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Keywords: graft copolymerization, cassava starch, acrylamide, Water based mud rheological properties.

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ABSTRACT

Modified starches have gained popularity in recent years due to the availability of starch. Graft polymerization is one of the ways to improve the properties of starch. Starch was obtained by process of wet milling from four genotypes Cassava tubers (TMS 96/1632, TMS 98/0581, TMS 07/ 0593 and TMS 01/1371), assigned as A, B, C, and D, respectively, were grafted with PAM and synthesized by polymerization method. Potassium Persulfate (PPS) was used as an initiator with ethanol-water solution used to remove the homo-polymer acrylamide (PAM). The starch-grafted copolymer was characterized by Fourier transform infrared spectroscopy. From the result, it was observed that the FTIR spectra for the four starches grafted onto Polyacrylamide Monomer showed the presence of PS-g copolymer with new absorption bands at the range of 1644cm^{-1} – 1654cm^{-1} this indicates a primary amide group in the polyacrylamides. The drilling fluids treated with the PS-g copolymer showed mud weights between 8.6 – 9.0 with genotype D S-g-PA copolymer presenting a better rheology properties and fluid loss control. All drilling fluids prepared with copolymerized starch exhibit shear thinning and pseudoplastic properties.

Keywords: graft copolymerization, cassava starch, acrylamide, Water based mud rheological properties.

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I. INTRODUCTION

Drilling fluids are an important part of drilling operations. Drilling fluids are complex mixtures of solids and liquids. During drilling operations, additives are added in order to enhance the properties of the drilling fluid. These additives perform different functions in drilling fluids such as increase mud weight, reduce viscosity, reduce fluid loss. However, most of these additives are currently not suitable for continuous use due to their adverse environmental effects resulting from high toxicity and poor biodegradability (S. Wysocki et al. 2005), Polymers have been used as additives and one of this is the use of polymeric additives and starch being the was the first and the most widely. Recent studies have used products obtained from biomaterials such as starch, xanthan gum, ginger and guar gum (Akinwumi et al. 2018) as suitable alternatives to the conventional additives to obtain desired rheological and filtration properties.

Starch is an effective colloid and it decreases the filtration of water by dispersing drilling fluids and increasing viscosity. Cassava (Manihot esculenta Crantz) , a polysaccharide polymer, is a root crop which has been investigated to exhibit different properties that have potential for a wide end use. In recent years, starch has been gaining significance in the oil industry as an additive in drilling fluid. Its use is widespread due to its availability and low cost (Peter Adewale et al. 2022). Native starch from corn, cassava, potato, and banana has been used as an additive in drilling fluids as a viscosifier and as an alternative to Carboxymethyl Cellulose and Polyanionic Cellulose.

Studies have shown that native starch degrades and destroys the integrity of the drilling fluid due to their poor temperature resistance (Shadfar et al. 2024). In response to the degradability and unstable nature of native starch, researchers have come up ways to improve the properties of starch via modification. Starch has been modified by different methods such as chemical modification (etherification, esterification, acid hydrolysis, oxidation), physical modification (Pregelatinization) and enzymatic modification (degradation) (Haq Hawaz et al. 2020).

Modified starch in drilling increases its resistance to bacterial attack and also improves the rheological properties of the drilling mud this starch is prepared with (Diana Soto et al. 2020). This study describes the development and evaluation of modified starch using graft polymerization method. The graft copolymerization of starch is highly necessary in order to reduce its limitations and applications and is employed to enhance its properties without altering its intrinsic properties (Haq Hawaz et al. 2020, Vidyagauri V. L et al. 2018) and it is one of the effective ways to improve the properties of natural polymers.

Graft copolymerization combines the properties of the starch and the homopolymer which then produces unique and advanced properties as opposed to the original materials (Monica R.N et al. 2022).

II. MATERIALS AND METHODS

Four (4) genotypes of native cassava, TMS 96/1632, TMS 98/0581, TMS 07/ 0593 and TMS 01/1371 were obtained from IITA (International Institute of Tropical Agriculture), Ibadan, Nigeria. Acrylic acid, Potassium Persulfate and Ethanol were all analytical grade and obtained from Oarsman chemicals in Ibadan, Nigeria.

2.1 Cassava starch

The cassava tubers were processed using the wet milling method

2.2 Copolymerization of Cassava Starch

Preparation of S-gPAM copolymer was performed using the following 20g of starch obtained from cassava stems was dissolved in 100ml distilled water and heated in a water bath at 50 °C for 15 min. 10 g of acrylic acid was added and 2% of initiator (Potassium Per sulfate) was added. Temperature was kept at 50 °C for 120 min under reflux with vigorous stirring on magnetic stirrer. Reaction product was precipitated in 200 mL of ethanol. After filtration, precipitate was washed with 20 mL of ethanol at ambient temperature and then 4 times with ethanol-water solution (80:20) to ensure complete removal of homo-polymer acrylamide (PAM). The resulting starch grafted polyacrylamide (S-gPAM) was oven-dried for 24hours to remove water. This procedure was carried out for the 4 genotypes of cassava starch (Vidyagauri & Savita, 2021)

2.3 Characterization of the Grafted Starches

The determination of carbonyl, carboxyl and other group contents present in the copolymerized starch was carried out using the Fourier Transform Infrared Spectroscopy. The analysis was carried on both the native starch and the copolymerized starch for evidence of grafting. The spectra were obtained using a FTIR spectrophometer (spectrum one, Perkin Elmer/PIKE MIRacle^{TM} Technologies, Wellesley, MA, USA) using KBr pellets and measured in ATR mode with a special resolution of 0.5cm^{-1} used.

