# Potential Evaluation and Optimization of Natural Biopolymers in Water-Based Drilling Mud

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## Abstract

Drilling cost optimization has always been an important issue in the petroleum industry. In order to save costs and create new markets for local materials, Detarium microcarpum also known as ofor and food gum (Cissus populnea) powders were evaluated in this study at high temperature as an alternative to imported chemical additives in waterbased drilling fluid. The base mud composed of alkali beneficiated local clay, Brachystegia eurycoma also known as achi, corn and cocoanut fibers whose viscosity, yield point and gel strength fell short the recommended API standard from preliminary analysis. The two factors were combined using experimental design technique and mud properties optimized numerically using desirability function. At optimum conditions, The mud's properties are as following: Plastic viscosity, PV (18.4 ± 0.63 cp), Yield point, Yp (15.7 ± 0.9 lbf/100ft2), Fluid loss (FL) (12.1 ± 0.37 ml) and 10 min Gel strength (5.6 ± 0.05 lbf/100ft2). These values are in good agreement with the API recommended standard. Both biopolymers exhibited high potential at low and moderate temperatures. However, food gum is thermally stable, a good rheology stabilizer and filtrate reducer up to the test temperature of 185 °F. The presence and nature of salts in solution influences differently the viscosity of the two biopolymers.

## **Keywords**

API filtrate loss; Biopolymer; Experimental design; Gel strength; Optimization; Rheology.

## 1. Introduction

Filtration control is primarily the main reason for using polymers as supplement additives to bentonite in drilling muds. Notwith-

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standing, polymers are also added as viscosity booster or shale stabilizer. Because water-based mud (WBM) is the most common drilling fluid for both offshore and onshore, water-soluble polymers (WSPs) are preferred in drilling fluids. The WSPs are classified into two types; synthetic and natural polymers. Examples of synthetic polymers are hydrolyzed polyacrylate and vinyl acetate-maleic anhydride co-polymers. There are two main drawbacks in using synthetic polymers; (a) reaction with calcium and (b) severe viscosity and gelation of the mud [1]. Asides these, synthetic polymers are expensive and generally salt- and pH-sensitive as oppose to natural polymers. The importation of these chemicals apart from adding to OPEX may not allow indigenous companies in the oil and gas business to compete effectively with their foreign counterparts.

Natural polymers are able to absorb water and increase the viscosity of water (i.e., thickening water) due to their molecular size and shape. They rely on chain extension and physical entanglement of solvated chains for viscosity enhancement. They are non-charged and therefore less sensitive to salinity in contrast to the synthetic ones [2]. Long and complicated molecular chains of water-based polymers tie up the water and can build viscosity without solidified [3]. Examples of natural polymers include starch, carboxymethyl cellulose (CMC) and Hydroxyethyl cellulose (HEC). Some hemicelluloses have also been utilized for a long time due to their nontoxicity, solubility in water and ability to form gels. Typical examples are the mannans such as guar gum, locust bean gum, and konjac glucomannan, which are commercial emulsifiers and thickening agents [4]. Examples of gums include exudate (Arabic, Karaya), microbial fermentation (pullulan, xanthan gum, dextran and gellan gum) and seed gums (guar gum) [5].

Polysaccharides are water-soluble and have found applications in many oilfield operations including drilling, polymer-augmented water flooding, chemical flooding and profile modification. Different natural polymers such as starch, soy protein isolate, Guar gum, Xanthan gum, and cellulose derivative have been applied to improve the rheological and filtration performances of WBMs [6-10]. Starch properties in drilling mud depend on the source, granule size distribution and morphology, amylose/amylopectin ratio and other factors such as composition, pH, and nature of chemical modifications [11]. The stability and texture of starch-based products depend on gelatinization and reorganization behaviors [12]. According to Omotomiowa et al., [13], locally sourced cassava starch at 4% concentration was enough to improve rheology of WBM significantly. Gray and Darly, [14], studied the use of guar gum, carboxymethyl cellulose (CMC), and hydroxypropyl starch as filtration control agents and as viscosifiers. They concluded that filtration parameters like sorptivity and diffusivity of these polymers are dependent on temperature.

Scleroglucan, also known as schizophyllan was reported to exhibited high viscosity at low concentrations [15]. Compared with other biopolymers, Scleroglucan was reported to be more stable thermally, more tolerance to divalent and trivalent cations such as Ca<sup>2+</sup>, Mg<sup>2+</sup> and Fe<sup>3+</sup> and characterized by excellent carrying capacity. However, it is highly sensitive to chemically reactive additives and geological formations at high temperature [15].

