Research Article / Review Article FUPRE JOURNAL 9(2):20-31(2025)

FUPRE Journal

of

Scientific and Industrial Research

ISSN: 2579-1184(Print) ISSN: 2578-1129 (Online)

http://fupre.edu.ng/journal

Development and Characterization of an Oil-Soluble Viscosifier for an Oil-Based Drilling Fluid Preparation with Sugarcane Bagasse

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ARTICLE INFO

Received: 09/02/2025
Accepted: 23/07/2025

Keywords

Drilling fluid, viscosifier, cuttings, obms, rheological properties

ABSTRACT

Drilling fluids are essential in drilling operations, transporting cuttings to the surface, and providing requirements to prevent catastrophic events during drilling operations. This work is aimed at developing a suitable viscosifier, from and agro-waste product (sugarcane bagasse), which contains a considerable amount of wax, which is soluble in the oil phase of the OBM, thereby improving the rheological properties of the entire mud. This was achieved by Solvent Extraction of processed sugarcane bagasse, using carbon tetrachloride as a solvent, Isopropyl alcohol and activated carbon for the treatment of obtained wax. The activated carbon was also produced from 50% of the air-dried bagasse, and wax was extracted from (60g, 75g, 80g, and 85g) of powdered bagasse sample, having a yield of an average of 11.3% (wt. /wt.) basis. Obtained wax was characterized by Fourier Transform Infrared Spectroscopy (FT-IR), indicating the presence of alkanes, ketones, alcohols, aldehydes and carboxylic acids respectively. Oil-based mud having a mud weight of 10.00ppg was prepared, using Diesel oil as base oil with an oil/water ratio of 70/20. It was tested with varying concentrations of the developed sugarcane wax viscosifier (0%, 0.46%, 0.91%, 1.35%, 1.80% and 2.24%), to observe trends in rheological properties such as Apparent Viscosity, Plastic Viscosity, Yield Point, Gel Strengths and Marsh Funnel Viscosity, which revealed a positive increasing trend of rheological properties with an increase in concentration of the viscosifier; carried out at room temperature. OBM Electrical Stabilities were also obtained and revealed a decreasing trend with an increase in viscosifier concentration. Therefore, Sugarcane waxes can serve as substitutes for Viscosifiers in OBMs.

1. INTRODUCTION

Oil and gas exploration is now becoming a huge investment, even as the world advances daily. However, these explorations involve drilling processes which require the use of a drilling fluid (mud), which is a mixture of fluids used to drill boreholes (Juan *et al*., 2018). According to the American Petroleum Institute (API), it is a circulatory fluid used in rotary drilling to perform any or all the various functions required in drilling operations. The major importance of these drilling fluids in the drilling process ranges from transporting drill cuttings which are bits of rocks that break away due to the action of the bit teeth which have to be transported to the surface, and this is to prevent the bit from being stuck, as well as reducing drag and bit wear, to adding pressure control, which is most times referred to as mud window, which is a range of pressures that avoid drilling

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problems (Shah *et al*., 2010).

These fluids serve as means of cooling and lubrication, and many more top-notch functions. These drilling fluids are classified based on preparations which are majorly three, namely;

- Oil-Based Drilling Fluids
- Water-Based Drilling Fluids
- Pneumatic Drilling Fluids

The oil-based drilling fluid/mud can be viewed as a drilling fluid that has oil as its continuous external phase and the water (if present), is dispersed or in the internal phase, and all the solids (if present) in the oil-based drilling fluid are oil-wet. All drilling additives are all oil-dispersible and soluble, as its filtrate is oil, the water (if present) is emulsified in the oil phase (Chinese Patent, 2015). There are also certain oil-based drilling fluids/muds that are referred to as "Conventional All-Oil Muds", because they have oil as the external phase, but they are designed to be free of water when formulated or in use, the nominal oil-based drilling mud (OBMs/OBFs) has namely 95-100% of its liquid phase as oil, with 0-5% as unintentional water (Agwu *et al*., 2015). It can therefore be understood that, oil-based drilling muds have major applications due to its copious advantages over the other types of drilling fluids. These fluids possess special properties and qualities that are essential in borehole drilling. In high-pressure and high-temperature applications, a unique drilling fluid composition is necessary to fulfil the crucial downhole conditions (Ferdous, 2013). Generally, drilling fluids can be classified into oil-based fluids, water-based fluids and pneumatic fluids, depending on their nature and composition. The drilling fluid composition is paramount in reducing drilling time and expense. The essence and overall importance of drilling fluid is to keep the well from blowout and to keep the well in good condition, such that the drilling operations could continue up to the desired extent. The performance of the drilling fluid determines the success or failure of the drilling activity (Shah *et al*., 2010).

