Fluid Loss and Filtration Properties of a Citrus Sinensis Pectin Extract-based Drilling Mud

Chike K. O. Iheaturu N. C. Aharanwa B. C. Ezeamaku L. U. Ojinmah N. E. Uwakwe C. Ojiaku C. I. Department of Polymer & Textile Engineering, Federal University of Technology, P.M.B 1526, Owerri, Imo State, Nigeria

Abstract

The production of drilling mud using pectin extracted from Citrus Sinensis peels was carried out. The extraction was carried out using water-hot acid technique which is a conventional method, and the extracted pectin was pregelatinized using calcium water. The pre-gelatinized pectin biopolymer was used to prepare drilling mud. Filter loss method was used to determine the filtration properties of the mud at 25°C and 200°C and 0.1g/mol concentration of pectin polymer was compared to the filtration behavior of hydroxyl propyl starch modified drilling mud. Our results showed that the pectin biopolymer mud (PPM) has better filtration control behavior than the hydroxyl propyl starch modified drilling mud (CMM). The study also showed that highest sorptivity value of 21.25 was obtained with PPM at 200°C, while the highest diffusivity value of 0.424 was obtained with CMM at 200°C. **Keywords:** Pectin, Citrus, Biopolymer, Sorptivity, Diffusivity, Drilling mud, Filtration **DOI:** 10.7176/CMR/12-3-03

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1.0 Introduction

Pectin, a naturally occurring biopolymer, is found in cell walls of fruits and vegetables (1–3). The various sources of pectin include citrus peels like orange, grapes, and tangerine, sugar beets, passion fruit and residues of mango, banana peels, guava, coffee, dried apple pomace, sunflower heads, papaya, and cocoa processing (4,5). The middle lamellae of citrus peels and apple fruits accounts for the richest sources of pectin (6,7). It has been spaciously applied as a gelling agent in jam manufacture. Today, in addition to its usage in the food and beverage industry as a thickener or gelling agent and as a colloidal stabilizer (8), it has found use also in the pharmaceutical and biotechnology industries (9,10). Though pectin occurs in the middle lamellae of most plant tissues, there is inadequate measure of pectin that may be utilized from those plants for commercial purposes (11,12). New applications have emerged for pectin, not just a gelling agent, but may also be used in drilling fluids (8). Many of earth's valuable resources such as oil and mineral water can be found deep beneath the surface. Drilling has been used to obtain these resources from the earth's crust and this has led to the existence of drilling fluids to facilitate the process. The earliest form of drilling fluid was made with water, which has the basic functions of softening rock and bringing the cuttings out of the well (13). With the advances in technology, drilling fluids are far more functional than in earlier productions. It now has the ability to remove drill cuttings from well, cool and lubricate the drill bit, stabilize the borehole wall, lubricate the drill pipe, reduce fluid loss the formation and suspend (14). Different types of drilling fluids have been used over the years such as water based fluid (WBF) and non-aqueous fluids (NAF) (15-17).

Drilling oil and gas produces wastes classified into liquid waste involving produced water and solids involving mud, clay, shale, sand and cuttings. In most cases, disposal of this waste possess a big threat to the environment, more so when most of the additives used in formulating drilling muds are toxic, with different levels of toxicity (18), making it imperative that the environment where the muds are used should be protected from serious harm and the habitat preserved. There is the need for additives that is environmentally friendly (16,19). Drilling muds should be biodegradable, less toxic with no aromatic compounds. In this work, pectin extracted from Citrus Sinensis peels was used to produce a drilling mud that is inherently biodegradable to ameliorate toxicity problem when released to the environment.

2.0 Materials and Methods

2.1 Materials

Citrus Sinensis peels, lemon juice, distilled water, calcium phosphate powder (rock mineral source), sodium hydroxide, clay (bentonite), hydroxyl propyl starch (standard grade) and methyl ethyl ketone containing 10% methanol (rubbing alcohol), all of analytical grade, were used in this experimental study.

2.2 Sample Preparation

2.2.1 Extraction of Pectin

The Citrus Sinensis s were peeled and the white membranous part of the peel was dried in open air. The peel was cut into slim strips and a knife was used to remove the white part of the peel, known as the pith. Water and 2-5 tea

spoons of lemon juice were mixed with the chopped pith. This mixture was left for an hour. In a large non-reactive (stainless or enamel) sauce pan, the mixture of water, pith and lemon juice was boiled for 10-20mins. After boiling for 10-20 minutes, the boiled mixture was poured into a jelly bag and allowed to drain.

2.2.2 Test for pectin

One (1) tea spoon of the liquid mixture and rubbing alcohol (isopropyl alcohol) were mixed in a small jar and stirred gently for proper mixing. The mixture was allowed to stand for a minute, which later formed a jelly-like mass showing the presence of pectin. Normally, if the liquid contains pectin, it will form a jelly like mass. If it forms doted lumps, it has no pectin.

