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STRUCTURE-PROPERTY RELATIONSHIP OF PECTIN BIOPOLYMER FOR ADVANCED FILTRATION CONTROL IN WATER-BASED DRILLING MUD

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ABSTRACT

In this work, water-based drilling mud was prepared, using pectin biopolymer obtained from citrus peels and modified with calcium carbonate obtained from snail shells to maintain the sample's bio-based nature. Pectin was extracted from orange peels by acid extraction method, in a Hydrochloric (HCL) acid solution of 0.05M concentration, at 100°C. It was filtered and coagulated with Isopropanol in the ratio of 1:2. Calcium Carbonate was obtained from Calcined Snail shells at 400°C ± 20°C for 60 minutes in the Laboratory Furnace. The Pectin Was modified with Calcium Carbonate solution in the ratio of 1:5. The samples were analyzed by Fourier Transform Infrared Spectroscopy (FTIR), and Atomic Force Microscopy (AFM). The modified Pectin was used to prepare a polymer water-based drilling mud tagged MCP. A standard mud was also prepared from Chemically modified carboxymethyl starch Tagged MMS. The filtration properties of both muds were obtained by filter loss method at room temperature of 25°C and high temperature of 270°C. The FTIR peaks showed the presence of hydroxyl, carbonyl, C-N or C-O bonds, which adds to the stability and flow properties of the drilling mud. AFM showed the size, shape and distribution of the grains which aids surface reactivity and adsorption. From the results, Fluid Sorptivity for MCP = 29.619 ml/min. Fluid Sorptivity for MMS = 28.00 ml/min. This is proving that MCP has better fluid loss control than MMS, at the temperatures under study. It also shows that MCP has better ability to maintain wellbore stability during Drilling.

Keywords: Biopolymer, Drilling Mud, Filtration, Pectin, Sorptivity, Structure-Property.

1. INTRODUCTION

The oil and gas industries produce a huge amount of spent drilling fluid, produced water, and accumulated drill cuttings from drilling operations, which are the sources of environmental pollution (Effiom, 2023; Borah & Das, 2021). Today's interest in protecting nature is encouraging researchers and the oil and gas industry to move towards more environmentally friendly practices (Udeagbara et al., 2021; Taleb et al., 2020). By this, biopolymers are used as drilling fluid additives in drilling operations (Lalji et al., 2023; Behnamanhar et al., 2014; Fend & Pehils, 2012). The development of special types of fluids and additives to cure all sorts of downhole problems bring its own difficulties (Li et al., 2022). In this project, two biopolymers (Pectin, Calcium Carbonate) have been used to improve the biodegradability of drilling mud, enhance sorptivity, and reduce environmental damage. Pectin is a natural heteropolysaccharide that which is found in the cell wall of all higher plants (Abeline et al., 2021; Müller-Maatsch et al., 2016). It has been used for its gel formation, thickening and stabilizing properties in a wide range of applications from food to the pharmaceutical and cosmetic industries (Alamineh, 2018). They are present in the cell walls located in the middle lamella, and primary and secondary cell walls (Müller-Maatsch et al., 2016). Pectin serves as an excellent viscosifier in drilling mud (Halvorsen et al., 2019; Fend & Pehils, 2012). d-Galacturonic acid is the main chemical component of pectin, where it exists as the polymer poly-galacturonic acid. It has an aldehyde group at C1 and a carboxylic acid group at C6 in the purest form. Typical levels of pectin in fresh fruits and vegetables include; Apples, 1–1.5%, Apricots, 1%, Cherries, 0.4%, Oranges, 0.5–3.5%, Carrots 1.4%, Citrus peels, 30%, Rose hips, 15% (Chike et al., 2020). Orange is a perennial plant with the botanical name *Citrus Sinensis*. The major regions in Nigeria known for high orange production include states like Kano, Sokoto, Niger, Kaduna, and Adamawa (Udeagbara et al., 2021; Chike et al., 2020). Sokoto state located in the northwestern part of Nigeria is known particularly for its significant orange production. Orange trees typically take about three to four years to start producing fruits after being planted. When an orange juice producer is done squishing its oranges, a lot of orange peel is left unused (Chike et al., 2020). This peel is considered by some farmers to be waste and may be used to feed the cattle or perhaps as fertilizer. This orange peel is the raw material for this work. Snails are freshwater gastropod mollusks with single shells typically spiral in shape. They are found in a wide range of environments, inter-tidal zones to deep ocean floors and freshwater habitats. Snails are of various sizes. The shells of snails are made of calcium carbonate (Xintong et al., 2021; Zhu et al., 2019; Xue et al., 2018). When the calcium carbonate shells of snails are processed, they yield nanomaterials that are fit as additives for drilling mud. Calcium carbonate is an organic compound with the chemical formula CaCO_3 and it is very abundantly available in nature, obtained from sources like shells (Medved & Gaurina-me, 2022; Michael et al., 2021). It has a role as an antacid, a food coloring, a food firming agent, and a fertilizer. It is a calcium salt, a carbonate salt, a one-carbon compound, and an inorganic calcium salt (Chike-Onyegbula et al., 2012; Hassim & Rachmawati,

