Available online www.ejaet.com

European Journal of Advances in Engineering and Technology, 2020, 7(4):28-35

Research Article **ISSN: 2394 - 658X**

Determination of the inhibitive potassium ion in Detarium microcarpum and its effects on swelling shale

S.U. Eke, A. O. Ejiofor, F. E. Ekesi and K.C. Igwilo

Department of Petroleum Engineering, Federal University of Technology Owerri,
Imo State, Nigeria
solomon.eke@yahoo.com

ABSTRACT

This research involves the determination of the amount of inhibitive potassium ion in Detarium microcarpum and how it affects shale swelling. In this research the physical properties and major oxide composition of two different shales were determined using AA320N Spectrophotometer. The amount of inhibitive potassium ion in Detarium microcarpum was determined using Flame Photometer. The clay contents of the shales were also determined and swell test was carried out on the shale with higher clay content by measuring the increment in volume of the shale when water was added to it. The results obtained indicate that the shales differ in their clay contents and have different swelling tendencies. A locally sourced material, Detarium microcarpum was obtained and was separated into two, one portion was shelled and the other was unshelled. Each of the portions was prepared and tested for potassium ion using flame photometer and it was discovered that the unshelled portion contained higher amount of potassium ion. The potassium ion was extracted and was used to carry out inhibition test on the shale by using different concentration of the extract. The results obtained indicate that the shale swelling was inhibited. This work therefore proposes the use of Detarium Microcarpum as an additive in water based drilling fluid for shale swelling inhibition.

Key words: Spectrophotometer, Detarium Microcarpum, Flame Photometer, Potassium ion, Inhibition

INTRODUCTION

Shales contain clays that swell, dispersed and slough into the wellbore resulting in wellbore instability which leads to hole enlargement, tight hole, decreased penetration rate, lost circulation, bit balling, stuck pipe and other difficulties in drilling and completion of wells. Shales that cause the most difficulty contain a high percentage of clays. The difficulties occur when the shale swells after being exposed to the drilling fluid. Shale instability has been a major problem in the oil and gas drilling industries. Maintaining a stable borehole is one of the major tasks encountered in the oil and gas industry due to the fact that wellbore instability-related problems will result in additional high drilling costs and have a severe impact on drilling schedule [1].

A lot of works have been published with the intention to clarify the uncertainties about shale-fluid interaction. The study of shale fluid interactions became popular in the seventies after many publications by Chenevert that revealed the importance of drilling fluid activity in the successful inhibition of shale formations. In his first researched work, He experimentally tested a wide range of shales hence expanding the knowledge of shale characteristics [2].

Ballard developed an experimental technique using radioactive tracer to monitor water and ion movement in shales [3]. He found it to be diffusion dominated process when no force is applied and the driving force for the transfer of the ions in and out of shale is concentration gradient. Van pointed out that two mechanisms are responsible for the swelling of clays [4]. These are surface hydration and osmotic swelling. Surface hydration shows little signs of swelling but the hydration energy is high and large amounts of pressure are required to desorp surface hydration water. Osmotic swelling occurs when the concentration of ions at the wellbore wall is higher than that of the drilling fluid. When this is the case, water moves toward the clay surface causing swelling. The amount of swelling depends on concentration of salts in the shale relative to that of the drilling fluid. This means that osmotic swelling could be controlled if the concentration of the salts in the drilling fluid is higher than that in the shale. Problem shales were classified by O'Brien and Chenevert according to the dominant clay mineralogy; these clays included Montmorillionite, Illite and Chlorite [5]. They also highlighted three major factors known to cause shale problems. These factors include shale swelling, shale cutting dispersion, and abnormal pressure. When not properly managed, these factors further result in borehole washout, bit balling, caving, sloughing and heaving, stuck pipe, high torque and drag among others [1, 6]. Dispersion causes shale particles to disintegrate into the drilling fluid. These solids are difficult to remove and cause problems that could lead to hole washout.

