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## **Effect of Sodium Carboxymethyl Cellulose from *Delonix regia* Sawdust on Rheological and Filtration Properties of Water Based Drilling Fluid**

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### **Abstract**

Over the past years, there has been an increase in the importation of Sodium Carboxymethyl cellulose (CMC), an important drilling mud polymer additive, in the Nigerian oil and gas industry. However, the ripple effects of the importation of this polymer and other oilfield chemicals on the Nigeria oil and gas industry includes rising cost of oil and gas field development, limited oil and gas industry growth, and capital flight. In order to mitigate this trend, studies on the use of local substitutes such as starch and its derivatives have gathered momentum with risk such as competition with food supply and increase in food cost. The use of sawdust wastes which offers a non-competing and a cheap source of feedstock in the production of CMC have rarely been investigated.

The study therefore investigated production of CMC from sawdust waste of a highly underutilized wood (*Delonix regia*), after which drilling mud tests were conducted to determine the rheological and filtration properties of mud treated with the CMC products. The CMC production adopted the Williamson ether synthesis process in a slurry medium involving two main reactions of mercerization and etherification. All reaction parameters were held constant except the etherifying agent concentration. The CMC products were characterized using FTIR Spectroscopy.

The synthesized carboxymethyl cellulose products yielded good filtration and rheological properties suitable for drilling fluid applications. The use of low concentrations of about 0.5g to 1.0g of the synthesized products per laboratory barrel of mud could reduce filtration volume by 11.4% to 32.9% at low temperature and pressure conditions. The synthesized CMC products obtained from this work can be used as local substitute of low viscosity foreign CMC products.

### **Introduction**

Sodium carboxymethyl cellulose is a vital polyanionic cellulose ether used alone or in combination with bentonite clay in the improvement of the filtration and rheological characteristic of a water based drilling fluid. It is the most widely used cellulosic in the drilling sector. Carboxymethyl Cellulose (CMC as it is usually called) was first prepared in Germany in 1918 by German E. Jansen and was produced commercially in the early 1920's at IG Farbenindustrie AG, Germany now known as BASF. Since 1947 CMC has been

used as a viscosifier and fluid loss reducer in water based fluids which indicates its reliability in drilling mud (Young & Maas, 2001).

A survey of literature has shown that several wastes from wood and non-wood sources have been used to synthesize CMC by different methods in order to fully exploit the possibility of converting these wastes to useful chemical products. Wastes from wood sources typically involves the use of sawdust while wastes from non-wood sources include agave, bagasse, banana stem, cotton fibers and linters, lantana camara, orange peel, paper sludge, rice straw, sago waste, beat pulp, water hyacinth, palm oil empty fruit bunch and so on (Singh & Singh, 2012). The industrial production of CMC is majorly obtained from wood pulp and cotton as the cellulose content of cotton is 90% and that of wood is 40-50%. The improvement of CMC product quality have made it possible to use in an increasing number of applications such as food and beverages, pulp and paper, textile production, tooth paste, detergent/laundry, pharmaceuticals, paint industries, cosmetics and personal care, and oil well drilling fluids. In oil well drilling applications, CMC is used to control Mud rheological and filtration properties.

In Nigeria, wood wastes such as sawdust generated in various sectors of the wood industries is enormous. It is estimated that Nigeria generates 5.2 million tons of wood wastes and 1.8 million tons of sawdust annually (Owoyemi et al, 2016). Although over the years, more advanced and improved methods of sawdust waste utilization have evolved a larger chunk of sawdust waste are still improperly disposed leading to environmental hazards. The recycling and reuse of wood wastes help to curb these environmental hazards and convert waste to wealth. The conversion of waste sawdust to useful chemical products such as sodium carboxymethyl cellulose offers a way to recycle and reuse sawdust from the waste industry. *Delonix regia* sawdust as a wood waste is often under-utilized in Nigeria and no known research has been conducted on the conversion of its huge sawdust waste to sodium carboxymethyl cellulose. The essence of this research is to use sawdust from *Delonix regia* wood specie to synthesize various Sodium Carboxymethyl cellulose products, characterize it by means of a Fourier Transform Infrared Spectrophotometer and investigate its impact on drilling mud rheological and filtration properties in order to determine its suitability as substitute for foreign CMC in drilling mud formulation.