2010). Its primary use today is as a bridging material in drill-in, completion, and workover fluids (Jamrozik et al., 2016).

2. MATERIALS

The materials used in the extraction of Pectin, Calcium Carbonate, and preparation of the drilling mud include; Citrus (orange) peels, snail shells, Double distilled water (H₂O), Isopropanol (C₃H₈O), Hydrochloric acid (HCL), Soda ash, Caustic soda (NaOH), Borax, Potassium chloride, Barite, Xanthan gum, Carboxymethyl starch (CMS), Grinding machine, Weighing balance, Sieve/Mesh, Knives, Water bath, Oven, Measuring cylinder, Conical flask, Beakers, Tray, Filter paper, White filter fabric, pH meter, Bowl/Basin, Standard filter press, Stirrer, Thermometer, Stop watch, Refrigerator, Furnace, Perforated sheet, Mixer.

3. METHODS

3.1 Sample Preparation Methods

The procedure for this study involves sample preparation of the citrus peel, the extraction of pectin from citrus peels using acid extraction method, extraction of CaCO₃ from snail shell using calcination method, modification of the extracted pectin, preparation of drilling mud, and experimental method (filter loss method).

3.1.1 Extraction of Pectin from Orange Citrus Peels

Appropriate quantities of oranges were gathered from the local market at Ihiagwa in Imo State. The oranges were neatly washed with clean water and then peeled using a knife. After seven days of sun drying the orange peels, the peels were further ground into smaller particles using a grinding machine and the particle size was further reduced into smaller finer powder through sieving. A quantity of powdered orange peels was added to an adequate quantity of a hydrochloric acid (HCL) solution of concentration 0.05M. The mixture was heated in a water bath at a constant temperature of 100°C for 1hr while continuously stirred for 2 minutes after boiling. Subsequently, the mixture was filtered using a filter fabric and then a filter paper to extract the filtrate which is a brown viscous liquid from the residue. This process was repeated to obtain an adequate amount of viscose filtrate. To coagulate the filtrate, a good quantity of isopropanol was added, and the mixture was stirred resulting in the emergence of a solid or gel-like mass (coagulum) confirmed to be pectin. The coagulated pectin in the isopropanol was refrigerated at a controlled cooling temperature for cooling and sedimentation which was later collected, dried, shredded and stored in a vacuum.

3.1.2 Extraction of Calcium Carbonate (CaCO₃) from Snail Shell

Good quantities of snail shells were gathered from the local market and the shells were then washed thoroughly with water to remove debris or organic materials (dirt). The shell was sun dried on a flat perforated surface for several hours, and later was crushed into smaller particles using a grinder. The crushed shells were sieved into finer particles and calcinated at 400°C ± 20°C for 60 mins (1hr) using the laboratory furnace. The result (a white powder) was confirmed to be CaCO₃.

3.1.3 Modification of Pectin

The modification was done to pre-gelatinize the extracted pectin. During the modification, some calcium water was firstly prepared by mixing the calcium Carbonate and water in the ratio 1:5, and this meant that 100g of calcium Carbonate was added to 500ml of distilled water and was well stirred. The stirring was done at an interval of 10 minutes for about 40 minutes. The extracted pectin of weight 250g was added to the mixture and the whole solution was stirred in a mixer at intervals for about 12 hours. At this point, there was formation of a gel, which was allowed to dry and solidify under atmospheric temperature. The solidified gel was transformed into powdered form by grinding.

3.1.4 Preparation of Drilling Mud

The pectin polymer in powdered form was used to prepare water-based drilling mud. The water-based polymer drilling mud tagged MCP in this work was prepared by mixing 4.0g, 8.0g, 15.0g, 20.0g and 25.0g of the powdered modified citrus pectin polymer respectively in 520ml of water to obtain 0.01-0.05g/ml concentrations of pectin in water for the MCP mud sample. Firstly, the mud (MCP) was prepared by mixing bentonite clay and water, and then the pectin polymer was added slowly to avoid formation of lumps, while stirring takes place. Soda ash, caustic soda and borax were included. Also, for comparison, chemically modified Carboxymethyl starch was used to prepare standard water-based drilling mud of the same concentrations and more additives, and the standard mud is tagged MMS in this work.