Many indigenous polymers have become very popular for their use in drilling fluid due to their ability to modify rheological properties of clay suspension and their environmental friendliness [16]. The common challenges experienced are excessive fluid loss, low gel strength and the need to formulate a fluid with desirable rheological properties to withstand increasing temperature and pressure conditions [17]. An example of such a polymer is Welan gum (WLG). WLG is widely as a thickener in the food industry. Its molecule consists of repeating tetrasaccharide units with single branches of L-mannose or L-rhamnose [18]. WLG exhibits good viscosity in at elevated temperature and in the presence of sodium chloride [19]. However, the use of WLG gum in drilling mud formulations was greeted by excessive fluid loss in drilling mud [20].

Considering the economics, sustainability, and environmental effect, the use of natural polymers is highly recommended. Some efforts using wholly or partially substituted local materials have been reported in the design of muds for drilling [17, 21, 22]. The limitations that highlighted some of the findings could be summarized thus: (1) excessive fluid loss, (2) low gel strength and (3) thermal instability and (4) sensitivity to sodium, potassium and calcium salts. In this study, two lesser-known natural polymers (Cissus populnea and Detarium micocarpum) were examined experimentally on the basis of rheology, filtration control, thermal stability and sensitivity to Na<sup>+</sup> and Ca<sup>+2</sup> salts [23].

## 2. Experimental

## 2.1 Material

The polymers (Cissus populnea and Detarium micocarpum) were locally sourced from markets in Lagos, South-west Nigeria. Analar grade Na-HCO<sub>3</sub>, NaCl and CaCl<sub>2</sub> were purchased from local suppliers. The bentonite clay was sourced locally from Ohia ( $007^{\circ} 25'' \text{ N}/007^{\circ} 47'' \text{ E}$ ) in Abia state, Nigeria. The description of the polymers is given as follows:

## 2.1.1 Cissus populnea

C. populnea plant also called food gum plant can be found in western part of Africa. The Cissus gum extracted from the plant had been reported to be used as a soup thickener, treatment of venereal diseases, indigestion [24, 25] and drug binder [26]. The phytochemical constituents of the extracted liquor have been reported and used in ethnomedicine for treatment of male infertility [27]. As shown in Table 1, food gum plant contains more than 60 % cellulose and about 15% Hemicelluloses by weight.

	Table 1.	Proximate	composition	of C. po	pulnea fibers
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Composition	Percentage (w/w %)
Moisture	3.94 ± 0.23
Dry matter	96.06 ± 0.2
Ash	$1.59 \pm 0.14$
Wax	$2.94 \pm 0.31$
Water soluble	$2.33 \pm 0.27$
Pectins	$1.14 \pm 0.03$
Lignins	$11.52 \pm 0.27$
Hemicelluloses	$14.74 \pm 0.42$
Celluloses	$61.80 \pm 0.45$

### 2.1.2 Detarium micocarpum

D. micocarpum is a biopolymer confined to West and Central Africa. It is typically a species of dry savanna [28]. It is popular among the Ibo tribe of South-eastern Nigeria. D. microcarpum bears different local names among socio-cultural groups of different countries. For examples, socio-cultural groups like Yoruba, Igbo, Kanuri and Hausa in Nigeria named the plant as Ogbogbo, Ofo, Gatapo and Taure while Fulbe, Sonrai, and Soninke in Mali called it Doli, Tambacounba, and Fantu respectively [29]. It is the most investigated species of the genus because of its importance in African traditional medicine. The legume is very rich in polysaccharide gum. Table 2 shows the proximate composition of D. micocarpum fruit. The seed polysaccharide was described as a stabilizer and gelling agent in some processed fruit products [30].

#### 2.2 Clay processing and analysis

The microscopic structure of the raw clay was analyzed using X-ray diffraction (XRD). The diffraction of X-rays represented in Fig. 1 showed that the clav is predominantly composed of kaolinite, illite, and quartz. Kaolinite, Al<sub>2</sub>Si<sub>2</sub>O<sub>5</sub>(OH)<sub>4</sub>, is a layered aluminosilicate with a dioctahedral 1:1 layer structure consisting of tetrahedral silicate sheets and octahedral aluminum hydroxide sheets [32]. The chemical composition of the samples was determined after beneficiation using sodium bicarbonate (NaHCO<sub>3</sub>) with X-ray fluorescence spectroscopy (XRF) using a Philips X-Unique spectrometer. The description of the beneficiation is available elsewhere [33]. The physicochemical properties of the raw and the modified samples are shown in Tables 3. The major oxides are oxides of silica and alumina. Comparing the result with the result reported by Bailey [32], the silica content (68.9%) is comparable to that of Wyoming bentonite (68.0%). However, the  $Fe_2O_3$  and  $TiO_2$  (8.5 and 7.67%) contents were higher than the reported Wyoming bentonite (3.94 and 0.16%, respectively).