A blowout is the uncontrolled release of crude oil and/or natural gas from an oil-well or gas well after pressure control systems have failed. In an oil-based drilling fluid preparation, the development and characterization of some of the compositions of the drilling fluids play a major role, in which a viscosifier stands out. The composition of drilling fluids depends on the additives and components added to the mud, and this aids to achieve the desired parameters and have stable operation while drilling (Fakoya and Ahmed, 2018). Clays and polymers are the most common types of viscosifiers (John, 2018). There are many drilling fluid additives which are used to develop key properties of the drilling fluid, and the most common types of additives used in both water and oil muds include: weighting materials, viscosifiers, filtration control materials, rheology control materials, alkalinity and pH control materials, lost circulation control materials, lubricating materials and the shale stability materials (Shah *et al*., 2010).

In the formulation and development of a suitable viscosifier, locally sourced materials such as sugarcane are used. Sugarcane (*Saccharum officinarum L*.), provides raw material for the second largest agro-based industry after textile. It is considered as one of the best converters of solar energy into biomass and sugar, it is a rich source of fiber (cellulose), fodder (green top, bagasse, molasses), fuel and chemicals (bagasse, molasses and alcohol). During the process of sugar production, the main by-product of cane sugar industry is bagasse, molasses and press mud. The other co-products and by-products of less commercial value are green leaves, green tops, trash, boiler ash and effluents which are generated by sugar industry and distillery (Parameswaran, 2013). Sugarcane bagasse contains considerable amount of

wax, this has good techno-economic potential. This wax is a whitish to dark-yellowish coating on the surface of the sugar cane, which is extracted and separated along with press mud during crushing and processing of the cane juice. This wax portion thus finds applications in cosmetics, paper coating, textiles, fruit and vegetable coating, leather sizing, lubricants, adhesives, polishes and pharmaceutical industry (Gowda *et al*., 2012). Sugarcane wax is extracted from bagasse, a by-product filter residue from the production of sugar from stalks of the sugar cane plant. Sugar cane wax comprises mostly of long-chain fatty alcohols and some fatty acids also, the wax in its refined form has a light yellowish colour; it is relatively hard and has a melting point of 80◦C (Mohan *et al*., 2021).

2. MATERIALS AND METHODS

2.1 Materials

The materials used are, sugar cane bagasse, isopropyl alcohol (IPA), activated carbon, carbon tetrachloride (CCl_4), phosphoric acid (H_3PO_4), conical flask, beakers, soxhlet apparatus, kheldjar digester, condenser, measuring cylinders, electronic weighing balance, oven, furnace, laboratory mortar and pestle, spatula, retort stand, heating mantle, grinder, electrical stability tester (EST), and sieves.

2.2 Methods

The protocols for extraction of sugarcane bagasse wax, were as contained in Chinese Patent (2015), and in Wang (2012). It entails purchasing of 50 stems of sugarcanes (each 24.5cm long) from a market located in Effurun, Warri, Delta State. The sugarcanes were crushed via chewing to obtain bagasse as waste. The obtained bagasse and peels were air-dried for 2 weeks, to reduce its moisture content. Fig. 1 show a typical process.