Table 1: Formulation for MMS mud (Standard mud)

S/N	Materials	Quantity/Weight	Functions
1	Water	630ml	Base Fluid
2	Xanthan gum	3.4g	Viscosifiers
3	Potassium Chloride	15g	Inhibitor
4	Barite	38.4g	Weighting agent
5	Bentonite	20.0g	Weighting agent
6	Caustic Soda	0.4g	pH adjustment

7	Borax	1.5g	Preservative
8	Soda ash	0.4g	Hardness control
9	Carboxymethyl starch	4.0g, 8.0g, 15.0g,	Filtration/fluid loss control, Viscosifiers, and inhibitors.
		20.0g, 25.0g	

Table 2: Formulation for MCP mud (New mud)

S/N	Materials	Quantity/Weight	Functions
1	Water	630ml	Base Fluid
2	Bentonite	20.0g	Weighting agent
3	Caustic Soda	0.4g	pH adjustment
4	Soda ash	0.4g	Hardness control
5	Borax	1.5g	Preservative
6	Pectin polymer	4.0g, 8.0g, 15.0g,	Filtration/Fluid loss control, viscosifiers, and inhibitor
		20.0g, 25.0g	

3.2 Experimental Methods

3.2.1 AFM and FTIR Methods for Characterizing the Extracted Pectin and CaCO₃

Atomic Force Microscopy (AFM) and Fourier Transform Infrared Spectroscopy (FTIR) are the two techniques used to characterize the structure and properties of both the pectin and CaCO₃ at molecular level. AFM allows high resolution imaging of the surface topography of both pectin and CaCO₃ molecules, providing information about the size, shape, and surface roughness of the samples (Xue et al., 2018; Youlin, 2013). FTIR spectroscopy was used to analyze the chemical composition and molecular structures of the samples by measuring the absorption of infrared light by the samples (pectin and CaCO₃) molecules.

3.2.2 Filter Loss Method for determining the Filtration Properties

In the determination of the filtration properties of the mud, filter loss method was used, as described in previous work (Ismail et al., 2017; Jamal et al., 2016; Hall & Hoff, 2012). The two stages of the experiment were performed at room temperature of 25°C and high temperature of 270°C respectively. During the experimental method, 550ml of MCP mud was poured into the chamber of the standard filter press at a constant pressure of 100 psi at room temperature (25°C). The filtration test was run at different time intervals and the filtrate or filter loss, or fluid loss volume was read by measuring in a graduated measuring cylinder. The same quantity of mud was heated in an oven at the higher temperature of 270°C, and the same filtration test was run on it. The same experimental procedures were carried out on the

MMS mud, and the fluid loss volume was read from the graduated cylinder each time, and the readings were recorded and tabulated.

4. RESULTS AND DISCUSSION

4.1 GRAIN ANALYSIS REPORT FOR PECTIN AND CaCO₃ USING AFM

Table 3: Grain Analysis Report for Pectin and CaCO₃ using AFM

	Pectin	CaCO₃
No. of Grains	37	72
Density	141.144 μm^{-2}	274.658 μm^{-2}
Total Projected Area (abs.)	254772 nm^2	252121 nm^2
Total Projected Area (rel.)	97.19%	96.18%
Mean Grain Area	6885.73 nm^2	3501.68 nm^2
Mean Grain Size	100.2313 nm	24.4510 nm
Total grain volume (zero)	105703 nm^3	24690.9 nm
Total grain volume (minimum)	75453.4 nm^3	24690.9 nm^3
Total grain volume (Laplace)	3259.54 nm^3	1667.62 nm^3
Total projected boundary length	6.35042 μm	9.07751 μm

From Table 3, there was analysis of the parameters in the grain summary report of the pectin and calcium carbonate sample provides valuable insight into the size, shape, distribution, and volume characteristics of the grains. The detailed discussion of the parameters mentioned in the report (see Table 3) is given below:

Number of Grains: The total number of grains identified in the sample indicates the sample's granularity and the complexity of its microstructure. Analyzing the distribution and arrangement of these grains can provide information on the material's overall composition and properties.

Density: The density value reflects the spatial distribution of grains; it also quantifies the number of grains per unit area. A higher density suggests a more closely packed arrangement of grains, while a lower density suggests a more porous or dispersed arrangement of grains. Understanding density is crucial for assessing material packing and homogeneity.

