [10, 11]. There is yet another newer class of surfactants known as Gemini (or dimeric) surfactants which are considerably much superior to the conventional surfactants in many ways [12]. Gemini surfactants consist of two hydrophobic tails each attached to a hydrophilic head group connected at the level of head groups by a spacer group [13-16]. The length and type of this spacer moiety dictates the conformation of the dimeric molecule having a high diffusion rate, high surface activity, and low critical micelle concentration [17]. In recent years, studies on Gemini surfactants are directed towards changes associated not only with the variation in the length of the spacer group, but also with the introduction of various substituent groups within the spacer [18, 19].

Most naturally occurring surface active agents are nonionic surfactants. These non-ionic surfactants are manufactured entirely from natural, renewable resources such as plant and animal oils [20]. They have great environmental compatibility and are highly biodegradable. Nonionic bio-surfactants like saponins originate from plants. Structurally, surface active agents are diversified due to their emulsifying and foaming characteristics which are enhanced by their hydrophobic and hydrophilic nature [1].

Potash ($K_2CO_3 \cdot H_2O$) extracts of *Vernonia amygdalina* and *Terminalia mantaly* leaves which possess surfactant properties will be explored in this research as surface active agent, and will be used as an additive for formulating water-based drilling mud; thus the rheological properties of the formulated mud will be evaluated.

2. Materials and Methods

2.1 Plant materials collection

Vernonia amygdalina and *Terminalia mantaly* leaves samples were obtained from Choba area in Port Harcourt, Rivers state, Nigeria. Taxonomic identification of fresh green leaves was carried out at the Department of Plant Science and Biotechnology, University of Port Harcourt. The samples were preserved in a clean plastic bag which was then transported to the laboratory for the research. The leaves were shade-dried for six weeks and then crushed into fine powder using an electric milling machine. The powdered plant samples were kept in air tight containers and labeled prior to use.



Plate 2.1: Dried *Vernonia amygdalina* leaves



Plate 2.2: Dried *Terminalia mantaly* leaves

2.2 Preparation of alkaline solution.

Potash ($K_2CO_3 \cdot H_2O$) was used in preparation of an alkaline solution. 20 g of the potash was weighed into a 250 ml beaker, after which 200 ml of distilled water was also added into the 250 ml beaker. The beaker was stirred for 5 minutes using a magnetic stirrer and then allowed to settle. The mixture was then sieved using a filter paper and the filtrate were collected which will be used for the extraction process.



2.3 Extraction of plant material

50 g each of the crushed *V. amygdalina* and *T. mantaly* powder was soaked in 150 ml of the prepared alkaline solution at 50 °C for 40 minutes with occasional shaking. After filtration, the crude extracts were then transferred into corked containers and reserved for further analysis.

2.4 Characterization of K₂CO₃.H₂O extract of the plants

2.4.1 FTIR characterization

All the crude K₂CO₃.H₂O-extracts from the two plants were subjected to FTIR spectroscopy. Functional groups present in the crude K₂CO₃.H₂O-extracts of the plants were obtained through the spectral data [21].

2.4.2 pH

The pH of the extracts was measured using Hanna digital pH meter at room temperature (27 °C). The pH measurement was done in triplicate.

2.4.3 Conductivity

The conductivity of the extract was measured using an Oakton conductivity meter (Model: CON 150). The instrument was calibrated using a standard solution (Ethanol). The electrode was then removed from the standard solution and rinsed with distilled water. It was then dipped into the beaker containing the K₂CO₃.H₂O-extracts and the conductivity values were read from the conductivity meter. The conductivity measurement was also done in triplicate.

2.4.4 Surface tension

Surface tension of the K₂CO₃.H₂O-plant extracts was measured using Wilhelmy plate tensiometer. The plate was cleaned thoroughly in a burner flame and was then attached to a scale by means of a thin metal wire. The plate was moved downwards by a control knob, towards the K₂CO₃.H₂O-extract surface, until the meniscus connected with it. The force acting on the plate due to the wetting by the K₂CO₃.H₂O-extract was measured by a tensiometer and then recorded. However, the processes were performed in triplicate.