2.4 Mud Preparation and Tests

The drilling mud was prepared in accordance with the API 13A recommended practice. 15 mud samples were prepared using 21.5g of bentonite gradually added to 350 ml of distilled water and mixed to obtain a homogenous mixture using a Hamilton Beach mixer. The samples were labelled A, B, C, D and CMC was allowed to stay from 16hours allowing for proper hydration before an electronic weighing balance was used to measure 10g of barite into each mud suspension before varying concentration of the modified starches (2g. 4g and 6g) into the labelled mud samples the suspension was thoroughly mixed for about 20minutes, with a spatula used to scrap the polymer clinging to the beaker walls The suspension was poured into the Fann35A viscometer cup and sheared, and dial readings at 600, 300, 200, 100, 60, 30, 6, and 3 rpm were taken

2.5 Physico-Chemical, Rheological and Filtering Properties

The Physico-chemical, rheological and filtering properties of the different Water -Based Muds were determined. The density of the drilling fluids was determined using a mud balance. The pH was determined using a pH meter.

2.5.1 Determination of Mud Sample Density and pH

The density of the drilling fluids was determined using a Bariod Mud Balance while the pH was determined using the pH meter.

2.5.2 Rheological Properties

The readings obtained from the Fann 35A viscometer were used to determine the plastic viscosity, apparent viscosity and yield point. at 20 °C, 40°C and 70°C. using the equations 1.0,2.0 and 3.0, respectively.

$$PV(cp) = \theta_{600} - \theta_{300} \qquad 1.0$$

$$AV = \frac{\theta_{600}}{2} \qquad 2.0$$

$$YP(lbs/100ft) = \theta_{300} - PV \qquad 3.0$$

Power law model

This is a two parameter model that relates shear stress to shear rate in a non-linear manner (Alderman et al. 1988; Okafor & Evers, 1992). The model does not consider an excess yield stress and states the relation in equation 4 as;

$$\tau = K\gamma^{n} \qquad 4.0$$

Where K and n are consistency index and flow index respectively, τ is the shear stress and γ is the shear rate.

2.5.3 Filtration Properties

The American Petroleum Institute (API) filter press was used to determine the filtrate loss of the drilling fluids. The filtrate loss test is used to determine the quantity of fluid lost during drilling and the type of mud cake formed. Drilling fluids with poor filtrate loss control produce thick filter cakes that can pose issues in the wellbore. Such issues include but are not limited to; reduced rate of penetration, stuck pipe, excessive torque. Minimizing fluid loss and forming a thin permeable mud cake is crucial in drilling activities (Hanyi Zhong et al., 2022)

III. RESULTS AND DISCUSSION

3.1 Starch Characterization: Fourier Transform Infrared Spectroscopy

The Fourier Transform Infrared (FTIR) spectra in Figures 1.0 to 5.0 were obtained for a native starch (TMS 95/0289) and the copolymerized cassava starches from TMS 96/1632(A), TMS 98/0581 (B), TMS 07/ 0593 (C) and TMS 01/1371(D). The Figure 1.0 presents the spectrum of native cassava starch showing peaks at 3200cm^{-1} to 3500cm^{-1} and 2923cm^{-1} corresponding to OH and CH stretching while peaks at 1639cm^{-1} and 1416cm^{-1} correspond to OH and CH bonding. The native cassava starch also showed the short range order structures of the starch double helix at 1020 cm^{-1} and 930cm^{-1}. while those of the others S-gPAM showed a strong absorption band at 3300cm^{-1} - 3500cm^{-1} which is attributed to NH stretching vibration of the NH_2 group.

The band for OH stretching vibration was seen around 2908cm^{-1} – 2938cm^{-1}. New absorption bands at 1644cm^{-1} – 1654cm^{-1} indicates a primary amide group in polyacrylamides. NO asymmetric stretch bands were also observed around 1530cm^{-1}. Absorption bands at 1411cm^{-1} to 1416cm^{-1} shows scissoring vibrations of $-CH_2$ group. Absorption bands around 1080cm^{-1} corresponds to valence vibrations $-OCH-O-CH_2$ groups. The absence of the strong 1639cm^{-1} (C=N) absorption band in the copolymers indicate that copolymerization has taken place (Vidyagauri and Savita, 2021).