The Total Projected Area: The total projected area provides information on the overall coverage of grains in the image. A larger projected area indicates a sample with a higher surface coverage, which can affect properties related to surface reactivity and adsorption (Xue et al., 2018; Youlin, 2013).

The Mean Grain Area & Size: The mean grain area and size offer insights into the average size of grains in the sample. Understanding the size distribution is crucial for predicting properties like particle packing, strength, surface area, reactivity, mechanical and thermal properties based on grain size effects.

Total Grain Volume: This report provides multiple values for total grain volume obtained using different methods (zero, minimum, Laplace).

These volume measurements offer insights into the overall volume occupied by the grains and can help assess material density, porosity, and structural integrity.

The total projected boundary length: This indicates the cumulative length of grain boundaries. Longer boundary lengths may suggest complex grain shapes, which can influence properties like surface roughness and inter-particle interactions. Understanding the morphology and structural properties of the grains is essential for tailoring material synthesis processes, optimizing performance, and predicting behavior in specific applications (Xue et al., 2018). Researchers can use this information to design materials with desired properties and functionalities based on the characteristics revealed by the grain analysis.

4.2 FTIR PEAK RESULT FOR PECTIN

Table 4: FTIR Peak Table Result for Pectin

PEAK TABLE		
Peak Number	X (cm 1)	Y (%T)
1	3345.57	95.5
2	1737.77	94.49
3	1226.42	94.02
4	1012.33	94.02

Result Spectrum

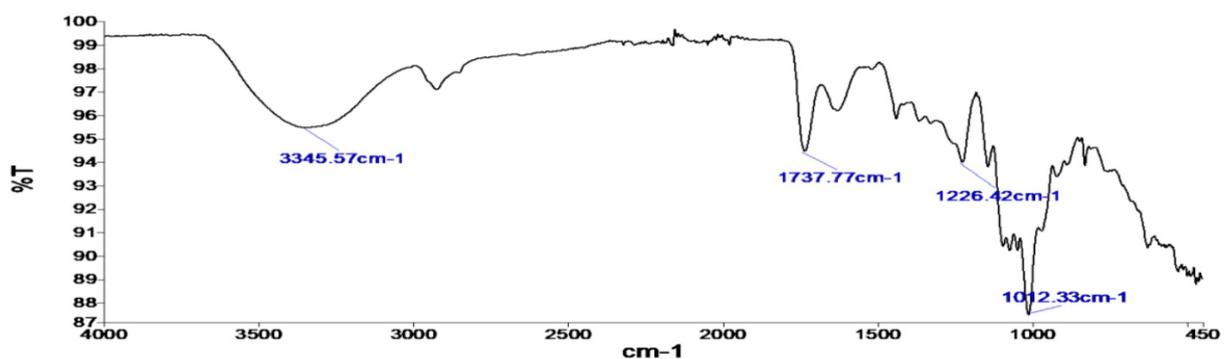


Fig 1: FTIR spectra of pectin extracted from citrus peels.

From Fig 1, and based on the peak table (see Table 4) provided in the report for Pectin, here is an analysis of the peaks observed in the FTIR spectrum:

Peak Number 1: Wavenumber: 3345.57 cm⁻¹, Transmittance: 95.50%. This peak is likely associated with the stretching vibration of the O-H (hydroxyl) functional group, which is commonly found in alcohols, phenols, and carboxylic acids. The high transmittance value suggests a strong absorption at this wavenumber.

Peak Number 2: Wavenumber: 1737.77 cm⁻¹, Transmittance: 94.49%. This peak corresponds to a carbonyl (C=O) stretching vibration, which is a characteristic feature of compounds such as ketones, aldehydes, esters, and carboxylic acids. The high transmittance indicates a significant presence of this functional group in the sample.

Peak Number 3: Wavenumber: 1226.42 cm⁻¹, Transmittance: 94.02%. This peak is likely associated with C-N stretching vibrations in secondary amines or C-O stretching vibrations in alcohols, ethers, or esters. The transmittance value suggests moderate absorption intensity at this wavenumber.

Peak Number 4: Wavenumber: 1012.33 cm⁻¹, Transmittance: 87.48%. This peak falls in the fingerprint region of the spectrum, indicating complex bending vibrations and skeletal motions of the molecule. The moderate transmittance value suggests some structural complexity in this region.

It's necessary to note that by analyzing the peak table, we can infer the presence of specific functional groups in the Pectin sample, such as hydroxyl groups, carbonyl groups, and possibly C-N or C-O bonds. Further interpretation and comparison with known spectra of relevant compounds can provide more insights into the chemical composition and structure of Pectin.