Water-Based Mud and Inhibitive Fluids

For many years, attempts have been made to overcome the difficulties encountered in drilling shale formations. Several researchers have designed both water and oil-based drilling fluids to increase wellbore stability. Mondshine developed a technique that determined salinity requirements of an oil-based mud in order to provide adequate inhibition [7]. Despite the fact that success was achieved using oil based muds, cost and environmental factors has made it necessary to design a locally made water-based mud additive to control shale instability. O'Brien and Chenevert demonstrated the effectiveness of using Potassium Chloride as a shale inhibitor [5]. Steiger suggested the use of potassium/polymer drilling fluids for shale inhibition [8]. These muds are less expensive and easier to use than oil-based muds. Several salts, polymers and their combinations are among the earliest mud systems recommended for the inhibition of shale swelling [9]. Salts become ions in aqueous solution and are free to move into the clay structure. Here, they occupy spaces which would otherwise be engaged by hydrogen ions from water molecules. The potassium ion in particular is able to inhibit shale reactivity due to its ionic size. This is mainly because the ionic size of the potassium ion, 2.66 Ǻ is closer to that of the space between the clay platelets, 2.8 Ǻ than any other exchangeable cation [5].

Plasticity Index of Shale

Plasticity Index (PI) is the range of water content over which shale has a plastic consistency. The consistency of most shale will be either plastic or semi-solid. Shale strength and stiffness behaviour are related to the range of plastic consistency [10]. Plasticity Index (PI) is the difference between the liquid limit and the plastic limit.

PI = Liquid Limit (LL) – Plastic Limit (PL)

PI = LL – PL

Clays are distinguished from Silts based on plasticity index. Shales with a high PI tend to be clay, those with a lower PI tend to be silt, and those with a PI of 0 (non-plastic) tend to have little or no silt or clay.

Rock samples with higher clay contents exhibited higher swelling tendencies [11]. Figure 1.0 shows the modified plasticity chart with plot of samples containing fine particles.

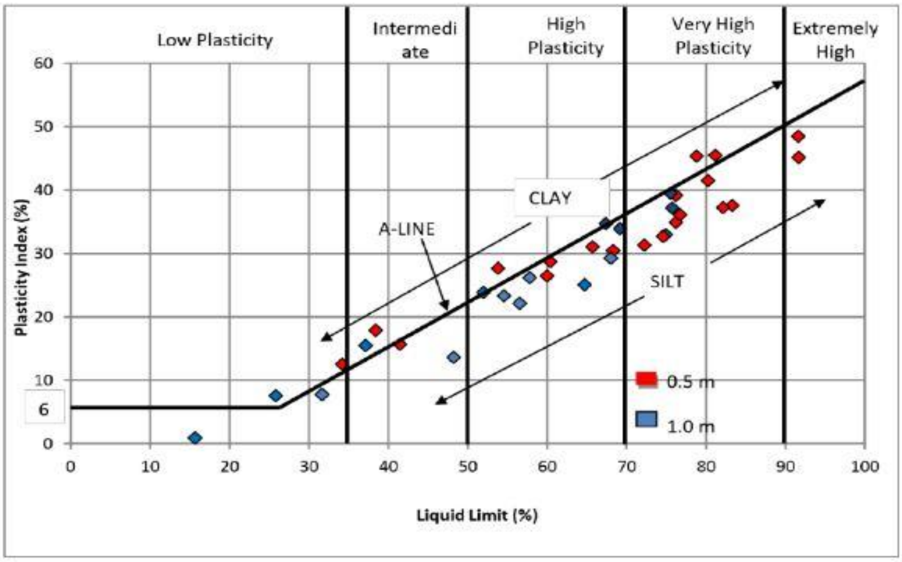

Fig. 1 Modified plasticity chart with plots of the samples containing fine particles [12]

Detarium Microcarpum

Detarium microcarpum is an African tree with height reaching up to 15 m, with distinguished grey bark, with dark green leaves, the tree can produce about 7 kg sweet fruits. It happens normally in the drier districts of Africa [13]. It belongs to the family caesalpiniceae, phylum spermatophyte and the order fabacea. It is particularly associated with dry savannah countries. It is known to flower throughout the wet season and fruits between November and January. The fruits are fleshy and quite edible [14]. The seed of *Detarium microcarpum* has high amount of potassium (105mg g^{-1}), sulphur (1.63mg g-1) and iron (3.12mg g-1). That means that the highest chemical content in Detarium microcarpum is potassium. The chemical constituents of *Detarium microcarpum* (Guill&Perr) plant was investigated for a comparison of the biological actions of the chemical composition of its leaves stem and root barks. Extracts from the leaves, stem and root barks were analyzed for feeding deterrent and contact toxicity activities. [15].