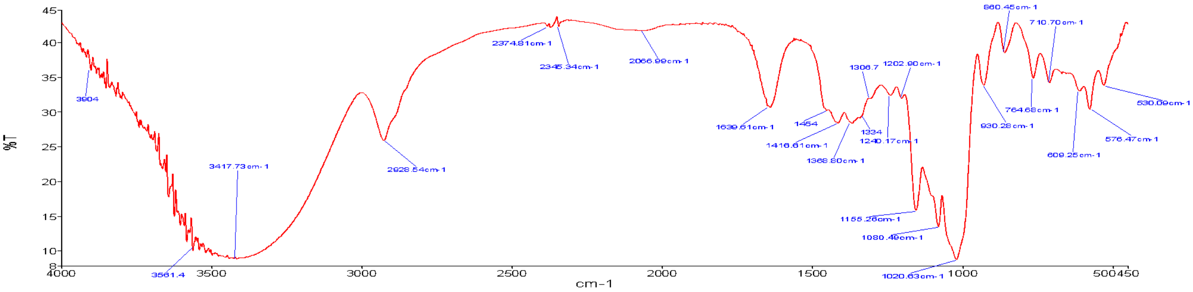

Figure 1.0: FTIR spectra of native starch

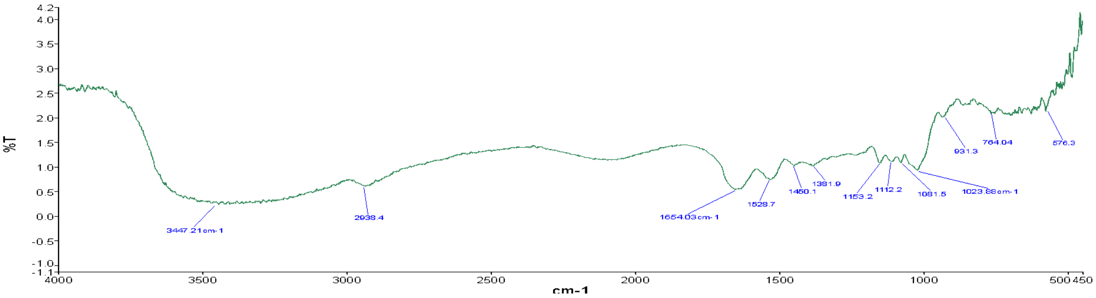

Figure 2.0: FTIR spectra of sample A

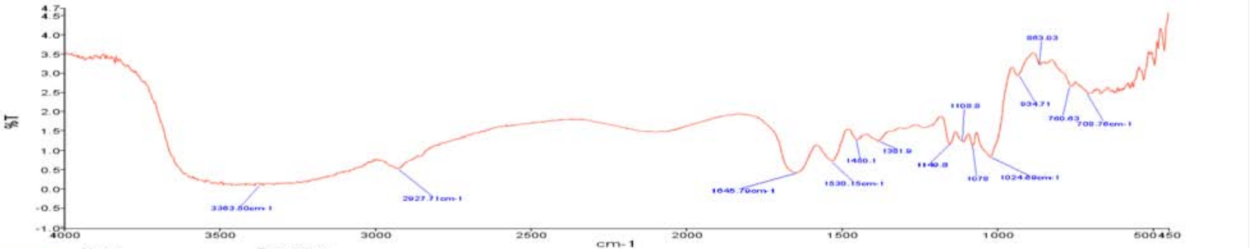

Figure 3.0: FTIR spectra of B

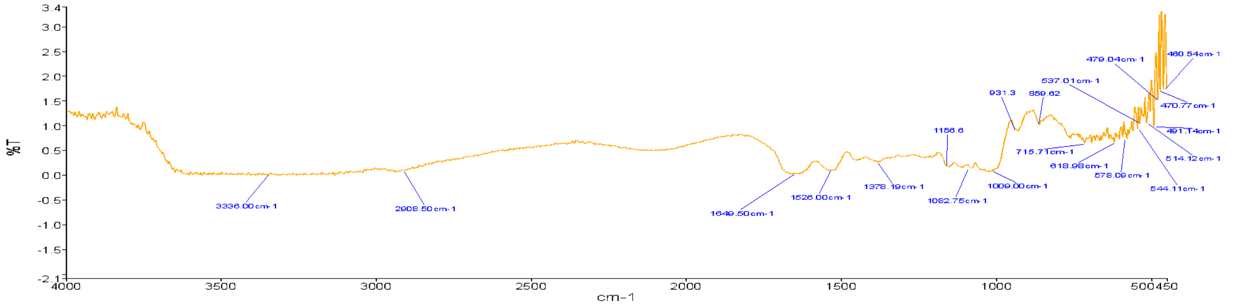

Figure 4.0: FTIR spectra of C

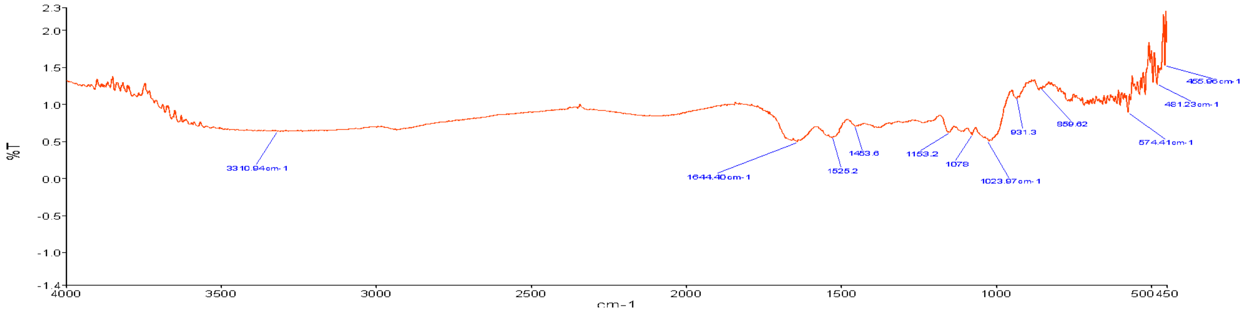

Figure 5.0: FTIR spectra of D

3.2 Mud weight

The Figure 6.0 presents the result of the mud weight of all the mud samples. from the result it can be observed that mud samples treated with polymerized starch had weights higher than the control mud. Also there is a minimal increase in the mud weight as the weight of the additive, copolymerized starch, increases.