2.2.1 Preparation of activated carbon

30g of activated carbon was prepared in the laboratory from the sugarcane bagasse, following these procedures. Carbonization was carried out with the aid of a furnace; 50% of the air-dried sugarcane bagasse and peels were carbonized at 400^0C for 2 hours for each batch (4 Batches, in total). The resulting batches of sugarcane bio chars were combined, and size reduction was achieved with the aid of a laboratory mortar and pestle. The resulting bio char was further reduced in size using a 1mm sieve. Activation was done by weighing 30g of the biochar, and mixing with the 0.5M of phosphoric acid, to form a slurry mixture, which was kept in an air-tight container for 24 hours. Thereafter, the slurry mixture was filtered to remove the excess phosphoric acid used for the activation. The sieved activated carbon was neutralized by rinsing several times with warm water. The activated carbon was then kept in the oven at a temperature of 105^0C, for 10 hours, for moisture to be completely removed. The dried activated carbon was then sieved to obtain a smooth fine particle size of 150 microns, which was then stored in an air-tight container for further use.

2.3 Development/Extraction of Viscosifer (Sugarcane Bagasse Extract/Wax)

Sugarcane bagasse wax was extracted using the Soxhlet extraction method.

a) Air-dried sugarcane bagasse and peels were grinded to powder and sieved to 150 microns using a sieve.
b) 60g, 75g, 80g and 85g of the powder were measured and poured into an improvised thimble respectively.
c) The designed thimble containing the sample was placed into the Soxhlet extractor.
d) 500ml of carbon tetrachloride was added to the solvent reservoir flask.
e) The assembled extractor was mounted upon a heating mantle (set to 76.7^0C).
f) Wax extraction was allowed for 8 hours**,** on three different soxhlet extractors setup.

g) The resulting mixture of solvent and sugarcane extract was concentrated by evaporation, to remove the solvent, leaving behind the waxy residue.
h) Residue wax, which contained some impurities was dissolved in isopropyl alcohol (IPA), and was refluxed with the prepared activated carbon for 2 hours to remove any undesirable colour/pigment present.
i) The extract was then filtered with the aid of filter papers, to remove the activated carbon.
j) Wax solutions still containing IPA after filtration were concentrated to obtain crude wax.

Fig. 1: Obtained Crude Wax

2.4 Rheological Tests

2.4.1 Oil-based drilling mud preparation

Table 2.1: The Components used in the oil-based drilling mud preparation and their quantities.

COMPONENTS	FUNCTIONS	QUANTITY
Diesel	Base oil	192ml
Water	Liquid phase	48ml
Organophilic Bentonite	Viscosity enhancer	8.5g
Barite	Weighting agent	85g
Slaked Lime $Ca(OH)_2$	pH controller	5g
CPMUL-P	Primary emulsifier	4g
CPMUL-S	Secondary emulsifier	2g
25% $CaCl_2$ solution	Reactive clay stabilizer	40ml

Without the addition of the new sugarcane extract, the drilling fluid was prepared as follows:

a) 25% $CaCl_2$ Pre-Mix:
25g anhydrous $CaCl_2$ was dissolved into 75ml distilled water, and was stored for further use.
b) With the aid of a measuring cylinder, 192ml of diesel oil was measured and poured into a mixing bowl of the mud mixer.
c) While stirring on the mixer at low rpm (gear 1), CPMUL-P and CPMUL-S were added, while ensuring that they were fully dissolved in the oil.
d) The stirring speed of the mixer, was then increased to the highest rpm (gear 3), and the prepared 25% $CaCl_2$ solution was slowly added to the oil.
e) The remaining additives were then added in the order:
- Lime
- Organophilic Bentonite
- Slaked lime
- Barite

f) The mixture was further stirred for 30 minutes to obtain desired mud

2.4.2 Determination of oil-based drilling mud density

The mud weight was determined with the aid of a mud balance as follows:

a) Calibration
- The lid was removed from the cup and the cup was completely filled with water.
- The lid was then replaced and wiped dry.
- The balance arm was replaced on the base, with the extension resting on the fulcrum.

- The rider was adjusted gradually until a balance point was attained at 8.33ppg.

b) After calibration of the mud balance with water, the water is poured out, wiped dry for mud weight measurement.
c) The cup was subsequently filled with the oil-based mud (without viscosifier).
d) The lid was replaced and rotated, until it was firmly seated, while ensuring that some mud is expelled through the hole in the cup.
e) The expelled mud outside the cup was wiped.
f) The balanced arm was then placed on the base, with the extension resting on the fulcrum.
g) The rider was moved continuously, until the graduated arm was levelled, which was indicated by the level vial on the beam.
h) The density of the oil-based mud was then read and recorded from the left-hand edge of the rider, without disturbing it.
i) The mud temperature corresponding to density was then measured and recorded.