2.3 Modification of Pectin by Pre-Gelatinization

The pre-gelatinization was carried out by firstly preparing calcium phosphate dissolved in water in the ratio, 1:3 150g of calcium phosphate powder was added to 450ml of double distilled water and stirred well. The solution was stirred for 30 minutes at intervals of 5minutes and then the extracted pectin (100g by weight) was added. The whole solution was well stirred at intervals in a mixer for about 24 hours, and gel was formed. The formed gel was allowed to dry and solidify. It was grounded into powdered form. The product was the pre-gelatinized pectin.

2.4 Preparation of Water-based Drilling Mud

The water-based drilling mud was prepared by mixing 100g of the pre-gelatinized pectin in 500ml of water to obtain 0.1g/ml concentration of the pectin in water. To prepare the drilling mud, clay was firstly put in water and stirred vigorously. Later, the pectin solution was added slowly and stirred constantly for hour. The pH level of the mud was improved by adding sodium hydroxide (NaOH). Which was later compared to a chemically modified mud.

2.5 Determination of mud filtration properties

Filter loss was used to determine the filtration properties of the muds at 25°C and 200°C. 500ml of the mud PPM was poured into the chamber of the standard filter press at a constant pressure of 100psi and room temperature of 25°C. The filtration property of the mud was tested and a quantity was collected at different time intervals in minutes. Also, the same quantity of mud was heated in an oven at 200°C. The mud was re-mixed; further filtration test was done on it to collect more filtrate which was measured with graduated cylinder. The same experimental test was run on the standard mud CMM and all readings were recorded and tabulated.

2.6 Sorptivity and diffusivity of the pectin biopolymer mud

Sorptivity is the ability of a material to absorb or desorbs liquid by capillarity. It is the component of the flow processes and needs to be incorporated in any application where adsorption or desorption of a fluid from a porous media occurs due to a latent change at a surface boundary (**Cook**, 2008). It is also a measure of the resistive force against the fluid flowing through a filter cake. The slopes from the plots of fluid loss against square root of time give the fluid sorptivity. Filtration model for fluid loss, according to American Petroleum Institute (API 2000) is shown in the equation below;

$V = St^{1/2} \text{ or } V = S\sqrt{t}$

(2.1)

Where; V= volume of fluid loss (ml); S = Sorptivity of fluid (ml/min) (obtained as slope of the plot of V versus $t^{1/2}$; t = time of filtration (min).

Diffusivity is the rate of internal circulation of fluid within a system. It is also said to be a measure of the ability of a substance to transmit due to temperature differential. It is expressed as the quotient of the thermal conductivity divided by the product of specific heat capacity and density (Hall and Hoff, 2012). The slope of the plots of rate of filtration against time gives the diffusivity as shown below: $\Phi (R) = \Phi_0 exp^{-Dt}$ (2.2)

Where; Φ_0 and Φ = initial and final filtration rates (sec) respectively; D = diffusivity of fluid; t = time (min).

3.0 Results and Discussion

3.1 The filtration properties of the muds at 25°C

Table 3.1 is presented below from where fluid loss against the square root of time was plotted in Figure 3.1. Table 3.1 shows results of the experiment for formulated pectin based muds at 25°C.

| Table 3.1: Results from the experiment run for the muds at 25°C | | | | | | |
|---|---|---------------------------------|---|------------------------------|--|--|
| | Square | - | PPM | CMM-MUD | | |
| Time (mins) | root of time, t ^{1/2} (mins) | Fluid Loss Volume, V (ml) | Rate of Filtration, dv/dt (volume/ time) | Fluid Loss Volume, V (ml) | Rate of Filtration, dv/dt (volume/ time) | |
| 5 | 2.236 | 101 | 20.200 | 109 | 21.800 | |
| 10 | 3.162 | 115 | 11.500 | 122 | 12.200 | |
| 15 | 3.873 | 127 | 8.467 | 134 | 8.933 | |
| 20 | 4.472 | 151 | 7.550 | 156 | 7.800 | |
| 25 | 5.000 | 160 | 6.400 | 168 | 6.750 | |
| 30 | 5.477 | 166 | 5.533 | 175 | 5.833 | |
| 35 | 5.916 | 171 | 4.886 | 177 | 5.057 | |
| 40 | 6.325 | 174 | 4.350 | 180 | 4.500 | |
| 45 | 6.708 | 175 | 3.889 | 184 | 4.000 | |
| 50 | 7.071 | 178 | 3.560 | 185 | 3.700 | |



Figure 3.1: Plot of fluid loss against square root of time for the pectin based muds at 25°C. From the Table 3.1 and Figure 3.1 at 25°C, it was observed that fluid loss volume of CMM is slightly higher than that of the PPM. This shows that there is a close relationship in the fluid loss behavior of both muds. It is observed from the figure that the fluid loss increased with increase in square root of time.

