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**Applied Journal of Physical Science**
Volume 6(3), pages 50-61, June 2025
Article Number: 4B86DE3F1
ISSN: 2756-6684
https://doi.org/10.31248/AJPS2025.113
https://integrityresjournals.org/journal/AJPS

*Full Length Research*

# Effect of acrylamide-grafted cassava starch on the properties of a water-based mud

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Received 21st March 2025 Accepted 8th May 2025

**ABSTRACT:** Modified starch has 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 the process of wet milling from four genotypes of 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 homopolymer ACRYLAMIDE (PAM). The starch-grafted copolymer was characterised 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 in the range of 1644 – 1654 $cm^{-1,}$ which 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 better RHEOLOGY PROPERTIES and fluid loss control. All DRILLING FLUIDS prepared with copolymerized starch exhibit SHEAR-THINNING and pseudoplastic properties.

**Keywords:** Graft polymerisation, cassava starch, acrylamide, Rheological properties, drilling fluids, shear-thinning.

## 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 to enhance the properties of the drilling fluid. These additives perform different functions in drilling fluids, such as increasing mud weight, reducing viscosity, and reducing 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 (Wysocki *et al*., 2005), Polymers have been used as additives and one of this is the use of polymeric additives and starch being the first and the most widely. Recent studies have used products obtained from biomaterials such as starch, xanthan gum, ginger and guar gum (Akinade *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 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 (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 its poor temperature resistance (Davoodi *et al.,* 2024). In response to the degradability and unstable nature of native starch, researchers have come up with 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 (pre-gelatinisation) and enzymatic modification (degradation) (Nawaz *et al.*, 2020).

Modified starch in drilling increases its resistance to bacterial attack and improves the rheological properties of the drilling mud. This starch is prepared with (Soto *et al.,* 2020). This study describes the development and evaluation of modified starch using the graft polymerisation method. The graft copolymerization of starch is highly necessary to reduce its limitations and applications and is employed to enhance its properties without altering its intrinsic properties (Nawaz *et al.,* 2020, Lele *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 (Nemțanu *et al.,* 2022).

The use of acrylamide-grafted starch in water-based muds is a promising area of research, particularly for its potential to enhance the properties of drilling fluids. Acrylamide-grafted starch is a modified form of starch that can improve the rheological and filtration properties of drilling muds, which are crucial for efficient drilling operations. Modified starches, such as dually modified starch, have been shown to significantly reduce filtrate volume and cake thickness, achieving up to an 81% decrease in filtrate volume at certain concentrations. This demonstrates the potential of modified starches as cost-effective alternatives to traditional chemical additives in drilling muds, contributing to improved borehole stability and reduced operational costs (Ali *et al.*, 2024). Similarly, the use of unmodified and modified starches, such as those modified with ethylenediaminetetraacetic acid (EDTA-DSD), has been evaluated for their effectiveness as fluid-loss control additives. These starches have shown favourable results in terms of plastic viscosity and yield point, making them suitable for high-pressure, high-temperature conditions (Dike *et al.,* 2024).

Corn starch, when modified, has also been identified as an environmentally friendly option for enhancing the rheological properties and fluid loss control of water-based muds. Modified corn starch has been found to outperform traditional additives like polyanionic cellulose, particularly at high temperatures, due to its improved entanglement in the mud system (Ricky *et al.,* 2022). Cationic starches, which contain fixed nitrogen content, have been used in various industrial applications, including as additives in water treatment processes. These starches offer unique properties that can be leveraged in drilling mud formulations to improve viscosity and fluid loss control (Merle *et al.,* 2000).

Cassava starch is another locally sourced starch that has been extensively studied for its ability to improve the rheological properties of water-based muds. Research has shown that cassava starch can enhance the viscosity and suspension ability of drilling fluids, making it a viable alternative to more expensive imported additives (Omotioma *et al.*, 2015). Micronised starch, with its reduced particle size, has been shown to significantly improve the rheological and filtration properties of drilling fluids, offering a new formulation that can help avoid common drilling issues such as formation damage (Elkatatny, 2019).

This research analyses how this grafted starch influences drilling performance, aiming for better drilling and greener solutions.

## 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.

### Cassava starch

The cassava tubers were processed using the wet milling method. The wet milling method for processing cassava tubers involves several key steps that transform raw cassava roots into a usable product, typically cassava flour. This method is essential for removing undesired components and ensuring the final product is suitable for consumption or further processing. The process is characterised by its ability to efficiently separate components and produce high-quality product mass. Below, the method is broken down into its main components and considerations.

#### *Grating and slurry formation*

The initial step in the wet milling process involves grating the cassava roots. This is crucial for breaking down the tubers into smaller particles, which facilitates the subsequent processing steps (Sanders *et al.,* 2005). Water is then added to the grated cassava to form a slurry. This slurry acts as a medium for separating the desired starch from other components, such as fibres and proteins (Sanders *et al.,* 2005).

#### *Removal of undesired components*

The slurry undergoes a separation process to remove undesired components, including proteins and fibres. This step is vital for ensuring the purity and quality of the cassava flour (Sanders *et al*., 2005).

The separation process can involve various techniques, such as sieving or centrifugation, to achieve a high-quality product mass (Sanders *et al*., 2005).

***Drying and final product formation***

After the undesired components are removed, the product mass is dried. This step is essential for reducing moisture content and ensuring the stability and shelf-life of the cassava flour (Sanders *et al.,* 2005). The drying process can be conducted using different methods, such as sun drying or mechanical drying, depending on the available resources and desired product characteristics (Sanders *et al.,* 2005). The wet milling method is effective in producing high-quality cassava flour, but it requires careful management of water usage and waste disposal to minimise environmental impact (Sanders *et al.,* 2005). Additionally, the process must be optimised to ensure the removal of cyanogenic compounds, which are naturally present in cassava and can be toxic if not adequately reduced.