2.4.3 Viscosity test

a) The newly mixed mud (without the addition of viscosifier), was poured into the sample cup to the marked line (350 ml).
b) The cup was then placed on the tray immediately.
c) The tray was lifted to make the liquid level of the inner cup reach the marked point of the outer rotary cylinder.
d) The speed was adjusted from high to low (600rpm and 300rpm) for the respective measurements.
e) The reading under each chosen speed was recorded, after the reading of the dial was stable.
f) Steps (a –e) were then repeated, with the intermittent addition of varying volumes of the prepared sugarcane extract as viscosifier.

2.4.4 Determination of gel strength of OBM

a) The samples (A-E, Z), were stirred at 600rpm for 15 seconds
b) The stirred samples were allowed for the desired rest time of 10 seconds and 10 minutes respectively.
c) Subsequently, the RPM knob was switched to the GEL position.
d) The maximum deflection of the dial before breakage of the gel was observed and recorded in $lb/100ft^2$.

2.4.5 Determination of marsh funnel viscosity

a) Calibration
The funnel was first filled to the bottom of the screen (1500ml) with water at 70^0F (21.1^0C), and the time of outflow of the quart (946ml), was modified to be 26 seconds (with a range of plus or minus ½ seconds).
b) Ensuring the funnel was in an upright position, the orifice was covered using the fingertip, and the freshly collected samples (labelled A-E, Z) for different intervals were poured through the screen into a clean dry funnel, until the fluid level reached the bottom of the screen (equivalent to 1500ml).
c) Fingertip was immediately removed from the outlet, and the time required for the OBM to fill the receiving vessel to 1-quart (946ml) level was recorded in

seconds as the Marsh Funnel Viscosity.

2.5 Electrical Stability Test (EST)

a) The mud (containing complete additives), was stirred at 6000rpm for 10 minutes.
b) Using an ES/Resistivity meter, the electrical stability of the mud was measured at 49^0C (120^0F), and recorded.

2.6 Fourier Transform Infrared Spectroscopy Analysis (FT-IR)

With the aid of an FT-IR Spectrometer, the FT-IR spectra of the wax extracts were recorded at 30^0C, with the FT-IR spectrometer scanned over the frequency range of 3500-1000cm^{-1}, and a resolution of 8cm^{-1}.

3. RESULTS

3.1 Viscosifier Yield

Feasible yield of sugarcane bagasse extract, finds its expression with Soxhlet Extraction Method. Experiments conducted reveal that proper condensation medium is required for adequate yield of wax.

3.1.1 Viscosifier experimental results

In accordance with the existing principles of Soxhlet extraction, wax yield with this method was found to be 11.31% (wt/wt) of the air-dried sugarcane bagasse and peels. Compared to wax extracted from Press mud (48.5%). Hence this is attributed to the huge quantity of sugarcane juice that gets filtered during pressing, resulting press mud having higher concentration of wax, whereas, sugarcane peels are produced by shredding of surface of sugarcane before crushing, this affects the total yield.

Standard principles according to Chinese Patent, (2015) and Wang (2012) were followed, revealing the paramount role of size reduction of samples for adequate extraction.

From Figures. 2 and 3, the number of runs describes the amount of contact time allowed for the solvent and the sample, for complete extraction which is known by the clear extracts that indicates completion of extraction.

3.1.2 Extraction experiment specifications

The experimental requirements are:
Mass of raw material (Air-dried sugarcane bagasse) = 400g