## Materials and methods

### Materials

**Raw Materials and Reagents for Cellulose Extraction and CMC synthesis.** *Delonix regia* sawdust used as feedstock for cellulose extraction and CMC synthesis, Reagent grade Sodium hydroxide pellets-used for soda pulping and cellulose mercerization, 3.5% w/v Sodium hypochlorite Solution for bleaching of pulped *Delonix regia* sawdust, 99.7% Iso-propanol-used in a diluted state as a solvent for heat exchange and mass transfer in CMC synthesis, Glacial acetic acid-used in a diluted state for the neutralization of the reaction mixture after CMC synthesis, Absolute ethanol and methanol-is used in a pure form or dilute state to wash and purify the CMC after production, Hydrochloric acid, nitric acid, sodium hydroxide, ethanol, methanol, distilled water and phenolphthalein indicator-used for determination of degree of substitution.

**Laboratory Equipment and Devices for Cellulose Extraction and CMC synthesis.** The equipments and devices used in this study for cellulose extraction and CMC synthesis includes; Grinding machine-for the comminution of *Delonix regia* sawdust, laboratory milling machine-to pulverize extracted cellulose, Sieves with aperture sizes of 500  $\mu\text{m}$  and 75 $\mu\text{m}$  for the screening of grinded *Delonix regia* sawdust and cellulose fibers respectively, a thermostatic hotplate with magnetic stirrer, Digital PH meter-to adjust the PH of CMC reaction mixture after synthesis, A vacuum filtration apparatus for quick filtration, a 100°C mercury-in-glass thermometer-for temperature determination and confirmation, Oven-to dry, Glassware such as Measuring cylinders, flat bottom flask, Erlenmeyer flask, glass rod, funnel, beakers and volumetric flask.

**Materials, Devices and Equipments for Drilling fluid Test.** The materials used in drilling tests include Bentonite clay-the primary additive used in drilling mud formulation and synthesized *Delonix regia* CMC-whose performance as a secondary additive in drilling mud rheology and fluid loss control is evaluated in this project. The devices and equipment used for drilling fluid test includes; Mud Balance-used in the measurement of mud density, An OFITE 8-speed viscometer-used in mud rheology measurement, A multi-cell filter press with a 100 psi air compressor-used in filtration test, Whatman50 filter paper, A multi-mixer for high shear mixing of water, clay and additives. Other materials used are 10ml measuring cylinders, mixer cups, aing containers, tissue paper and distilled water.

## Methods

**Sample Collection and Preparation.** The sawdust used in this work was collected from *Delonix regia* wood felled adjacent the University Clinic by the University of Ibadan Biodiversity Management Committee. The sample was carefully collected to avoid the inclusion of abrasive sand materials that will reduce the quality of the work. The collected sawdust sample is sun-dried under ambient conditions to remove moisture and facilitate easy comminution. The sun-dried sawdust is grounded to fine particles in order to increase its surface area and ensure the easy breakdown of the lignin-hemicelluloses complexes during extraction. The grounded sawdust is passed through a 30 mesh sieve and stored for the next stage.

**Cellulose Extraction.** The extraction of cellulose was done through pulping and bleaching of sawdust obtained from the grinding and sieving stages above. 40g of the sample was cooked in an aqueous solution of 8 % NaOH at sample to solvent ratio of 1:20 (w/v) for 3.5 h at 95°C. The obtained dark brown slurry was filtered by vacuum filtration and solid material was washed several times using copious amount of distilled water. The residue obtained from the pulping step above is subjected to bleaching in two stages. In the first bleaching stage, the residue was treated with 400ml of 3.5%w/v sodium hypochlorite solution at 80°C for 30 minutes under magnetic stirring and the product was then filtered and washed with water to remove the degraded lignin-hemicelluloses complexes. To obtain a brighter pulp, the product from the first bleaching stage was suspended in 300ml of 2.3% w/v sodium hypochlorite solution and stirred for 15mins under ambient conditions. The product was filtered and washed with water until the odour of hypochlorite could no longer be detected. The product obtained from this step is known as *Delonix regia* cellulose. The cellulose obtained is dried in an oven at 60°C to a constant weight. The extraction was repeated three times and the average yield was determined after which the cellulose was grounded and screened using a sieve with an aperture size of 75µm. The grounded and sieved cellulose was stored for the next stage.