2.4.5 Density

A 50 ml density bottle was measured using an electronic weighing balance. 50 ml of the K₂CO₃.H₂O-plant extract was poured into it and the weight was recorded. The formula in equation 1 was used in calculating the density of the K₂CO₃.H₂O-extract.

$$\text{Density} = \frac{\text{Mass of extract}}{\text{Volume of extract weight}} \quad (1)$$

2.4.6 Viscosity

Viscosity of the K₂CO₃.H₂O-extracts was measured using the Ostwald viscometer, and the calibration was done using a distilled ethanol solution. The kinematic viscosity was first obtained using the Ostwald viscometer, while the dynamic viscosity was then deduced by multiplying the kinematic viscosity by the density of the fluid. The K₂CO₃.H₂O-plant extracts viscosity was also measured using same process and the temperature at which it was carried out was recorded.

2.5 Mud preparation

Water based drilling mud formulated for the research involves the following components: Water, Bentonite clay, Caustic soda, Soda ash, PAC L, XCD polymer, KCl salt, Barite, Commercial surfactant and K₂CO₃.H₂O-plant (*Vernonia amygdalina* and *Terminalia mantaly* leaves) extracts.

Pre-hydration of bentonite clay was done for 16 hours (340 ml of water + 22.5 g of bentonite), for proper yielding and hydration of the bentonite. Every other additives was added into the pre-hydrated bentonite at five minutes time intervals with constant agitation using a Hamilton beach mixer, following the order in Table 2.1 [22, 23].



Various drilling mud formulations were done using the different $K_2CO_3.H_2O$ -plant extracts, as well as the control mud (mud with commercial surfactant and mud without surfactant) and each mud formulation is indicated in Table 2.1. All the chemicals used were made available by Hamilton Technologies Limited.

Table 2.1: Drilling Mud Formulation

MUD COMPONENTS	CONTROL MUD (C1)	CONTROL MUD (C2)	PLANT EXTRACTS MUD	FUNCTION
Pre-hydrated bentonite	340 ml of water + 22.5g bentonite	340 ml of water + 22.5g bentonite	340 ml of water + 22.5g bentonite	Base fluid and viscosifier
Soda ash	0.5 g	0.5 g	0.5 g	Calcium precipitant in low pH mud
Caustic soda	0.5 g	0.5 g	0.5 g	Alkalinity stabilizers
PAC L	1.25 g	1.25 g	1.25 g	Fluid loss and viscosifier
XCD polymer	3 g	3 g	3 g	Fluid loss and viscosifier
KCl salt	5 g	5 g	5 g	Inhibitor
Barite	56 g	56 g	56 g	Weighting agent.
Commercial surfactant	5 ml	-	-	Emulsifiers/ rheology modifier
$K_2CO_3.H_2O$ -Plant extracts	-	-	5 ml	Emulsifiers/ rheology modifier

2.6 Evaluation of the formulated drilling mud properties

2.6.1 Mud specific gravity and pH

Specific gravity was determined using the mud balance for the various muds formulated. The results obtained should match the specific gravity necessary to check formation pressure without fracturing or damaging the formation [24].

Hanna digital pH meter was used to determine the alkalinity of the various formulated drilling mud. Increased alkalinity of drilling mud will result in the use of excess additives, thus, increasing the cost effect on the formulation [25].

2.6.2 Rheological properties

Plastic viscosity, yield point and gel strength were evaluated at temperatures 30, 49 and 60 °C for the different formulated mud. The American Petroleum Institute (API) standard procedure was used to determine the rheological properties of the formulated mud [26]. FANN viscometer was used to measure Plastic viscosity (PV), Yield point (YP) and gel strength [27]. For gel strength testing, gel strength after 10 seconds and 10 minutes according to [28] were measured.

2.6.3 Filtration loss

The filtrate loss volume was measured to determine the filtration characteristics of the mud. API fluid loss apparatus was used at ambient temperature. The filtrate was collected in 30 minutes time ($\Delta P = 100$ psi, no. 5 Whatman filter paper). The volume of filtrate loss was recorded from the graduated cylinder at the end of 30 minutes [29].