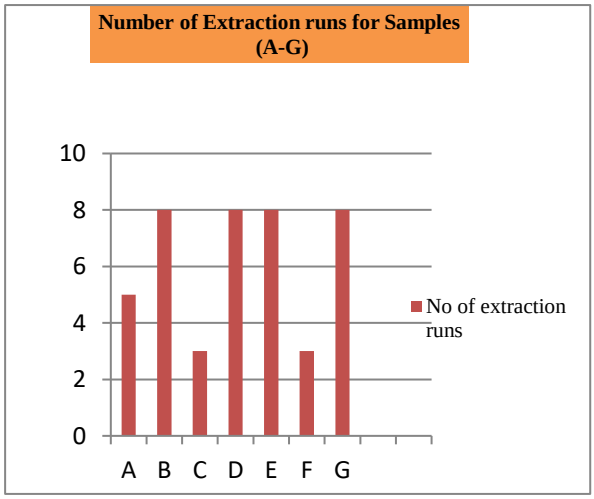

Figure 2: Number of extractions runs for each sugarcane bagasse sample

Volume of CCl_4 = 3L
Volume of IPA = 2L
Mass of activated carbon = 30gm

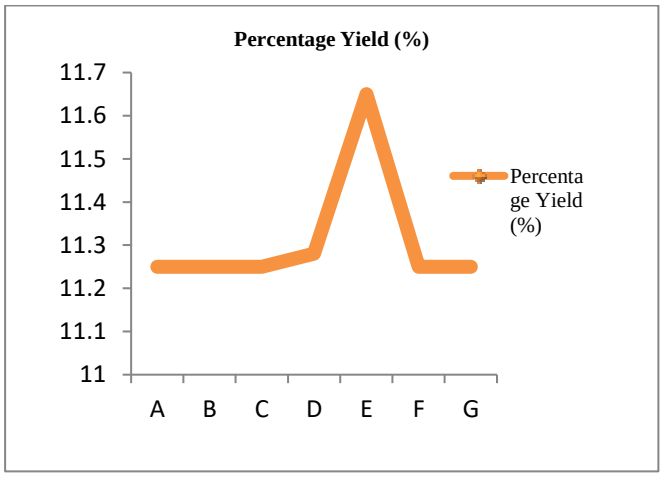

Figure 3: Percentage yield of sugarcane bagasse extracts

3.2 Fourier Transform Infrared Spectroscopy (FT-IR) result of Sugarcane Wax

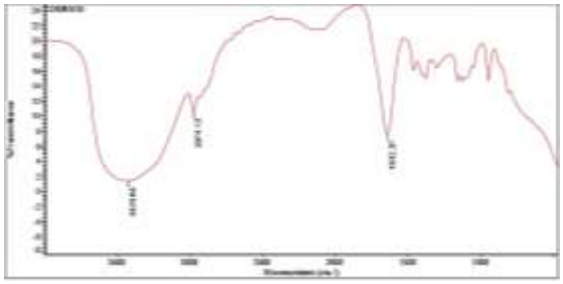

Figure 4 FT-IR Analysis of Sugarcane Bagasse Wax

Table 1: Wavenumbers of Sugarcane Wax Spectra, and their Corresponding Functional Groups they represent.

Wave numbers	Functional Group	Intensity
3423.84	- OH Stretch	1.426
2974.13	- CH Stretch	10.478
1642.37	- C=O Stretch	7.678
1462.44	- CH_2 Stretch	16.021
1374.96	- CH_3 Stretch	18.124

From Fig. 4 and Table 1, the FT-IR analysis of sugarcane bagasse wax, revealed the presence of several organic functional groups, indicating their respective compounds. Also, the absorption bands in the FT-IR Spectra have different intensities that is usually referred to as strong (s), medium (m), weak (w), broad and sharp. The intensity of the absorption band depended on the polarity of the bond, and thus, the bonds with higher polarity showed more intense absorption band. Hence, the sugarcane wax sample produced was found to predominantly contain five main classes of compounds namely; alkanes, fatty acids, alcohol, aldehyde and esters.

3.3 Nature of Prepared Oil-Based Mud

The oil/water ratio for both samples of OBMs was prepared with oil/water ratio of (70:30). An oil-based drilling mud can function well with an oil/water ratio ranging from 65/35 to 95/5, though the most commonly used range is from 70/30 to 90/10. Mud sample I was prepared with a commercially available viscosifier for OBMs (organophilic bentonite), while Mud sample II contains the developed viscosifier (sugarcane wax), which was varied and added in percentages to observe trends in varying temperatures.