METHODOLOGY

Experimental Site

This research was carried out in chemistry laboratory and erosion study laboratory at Federal University of Technology Owerri (FUTO) Imo State, Nigeria.

The area is geographically located between latitude 05°27^1 N and longitude 07°02^1 E at an altitude of 91m above sea level. The area has a mean annual rainfall of 2300-2700mm and average minimum temperature of 18°C and maximum of 33°C.

Sample Collection and Preparation

Detarium microcarpum was obtained locally from Owerri main market in Imo State. It was divided into two portions, one was shelled (back cover was removed) and the other was unshelled (back cover was not removed) as shown in the Figure 2.0.

- The unshelled *Detarium microcarpum* was labeled sample A
- The shelled *Detarium microcarpum* was labeled sample B

Fig. 2 Detarium microcarpum; Sample A (shelled portion) and Sample B (unshelled portion)

Shales used for this research was obtained from two different locations namely; Ihube and Ezinachi in Okigwe Local Government Area, Imo State. These shales were tested to determine their physical properties. Figure 3 shows the two shales used for this research and labeled as follows;

- The Ihugbe shale labeled sample C
- The Ezinachi shale labeled sample D

Sample C (Ihube shale)

Sample D (Ezinachi shale)

Fig. 3 Shales obtained from Ihube and Ezinachi locations, labelled as Sample C and D resepectively

MATERIALS AND METHODS

Table 1 shows the apparatus used for the determination of the physical properties of the shales and the test for potassium ion in *Detarium microcarpum*.

Table -1 Apparatus used for the research and their function

Name of Apparatus	**Function**
Pestle and mortar	Used to grind the samples
Mettler pin 163 TLAB	Used to measure the mass of samples
Hot Plate	Used for ashing of samples
Spatula	Used to evenly turn the sample while ashing
Filtrate paper and Funnel, 3 inch	Used to filter the sample
Flame analyzer (photometer)	Used to determine the Potassium ion (K^+)
AA320N spectrophotometer	Used to determine the oxides in the sample
Beaker, 250 ml, glass	Used to measure the volume of liquid used for analysis
Sieve 425μm	Used to sieve the samples

Test for Potassium Ion

3.0g each of the samples were measured separately (using Mettler pin 163 Top Loading Analytical Balance), ashed for about 30minutes and allowed to cool. 10ml each of concentrated Hydrochloric acid (HCl) and Trioxonitrate (v) acid (HNO_3) was added into each of the samples and allowed to stand for about 60minutes. The mixture was heated for about 5minutes to evaporate some of the solvent but not to dryness and was allowed to cool for about 5 minutes. 50ml of distilled water was added into each of the mixtures stirred and filtered. The filtrates from each of the samples were sucked into the flame photometer and the amount of potassium ion in each of the samples was determined and recorded.

Tests for major oxides in Shales

The shale samples were dried at 105°C for 1 hour in the oven. 1g of each of the samples was measure and grinded with pestle and mortar. 50% HNO_3 solution was prepare, 10ml of the prepared acid was added into grinded samples. The solution was boiled for 30minutes and allowed to cool, 5ml of Hydrogen peroxide (H_2O_2) solution and 2ml of distilled water was added then boil for 10minutes. The solution was filtered into a 50ml volumetric flask and made up to the 50ml mark. The sample was analyzed with AA320N spectrophotometer.

Test for liquid limit of the shales

The shale samples were crushed and sieved using a 425μm sieve size then mixed with distilled water. The mixture was allowed to stand for 3 days (maturation). The mixture was then remixed and some portion was placed in the bowl on casagrande apparatus. The sample was cut into two equal halves (grooved). Blows were repeatedly applied to the sample and the number of blows required to join the grooved sample together was recorded. The procedure was repeated and the number of blows recorded in each case.