3. Results and Discussion

Table 3.1: FTIR Absorption bands and their possible group assignments with $K_2CO_3.H_2O$ -plant extracts

POSSIBLE FUNCTIONAL GROUPS	ABSORPTION BANDS (cm^{-1})	
	Bitter leaf extract	Almond leaf extract
O-H stretching	3394.83, 3302.24	3356.25
C-O stretching	1365.65	1365.65
C-H stretching	2322.37, 2098.62	2114.00
C=C stretching	1643.41	1643.41
O-C=O stretching	1411.94	1411.94

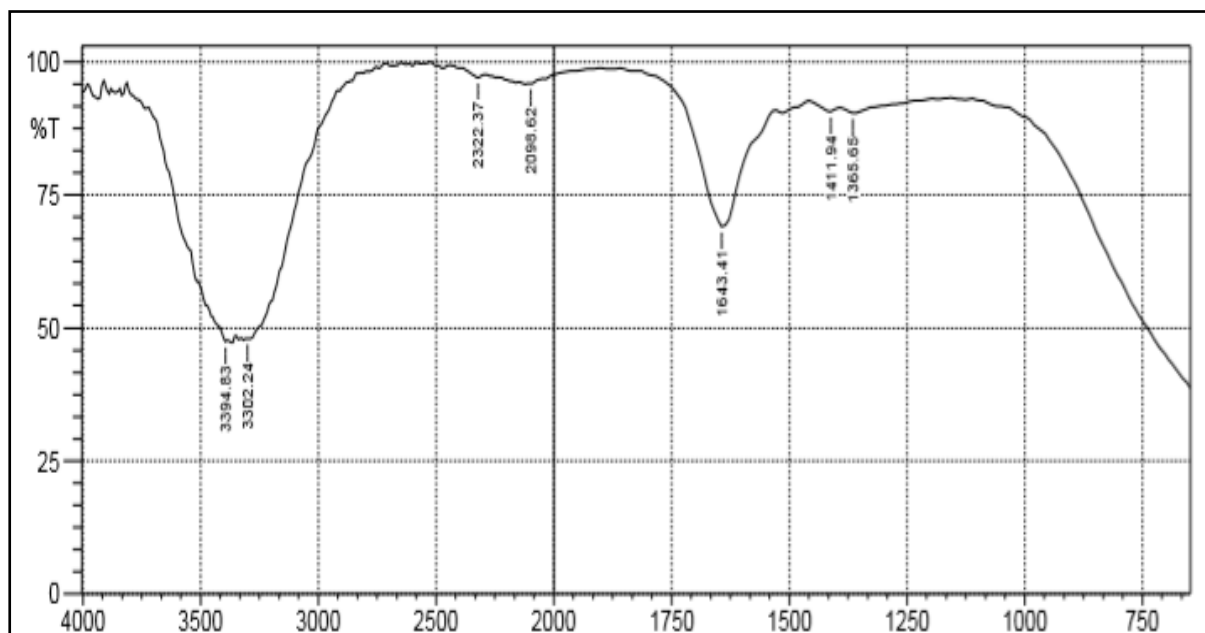


Figure 3.1: FTIR spectrum for $K_2CO_3.H_2O$ -bitter leaf extract

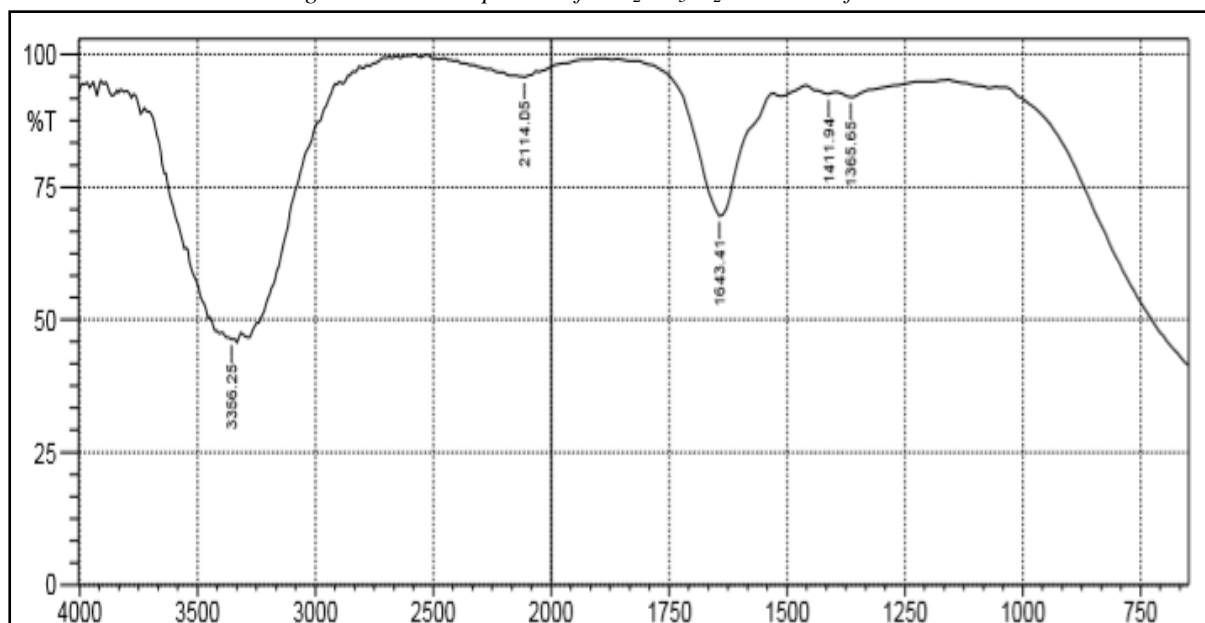


Figure 3.2: FTIR spectrum for $K_2CO_3.H_2O$ -almond leaf extract



Table 3.2: Physicochemical properties of $K_2CO_3.H_2O$ -plant extracts

PHYSICOCHEMICAL PROPERTIES	$K_2CO_3.H_2O$ -PLANT EXTRACTS	
	Bitter-leaf extract	Almond-leaf extract
pH at 27 °C	8.14	7.57
Conductivity (ms/cm) at 26.7 °C	22.40	17.60
Surface tension (mN/m) at 32.5 °C	39.00	54.40
Density (g/cm^3) at 32.5 °C	1.03	1.02
Kinematic viscosity (cSt) at 32.5 °C	0.95	1.02
Dynamic viscosity (cP) at 32.5 °C	0.98	1.04

3.2 Rheological properties

Rheology of drilling mud must be maintained at sufficient values to be able to give optimum results when in use. The rheological properties include plastic viscosity, yield point and gel strength; and were measured at three different temperatures (30, 49 and 60 °C) in this study. Determining and designing these properties is beneficial in producing a good drilling mud will possess certain characteristics and functions such as: removing the drilling cuttings, holding drilling cuttings and weight material in suspension when not circulating, reducing to a minimum any adverse effect upon the well bore, and releasing cuttings at the surface [30].

3.2.1 Plastic Viscosity

Viscosity of drilling mud contributed by the solid and liquid phases of drilling mud is termed plastic viscosity. It can be calculated by the difference between dial reading at 600 and 300 RPM. Plastic viscosity of drilling muds decreases as its temperature increases since the viscosity of its base fluid decreases [31].

Results presented in Table 3.3 show that the plastic viscosities of the various formulated water base mud decreased slightly when the test temperatures were increasing except for ALEM. A decrease in PV value as temperature increases was also observed by [23]. BLEM at 30 °C and ALEM at 49 °C were observed to give the highest PV values (17 cP) compared to other formulated muds. A high PV value causes an increase in pressure drop which eventually reduces flow rate. C2 was also observed to display the lowest PV values (9, 8 and 6 cP) at the different test temperatures (30, 49 and 60 °C) respectively. Degradation of some of the mud additives especially the breaking down of long chain molecules of surfactant into short chain molecules can lead to the decrease in PV values as temperature increases.