From Figure 3.1 at 25°C, it was observed that the rate of filtration according to the curves of the two muds were close, showing that they have close relationship but the rate at which Filtration occurs in chemically modified mud is less than that of pectin polymer mud. Therefore, it is clearly seen that pectin polymer mud gives a better filtration than chemically modified mud.



Figure 3.2: Plot of Rate of Filtration against time for the muds at 25°C.

3.2 The filtration properties of the muds at 200°C

Table 3.2 presents the filtration properties of the pectin based muds at a higher temperature of 200°C while Figure 3.3 is the plot of the fluid loss against the square root of time.

| | Square | F | PPM | CMM-MUD | | |
|----------------|----------------------------------|------------------------------|--|------------------------------|---|--|
| Time (mins) | root of time, t 1/2 (mins) | Fluid Loss Volume, V (ml) | Rate of Filtration, dv/dt (volume/ time) | Fluid Loss Volume, V (ml) | Rate of Filtration, dv/dt (volume/ time) | |
| 5 | 2.236 | 126 | 25.200 | 142 | 28.400 | |
| 10 | 3.162 | 155 | 15.500 | 173 | 17.300 | |
| 15 | 3.873 | 163 | 10.867 | 194 | 12.933 | |
| 20 | 4.472 | 181 | 9.050 | 211 | 10.550 | |
| 25 | 5.000 | 209 | 8.360 | 220 | 8.800 | |
| 30 | 5.477 | 213 | 7.100 | 222 | 7.400 | |
| 35 | 5.916 | 215 | 6.143 | 225 | 6.429 | |
| 40 | 6.325 | 220 | 5.500 | 227 | 5.675 | |
| 45 | 6.708 | 223 | 4.956 | 228 | 5.067 | |
| 50 | 7.071 | 226 | 4.520 | 229 | 4.580 | |

| | L | 0 | 1 | | | | |
|-----------|------------------|--------------|---------|----------------|---------------|-----------------|----------------------|
| Table 3.2 | : Result from ex | periment run | for the | filtration pro | operties of t | he muds at higł | n temperature, 200°C |



Square root of time t^{1/2} (mins)^{1/2}

Figure 3.3: Plot of Fluid Loss against Square root of time for the muds at high temperature. From the Table 3.2 and Figure 3.3, it is observed that as the square root of time increases, the curves of the two muds become closer. That means the muds' filtrate or fluid loss volume increases gradually with respect to the square root of time, t^{1/2}. The value at which PPM reduces fluid loss at 200°C is higher when compared to the CMM. Therefore, it is clearly seen that at higher temperature PPM is a better fluid loss controlling agent than CMM.

Figure 3.4 is the plot of rate of filtration against square root of time for the pectin based mud at 200°C.





From the Figure 3.4, it was observed that the rate of filtration for both PPM and CMM samples decreased speedily initially with increase in time and then slowly. The rate at which filtration occurs in CMM is higher than that of PPM which is an indication that filtration is better in PPM.

3.3 Results of sorptivity and diffusivity

The values of sorptivity (S) and diffusivity obtained from the plots are already given in Tables 3.3 and 3.4 respectively.

Table 3.3: Sorptivity (S) values of the mud samples at different temperatures

| Mud Samples | 25°C | 200°C |
|-------------|-------|-------|
| PPM | 18.1 | 21.25 |
| CMM | 17.01 | 17.09 |

Table 3.4: Diffusivity (D) values of the mud samples at different temperatures

| Mud Samples | 25°C | 200°C |
|-------------|-------|-------|
| PPM | 0.201 | 0.359 |
| CMM | 0.281 | 0.424 |
| CIVIIVI | 0.201 | 0.424 |

From Table 3.3, PPM has a higher sorptivity property at 25°C and 200°C which makes it a better fluid loss reducing agent than CMM, while from Table 3.4, PPM has a lower diffusivity at 25°C and 200°C which makes it better fluid for filtration than CMM.

4.0 Conclusion

Drilling mud prepared with calcium-water pre-gelatinized biodegradable pectin extract from Citrus Sinensis peels, has shown that the pectin biopolymer mud (PPM) has better filtration control behavior than the hydroxyl propyl starch modified drilling mud (CMM). The study also showed that highest sorptivity value of 21.25 was obtained with PPM at 200°C, while the highest diffusivity value of 0.424 was obtained with CMM at 200°C. This implies that the PPM has more fluid loss control capacity, and lower diffusivity at higher temperature than the widely applied mud prepared with hydroxy propyl starch. The low cost and accessibility of the Citrus Sinensis peels which is organic waste material may proffer reduced oil-well drilling cost, non-toxicity and environmentally adaptable solutions to the oil and gas industry.

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