4.3 FTIR PEAK TABLE RESULT FOR Calcium Carbonate, CaCO₃

Table 5: FTIR Peak Table Result for CaCO₃

PEAK TABLE		
Peak Number	X (cm 1)	Y (%T)
1	1467.81	53.58
2	1083.13	89.09
3	858.27	66.07
4	712.5	79.39
5	700.03	89.07

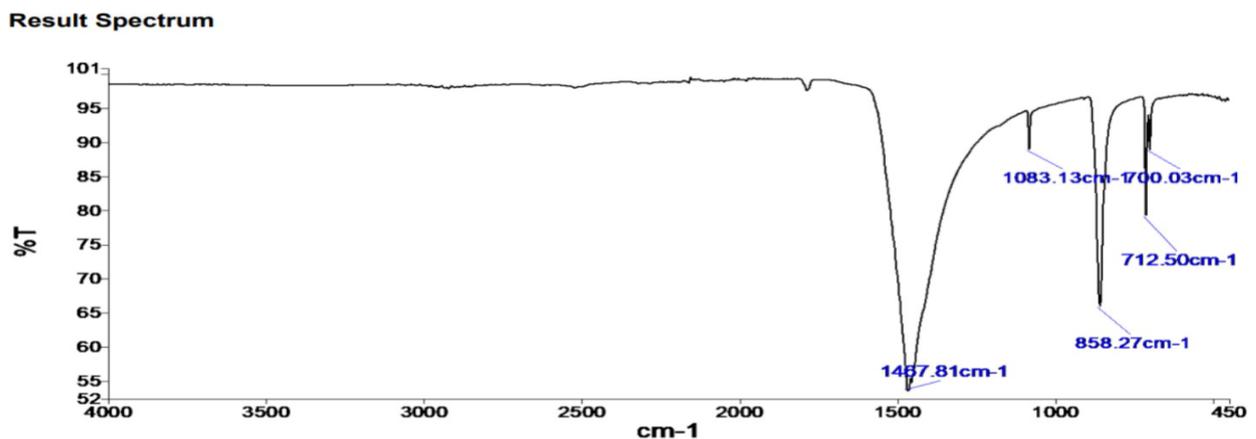


Fig 2: FTIR spectra of CaCO₃ extracted from snail shells

Fig 2 and the peak table (see Table 5) provided in the report for the Calcium carbonate sample offer valuable information about the absorption peaks observed in the FTIR spectrum. Here is an analysis of the peak table results:

Peak Number 1: (1467.81 cm⁻¹, 53.58%T), this peak corresponds to the bending vibration of carbonate (CO₃) ions in the Calcium carbonate sample. The presence of this peak indicates the presence of carbonate groups in the sample.

Peak Number 2: (1083.13 cm⁻¹, 89.09%T), this peak is characteristic of the symmetric stretching vibration of carbonate (CO₃) ions. The high transmittance value suggests that this vibration is strong in the sample, further confirming the presence of carbonate groups.

Peak Number 3: (858.27 cm⁻¹, 66.07%T), the peak at 858.27 cm⁻¹ is associated with the out-of-plane bending vibration of carbonate (CO₃) ions. The moderate transmittance value indicates the presence of this vibration mode in the sample.

Peak Number 4: (712.50 cm⁻¹, 79.39%T), this peak could be attributed to the bending vibration of the carbonate (CO₃) ions or may indicate the presence of impurities or other functional groups in the sample. Further analysis or comparison with reference spectra may be needed to confirm the exact nature of this peak.

Peak Number 5: (700.03 cm⁻¹, 89.07%T), the peak at 700.03 cm⁻¹ could also be related to the bending vibration of carbonate (CO₃) ions or other structural features present in the Calcium carbonate sample. The high transmittance value suggests a significant presence of this vibration mode.

Overall, the peak table analysis confirms the presence of characteristic carbonate.

(CO₃) vibrations in the Calcium carbonate sample, which is in line with the expected composition of Calcium carbonate. The absence of warnings in the quality check indicates a successful analysis with reliable results (Hassim & Rachmawati, 2010).