Table 3.3: Rheological properties of the various formulated water-based drilling mud at different test temperatures

Sample	Temperature (°C)	Plastic viscosity (cP)	Yield point (lb/100 ft ²)	Gel strength 10 minutes (lb/100 ft ²)	Gel strength 10 seconds (lb/100 ft ²)
Bitter leaf extract mud (BLEM)	30	17	25	5	4
	49	14	30	4	4
	60	13	20	3	3
Almond leaf extract mud (ALEM)	30	15	20	5	4
	49	17	30	6	5
	60	15	20	5	4
Mud with commercial emulsifier (C1)	30	16	22	5	4
	49	14	23	4	3
	60	13	23	4	2
Mud without emulsifier or plant extract (C2)	30	9	70	18	19
	49	8	64	18	18
	60	6	60	16	17



3.2.2 Yield Point

Cleaning a drilled hole and controlling of pressure are the major functions of yield point associated with drilling muds. A high yield point encourages the solid carrying capacity of a drilling mud [32], and it must be high enough to carry weighting materials and also drilling cuttings out of a well-bore, but not too high to generate excessive pump pressure when the pump starts pumping drilling mud [33].

Table 3.3 shows that the YP of C2 muds are higher than C1, BLEM and ALEM. These very high YP values (70, 64 and 60 lb/100 ft² at 30, 49 and 60 °C respectively) can be as a result of the absent of surfactant (surface active agent) molecules in the mud formulation. Absence of surface active agents in mud formulation encourages rapid flocculation of clay solid thus, yielding very high YP values.

The presence of surfactants; K₂CO₃.H₂O-BL extracts, K₂CO₃.H₂O-AL extracts and Ham mul in BLEM, ALEM and C1 respectively, gave desirable YP results when compare to C2. Also, it was observed that the YP values of the various formulated muds did not follow any definite trend as the test temperatures were increasing but C1 gave constant PV values (23 lb/100 ft²) at 49 and 60 °C test temperatures and also ALEM (20 lb/100 ft²) at 30 and 60 °C.

3.2.3 Gel strength

Measuring the attractive forces in a drilling mud system when at rest is termed gel strength. It entails the determination of the shear stress required to institute flow of a drilling mud that has been at rest for sometime [34]. Also, electrically charged particles in a drilling mud linking together to create a firm structure causes gel strength [35].

It can be observed from Table 3.3 that temperature also affects gel strength. The gel strength values at 10 sec and 10 min were gradually reducing as the test temperature increases for the various formulated mud except for ALEM which were inconsistent. However, gel strength at 10 min for C1 gave same gel strength values (4 lb/100 ft²) at 49 and 60 °C, and C2 gave also same gel strength value of 18 lb/100 ft² at 30 and 49 °C.

Also, C1 gave the lowest gel strength value (2 lb/100 ft²) at 10 second when compared to the values obtained by the other muds at 10 seconds gel. Swabbing, surging, difficulty in running log tools, entrapping of gases in muds and difficulty in removing of cuttings at the surface are caused by very high gel strength values [36].

3.3 Filtration loss

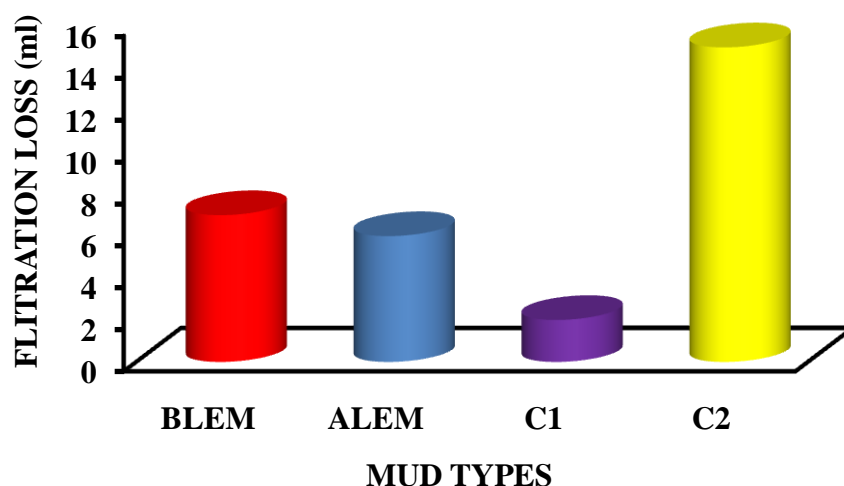


Figure 3.3: Filtration loss of the various formulated water base mud

Drilling mud filter cake texture can be used to estimate the filtration loss during drilling process. Additives in drilling mud influence the filter cake properties [37, 23]. From physical observation, it was noticed that the filter cakes of BLEM, ALEM and C1 were more compact and thin compared to C2. This observation can be as a result of surfactants present in ALEM, BLEM and C1, thus, the surfactant possesses long molecular chains that