Test for Plastic limit of the shales

The shale samples were crushed and sieved using a 425μm sieve size then mixed with distilled water. The mixture was allowed to stand for 3 days (maturation). The samples were remixed and was rolled into ball and rolled into threads of about 3mm diameter until it crumbles. As soon as the crumbled stage was reached, the crumbled samples were placed into a weighed moisture content container and reweighed. The sample was oven dried overnight, cooled and reweighed.

Shale Swelling Test and Inhibition

Sample C was oven dried and 10ml was measured into the measuring cylinder in five different sections without compacting or shaking the cylinder. Different masses of sample A were measured (5g, 10g, 15g and 20g). The first portion of sample C was tested with 50ml of distilled water by pouring the 10ml dried sample C slowly into the 50ml water contained in a 100ml cylinder and the new volume of sample C was recorded. Each of the remaining portions of sample C was tested with the different masses of sample A and the new volume of sample C was recorded.

RESULTS AND DISCUSSIONS

Potassium ion in Detarium microcarpum

The results obtained for the amount of potassium ion in *Detarium microcarpum* is shown on Table 2. The results indicate that the unshelled portion of *Detarium microcarpum* contains higher amount of potassium ion. This means that the potassium ion content is concentrated on the shell of the seeds as can be seen from the result. Therefore in order to use *Detarium microcarpum* to inhibit shale swelling the shell should not be removed.

Table -2 Amount of potassium (K^+) ion in sample A and B

Sample	Sample A (unshelled)	Sample B (shelled)
Amount of K^+ (mg/L)	7.0849	4.0597
Amount of K^+ (mg/kg)	118.08	67.66

Major Oxides in Shale

The two shales labeled sample C and D contain a wide range of major oxide as shown in Table 3 and 4 respectively. The shales are gray in colour and typically composed of variable amounts of clay minerals and quartz grains. Addition of variable amounts of minor constituents will alter the colour of the shale. Black shale results from the presence of greater than one percent carbonaceous material and indicates a reducing environment. The result obtained shows that the sample has highest percentage of Silicon (iv) oxide followed by Aluminum oxide. These indicate that they are typical gray shale with high quartz content. The variation in the properties is due to differences in the depositional environments of the two samples.

Table -3 Major Oxides compositions of sample C

Element	Composition of Sample C (ppm)	Composition of Sample C (%)
Na_2O	1.620	0.019
K_2O	Nil	Nil
CaO	0.239	0.017
MgO	0.632	0.052
MnO	0.150	0.001
P_2O_5	0.081	0.019
Fe_2O_3	1.692	0.1215
Al_2O_3	250	23.5
TiO_2	0.523	0.045
SiO_2	500	53.8

Table -4 Major Oxides composition of sample D

Element	Composition of Sample D (ppm)	Composition of Sample D (%)
Na_2O	0.947	0.025
K_2O	Nil	Nil
CaO	0.523	0.015
MgO	0.210	0.007
MnO	0.277	0.071
P_2O_5	0.093	0.009
Fe_2O_3	2.593	0.074
Al_2O_3	700	26.32
TiO_2	0.854	0.030
SiO_2	1200	51.36

Liquid limit of the Shales

The liquid limit of shale is the moisture content at which the shale will barely flow under applied force. Table 5 and Table 6 show the percentage water content of samples C and sample D respectively. The liquid limit was obtained by plotting the graph of water content against the number of blow as shown in Figure 4 and Figure 5 respectively. The liquid limit obtained for the two samples varied due to the differences in their clay contents. Initially wet shale reaches consistency at which it stops to behave as a liquid and begins to exhibit plastic behavior and this point is called the liquid limit. This property of shale is important because at this point the shale will form a thick film of water and there is a decrease in the cohesive strength of shale. This point enables a drilling engineer to control the characterize shale formation.