4.4 RESULTS FROM THE FILTER LOSS EXPERIMENT FOR MUDS AT ROOM TEMPERATURE, 25°C

Table 6: Results from the Filter Loss Experiment for Muds at RoomTemperature, 25°C

Time	Square Root of Time $T^{1/2}$	MCP		MMS	
(Mins)	(mins)	Fluid Loss volume V	Rate of	Fluid Loss volume V	Rate of
		(ml)	Filtration dv/dt	(ml)	Filtration dv/dt
			(ml/min)		(ml/min)
5	2.23	129	25.8	148	29.6
10	3.16	152	15.2	170	17
15	3.87	170	11.33	185	12.33
20	4.47	189	9.45	206	10.3
25	5	207	8.28	222	8.88
30	5.47	220	7.33	238	7.93
35	5.91	233	6.66	251	7.17
40	6.32	248	6.2	263	6.58
45	6.7	260	5.78	270	6
50	7.07	268	5.36	276	5.52

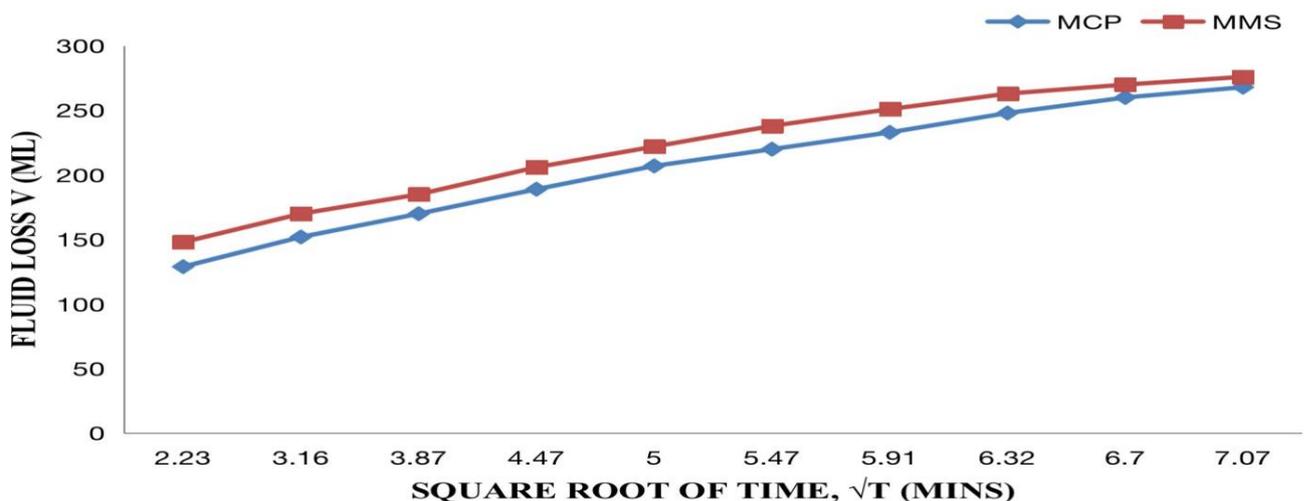


Fig 3: Plot of Fluid Loss Versus Square Root of Time for the Muds at Room Temperature 25°C.

From the graph above (fig 3), the fluid loss rate of MCP (pectin mud) is slightly lower than that of MMS (Carboxymethyl starch) standard mud at 25°C. This proves that MCP has a better fluid loss control when compared to MMS. In drilling operation, MCP has a better ability to maintain wellbore pressure and control wellbore instability over MMS at room temperature. It's important to note that the fluid sorptivity (at 25°C) is equal to the slope of the curve. The fluid sorptivity for each mud is calculated from the graph, and the value is given below;

Fluid Sorptivity for MCP = 29.619 ml/mins

Fluid Sorptivity for MMS = 28.00 ml/mins

The relationship between Fluid Loss Volume, Sorptivity and Square Root of time is given the equation (Shuvo et al., 2023; Maiti et al., 2021; Zengwei et al., 2019; Hall & Hoff, 2012):