make the mud more viscous and form a tight filter cake [38]. From Fig 3.3, it can be seen that the presence of surfactants have successfully enabled BLEM, ALEM and C1 obtain reduced filtration loss (7, 6 and 2 ml respectively), when compared to C2 with filtration loss of 15 ml. Very high filtration loss volume can lead to tight spot in the hole resulting to excessive drag, increase pressure surges when moving the pipe due to decreased hole diameter, differential pressure sticking of the drill string and formation damage from filtrate loss [39].

3.4 Mud pH and specific gravity

Table 3.4: pH and specific gravity of the various formulated water based drilling mud

MUD TYPES	pH	SPECIFIC GRAVITY
BLEM	9.1	1.08
ALEM	9.2	1.09
C1	8.3	1.08
C2	8.2	1.07

Table 3.4 shows the mud specific gravity and pH from the various formulated water based drilling muds. The pH values were measured using a pH meter, while a mud balance was use to measure the mud specific gravity. The pH values obtained from the various muds are in the order; ALEM (9.2) > BLEM (9.1) > C1 (8.3) > C2 (8.2). The muds were observed to be alkaline which is good for drilling muds. However, BLEM and C1 have same specific gravity value (1.08), while ALEM gave the highest specific gravity value of 1.09 and C2 gave the lowest value of 1.07.

4. CONCLUSION

This study was carried out to evaluate the surface active characteristics of *Vernonia amygdalina* and *Terminalia mantaly* leaves potash ($K_2CO_3 \cdot H_2O$) extracts, when used in the formulation of water based drilling fluids. The extracts acted as surfactant in the formulation of the mud; thus, the rheological properties (plastic viscosity, yield point and gel strength), filtration loss volume as well as mud pH and specific gravity were evaluated in other to determine the effects of the $K_2CO_3 \cdot H_2O$ -plant extracts in the muds. The results obtained were compared to mud formulated with a commercial surface active agent (C1) and mud without any surface active agent C2. Based on the analyses of the results obtained, the following can be concluded from this study:

1. The plastic viscosities of the various formulated muds were observed to be decreasing as the test temperatures increased except for ALEM which was inconsistent. The decrease in PV as temperature increased might have resulted from the degradation of the mud additives leading to deflocculating and dispersion of the mud particles.
2. BLEM and ALEM gave optimum yield point values when compared to the control muds used for the study. This relatively high YP values from literature is high enough to carry drilling cuttings and weighting materials out of the wellbore during drilling process. However, C2 gave excessive YP values (70, 64 and 60 lb/100 ft² at 30, 49 and 60 °C respectively).
3. Gel strength values obtained from the study showed that the different formulated mud exhibited flat gel strength, thus desirable during drilling operation. The gel strength at 10 sec and 10 min was observed to be decreasing as the test temperatures increased with the exception of ALEM. Flocculation in water based mud increases gel strength while deflocculation decreases the gel strength.
4. pH values obtained from the study are in the order: ALEM (9.2) > BLEM (9.1) > C1 (8.3) > C2 (8.2), while the specific gravity is in the order of ALEM (1.09) > C1 (1.08) > BLEM (1.08) > C2 (1.07).
5. The presence of surfactants ($K_2CO_3 \cdot H_2O$ -plant extracts) has successfully enabled BLEM and ALEM to exhibit reduced filtration loss (7 and 6 ml respectively), when compared to C2 with filtration loss of 15 ml.



Nomenclature list

ALEM	Almond leaf extract mud
BLEM	Bitter leaf extracts mud
C1	Mud with commercial surface active agent
C2	Mud without surface active agent
YP	Yield point
PV	Plastic Viscosity
API	American petroleum Institute
FTIR	Fourier-transform infrared spectroscopy
AL	Almond leaf
BL	Bitter leaf
PAC L	Polyanionic cellulose polymer
KCl	Potassium Chloride
XCD	Xanthan gum
K ₂ CO ₃ .H ₂ O	Potash

Declaration of conflicting interests

The author(s) declared no potential conflicts of interest with respect to the research, authorship and/or publication of this article.

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