**Synthesis of sodium carboxymethyl cellulose.** The production of CMC was carried out by heterogeneous synthesis in a slurry process involving two main reactions of mercerization and etherification. A one step carboxymethylation process was adopted in the etherification stage. 8g of cellulose powder is stirred vigorously in 90% isopropanol in a solid-liquor ratio of 1:28 over a 30 minutes period. In the mercerization step, 15%w/v NaOH at 2.45mol/AGU is added to the mixture and stirred for another 60 minutes at a temperature of 25°C. After the mercerization process is over, various amount of Monochloroacetic (MCA) 3.5, 5.0, 6.5, 8.0, 9.5 g per 8g cellulose was added and the temperature of the mixture was raised to 60°C and stirred using a magnetic stirrer. The reaction continued for 3 hrs. After the completion of the etherification reaction, the reaction mixture was neutralized with 90% acetic acid and then filtrated. The solid obtained as CMC was washed with 70% ethanol and absolute methanol to remove undesirable byproducts, filtrated and dried at 100°C in an oven.

**Characterization of Synthesized CMC products by FTIR spectroscopy.** The Fourier Transforms Infra Red (FT-IR) spectra of *delonix regia* sawdust, extracted cellulose and carboxymethyl cellulose products were obtained using Infra Red (IR) spectrometer. All measurements were carried out using the KBr method. The samples were dried in the oven at 60°C. About 0.2 mg of sample and 2 mg of KBr were mixed and ground

finely and the mixture was compressed to a form a transparent disk. Transmission was measured at the wave number range of 4000-400  $\text{cm}^{-1}$  for each product and the infrared spectra were recorded.

**Water-Based Mud Sample Preparation and Drilling Fluids Tests.** The experimental procedures used in this study in the preparation and testing of drilling muds conformed to the API 13B-1 standard. In each mud samples prepared, 22.5g of bentonite clay is added to 350ml of distilled water and stirred vigorously using a multi-mixer till all the lumps became invisible. Then, 0.5g, 1.0g and 1.5g of 4 varieties of delonix CMCs produced based on the variation of the MCA concentration was added to the bentonite mud separately into different cups and were stirred vigorously with the aid of a multi mixer until lumps CMC disappeared. In all, 16 samples of mud were required for this investigation which included 12 samples with delonix Na-CMC powder, one control mud sample without delonix Na-CMC powder and three control mud samples consisting of standard low viscosity foreign CMC used in the industry. After preparing a mud sample, it was poured into a container, properly covered and allowed to age for 24hrs at room temperature after which drilling mud tests were conducted. The table below shows the delonix CMC products used in WBM preparation, the quantity used in each mud sample and the mud samples used in the mud tests. Mud samples of controls used in mud tests are not included on the table.

**Table 2.1—Mud Samples and Quantity of Delonix CMC Products used in preparation.**

MCA QUANTITY USED IN CMC PRODUCT (g)	DELONIX CMC PRODUCT	CMC QUANTITY USED IN MUD SAMPLE (g)	MUD SAMPLE
3.5	CMC-35	0.5	A <sub>1</sub>
		1.0	A <sub>2</sub>
		1.5	A <sub>3</sub>
5.0	CMC-50	0.5	B <sub>1</sub>
		1.0	B <sub>2</sub>
		1.5	B <sub>3</sub>
6.5	CMC-65	0.5	C <sub>1</sub>
		1.0	C <sub>2</sub>
		1.5	C <sub>3</sub>
8.0	CMC-80	0.5	D <sub>1</sub>
		1.0	D <sub>2</sub>
		1.5	D <sub>3</sub>
9.5	CMC-95	-	-
		-	-
		-	-

### Mud Rheology Test

The drilling mud rheology test was conducted to investigate the effect of adding delonix Na-CMC on the rheological properties of the drilling fluid formulation at varying temperature conditions. The foreign CMC product was not included in this test. The rheological properties determined were, plastic viscosity, yield point, and 10s gel strength and were recorded with a Fann Viscometer Model 3500 at dial readings of 600, 300, 200, 100, 60, 30, 3 rpm as indicated in API 13B-1 for measuring mud properties. The following equations were utilized;

$$\text{Apparent Viscosity } (\mu_a) = \frac{\phi_{600}}{2}$$

$$\text{Plastic Viscosity } (\mu_p) = \phi_{600} - \phi_{300}$$

$$\text{Yield Point } (\mu_p) = \phi_{600} - \mu_p$$

### Mud Filtration Test

The filtration test was conducted at room temperature using a standard multi-cell filter press with a 100 psi air compressor. This test was conducted in order to evaluate the effect of adding the various synthesized delonix Na-CMC products on the filtration performance of the drilling fluid and compare the result with that of a low viscosity foreign CMC product. Therefore, filtration tests were conducted for the foreign CMC product and the synthesized product.