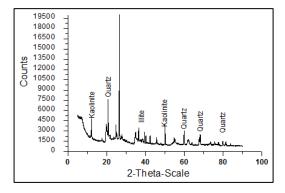
#### 2.3 Rheology and filtration

All rheological data were collected in triplicates using OFITE 8-speed viscometer. The viscometer measures the torque under 8 different rotational speeds, ranging from 600 to 3 rpm. The readings were translated into mud plastic viscosity, mud yield point, and mud apparent viscosity using Eq. 1 - 3. Mud filtrate was measured with OFITE Model 14030 filter press. Mud passes through a filter paper at 100 psi, and the filtrate volume after 10 second and 10 minutes of filtration were recorded. The pressure was provided by a cartridge containing pressurized carbon dioxide. Mud was prepared with high-speed mixer. The mud viscometer, filter press, and mud mixer are all industry-standard devices.

Composition	Mesocarp (%)	Seed (%)
Moisture	$15.0 \pm 0.01$	$5.0 \pm 0.01$
Crude fat	$10.5 \pm 0.01$	15.5 ± 0.02
Crude Ash	$3.3 \pm 0.01$	$3.5 \pm 0.02$
Crude fiber	$10.2 \pm 0.02$	$11.2 \pm 0.01$
Crude protein	$6.0 \pm 0.03$	$13.5 \pm 0.02$
Total Carbonhydrate	54.0 ± 0.01	50.5 ± 0.03

Table 2. Proximate composition of D. micocarpum [31]

\* Results are mean ± SD of duplicate determinations



**Figure 1.** Diffraction of X-rays: microscopic structure of the raw clay.

**Table 3.** XRF Result for the analysis of chemical composition of the raw samples of Nigeria Bentonite

		Oh	iia Clay
Oxides	Element	Raw	Treated
SiO <sub>2</sub>	Si	68.9	67.5
Al <sub>2</sub> O <sub>3</sub>	Al	11	10
Fe <sub>2</sub> O <sub>3</sub>	Fe	8.5	8.48
MnO	Mn	ND	ND
MgO	Mg	ND	ND
CaO	Са	ND	ND
Na <sub>2</sub> O	Na	0.08	3.12
K <sub>2</sub> O	К	1.4	ND
TiO <sub>2</sub>	Ti	7.67	7.87
P2O5	Р	ND	ND
LOI	LOI	2.15	3

The Plastic viscosity (PV) was calculated by the following formula:

$$PV = \theta_{600} - \theta_{300} (in \, cp) \tag{1}$$

The Apparent Viscosity (AV) was calculated by

$$AV = \frac{\theta_{600}}{2} (ln \, cp) \tag{2}$$

The yield point (Yp) was estimated by

$$Yp = \theta_{300} - PV \left( in \frac{lb}{100ft^2} \right)$$
(3)

 $\theta_{600}$  is the dial reading at 600 rpm and  $\theta_{300}$  is the reading at 300 rpm.

## 2.3 Influence of salts on rheology of biopolymers

A two-level factorial design of experiment was used to investigate the sensitivity of C. populnea and D. micocarpum to NaCl and CaCl<sub>2</sub> in the presence of other additives. A base mud of fixed volume was prepared by mixing 24 g of beneficiated Ohia clay into 350 ml of fresh water. Cocoanut fiber, Corn starch, C. populnea and D. micocarpum were added in a 16 runs experiment as shown in Table 4. The C. populnea and D. micocarpum were evaluated at 185 °F for their sensitivity to NaCl and CaCl<sub>2</sub> in WBM formulation. The effects on the plastic viscosity (PV) and yield point (YP) were examined using additives in the following ranges: Cocoanut fiber (1 - 2.5 g), Corn starch (2 - 5 g), Salts (9 - 11g), C. populnea (2 - 5 g) and D. micocarpum (2 – 5 g).