$$V = S\sqrt{T} \quad [1]$$

Where V = Fluid loss volume, S = Sorptivity and T = Square root of time

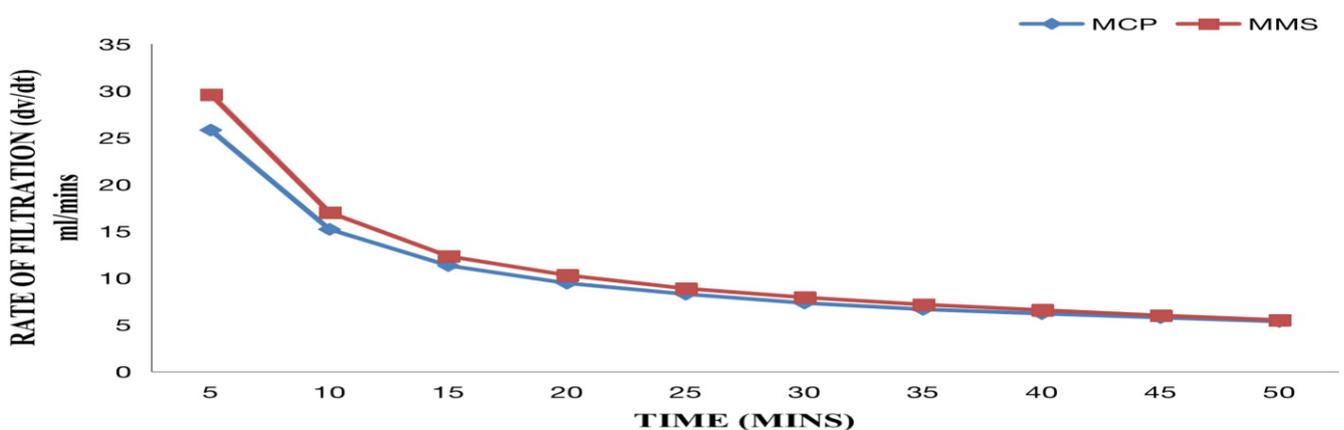


Fig 4: Plot of Rate of Filtration versus Time for the Muds at Room Temperature, 25°C

From the above plot, it is observed that the rate of filtration of MCP is slightly lower than the filtration rate of MMS as both decreases at increase in time. Rate of filtration is the speed at which the mud (MCP & MMS) loses liquid phase into the surrounding formation. The result from this plot shows high fluid

loss of MMS which leads to decrease in well stability when compared to MCP. High filtration rate causes formation weakening and wellbore collapse (Ebikapaye, 2018). MCP is a better option when compared to MMS in terms of filtration rate showing a more preserved permeability.

The slope of this plot shows the fluid diffusivity of the mud (MCP & MMS).

4.5 RESULTS FROM THE FILTER LOSS EXPERIMENT FOR MUDS AT HIGH TEMPERATURE, 270°C.

Table 7: Results from the Filter loss Experiment for Muds at High Temperature, 270°C

Time	Square Root of Time	MCP		MMS	
(MINS)	\sqrt{T} (MINS)	Fluid Loss volume V	Rate of Filtration dv/dt	Fluid Loss volume V	Rate of Filtration dv/dt
		(ML)	(ML/MIN)	(ML)	(ML/MIN)
5	2.23	156	31.2	187	37.4
10	3.16	184	18.4	210	21
15	3.87	211	14.07	234	15.6
20	4.47	227	11.35	255	12.75
25	5	240	9.6	270	10.8
30	5.47	252	8.4	289	9.63
35	5.91	262	7.49	294	8.4
40	6.32	271	6.78	263	6.58
45	6.7	277	6.16	302	6.71
50	7.07	28	5.66	308	6.16