**Copolymerization of cassava starch**

Preparation of S-g-PAM copolymer was performed using the following: 20 g of starch obtained from cassava stems was dissolved in 100 ml of 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 Persulfate) was added. The temperature was kept at 50°C for 120 min under reflux with vigorous stirring on the magnetic stirrer. The reaction product was precipitated in 200 mL of ethanol. After filtration, the 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-g-PAM) was oven-dried for 24 hours to remove water. This procedure was carried out for the 4 genotypes of cassava starch (Lele and Kumari, 2021).

**Characterisation 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 out on both the native starch and the copolymerized starch for evidence of grafting. The spectra were obtained using an FTIR spectrophometer (Spectrum One, Perkin Elmer/PIKE MIRacle™ Technologies, Wellesley, MA, USA) using KBr pellets and measured in ATR mode with a resolution of 0.5 $cm^{-1}$.

**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 for 16 hours allowing for proper hydration before an electronic weighing balance was used to measure 10g of barite into each mud suspension, then varying concentration of the modified starches (2, 4 and 6 g) was added into the labelled mud samples the suspension was thoroughly mixed for about 20 minutes, with a spatula used to scrape 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.

**Physicochemical, rheological and filtering properties**

The Physicochemical, 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.

**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 a pH meter. The Bariod mud balance measures the density of a fluid by balancing a sample of the fluid against a known weight. The balance consists of a graduated arm with a cup at one end to hold the fluid and a counterweight at the other end. The arm is balanced on a fulcrum, and the density is read directly from a scale on the arm. This method is widely used in drilling operations to ensure the mud has the correct density to maintain wellbore stability and control formation pressures (Winckler *et al*., 2021; Weber *et al.*, 2021; McKay *et al.*, 2019).
Procedure:

1. Fill the cup with the mud sample, ensuring no air bubbles are trapped.
2. Place the arm on the fulcrum and adjust until it is level.
3. Read the density directly from the scale, which is typically calibrated in pounds per gallon (lb/gal) or grams per cubic centimetre ($g/cm^3$).

A pH meter measures the hydrogen ion activity in a solution, providing a pH value that indicates the acidity or basicity of the sample. The device consists of a probe and a meter that displays the pH value. The procedure are as follows:

1. Calibrate the pH meter using standard buffer solutions before measurement.
2. Rinse the probe with distilled water and immerse it in the mud sample.
3. Allow the reading to stabilise before recording the pH value.

### Rheological properties

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

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

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

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

### 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 and Evers, 1992). The model does not consider excess yield stress and states the relation in equation 4 as;

$$\tau = K\gamma^n \quad 4.0$$

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

### *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, and excessive torque. Minimising fluid loss and forming a thin permeable mud cake is crucial in drilling activities (Zhong *et al*., 2022).

## RESULTS AND DISCUSSION

### Starch characterisation: Fourier Transform Infrared Spectroscopy

The Fourier Transform Infrared (FTIR) spectra in Figures 1 to 5 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). Figure 1 presents the spectrum of native cassava starch showing peaks at 3200 $cm^{-1}$ to 3500 $cm^{-1}$ and 2923 $cm^{-1}$ corresponding to OH and CH stretching, while peaks at 1639 $cm^{-1}$ and 1416 $cm^{-1}$ correspond to OH and CH bonding. The native cassava starch also showed the short-range order structures of the starch double helix at 1020 and 930 $cm^{-1}$. While those of the others S-gPAM showed a strong absorption band at 3300 – 3500 $cm^{-1}$, which is attributed to the NH stretching vibration of the $NH_2$ group.

The band for OH stretching vibration was seen around 2908 – 2938 $cm^{-1}$. New absorption bands at 1644 – 1654 $cm^{-1}$ indicate a primary amide group in polyacrylamides. NO asymmetric stretch bands were also observed around 1530 $cm^{-1}$. Absorption bands at 1411 to 1416 $cm^{-1}$ shows scissoring vibrations of $–CH_2$ group. Absorption bands around 1080 $cm^{-1}$ correspond to valence vibrations –OCH-O-$CH_2$ groups. The absence of the strong 1639 $cm^{-1}$ (C=N) absorption band in the copolymers indicates that copolymerization has taken place (Lele and Kumari, 2021). The reduction of peaks in the 1000 to 1500 $cm^{-1}$ range can be attributed to several factors, including changes in chemical bonding, sample composition, and structural modifications. This range often corresponds to the vibrational modes of functional groups such as Si-O, C-O, and C-C, which are sensitive to environmental and processing conditions.

### Mud Weight

Figure 6 presents the result of the mud weight of all the mud samples. From the result, it can be observed that mud samples treated with polymerised 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.

### pH of drilling fluids

The pH of the drilling fluids increased with increasing weight of the copolymerized starch as presented in Figure 7. The mud samples' pH is within the range of 8.6 to 8.9. This means that the modified starch reduced the hardness of water by precipitating the calcium ions in the water, thereby increasing the pH of the water. Therefore, modified starch can be used to reduce the mud acidity, which could lead to corrosion of bottom equipment during drilling (Sulaimon *et al*. 2020).

### The mud samples' rheological properties

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

### *Plastic viscosity*

The results for the plastic viscosities of the mud samples are shown in Figures 8 to 10. The API specification for high performance is below 35 cp, which the mud samples exhibited. Mud sample prepared with 6g of CMC at 40℃ had the highest PV at 18cp, followed by 2.0 g of D at 70℃ at 15cp.