# 2.4 Application of selected bio-polymers in WBM

#### 2.4.1 Mud composition

The composition of the base mud used for evaluation is available elsewhere (Salawudeen et al., 2016). It was noticed that the analysis from these authors was performed using fresh water and the fluid loss (12 ml/30 Mins/100 psi), plastic viscosity (5 cp), yield point (2 lbf/100ft<sup>2</sup>), 10 Sec/10 mins gel strength (1/1 gel), pH (7.35), mud weight (8.6 lbm/gal) and cake thickness (0.2 inch) were recorded. It is clear that these properties, except fluid loss, were below the recommended range (8-35 cp plastic viscosity,  $5 \le Yp \le$ 3PV, 2-5 lb/100ft<sup>2</sup> 10 sec. gel, 2-35 lb/100ft<sup>2</sup> 10 min. gel and 9.5-11.5 pH) according to the API [34]. D. micocarpum and C. populnea were applied to improve on these properties using an experimental design.

#### 2.4.2 Experimental Matrix

The 13 experimental runs for two (2) factors using the central composite sampling design (CCD) are shown in Table 5 and 6 for 85 and 185 °F, respectively. The choice of CCD is the need for reliable objective functions for the optimization study. To the base mud, D. micocarpum and C. populnea were added according to experimental design and different mud systems were prepared using API standard equipment. The rheological parameters were determined at 85 and 185 °F for each run. The physicochemical properties (pH and mud weight) were carried out according to the API chemical tests. API filtrate and gel (3 rpm dial reading after 10 seconds and 10 min of mixing) were determined using API recommendations.

							NaCl Salt			CaCl <sub>2</sub> Salt	
Run	CNF (g)	C. pop (g)	D. mico (g)	CasS (g)	Salt (g)	PV(cp)	Yp (lbf/100fft2)	AV (cp)	PV (cp)	Yp (lbf/100fft2)	AV (cp)
B. Mud	1	2	5	5	11	3	1	.5	7	12	13
B. Mud	1	2	5	2	9	4	0	4	4	14	11
B. Mud	1	2	2	5	9	6	0	6	5	2	6
B. Mud	1	2	2	2	11	4	0	4	6	0	6
B. Mud	2.5	2	2	2	9	3	1	3.5	7	1	7.5
B. Mud	2.5	2	5	5	9	4	2	5	6	2	7
B. Mud	2.5	5	2	2	11	15	13	21.5	9	3	10.5
B. Mud	2.5	5	5	2	9	11	5	13.5	10	1	9.5
B. Mud	1	5	2	5	11	11	4	13	23	23	34.5
B. Mud	1	5	5	5	9	25	26	38	15	9	19.5
B. Mud	2.5	5	2	5	9	20	18	29	26	26	39
B. Mud	2.5	2	5	2	11	4	4	6	6	2	7
B. Mud	2.5	2	2	5	11	5	0	5	3	11	8.5
B. Mud	2.5	5	5	5	11	21	24	33	21	21	31.5
B. Mud	1	5	5	2	11	13	7	16.5	16	10	21
B. Mud	1	5	2	2	9	15	12	21	12	5	14.5

Table 4. Factorial design matrix for biopolymer sensitivity to salinity at 185 °F

\* B. Mud = Base mud, CNF=Cocoanut fiber, CasS= Cassava starch

Table 5. Rheological, filtration and physicochemical properties of drilling fluids at 85  $^\circ\mathrm{F}$ 

Run A: C.p	op, g	B: D. mic, g	PV, cp	Yp, (lb/100 ft²)	Fluid loss, ml	10 Sec Gel (lb/ 100ft <sub>2</sub> )	10 mins Gel (lb/ 100ft²)	Cake thickness (mm)	MW (lbm/gal)
Base Mud	1	1	14	7	8.8	2	4	0.5	9.1
Base Mud	0	1	14	23	8	3	5	0.4	8.9
Base Mud	0	0	14	2	12.4	2	3	0.7	8.7
Base Mud	0	0	14	2	12.4	2	3	0.7	8.6
Base Mud	0	-1	10	1	15	1	2	0.6	8.7
Base Mud	-1	0	4	24	8	2	3	0.5	8.8
Base Mud	-1	-1	6	3	12	1	2	0.5	8.6
Base Mud	1	0	11	7	9.4	2	3	0.6	8.7
Base Mud	-1	1	19	26	8	3	6	0.7	8.7
Base Mud	0	0	14	2	12.4	2	3	0.7	8.6
Base Mud	1	-1	5	10	13	2	2	0.7	8.6
Base Mud	0	0	14	2	12.4	2	3	0.7	8.6
Base Mud	0	0	14	2	12.4	2	3	0.7	8.6