## Results and Discussion

### Cellulose Extraction and Etherification Reaction Yield

The product yield from cellulose extraction of delonix regia sawdust and the synthesis process of sodium carboxymethyl cellulose was measured based on oven dry weight.

#### Yield of Cellulose

The net oven dry weights of cellulose obtained from three extractions done are 16.8g, 16.71g and 16.9 g per 40g of sawdust used. The average percentage yield of cellulose (Cellulose %) extracted from the sawdust samples was calculated from equation 4.1 as given below;

$$\begin{aligned} \text{\% Yield of cellulose} &= \left( \frac{\text{Weight of Obtained cellulose (g)}}{\text{Weight of delonix sawdust sample used (g)}} \right) \times 100\% \\ \text{\% Yield of cellulose} &= \frac{50.41}{120} \times 100\% \\ \text{\% Yield of cellulose} &= 42\% \end{aligned} \quad (3.1)$$

#### Yield of delonix Na-CMC and effect of Reaction Parameters;

The percentage yield of delonix carboxymethyl cellulose products from the synthesis was calculated from Equation 3.2 based on the net dry weight of the reaction products obtained as presented in table 3.1.

$$\text{\% Yield of delonix Na-CMC} = \left( \frac{\text{Weight of dried Na-CMC (g)}}{\text{Weight of delonix sawdust sample used (g)}} \right) \times 100\% \quad (3.2)$$

**Table 3.1—Reaction Parameters and MCA concentration effect on Delonix Na-CMC yield**

Concentration of NaOH	Weight of Cellulose (grams)	Weight of MCA (grams)	Delonix CMC Product	Weight of dry delonix CMC (grams)	Percentage Yield (%)	Initial PH
15	8.0	3.5	CMC-35	9.25	115.6	13.52
15	8.0	5.0	CMC-50	9.04	113.0	13.23
15	8.0	6.5	CMC-65	9.48	118.5	12.46
15	8.0	8.0	CMC-80	8.85	110.6	11.85
15	8.0	9.5	CMC-95	7.60	95.0	9.98

The effect of varying the monochloroacetic acid concentration on the yield of the CMC was majorly determined as the other reaction conditions were held constants based on preliminary experiments and observations. In the tabulated results below, it is observed that increasing the MCA concentration between 3.5-8.0g per 8g of cellulose gave a yield above 100 percent while a poor yield was obtained when the MCA concentration is increased to 9.5gMCA per 8g of cellulose. Although the optimal NaOH concentration was

not determined in this work, it was observed that using a concentration of 3.25mol/AGU led to excessive gel that impeded the recovery of CMC products. Hence, a NaOH concentration of 2.45 mol/AGU was chosen and maintained throughout the course of this work.

### FTIR Spectra of Synthesized Carboxymethyl Cellulose Products.

The FTIR spectra of all the synthesized Na-CMC samples have shown typical absorption peaks of carboxymethylated cellulose as shown in Table 3.2 and Fig 3.1 According to report from literature, carboxyl groups and its salts have wave number about 1600-1640  $\text{cm}^{-1}$  and 1400-1450 $\text{cm}^{-1}$  (Adinugraha et al, 2005). The peaks at wave numbers of 1612.88-1637.00 and 1417.42-1421.14  $\text{cm}^{-1}$  of the synthesized CMC products indicated the presence of carboxymethyl substituent. The FTIR spectra of the samples show that the carboxyl, methyl and hydroxyl functional groups are found at wavelength of 1612.88-1637.00, 1417.42-1421.14  $\text{cm}^{-1}$  and 1330.75-1335.54.

Table 3.2—Assignment of main absorption bands in Synthesized Delonix CMC Products.