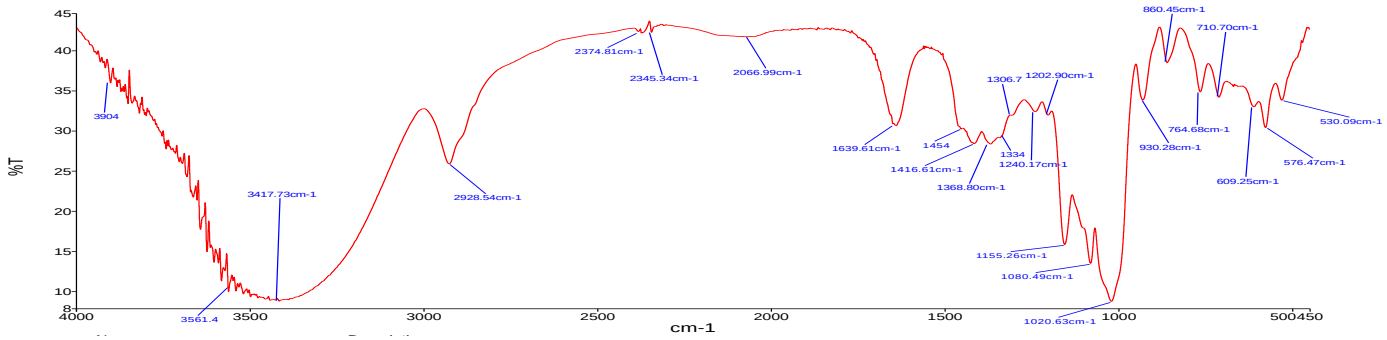


**Figure 1.** FTIR spectra of native starch.

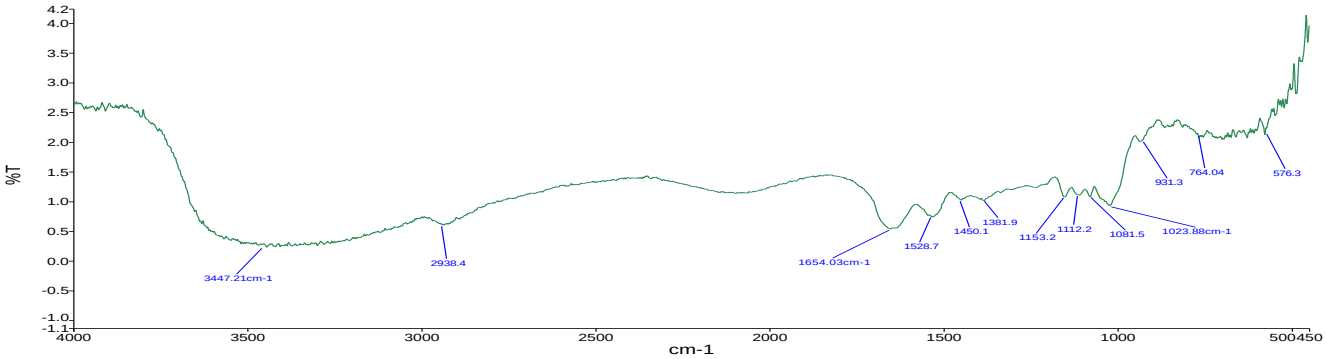


**Figure 2.** FTIR spectra of sample A.

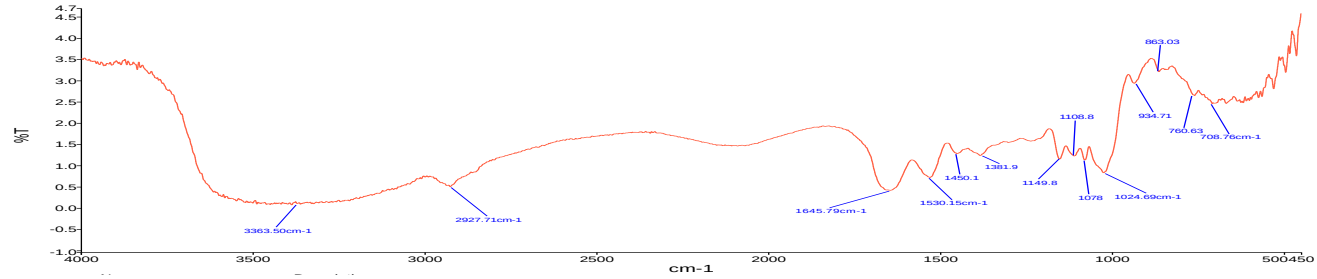


**Figure 3.** FTIR spectra of B.

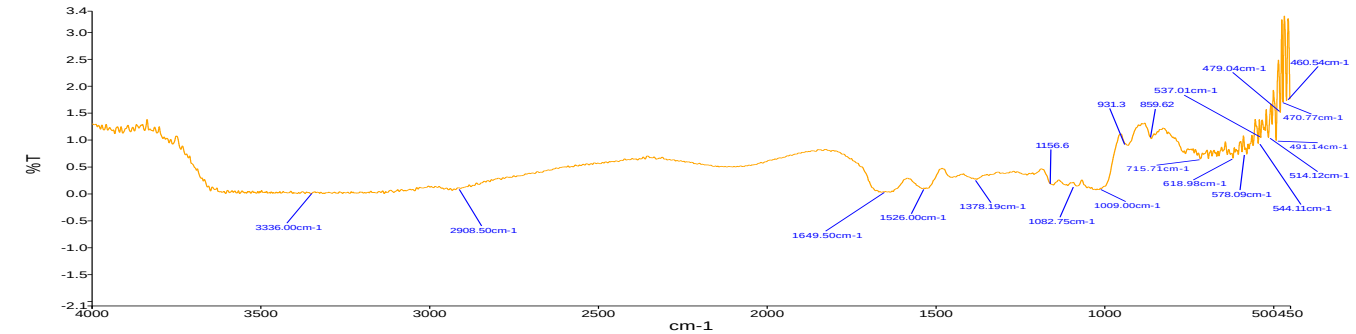


**Figure 4.** FTIR spectra of C.

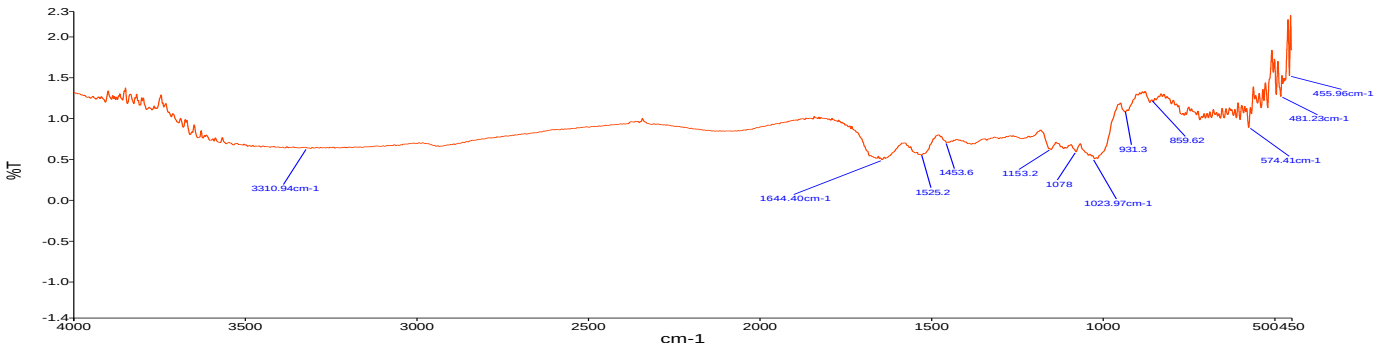


**Figure 5.** FTIR spectra of D.

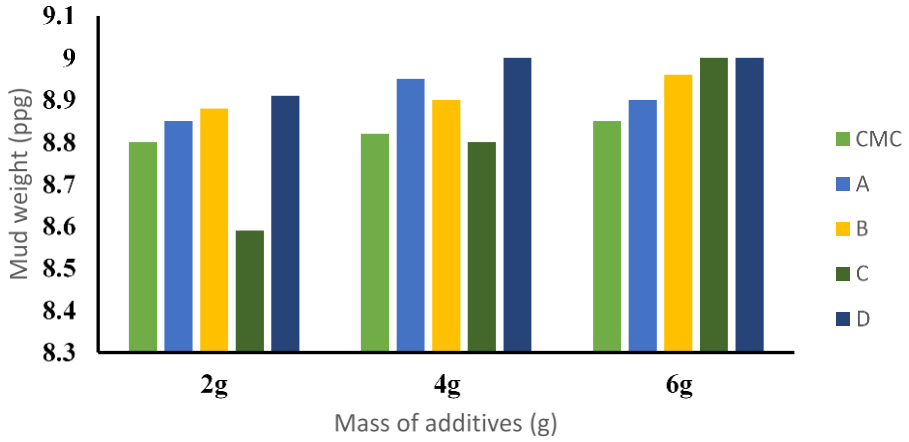


**Figure 6.** Mud weight of mud samples.

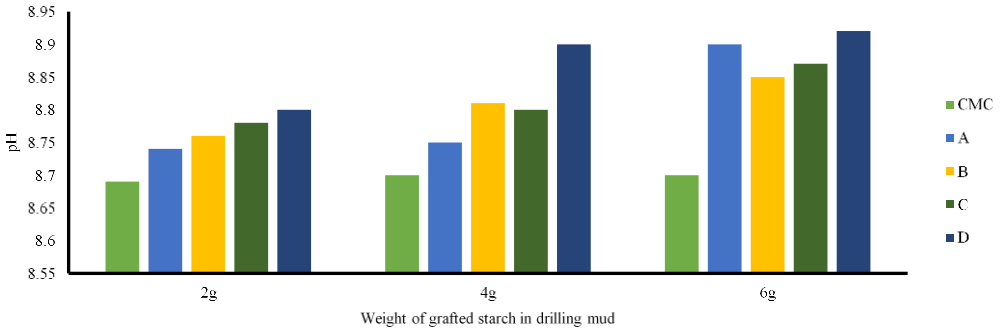


**Figure 7.** pH of mud samples.

### *Apparent viscosity*

The apparent viscosity of mud samples treated with copolymerized starch are presented in Figures 11 to 13. The apparent viscosity for all mud samples prepared with CMC had the highest values going as high as 47 cp. At 70℃, C and D had a value of 25cp while 6g of B at 40℃ had a value of 30cp.

### *Yield point*

The yield points 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 15-25 cp. The higher the YP, the better the mud lifts drill