\*High level (1), Medium level (0), Low level (-1)

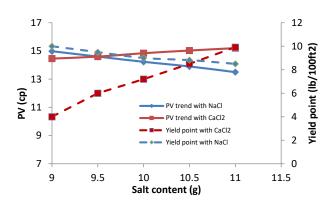
\*Base mud composition: 24 g Clay+350 ml water+1.25 g Corn fiber+0.25 g Cocoanut fiber+2 g B. eurycoma

Run	A: C. pop, g	B: D. mic, g	PV, cp	Yp, (lb/100 ft²)	Fluid loss, ml	10 Sec Gel (lb/100 ft <sup>2</sup> )	10 mins Gel (lb/100 ft²)	Cake thick- ness (mm)	MW (lbm/ gal)
Base Mud	1	1	21	25	12	2	5	1.5	9.1
Base Mud	0	1	24	17	12.6	3	4	1	8.9
Base Mud	0	0	7	2	12	1	2	1.4	8.7
Base Mud	0	0	7	2	12	1	2	1.4	8.6
Base Mud	0	-1	4	3	10.4	2	2	1.2	8.7
Base Mud	-1	0	10	6	8.2	2	3	1.1	8.8
Base Mud	-1	-1	6	1	9	1	2	1	8.6
Base Mud	1	0	22	1	10.6	3	4	0.8	8.7
Base Mud	-1	1	19	16	12.2	4	6	1	8.7
Base Mud	0	0	7	2	12	1	3	1.3	8.6
Base Mud	1	-1	8	3	13.6	3	4	1.4	8.6
Base Mud	0	0	7	2	12	1	2	1.5	8.6
Base Mud	0	0	7	2	12	1	2	1.4	8.6

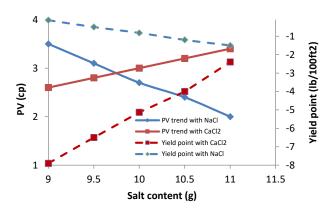
Table 6. Rheological, filtration and physicochemical properties of drilling fluids at 185 °F

\*High level (1), Medium level (0), Low level (-1)

\*Base mud composition: 24 g Clay+350 ml water+1.25 g Corn fiber+0.25 g Cocoanut fiber+2 g B. eurycoma



**Figure 2.** The trend of PV and Yield point of D. maccocarpum mud in the presence of Sodium and Calcium Chlorides.



**Figure 3.** The trend of PV and Yield point of C.populnea mud in the presence of Sodium and Calcium Chlorides.

## 3. Results and Discussions

### 3.1 Influence of salts on polymer performance

The presence of salts in solution influences the viscosity of the two polymers. Fig. 2 and 3 shows the viscosity and yield point trends when Sodium and Calcium chlorides were added to the mud containing 5 g D. maccocarpum and C. populnea respectively. It was observed that sodium ion has a degrading effect on biopolymer rheology. For example, when the amount of sodium chloride increased from 9 to 11 g, the viscosity and yield point of the mud dropped gently from 15 to 13.5 cP and 10 to 8.5 lb/100ft<sup>2</sup>, respectively. This effect may be attributed to the reduction in molecular dimensions resulting from diminished intermolecular electrostatic forces [35]. Also in the presence of salts, polymer will shrink and coil. The shrinkage of polymer chains can lead to a reduction in viscosity [36]. However, in the presence of a divalent salt (CaCl<sub>2</sub>), viscosity and yield point of both polymers increases (see Fig. 2 and 3). This effect is probably due to increased interaction between the polymer molecules [35, 37]. The viscosity increase observed with Ca<sup>2+</sup> is likely caused by hydrogen bonds that are able to form because of the screening of electrostatic repulsions between charged groups [38]. The salt molecules screen repulsive interactions between polymer chains thus bringing them closer to one another. The closer contact allows the formation of hydrogen bonds between polymer chains. The bonds increased the difficulty of the chains to

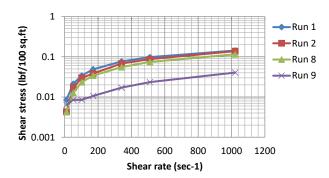
move past one another in solution, which is evidenced as an increase in viscosity. served with Runs number 6 and 7 (Bingham Plastic,  $R^2$ = 0.9791 and 0.9921).