Table -5 Liquid limit of sample C

Trial Number	1	2	3
Can Number	25S	36	42.0
Number of Blow	32.0	14.0	10.0
Weight of Can (g)	23.8	19.0	19.1
Weight of Can + Wet Sample (g)	41.9	34.4	39.2
Weight of Can + dry Sample (g)	34.6	27.6	30.6
Weight of Dry Sample (g)	10.8	8.60	11.5
Weight of Water (g)	7.30	6.80	8.60
Water Content (%)	67.6	79.1	74.8
Liquid Limit (%)	70.1	70.1	70.1

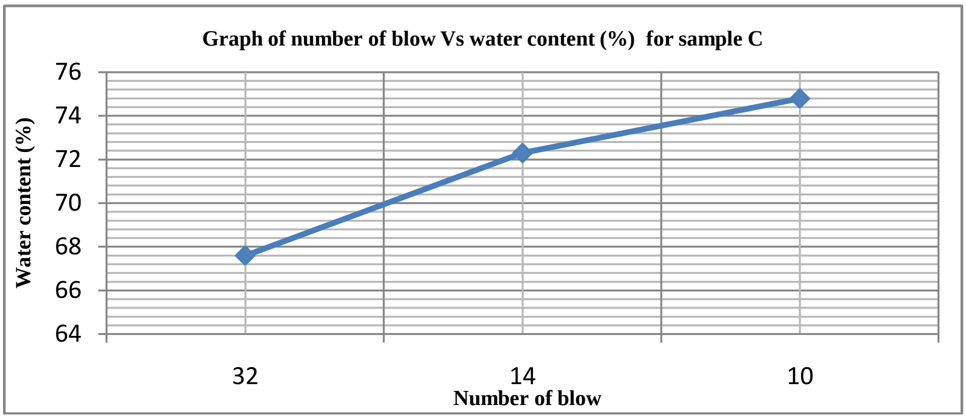

Fig. 4 Graph of water content against number of blows for sample C

Table -6 Liquid limit of sample D

Trial Number	**1**	**2**	**3**
Can Number	13X	11	5
Number of Blow	9.0	19.0	26.0
Weight of Can (g)	4.7	4.9	5.5
Weight of Can + Wet Sample (g)	15.7	15.3	22.9
Weight of Can + Dry Sample (g)	13.0	12.8	18.8
Weight of Dry Sample (g)	8.30	7.9	13.3
Weight of Water (g)	2.70	2.50	4.10
Water Content (%)	32.5	31.6	30.8
Liquid Limit (%)	30.9	30.9	30.9

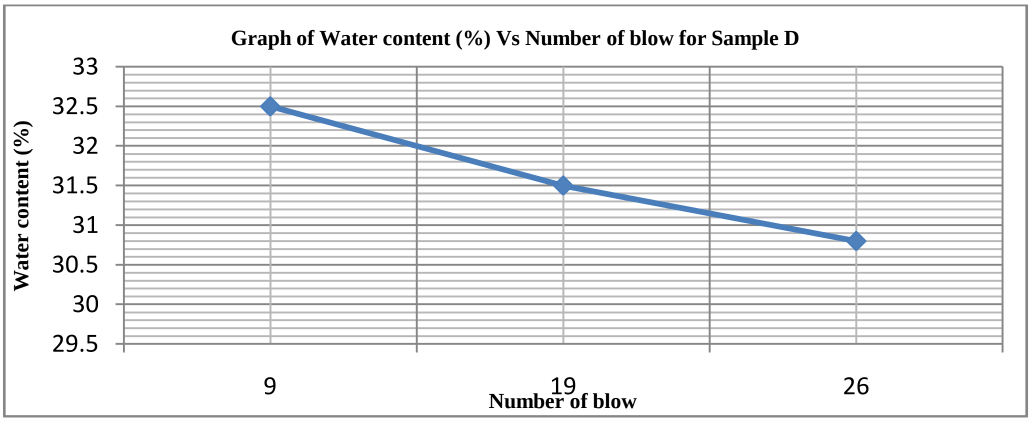

Fig. 5 Graph of water content against number of blow for sample D

Plastic limit of the shale

Plastic limit indicates the lowest moisture content at which the shale is plastic. At the plastic limit the shale can barely be rolled out into a wire form of 3mm diameter. The plastic limit of the two samples is shown in Table 7 and Table 8. The variation in the values is due to the differences in their clay content, organic matter content and depositional environments. Plasticity is a function of the content of the finer particles which determines the amount of surface available for water adsorption. Similarly organic matter has high adsorption capacity for water therefore plastic limit occurs at relatively high moisture content.