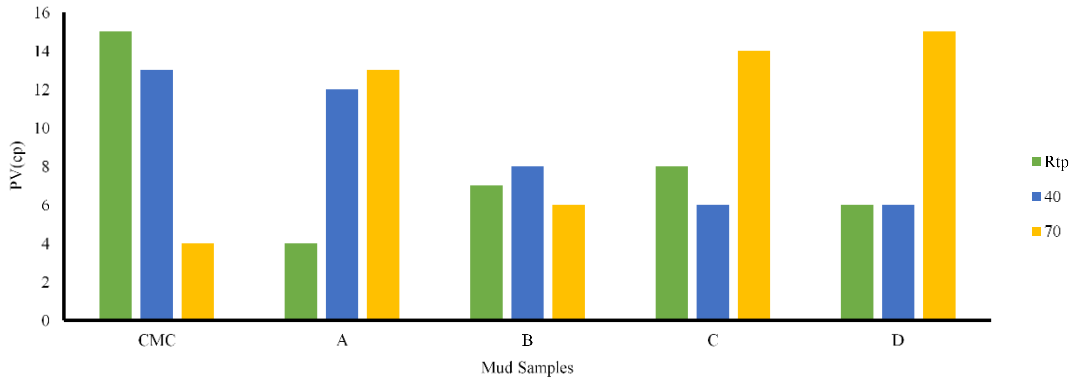


**Figure 8.** Plastic Viscosity of 2g of mud samples at Rtp, 40℃ and 70℃.

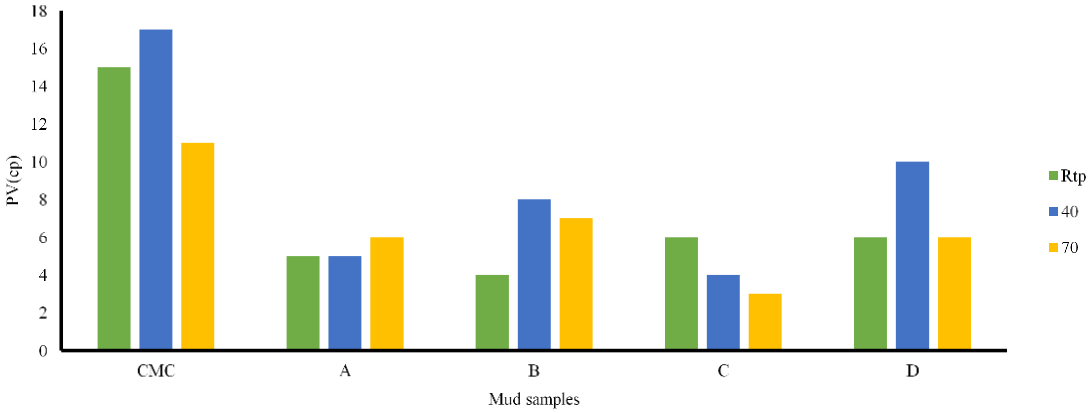


**Figure 9.** Plastic Viscosity of 4g of mud samples at Rtp, 40℃ and 70℃,.

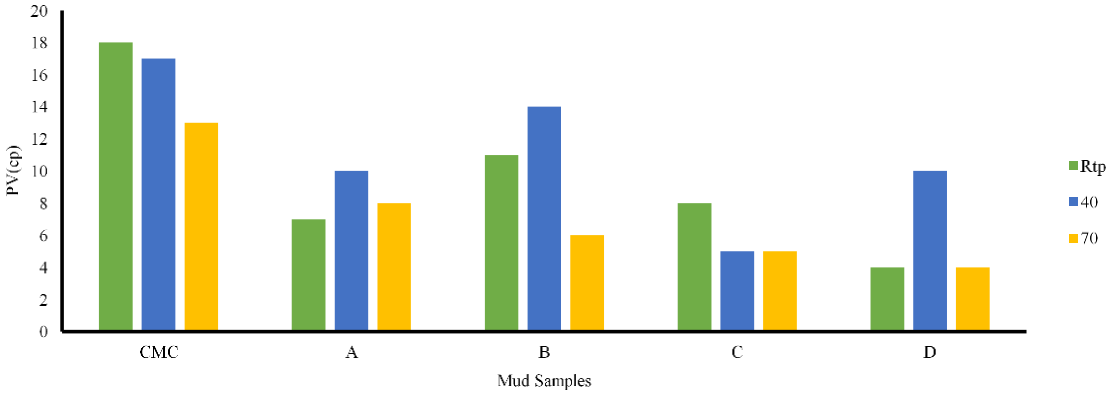


**Figure 10.** Plastic Viscosity of 6g of mud samples at Rtp, 40℃ and 70℃.

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. 2 g of B at r.t.p had the lowest YP at 11cp. All other mud samples had YP within the API specification except 2g of D at 40℃ with 32cp.