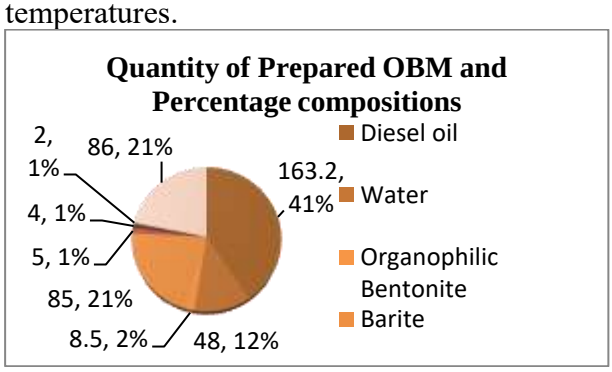

Figure 5: Representation of components, quantities in grams, and percentage compositions of prepared oil-based (Sample I)

The various percentage compositions, that the components of the prepared OBM (Sample I) occupy, are shown in Fig. 5. The percentage compositions were obtained by converting the volumes in ml to mass values in grams, to evaluate the compositions. The mass values were obtained by multiplying the volume in ml by the individual densities of the required components. Table 2 omits percentage composition, as the sugarcane wax quantity was varied to observe trends in rheological properties of the OBM, as shown below.

Table 2: Components of Mud (Sample II), with varied addition of developed sugarcane wax as viscosifier and their various quantities.

Mud Components	Quantity
Base oil	192ml
Water	48ml
Sugarcane wax (developed viscosifier)	Varied
Barite	85.0g
Slaked Lime ($Ca(OH_2)$)	5g
Cpmul-p	4g
Cpmul-s	2g
25% $CaCl_2$ solution	40ml

3.4 OBM Rheological Results

Calibration weight = 8.33 ppg
Mud Weight = 10.00 ppg
Mud samples (labelled **A – E**), each having a density of 10.00 ppg were analyzed with different quantities of sugarcane wax as a viscosifier. Sample Z is the initial mud sample without any added viscosifier, also with a mud weight of 10.00 ppg.

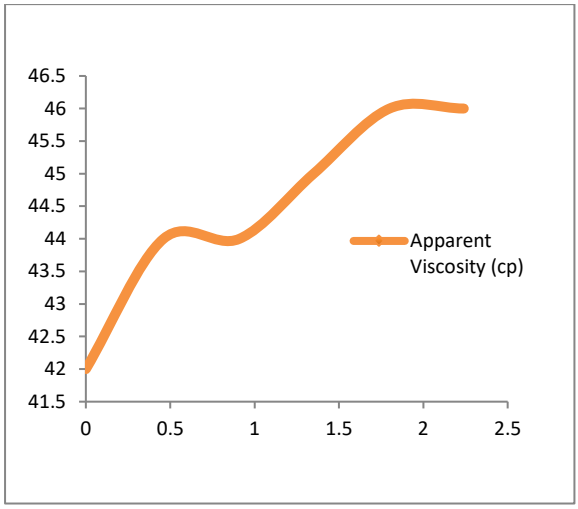

Figure 6: Effects of increasing concentration of sugarcane wax on the apparent viscosity.

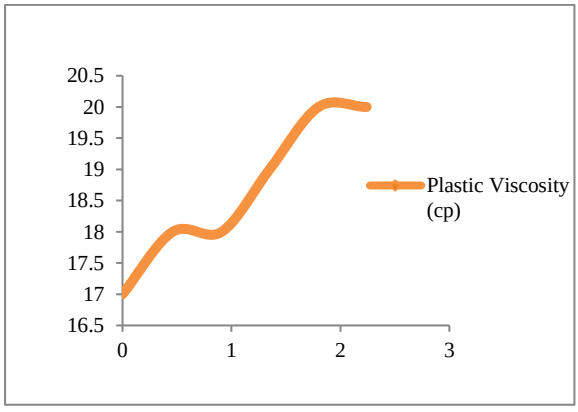

Figure 7: Effects of increasing concentration of sugarcane wax on plastic viscosity of OBM

3.5 Apparent Viscosity of Continuous Phase
Apart from the flow of OBM, the continuous phase, apparent viscosity is investigated in Fig. 6 to determine the suitable percentage of viscosifier that will be in a typical OBM. Investigations under different temperatures reveals a declination in viscosity,