Table -7 Plastic limit of sample C

Trial Number	**1**	**2**
Can Number	57	20C
Weight of Can (g)	8.1	13.6
Weight of Can + Wet Sample (g)	14.5	21.4
Weight of Can + Dry Sample (g)	12.8	19.4
Weight of Dry Sample (g)	4.70	5.8
Weight of Water (g)	1.7	2.00
Water Content (%)	36.2	34.5
Plastic Limit (%)	35.35	35.35

Table -8 Plastic Limit of sample D

Trial Number	1	2
Can Number	12	0W
Weight of Can (g)	13.2	13.4
Weight of Can + Wet Sample (g)	24.9	31.0
Weight of Can + dry Sample (g)	22.8	27.8
Weight of Dry Sample (g)	9.60	14.4
Weight of Water (g)	2.10	3.20
Water Content (%)	21.9	22.2
Plastic Limit (%)	22.05	22.05

Plasticity index of the Shale

For sample C, the liquid limit is obtained from Figure 4.0 at 25 blows the water content is 70.1% which is the liquid limit and the plastic limit of Sample C obtained from Table 7.0 is 35.5%. Therefore the plasticity index for this sample is 34.6%. This value shows that Sample C has high plasticity index hence, high clay content which will result in high swelling tendency. For sample D, the liquid limit is obtained from Figure 5.0 at 25 blows the water content is 30.9% which is the liquid limit and the plastic limit of Sample D obtained from Table 8.0 is 22.05%. Therefore the plasticity index for this sample is 8.85%. This value indicates that the Sample D has low plasticity index hence, low clay content which will result in low swelling tendency. Therefore only Sample C was used to carry out the swelling inhibition investigation.

Inhibition of Shale Swelling

Free swell test the increase in volume of the shale from loose dry powder form when it is poured into water expressed as percentage of the original volume. The result of the percentage swell of Sample C and the corresponding inhibition when different potassium ion concentrations of sample A was added to it is shown on Table 9.0 while the graph of percentage swell against potassium ion concentration is shown on Figure 6.0. The results indicates that the shale swell when water was added to it. This is due to the fact that the shale has a high plasticity index. When sample A was added, the swelling was inhibited according to the concentration of potassium ion in the sample.

Table -9 Swell test and inhibition of sample C using sample A

Concentration of k^+ in Sample A (g/50ml)	% Swell of Sample C
0.00	55
5.00	48
10.00	37
15.00	28
20.00	18

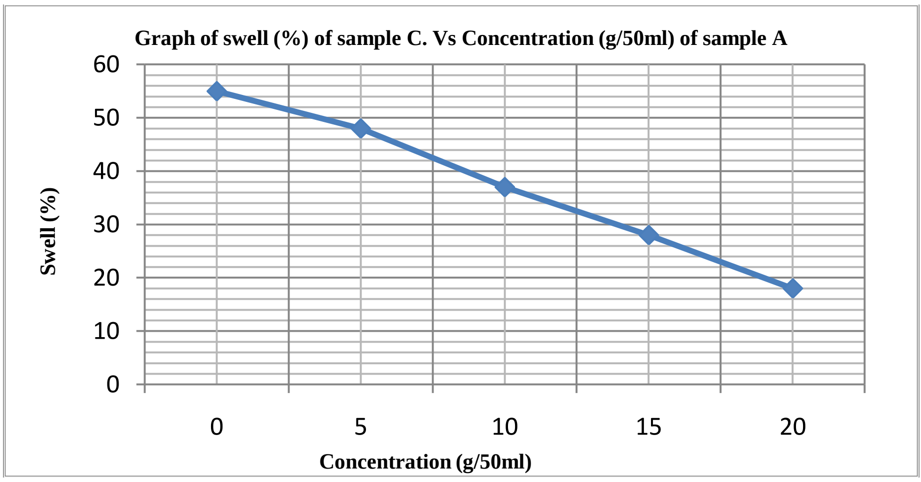

Fig. 6 Graph of percentage swell against potassium ion concentration

CONCLUSIONS AND RECOMMENDATIONS

Based on the analyses of the results, it is safe to conclude that *Detarium Microcarpun* has high amount of potassium ion determined as 118.08ml/kg. The major oxides in shales are Silicon and Aluminum and the swelling tendency of shale depends on its clay contents. Addition of Potassium ion to swelling shale inhibits the swelling.