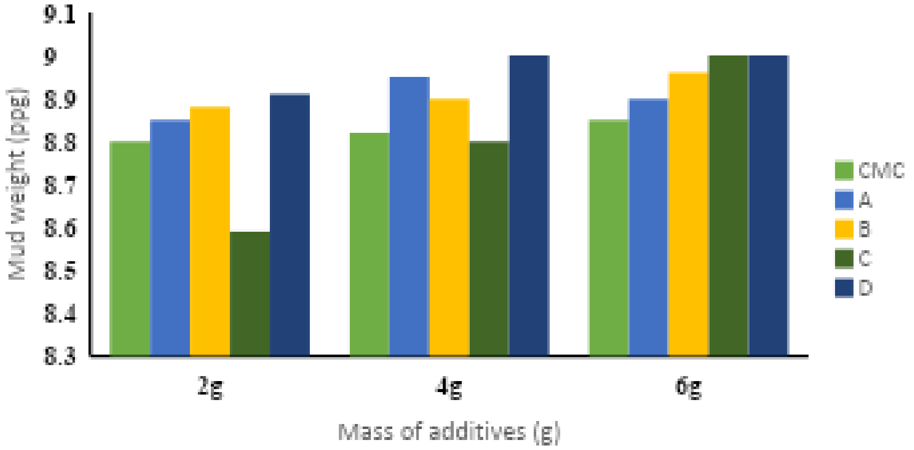

Figure 6.0: Mud weight of mud samples

3.3 pH of Drilling Fluids

The pH of the drilling fluids increased with increasing weight of the copolymerized starch as presented in the Figure 7.0. The mud samples pH is within the range of 8.6 to 8.9. These mean that the modified starch reduced the hardness of water by precipitating the calcium ions in the water thereby increasing the pH of water. Therefore, modified starch can be used to reduce the mud acidity which could lead to corrosion of bottom equipment during drilling (Aliyu, et al. 2020).

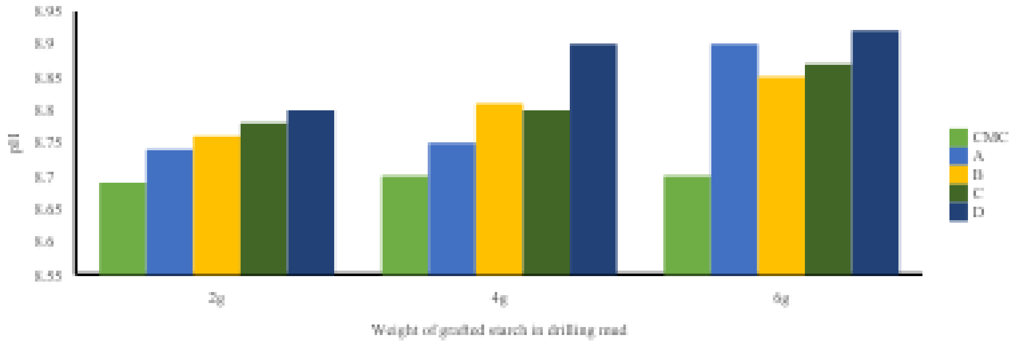

Figure 7.0: pH of mud samples

3.4 The Mud Samples Rheological Properties

The dial readings of the drilling fluid samples after the addition of copolymerized starch were taken.

3.4.1 Plastic Viscosity

The results for the plastic viscosities of the mud samples are shown in Figures 8.0 to 10.0.. The API specification for high performance is below 35cp which the mud samples exhibited. . Mud sample prepared with 6g of CMC at 40℃ had the highest PV at 18cp followed by 2.0g of D at 70℃ at 15cp.