### 3.2 Rheology

As shown in Table 5 and 6, the rheological parameters of different muds vary as a function of the added amount of D. micocarpum and C. populnea. It is apparent that high value of the main rheological parameters (yield point Yp and plastic viscosity Vp) of mud was recorded in experimental runs 1, 2, 8 and 9. Indeed, The Yp reached the maximum value (25 lb/100ft<sup>2</sup>) at first run when maximum amount of D. micocarpum and C. populnea were used. However, the lowest Yp (1  $lb/100ft^2$ ) was obtained when the amount of D. micocarpum was reduced by 50% (see Run 8). This indicates that the amount of D. micocarpum in the formulation must be above the mean value to reach a high yield of the mud. Similarly, highest plastic viscosity (24 cp) was observed with the mean value of C. populnea and high value of D. micocarpum. But, with minimum amount of C. populnea reduction was about 44%. Perhaps, this can be explained by the fact that C. populnea acted as a thermal stabilizer of PV at such high temperature of 185 °F.

#### 3.3 Rheological behavior

Fig. 4 shows the rheological behavior of the selected Runs (1, 2, 8 and 9) suspensions. The correlation coefficient (R<sup>2</sup>) was obtained for each curve-fitting and the highest coefficient determines the fluid model that better describes the particular fluid. According to rheograms (Fig. 3), all suspensions show non-Newtonian flow. Runs 1, 2 and 8 are best described by power-law with  $R^2 = 0.9985$ , 0.9954 and 0.9986, respectively. However, was described by Bingham plastic model with  $R^2 = 0.9971$ . This behavior could be attributed to the value of the gel strength with Run 9 having the highest (6 lb/100 ft<sup>2</sup>) and closest to the yield point then the Bingham Plastic model was adequate for the fluid rheology. Runs 1, 2 and 8 are characterized by relatively lower gel strength compared with their corresponding yield points; hence their behavior was similar to power-law fulids. It is very clear that at low guantites of C. populnea, the amount of C. populnea was responsible for the Bingham Plastic behaviour of Run 9. Similar behavior was ob-



**Figure 4.** Consistency Plot of Base fluid + Run 1, 2, 8 and 9 D. micocarpum and C. populnea composition with linear and power correlations.

#### 3.4 Interaction effects of the polymers

To study the factor effects, main or interaction (D. micocarpum and C. populnea) in the proposed mud formulation, a response surface methodology involving analysis of variance (ANOVA) was performed at 85 and 185 °F and model equations that best described the experiment were developed for optimization study.

#### 3.4.1 Diagnostic checking of the fitted models

All main effects, linear, quadratic and interaction were calculated for each model. The summary statistics obtained from the analysis of variance at 95 percent confidence interval ( $\alpha = 0.05$ ) are shown in Table 7, as well as the correlation coefficient obtained for PV, Yp, AFL and Gel model. The correlation coefficients for the responses (R<sup>2</sup> = 0.941, 0.966, 0.988 and 0.982, respectively) were quite high for response surfaces and indicated that the fitted quadratic models accounted for more than 90% of the variance in the experimental data, which were found to be highly significant. Based on F-statistics, the only regression coefficients not significant at 95% is C. populnea for PV and Yp however highly significant for API filtrate and Gel.

#### 3.4.2 Influence of temperature on plastic viscosity

Fig. 5 shows the evolution of the plastic viscosity of muds as a function of the D. micocarpum for a fixed value of C. populnea. It is noteworthy that the plastic viscosity of mud increases with increase in the amount of D. micocarpum in the mud for fixed values of C. populnea. With C. populnea fixed at a minimum value of 1.5 g (black ts lowest value (see Fig. 6). Thus, compared to the C. populnea, D. micocarpum is not stable at high temperature to maintain mud viscosity. colored curve in Fig. 5), the PV slightly increases with D. micocarpum. However, with C. populnea content increased to its maximum value of 4 g, a remarkable increase in PV was observed (red colored curve in Fig. 5). The dominant of C. populnea as a mud viscofier at high temperature (185 °F) became very clear. At low temperature (85 °F), D. micocarpum dominated and stabilizes the viscosity even with C. populnea fixed at i