Therefore, it is recommended that the use of locally sourced *Detarium Microcarpun* as water based drilling fluid additive should be considered. However, further work should be done on the cost of using *Detarium microcarpum* as compared with other drilling fluid additives for robust decision making. Finally, the stability of *Detarium Microcarpum* at high sub surface temperature and pressure should be thoroughly investigated.

REFERENCES

[1]. X. Chan, C.P. Tan and C. Detounay, A study on wellbore stability in fractured rock masses with impact of mud infiltration, Journal of Petroleum Science and Engineering, 2003, Vol. 38, Pp 145-154.

[2]. M.E. Chenevert, Shale Alteration by Water Adsorption, *Journal of Petroleum Technology*, 1970, Vol. 22, pp 1141-1148.

[3]. T.J. Ballard, S.P. Beare, T.A. Laelss, Fundamentals of shale stabilization, water transport through shale, *IADC/SPE24974 Presented at the European petroleum conference held in cannes,* France. 1992, 16-18.

[4]. O.H. Van, Compaction of Clay Sediments in the Range of Molecular Particle Distances Clays-Clay Minerals, 1963 Vol. 11, pp 178-187.

[5]. D.E. O'Brien and M.E. Chenevert, Stabilizing Sensitive Shales with Inhibited, Potassium-Based Drilling Fluids, *Journal of Petroleum Technology*. 1973 Vol. 25, pp 1089-1100.

[6]. R.P. Steiger and P.K. Leung, Quantitative Determination of the Mechanical Properties of Shales, *SPE Drilling Engineering*, 1992, Vol.7(3), Pp 1-5

[7]. T.C. Mondshine, New Technique Determines Oil Mud Salinity Needs in Shale Drilling, Oil and Gas Journal, 1969, pp 70-75.

[8]. R.P. Steiger, Fundamentals and Use of Potassium/Polymer Drilling Fluids to Minimize Drilling and Completion Problems Associated with Hydratable Clays, SPE annual technical conference and exhibition, San Antonio, TX, USA, 1982, Pp 1661.

[9]. A. Patel, E. Stamatakis, S. Young and J. Friedheim, Advances in Inhibitive Water-Based Drilling Fluids - Can They Replace Oil-Based Muds, *SPE 106476 prepared for presentation at the SPE International Symposium on Oilfield Chemistry held in Houston, Texas*, U.S.A.,2007

[10]. J. Atkinson, Soil description and classification, Geotechnical Reference package, Web. https://environment.uwe.ac.uk/geocal/soilMech/classification/default.htm, 2000.

[11]. C.M.O Nwaiwi and I. Nuhu, Evaluation and Prediction of the Swelling Characteristics of Nigerian Black Clays, Geotechnical and Geological Engineering Journal, 2006, Vol. 24, Pp 45-56

[12]. E.A. Gyamera, J.S. Kuma, D. Okae-Anti, Classification of Soils and Their Plasticity Index of the University of Cape Coast Research Station at Twifo Wamaso, International Journal of Soil and Crop Sciences, 2014, Vol. 2(1) Pp 025-032.

[13]. A.A. Mariod, H.E. Tahir, M.G. Komla, *Detarium microcarpum*: Chemical Composition, Bioactivities and Uses. In: Mariod A. (eds) Wild Fruits: Composition, Nutritional Value and Products. Springer, Cham, 2019, Pp 207-217.

[14]. R.W.J., Keay, C.F.A. Onochie and D.P. Standfield, Nigeria Trees, *Science and Education Publishing*. 1964, Vol. 2, Pp. 16-187.

[15]. B.A. Anhwange, V. O. Ajibola, Amino acid composition of seed of moringa oleifera, Detarium microcarpum, and bauhinia monandra, *ChemClass Journal*, 2004. Pp 9-13.