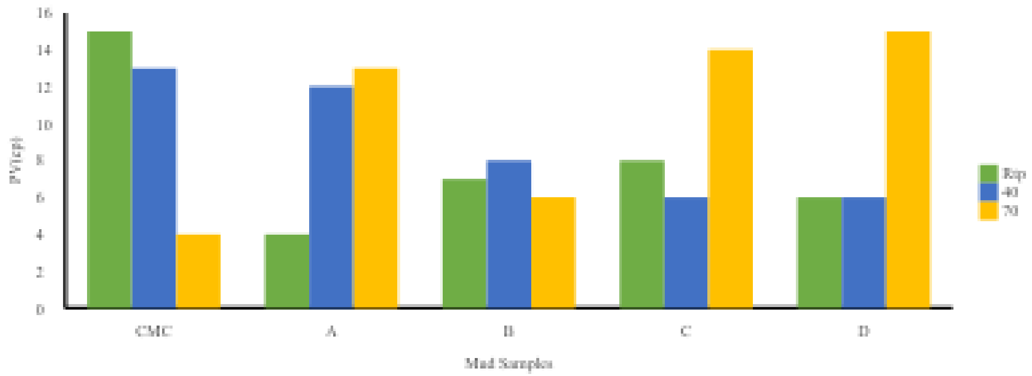

Figure 8: Plastic Viscosity of 2g of mud samples at Rtp, 40℃ and 70℃

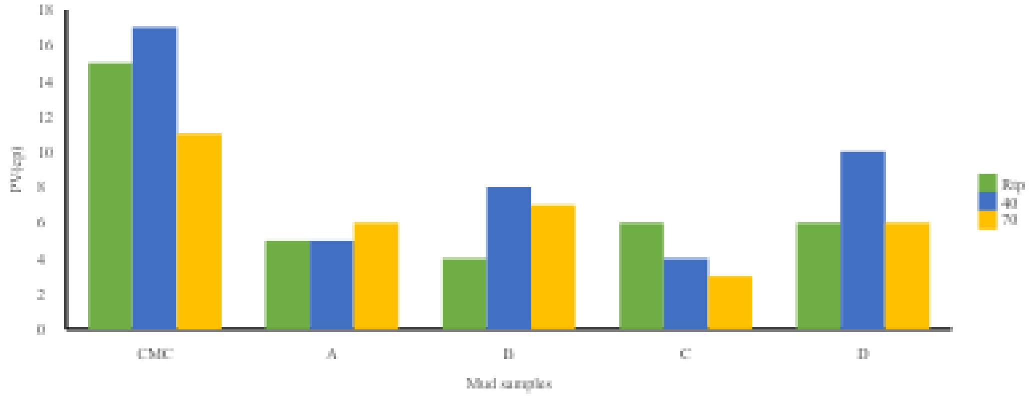

Figure 9: Plastic Viscosity of 4g of mud samples at Rtp, 40℃ and 70℃

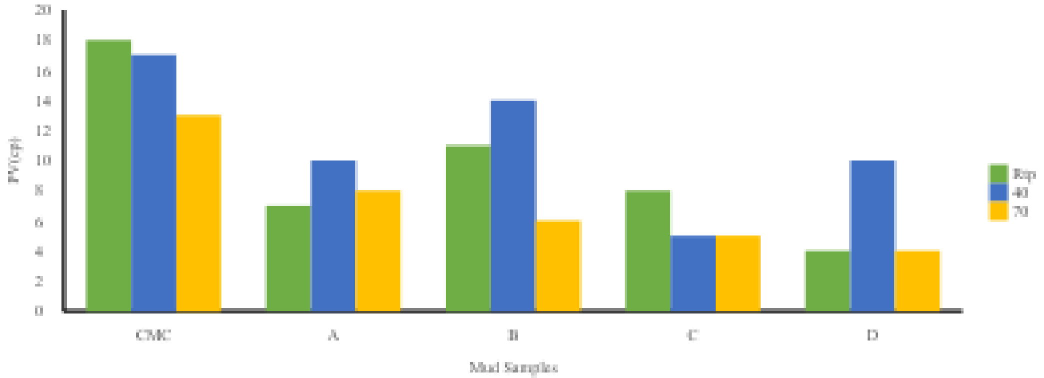

Figure 10: Plastic Viscosity of 6g of mud samples at Rtp, 40℃ and 70℃

3.4.2 Apparent Viscosity

The apparent viscosity of mud samples treated with copolymerized starch are presented in figures 11.0 to 13.0. The apparent viscosity for all mud samples prepared with CMC had the highest values going as high as 47cp. At 70℃, C and D had a value of 25cp while 6g of B at 40℃ had a value of 30cp.