	PV (cp)		Yp (lb/10	Yp (lb/100ft <sup>2</sup> )		API Filtrate (ml)		el
Parameter	F-value	P>F	F-value	P>F	F- value	P>F	F- value	P>F
Model	36.14	0.0002	64.7	< 0.0001	1819.73	< 0.0001	123.60	< 0.0001
С.рор	2.36	0.1754 <sup>a</sup>	0.006	0.9408ª	2890.00	< 0.0001	19.20	0.0047
D.mic	105.28	< 0.0001	207.09	< 0.0001	240.00	< 0.0001	235.20	< 0.0001
C.pop <sup>2</sup>	42.54	0.0006	18.37	0.0052	1163.64	< 0.0001	240.00	< 0.0001
D.mic <sup>2</sup>	11.81	0.0139	78.40	0.0001	2645.00	< 0.0001	58.80	0.0003
C.pop*D.mic	18.73	0.0049	19.69	0.0044	2160.00	< 0.0001	64.80	0.0002
Multiple. R <sup>2</sup>	0.941		0.966		0.998		0.982	

\*a = not significant at  $\alpha$  = 0.005 (95 confidence level)

\*P>F values less than 0.05 indicate significant model and model terms

The plastic viscosity (PV), Yield point (Yp), API filtrate loss (AFL) and 10 mins Gel (Gel) at the

test condition are best approximated by equations 4 – 7.

$PV = 56.03075 - 29.56347 * CP - 15.84901 * DM + 4.47123 * C.Pop^{2} + 2.21613 * D.mic^{2} + 2.84201 * C.pop * D.mic $ (4)	PV = 56.03075 -	29.56347 * CP ·	– 15.84901 * <i>l</i>	DM + 4.47123 *	$C.Pop^2 + 2.21613 *$	$D.mic^2 + 2.84201$	* C.pop * D.mic	(4)
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 $Yp = 77.9065 - 19.74612 * CP - 31.75769 * DM + 1.81918 * C.Pop^{2} + 4.15221 * D.mic^{2} + 2.54977 * C.pop * D.mic$ (5)

 $FL = 2.35156 + 12.29867 * CP - 5.12889 * DM - 1.664 * C. Pop^{2} + 1.02222 * D. mic^{2} - 0.64 * C. pop * D. mic$ (6)

 $Gel = 6.155 - 3.1733 * CP - 0.8444 * DM + 0.88 * C. Pop^{2} + 0.3889 * D.mic^{2} - 0.4 * C. pop * D.mic$ (7)

These equations are applicable within the following range of parameters: D. micocarpum (2 - 5 g) and C. populnea (1.5 - 4 g).

## 3.4.3 Effects of polymer on yield point at 185 °F

Fig. 7 shows the evolution of the yield point with increase in the concentration of C. populnea for fixed values of D. micocarpum. The maximum attainable Yp when D. micocarpum was fixed at minimum (2 g) is approximately 12.4 cp. This value decreases as the concentration of C. populnea increases from 1.5 to 4.0 g. Thus to main-

tain the yield, a relatively small amount of C. populnea is needed. However, an increase in the D. micocarpum component in the mud to a fixed value of 5 g, a dramatic raise in yield point (18  $lbf/100 ft^2$ ) was observed. This value increased as the concentration of C. populnea increased from 1.5 g (12  $lbf/100 ft^2$ ) to 4 g (25 cp).

## 3.4.4 Effects of polymer on Gel strength at 185 °F

Fig. 8 shows a 3-Dimensional plot of the effect of the C. populnea and D. micocarpum on the 10 min Gel of WBM at high temperature. The gel strength of drilling fluid determines the ability of the mud to suspend the cuttings and transporting them to the surface. It is evident that with the increase of the concentration of D. micocarpum (about 5.0 g) in the mud, the gel 10 was at the maximum value (5.8 lb/100ft<sup>2</sup>) when the amount of C. populnea was at its minimum value (1.5 g). For a higher gel, the concentration of D. micocarpum may be increased while reducing the concentration of C. populnea in the mud.

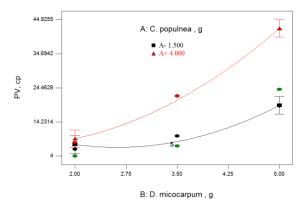


Figure 5. Effect of the D. micocarpum for a fixed value of C. populnea on the Plastic viscosity of WBM at  $185 \text{ }^\circ\text{F}$ 

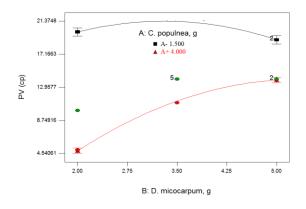


Figure 6. Effect of the D. micocarpum for fixed value of C. populnea on the Plastic viscosity of WBM at 85  $^\circ F$ 