Wave Number ( $\text{cm}^{-1}$ ) of Delonix CMC Products					Assignment
CMC-35	CMC-50	CMC-65	CMC-80	CMC-95	
3856.00	3868.00	3866.00	-	3862.00	Free OH
37401.7	3741.00	3742.58	3742.00	3743.00	Free OH
3434.00	3436.00	3441.00	3445.00	3455.00	OH stretching
2933.00	2932.50	2929.82	2928.42	2924.00	CH stretching of $\text{CH}_2$ & $\text{CH}_3$
2353.55	2354.05	2354.47	2354.53	2354.18	C-O asymmetric stretching vibration
1627.90	1612.88	1615.57	1623.00	1637.00	C=O stretching (Indicates CMC)
1417.42	1417.42	1420.58	1421.14	1420.11	$\text{CH}_2$ bending (methyl group present)
-	1335.54	1331.22	1330.75	1334.00	OH in plane bending
1039.25	1047.66	1039.00	1053.00	1061.63	C-O stretching of primary alcohol
633.33	634.50	664.47	662.17	664.00	OH out-of-plane bend

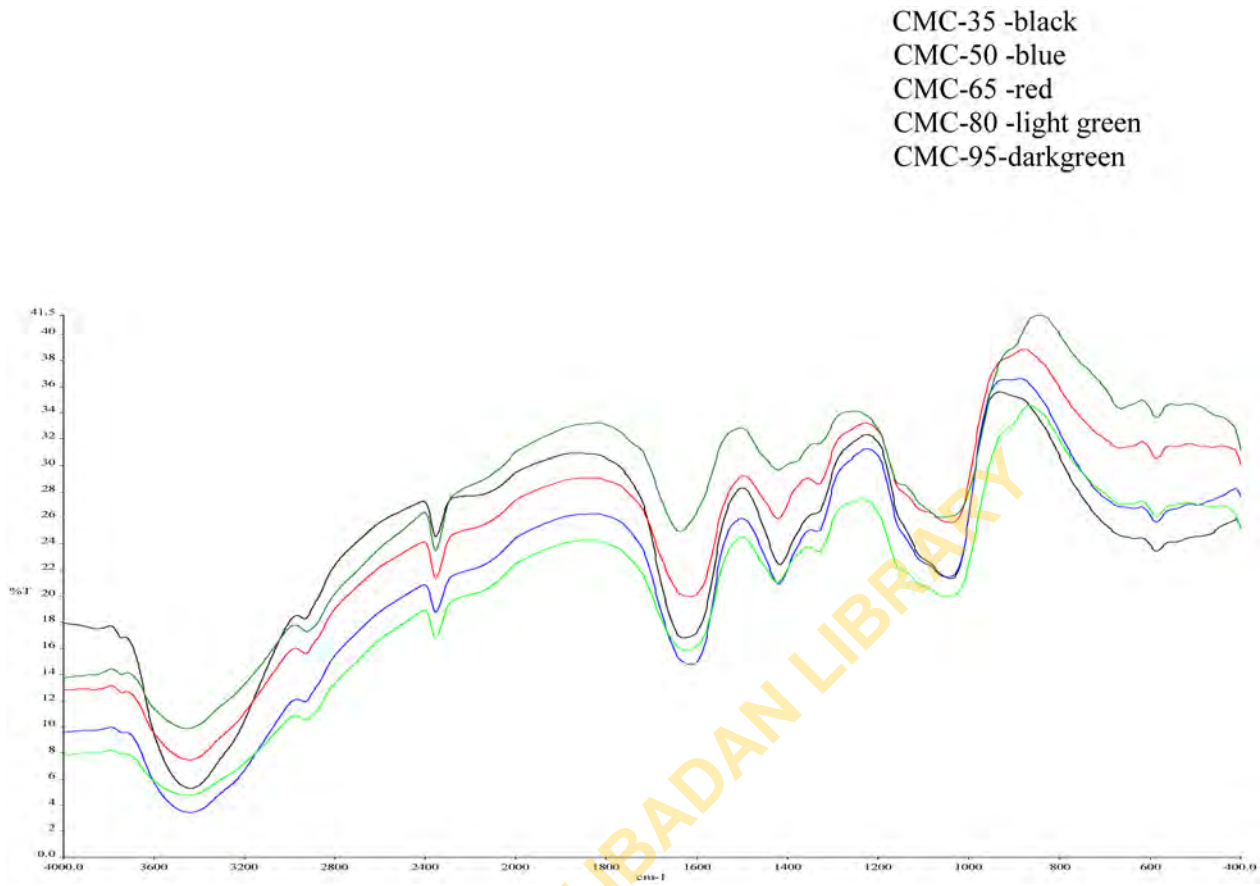


Figure 3.1—FTIR Spectra of Synthesized Sodium Carboxymethyl Cellulose products.