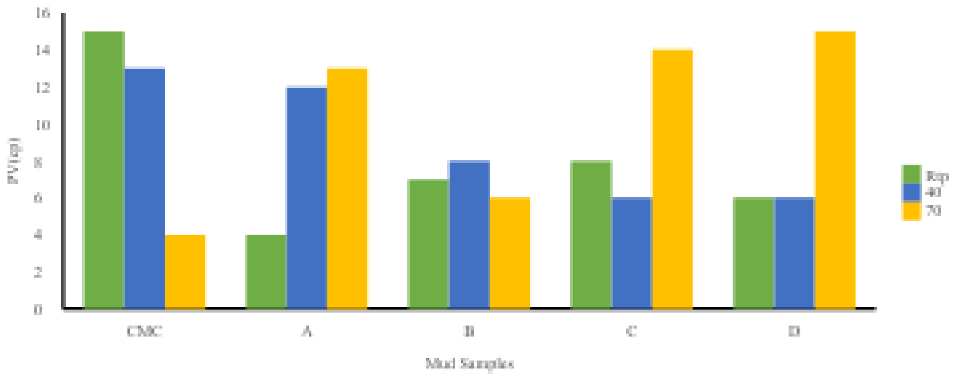

Figure 11.0: Apparent Viscosity of 2g of mud samples at Rtp, 40℃ and 70℃

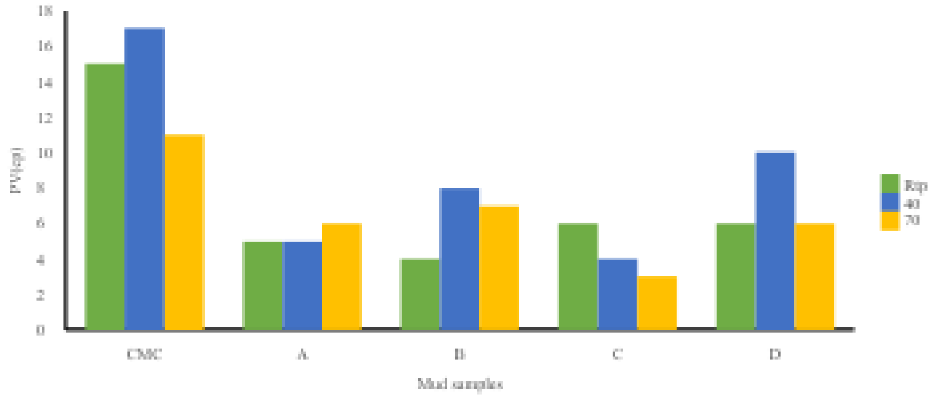

Figure 12: Apparent Viscosity of 4g of mud samples at Rtp, 40℃ and 70℃

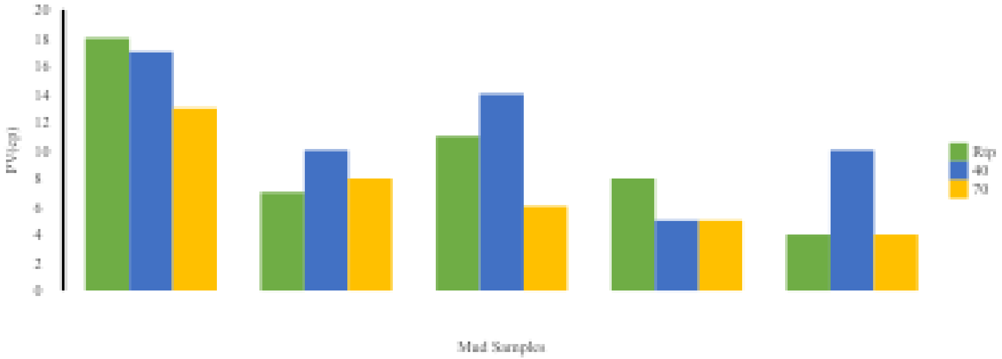

Figure 13: Apparent Viscosity of 6g of mud samples at Rtp, 40℃ and 70℃

3.4.3 Yield Point

The yield point of the mud samples in varying concentrations are shown in figures 14 to 16. High YP means an increase in mud rate. Yield point indicates the ability of drilling mud to lift drill cuttings from the wellbore. The API specification for high performance is between 15-25cp. The higher the YP, the better the mud lifts drill cuttings but excessive YP could lead to pressure loss during drilling mud circulation which could damage the formation. Mud samples prepared with 6g of CMC at all temperatures had the highest yield point with the highest at 58cp. 2g of B at r.tp had the lowest YP at 11cp. All other mud samples had YP within the API specification except 2g of D at 40℃ with 32cp.

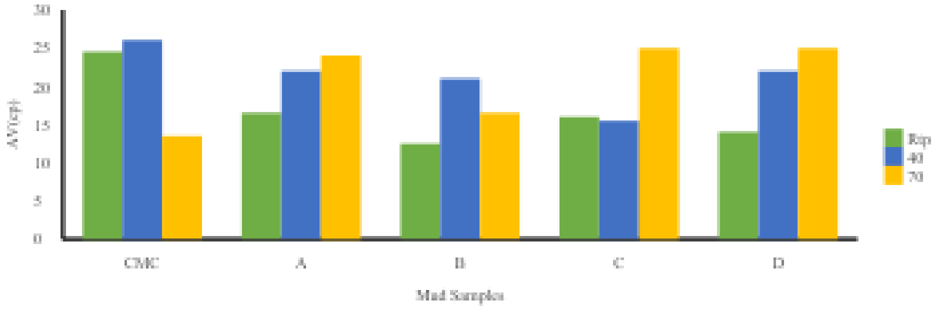

Figure 14: Yield Point of 2g of mud samples at Rtp, 40℃ and 70℃

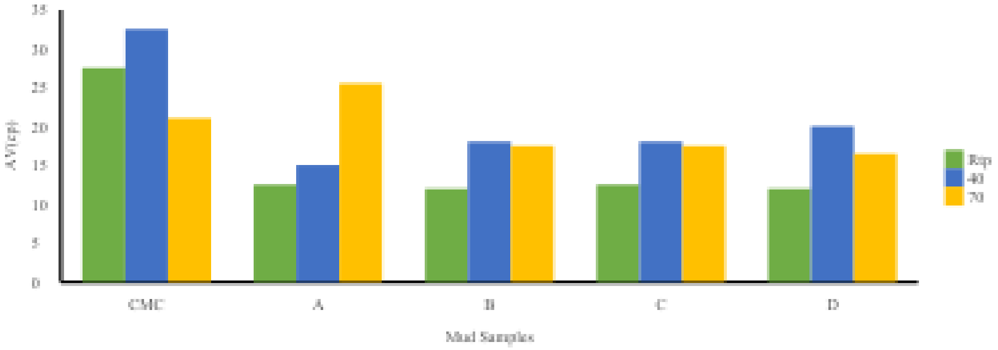

Figure 15: Yield Point of 4g of mud samples at Rtp, 40℃ and 70℃

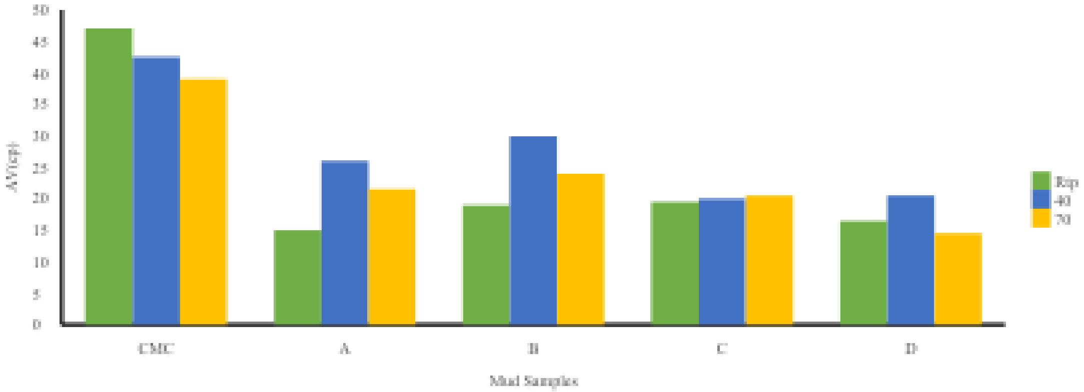

Figure 16: Yield Point of 6g of mud samples at Rtp, 40℃ and 70℃

2.4.4 Gel Strength

The API specification for the difference between the 10secs and 10mins gel strength is 20, if it exceeds 20, more pump power is needed to start the circulation after the static condition. Figures 17 to 19 show the gel strength values of mud samples taken at 10seconds and 10minutes at r.tp, 40℃ and 70℃. All copolymerized mud samples showed difference between 10secs and 10mins gel strength to be less than 20, therefore less pump power is needed to start the circulation after the static condition (Aliyu et al.