#### 3.4.5 The mud API fluid loss

The mud filtrate is the liquid portion of the mud system that is driven through a filter cake into the formation by the difference between the hydrostatic pressure of the mud column and the formation pressure. Fig. 9 shows the relation between mud filtrate and polymer concentration. It was noted that the filtrate increased from 8.9 to 12.2 ml with C. populnea fixed at its lowest value (1.5 g) while D. micocarpum increased from 2 to 5 g. The increment in fluid loss increases as the concentration of C. populnea increases by 50 %. Further increase in the amount of C. populnea above 3 g resulted in a decrease in fluid loss irrespective of the amount of D. micocarpum in the mud. However, a marginal reduction from 13.6 to 12 ml was observed with the amount of C. populnea fixed at 4 g while the amount of D. micocarpum varied between 2 and 5 g respectively.

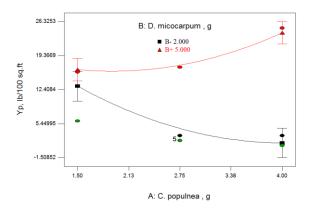


Figure 7. Effect of the C. populnea for a fixed value of D. micocarpum on the Yield point of WBM at 185  $^\circ F$ 

## 4. Optimization Study

### 4.1 Conditions for optimum responses

The models (PV, Yp, AFL, and Gel) were used to indicate the direction of changing variables for a specific purpose. For example, maximizing the Gel strength and minimizing the API filtrate. The multiple regression equations were solved numerically for the maximum Gel strength (6.0 lb/100ft<sup>2</sup>) and minimum API filtrate loss (8.2 ml) using desirability (D) objective function (Eq. 8). The overall desirability (D) is the geometric (multiplicative) mean of all individual desirabilities (di) that range from 0 (least) to 1 (most).

$$D = \left(\prod_{i=1}^{n} d_i\right)^{\frac{1}{n}} \tag{8}$$

Where n is the number of responses. The input variables (C. populnea and D. micocarpum) as shown in Fig. 10 were adjusted within a desired range that keeps the solution within the experimental boundaries. The PV and Yp were also set to be in range but with their lowest limit set to 10, guided by the API standard [34].

The optimum polymer concentrations predicted for all the response are 1.5 g of C. populnea and 4.79 g of D. micocarpum in the mud. These values are within the experimental range, indicating the validity of the selection of the variables range. The optimum conditions were experimentally tested, a PV of 18.4 ( $\pm$  0.63) cp, Yp of 15.7 ( $\pm$  0.9) lbf/100ft<sup>2</sup>, AFL of 12.1 ( $\pm$  0.37) ml and 10 min Gel value of 5.6 ( $\pm$  0.05) lb/100 ft<sup>2</sup> were obtained. These experimental values at the optimum compositions were in good agreement with the predicted values.

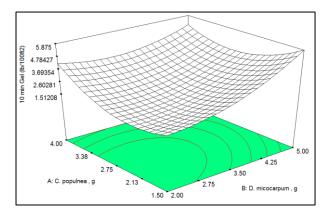


Figure 8. Effect of the C. populnea for a fixed value of D. micocarpum on the 10 min. Gel of WBM at 185  $^\circ F$ 

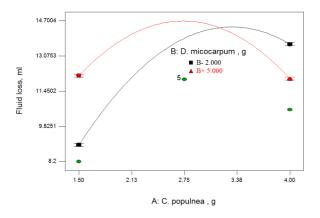


Figure 9. Effect of the C. populnea for a fixed value of D. micocarpum on the API filtrate of WBM at 185  $^\circ F$ 

## 5. Conclusions

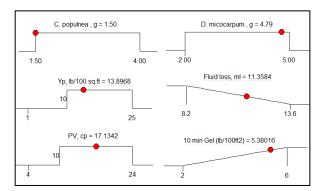
The major objective of this study was to evaluate and optimize two selected biopolymers on the viscosity of drilling muds without compromising physicochemical properties, Gel strength and API filtrate losses at high temperature. From the obtained results, it can be concluded that:

1. The presence and nature of salts in solution influences differently on the viscosity of the two evaluated biopolymer.

2. The physicochemical and rheological properties of base mud significantly improved in the presence of C. populnea and D. micocarpum biopolymers.

3. At high temperature, C. populnea was found to be thermally stable and a good rheology stabilizer and filtrate reducer at 185 °F.

4. Approximately, 85 % increase in Gel strength was achieved using C. populnea and D. mico-carpum biopolymers.



**Figure 10.** Ramps Report on numerical optimization of C. populnea and D. micocarpum for optimum PV, Yp, AFL and Gel strength

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