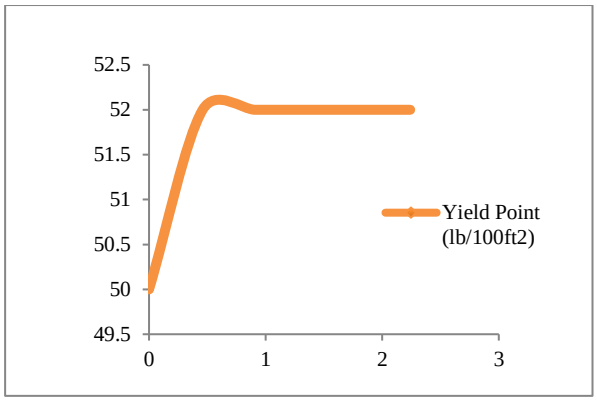

Figure 8: Effects of increasing concentration of sugarcane wax on yield point

as the viscosity of a fluid decreases with an increase in temperature. The main component of the continuous phase was diesel oil, and apparently, its reaction under any condition will primarily affect the flow properties of the continuous phase. As composition of viscosifier increases, viscous properties of the mud also increase. However, with increase in temperature, the strength of the intermolecular forces diminishes, which accounts for declination in viscous properties. Addition of various concentrations of Sugarcane Wax thereby, increases the Apparent Viscosity.

3.6 Plastic Viscosity and Yield Point of OBM

Similar trends are revealed in Fig.7, Fig. 8 and Fig. 9, as the concentration of the sugarcane wax increases from (0 – 2.24%). These same trends also surface, while using organophilic bentonite as viscosifier. The yield point is a function of the plastic viscosity as well. The yield point is the difference between the readings at 300rpm and the plastic viscosity. The 300rpm readings are also dependent on the plastic viscosity of the OBM, which accounts for the proportional trend at increasing viscosifier concentrations.

3.7 Initial and Final Gel Strength Result of OBM

In Fig. 10 and Fig. 11, the increasing Gel Strength is a function of inter-particle forces. The initial 10-second and 10-minute gel strength measurement, give an indication of gelation that occurs after circulation has ceased, and the mud remains static. Hence, the more the mud gels during shutdown periods, the more pump pressure will be required to initiate circulation again.

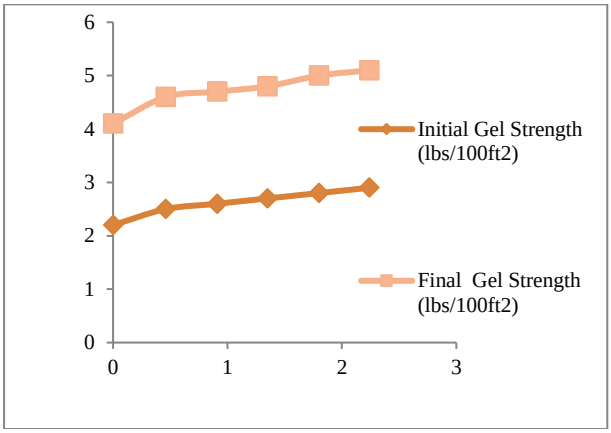

Figure 9*: Effects of Increasing Concentration of Sugarcane Wax on Initial Gel Strength and Final Gel Strength.*

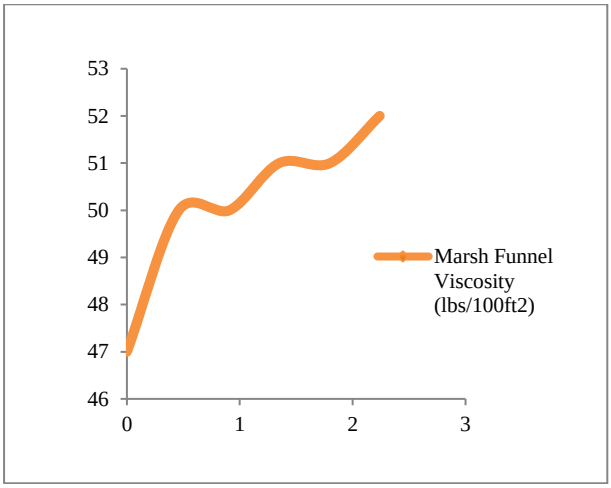

Figure 10: Effect of increasing concentration of sugarcane wax on the marsh funnel viscosity.