2020). The drilling mud sample prepared with 2g of copolymerized starch of genotype B at 40℃ had the same value at 10secs and 10min gel strength. The same was also observed for drilling mud sample prepared with 6g of copolymerized starch of genotype B at 70℃.

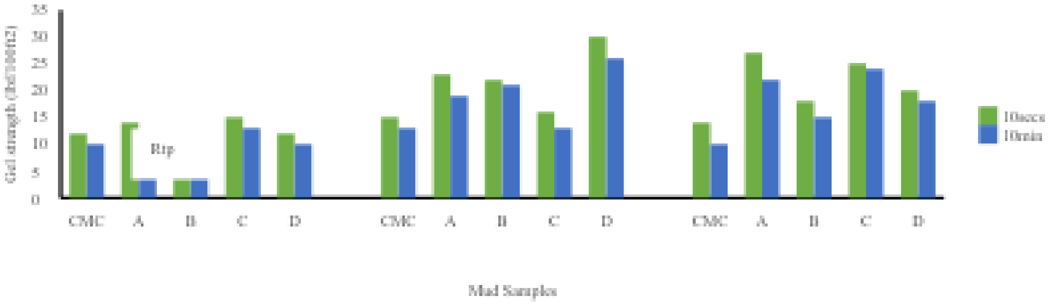

Figure 17: Gel strength of mud samples containing 2g of copolymerized starch at 10secs and 10mins

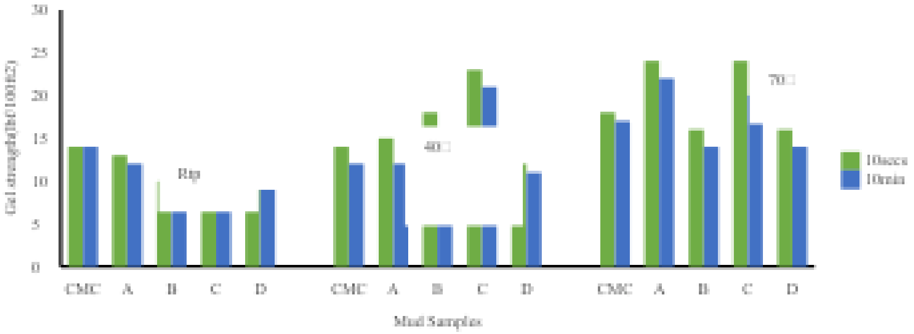

Figure 18: Gel strength of mud samples containing 4g of copolymerized starch at 10secs and 10mins

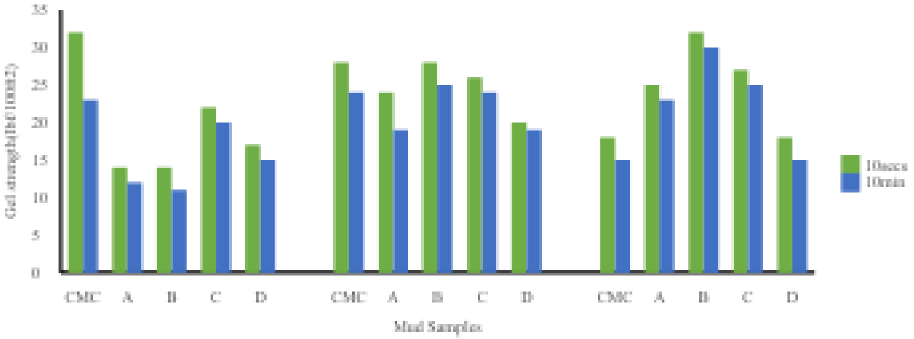

Figure 19: Gel strength of mud samples containing 6g of copolymerized starch at 10secs and 10mins

3.5 Filtration Properties and Mud Cake Thickness

The filtrate volume and cake thickness of all mud samples are shown in figures 20 to 22. The filtrate loss and mud cake properties of the samples were evaluated using the API filtration tests for 30mins. The test measured the quantity of water lost from the drilling mud at 30mins. All drilling mud samples produced thin permeable filter cake which indicates that formation damage will be minimized because of reduced fluid invasion (Imtiaz Ali et al. 2022). Drilling mud samples prepared with 2g of A, B and D all had filtrate volume ranging from 18.3 to 18.5ml while C sample had a filtrate loss value of 13.7ml.

For the 4g mud samples, D had the least filtrate loss volume at 15.5ml and the thinnest filter cake at 0.15ml.

Drilling mud samples prepared with 6g of the copolymerized starches all showed reduced filtrate loss volume with D showing the highest filtrate loss at 17.5ml while B showed the thickest filter cake at 0.5mm. Mud samples prepared with CMC at different weights all outperformed the drilling muds prepared with the copolymerized starches.