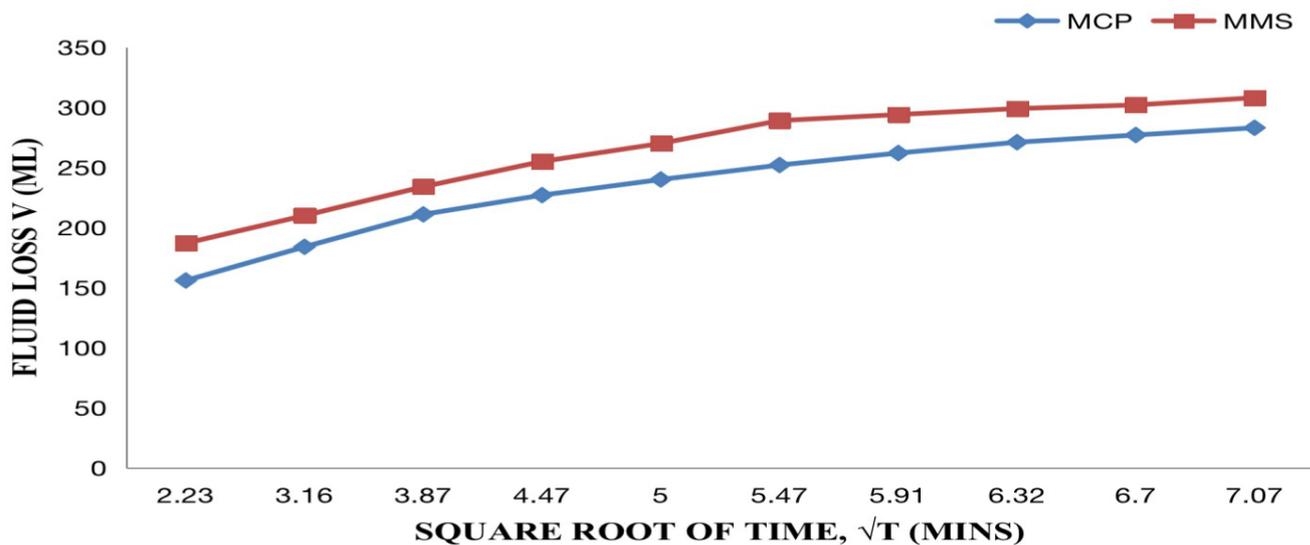


Fig 5: Plot of Fluid Loss Versus Square Root of Time for the Muds at High Temperature: 270°C

From table 7 and fig 5, it is observed that at high temperature (270°C), the fluid loss volume of MCP is slightly lower than that of MMS. The above result implies that the effect of high temperature drilling in fluid loss is lesser in MCP when compared to MMS. Therefore, it is important to note that the pectin mud (MCP) will give a better performance and wellbore integrity in high temperature drilling operations than the standard Carboxymethyl starch mud (MMS).

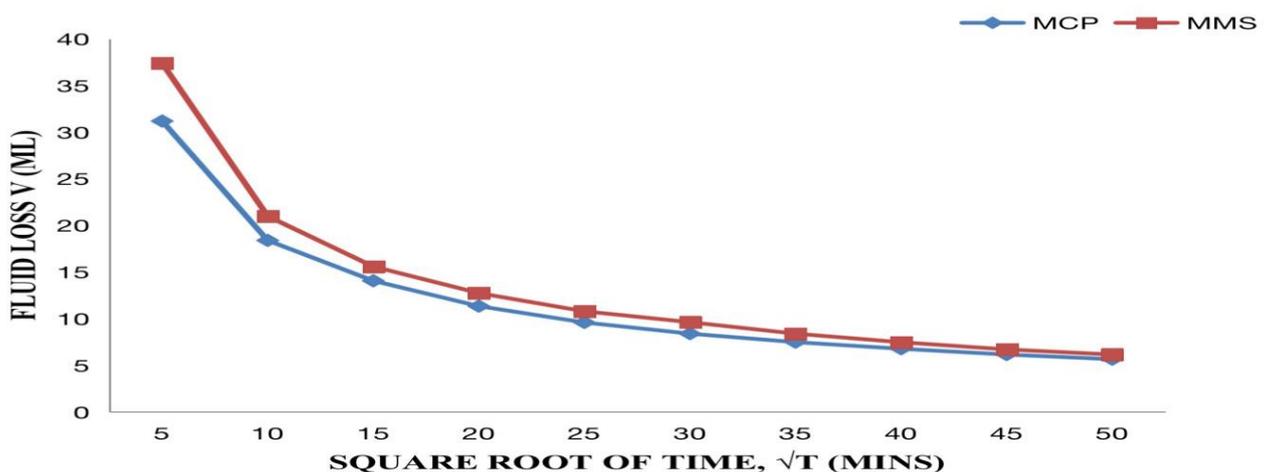


Fig 6: Plot of Fluid Loss Rate of Filtration versus Time for the Muds at High Temperature, 270°C

From fig 6, it is observed from the plot that the rate at which filtration occurs at high temperature in MMS is higher than that of MCP.

In high temperature drilling operation, the rate of mud filtration can be significantly influenced by elevated temperature leading to faster rate of mud filtration, resulting in more rapid fluid loss into the formation (Chike et al., 2020; Hamoodi et al., 2018). In summary, the Rate of Filtration versus Time graph indicates that filtration control is better in

MCP when compared to MMS.

5. CONCLUSION

The following conclusions were drawn from the work.

Pectin extracted from orange peels is suitable for preparing biodegradable polymer drilling mud. Biodegradable polymer drilling mud (fluid) can be prepared using pectin extracted from orange peels

which has been pre-gelatinized with calcium-water. The new biodegradable polymer mud has better fluid loss control behavior, better filtration rates, thus, the new mud has better fluid control agent. The low cost and accessibility of the orange peels which is a waste material can account for a reduced well cost. This biodegradable polymer drilling fluids prepared from pectin obtained from orange peels are environmentally friendly, non-toxic and are suitable for drilling operations in any environment.

The thermal stability of the new mud at 270°C implies that the mud is suitable for drilling well bores with bottom hole temperature as high as 270°C.

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The author(s) declare that it is not applicable.

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The author(s) declare that this is not applicable.

Competing interests

The author(s) declare that they have no competing interests.

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