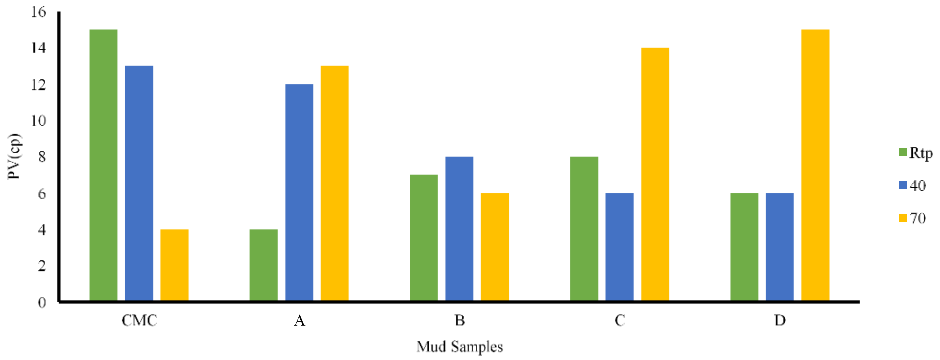


**Figure 11.** Apparent Viscosity of 2g of mud samples at Rtp, 40℃ and 70℃.

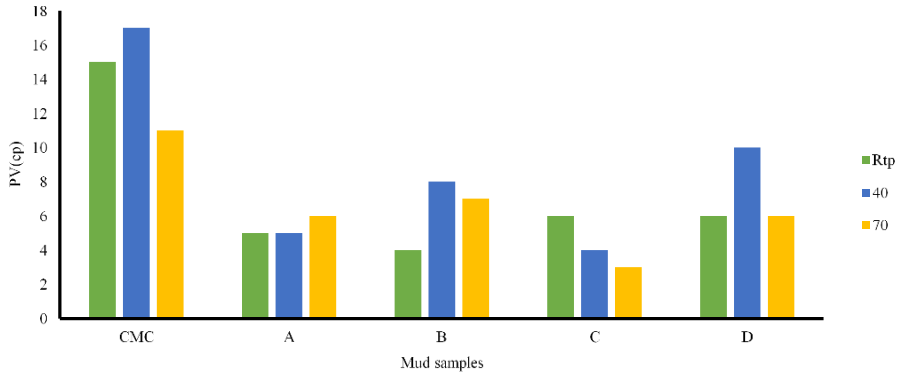


**Figure 12.** Apparent viscosity of 4 g of mud samples at Rtp, 40℃ and 70℃.

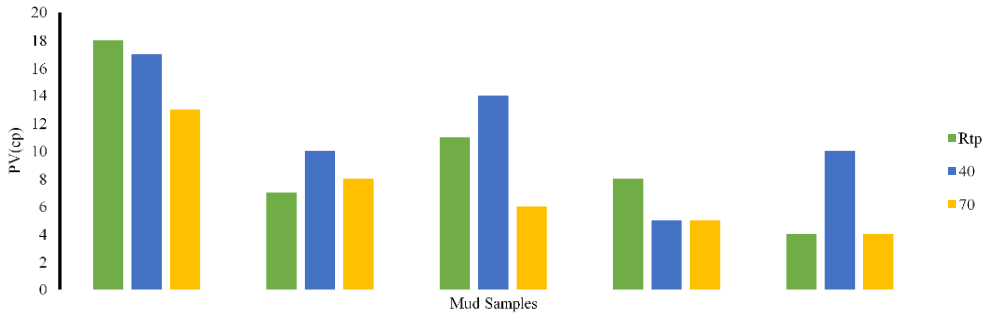


**Figure 13.** Apparent Viscosity of 6g of mud samples at Rtp, 40℃ and 70℃.

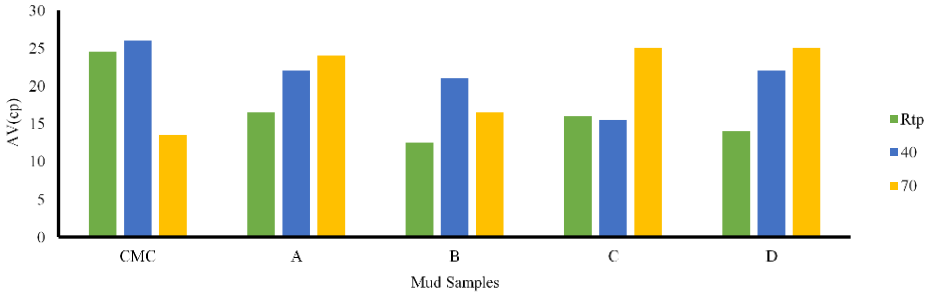


**Figure 14.** Yield Point of 2g of mud samples at Rtp, 40℃ and 70℃

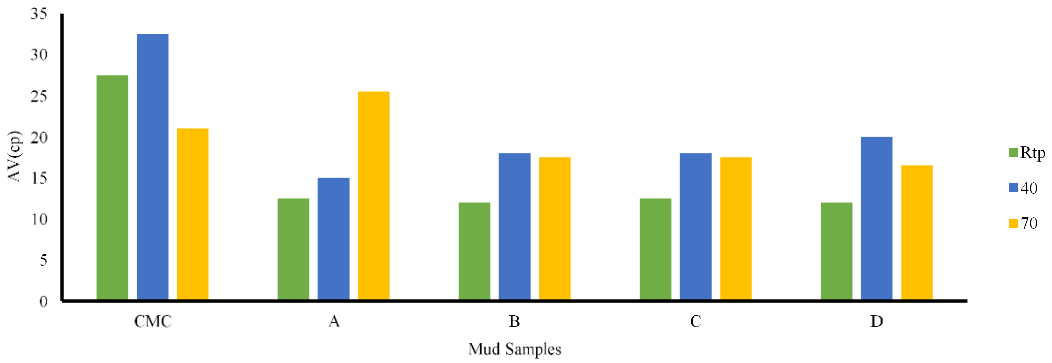


**Figure 15.** Yield Point of 4g of mud samples at Rtp, 40℃ and 70℃.

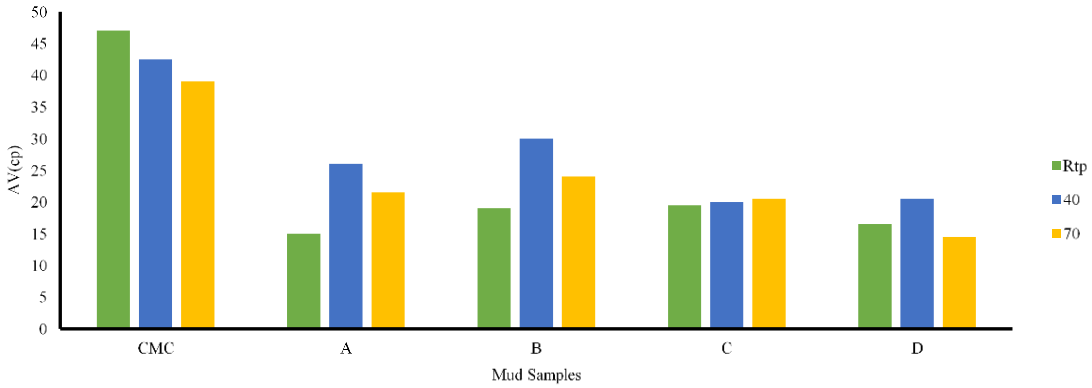


**Figure 16.** Yield Point of 6g of mud samples at Rtp, 40℃ and 70℃.

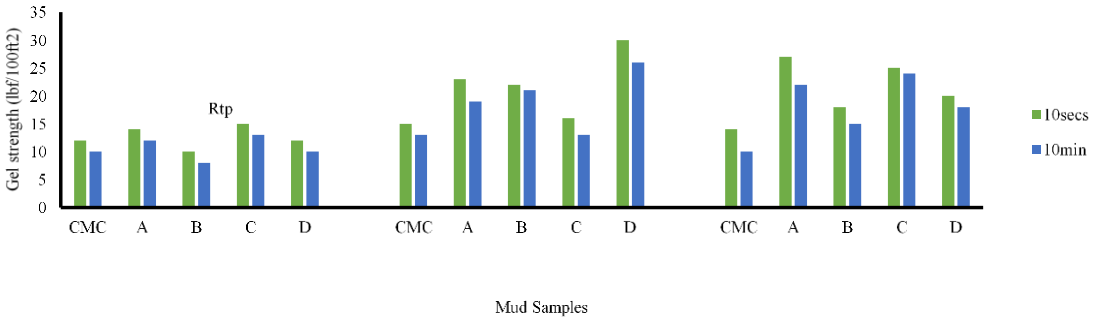


**Figure 17.** Gel strength of mud samples containing 2 g of copolymerized starch at 10 secs and 10mins.

### *Gel strength*

The API specification for the difference between the 10 seconds and 10 minutes 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 10 seconds and 10 minutes at r.tp, 40℃ and 70℃. All copolymerized mud samples showed the difference between 10 seconds and 10 minutes gel strength to be less than 20, therefore, less pump power is needed to start the circulation after the static condition (Sulaimon *et al.,* 2020). The drilling mud sample prepared with 2 g of copolymerized starch of genotype B at 40℃ had the same value at 10 seconds and 10 minutes gel strength. The same was also observed for the drilling mud sample prepared with 6 g of copolymerized starch of genotype B at 70℃.