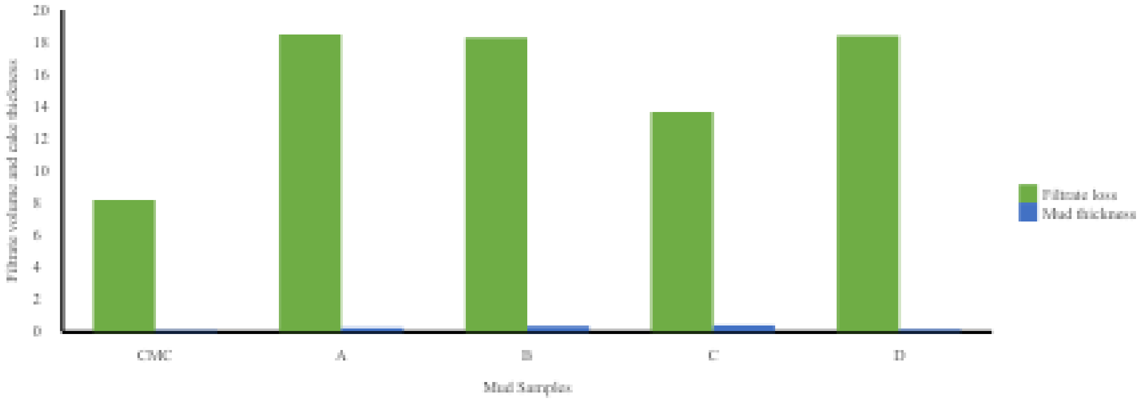

Figure 20: Filtrate volume and mud cake thickness for mud samples containing 2g of copolymerized starch

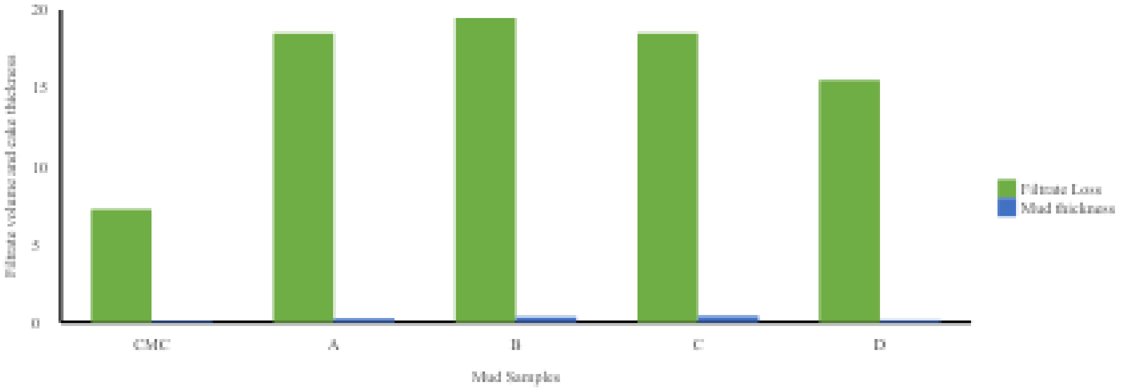

Figure 21: Filtrate volume and mud cake thickness for mud samples containing 4g of copolymerized starch

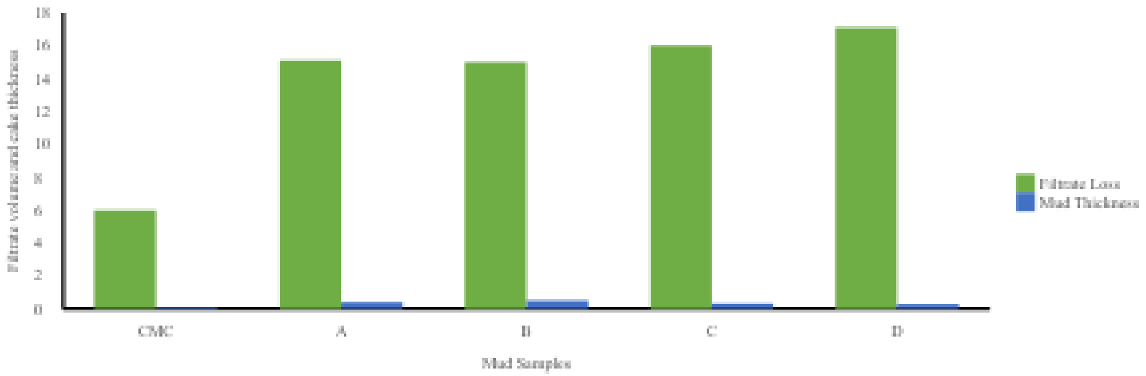

Figure 22: Filtrate volume and mud cake thickness for mud samples containing 6g of copolymerized starch

IV. CONCLUSION

Native starch was modified by graft copolymerization and the effect of these on the properties of water based mud was compared to mud formulated with CMC. All mud samples were alkaline in nature, the mud samples prepared with 2g of copolymerized starch had the lowest pH. The mud sample containing 2g of D at 70℃ had PV of 15cp and is capable of lifting drill cuttings.

Mud samples prepared with copolymerized D had the least filtrate loss volume at 15.5ml and the thinnest filter cake at 0.15ml, hence a good filtrate loss reducer compared to the other starch samples. Therefore, mud sample treated with D had better rheological properties and filtration loss control. From the results, increasing concentrations of the modified starch does not have a significant effect on the mud properties; however, there are differences in the mud properties based on genotype of the cassava. It was also observed that mud samples containing modified starch are shear thinning, pseudoplastic and therefore have acceptable flow characteristics.

Declaration of conflict of Interest

On behalf of all the co-authors, the corresponding author states that there is no conflict of interest

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