3.8 Marsh Funnel Viscosity Result of OBM

This is reported in seconds allowed to flow out of the funnel. It measures the apparent viscosity of the OBM. As the viscosity is increased due to the increased concentration of the sugarcane wax, the longer the time interval in (sec) it takes for mud to flow out of the funnel. Experiments were carried out at room temperature.

3.9 Electrical Stability (ES) Result

The ES of the OBM was found to be 250V at room temperature. The ES is dependent on the Oil/water ratio of the mud, and the ability of the oil phase to form an emulsion in water. Oil/water ratio of the mud was prepared to be **(**70/30**)**, with the oil phase in the higher ratio. As temperature increases, ES decreases, as it is dependent on the water phase of the OBM. The higher the water percentage in the mud, the higher the ES value, and the higher the emulsion potential. However, with a higher percentage of oil, the ES value drops from 250V.

3.10 Organophilic Bentonite and Sugarcane Bagasse Wax

All results show the suitability of sugarcane wax as viscosifier in place of organophilic bentonite in OBMs. Organophilic bentonite which is organoclay is an organically modified form of bentonite. Similarities are shown between the two in major ramifications.

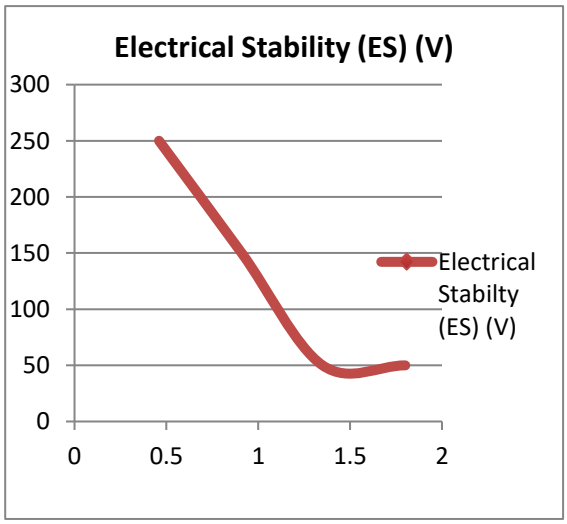

Figure 11: Effect of increased concentration of sugarcane wax on electrical stability (ES) of OBM.

4. CONCLUSION

The product, sugarcane bagasse wax can serve as a substitute for the imported

viscosifiers, and ultimately lead to economic gains for the nation. This project revealed the feasibility of the development and characterization of an oil-soluble viscosifier for an oil-based drilling fluid preparation. The sugarcane bagasse wax produced has proven to meet and nearly exceed the requirements for a standard viscosifier. From the results of the various experiments carried out, in the development of the wax, solvent extraction (soxhlet extraction) has proven to be highly efficient for the production of this viscosifier, and the physical properties of the sugarcane wax, is also an indication of other possible benefits. Also, the investigated rheological properties of the OBM using sugarcane wax as a viscosifier, revealed an increasing trend for every parameter, such as the apparent viscosity, yield point, plastic viscosity, gel strength. Characterization by FT-IR analysis revealed the wax's compatibility with the prepared OBMs, as it readily dissolves in the oil-phase of the mud, thereby making it feasible to mix properly with the entire mud components. The sugarcane bagasse wax was found to have possessed various proportions of long chain alkenes, hydrocarbons, fatty acids, ketones, aldehydes, alcohols, esters and steroids. It is naturally indigestible and also harmless to health, with a melting point of 75-80^{0}C. Hence it meets the aim, objectives and requirements of the study of being eco-friendly, locally sourced, oil-soluble and rheological compatible with OBM and can therefore be considered and applied as a viscosifier in drilling mud formulations for drilling operations.

Conflicts of interest

The authors declared that there is no conflict of interest.

Acknowledgments

The author(s) acknowledge(s) the Editor in chief and the Reviewers for their selfless services to improve this research work.

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