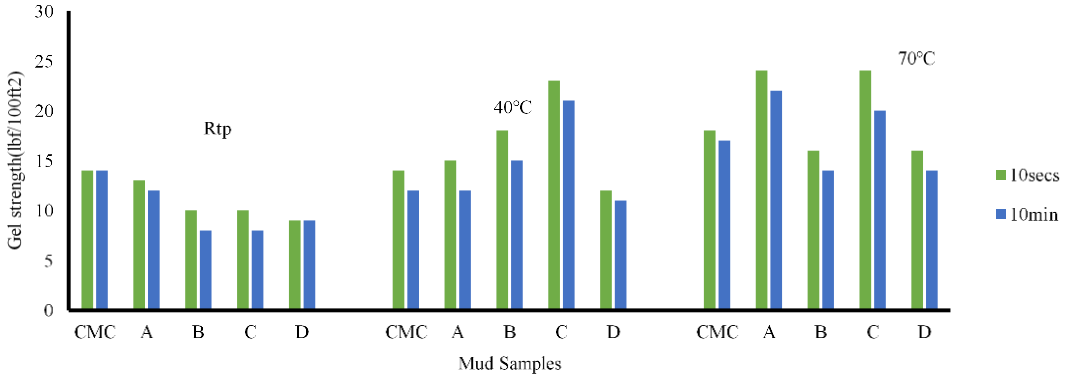


**Figure 18.** Gel strength of mud samples containing 4 g of copolymerized starch at 10secs and 10mins.

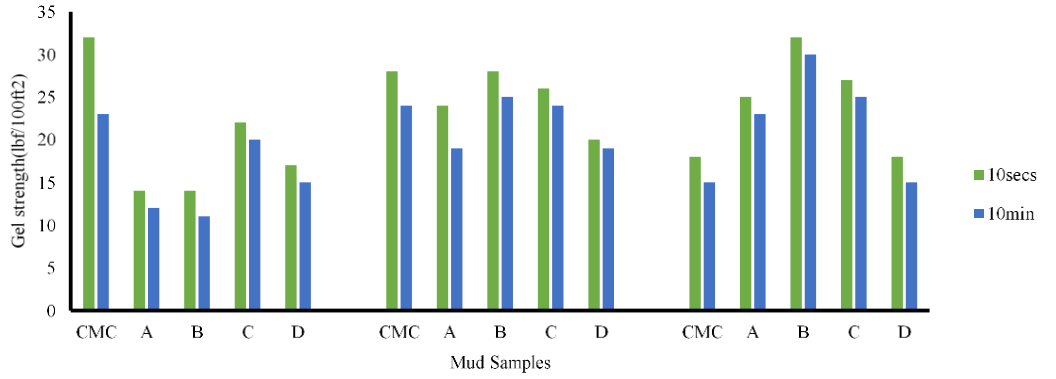


**Figure 19.** Gel strength of mud samples containing 6 g of copolymerized starch at 10secs and 10mins.

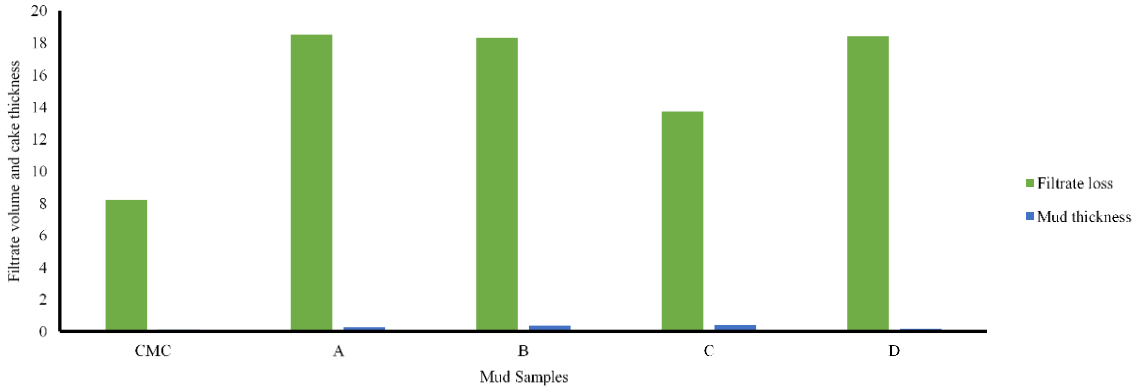


**Figure 20.** Filtrate volume and mud cake thickness for mud samples containing 2g of copolymerized starch.

## 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 30 minutes. The test measured the quantity of water lost from the drilling mud at 30 minutes. All drilling mud samples produced a thin permeable filter cake, which indicates that formation damage will be minimised because of reduced fluid invasion (Ali *et al.*, 2022). Drilling mud samples prepared with 2g of A, B and D all had filtrate volume ranging from 18.3 t0 18.5ml while

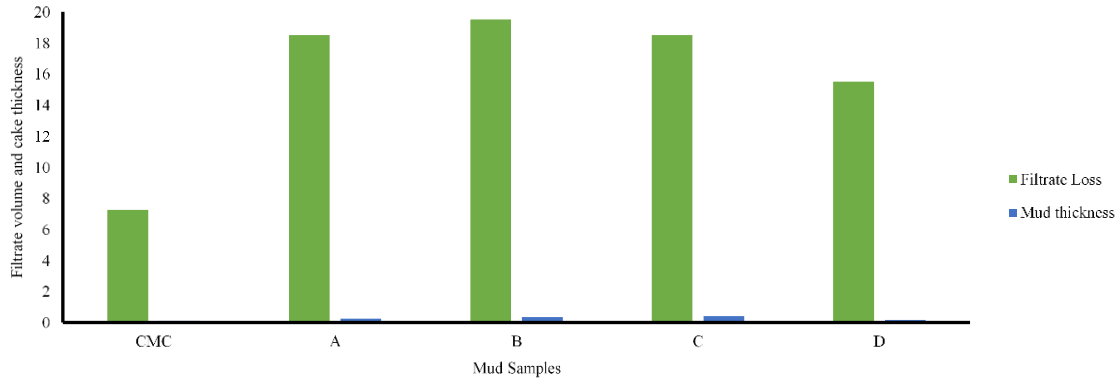


**Figure 21.** Filtrate volume and mud cake thickness for mud samples containing 4 g of copolymerized starch.

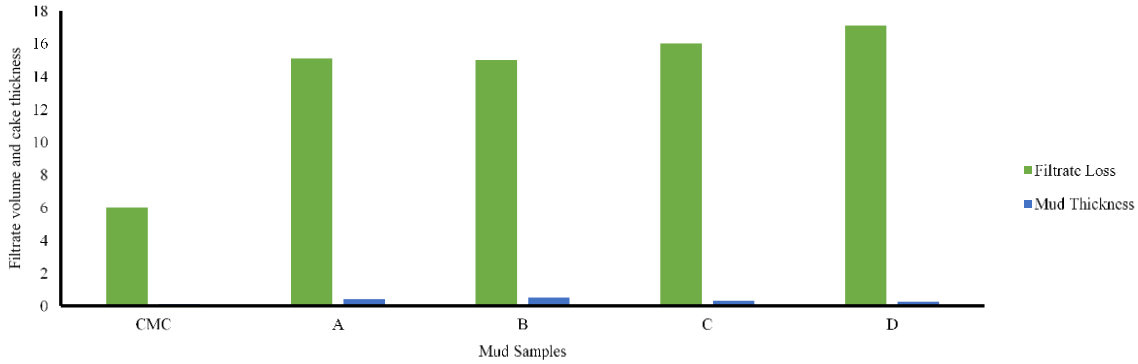


**Figure 22.** Filtrate volume and mud cake thickness for mud samples containing 6g of copolymerized starch.

the C sample had a filtrate loss value of 13.7 ml. For the 4g mud samples, D had the least filtrate loss volume at 15.5ml and the thinnest filter cake at 0.15 ml.

Drilling mud samples prepared with 6 g of the copolymerized starches all showed reduced filtrate loss volume, with D showing the highest filtrate loss at 17.5 ml 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.

## Conclusion

Native starch was modified by graft copolymerization, and the effect of these modifications 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°C had a PV of 15 cp and is capable of lifting drill cuttings. Mud sample 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, the mud sample treated with D had better rheological properties and filtration loss control. From the results, increasing concentrations of the modified starch do not have a significant effect on the mud properties; however, there are differences in the mud properties based on the genotype of the cassava. It was also observed that mud samples containing modified starch are shear-thinning, pseudoplastic and therefore have acceptable flow characteristics.

## Recommendation

The findings from this study on the “Effect of Acrylamide-Grafted Starch on the Properties of Water-Based Mud” provide a strong foundation for advancing sustainable drilling fluid technology. To extend these results, future research should compare the four cassava starch variants against other starch sources (e.g., corn, potato) to correlate amylose/amylopectin ratios with grafting efficiency and mud performance. Optimising grafting protocols through response surface methodology, exploring novel initiators, or irradiation techniques could enhance scalability and cost-effectiveness. Investigating synergistic blends with biopolymers like xanthan gum or guar gum under high-pressure, high-temperature condi-

tions may yield superior mud formulations.

## CONFLICT OF INTEREST

The author states that there is no conflict of interest.

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