**Effect of Synthesized CMC Products on Mud Rheology**

After the mud tests, the results obtained from the addition of varying concentrations of each of the synthesized CMC products were presented in tables. The tables are shown below;

**Plastic Viscosity.** The Plastic viscosity of the un-weighted water based mud samples prepared by the addition of the synthesized CMC products at concentrations of 0.5g, 1.0g and 1.5g was determined by taking the difference in dial readings from the 600 rpm and 300 rpm measurements at temperatures of 25°C (room temperature), 40°C, 60°C and 80°C. The results as shown in tables 3.3 to 3.7 presented in bar charts as shown in fig. 3.2, 3.3 and 3.4. A normal plastic viscosity measurement recommended for unweighted mud was obtained for all mud samples containing the synthesized CMC products when measured at 49°C (120°F). A normal range of plastic viscosity for an unweighted mud is 5-12 cp measured at 49°C (Bourgoyne et al, 1986). The range of plastic viscosity for all mud samples tested at 49°C were 6-8cp for 0.5g, 7-9 cp for 1.0g and 8-11cp for 1.5g of CMC used. The Plastic viscosity results obtained from the mud tests suggests that the synthesized CMC products can be used in the oil and gas industry for Water based mud formulation.

Table 3.3—Rheological parameters of control mud sample at varying temperature conditions

Sample	T (°c)	Speed and Dial Readings								Plastic Viscosity (μ <sub>p</sub> )	Yield Point lb/100ft <sup>2</sup>	Gel Strength lb/100ft <sup>2</sup>	
		600 rpm	300 rpm	200 rpm	100 rpm	60 rpm	30 rpm	6 rpm	3 rpm			⊖ <sub>600</sub> -⊖ <sub>300</sub>	⊖ <sub>300</sub> -μ <sub>p</sub>
Control Mud 1 (WBM + 0g CMC)	25	24	17	14	10	8	7	5	5	7	10	7	15
	40	22	16	14	11	10	9	7	7	6	10	9	20

Sample	T (°C)	Speed and Dial Readings								Plastic Viscosity ( $\mu_p$ )	Yield Point lb/100ft <sup>2</sup>	Gel Strength lb/100ft <sup>2</sup>	
		600 rpm	300 rpm	200 rpm	100 rpm	60 rpm	30 rpm	6 rpm	3 rpm			$\ominus_{600}-\ominus_{300}$	$\ominus_{300}-\mu_p$
	60	29	22	19	15	13	11	10	10	7	15	12	27
	80	35	27	25	21	20	18	17	17	8	19	20	43

Table 3.4—Rheological parameters of mud samples with 0.5g, 1.0g and 1.5g of CMC-35 at varying temperature conditions

Sample	T (°C)	Speed and Dial Readings								Plastic Viscosity ( $\mu_p$ )	Yield Point lb/100ft <sup>2</sup>	Gel Strength lb/100ft <sup>2</sup>	
		600 rpm	300 rpm	200 rpm	100 rpm	60 rpm	30 rpm	6 rpm	3 rpm			$\ominus_{600}-\ominus_{300}$	$\ominus_{300}-\mu_p$
A <sub>1</sub> (WBM + 0.5g CMC-35)	25	25	17	13	9	6	5	3	3	8	9	3	13
	40	19	12	10	7	6	5	3	3	7	5	3	15
	60	21	15	13	9	7	6	4	4	6	9	4	23
	80	28	21	19	15	15	11	8	8	7	14	8	28
A <sub>2</sub> (WBM + 1.0g CMC-35)	25	29	19	14	9	7	5	3	3	10	9	3	13
	40	20	13	11	7	6	5	4	3	7	6	4	13
	60	22	15	13	9	8	6	4	4	7	8	5	19
	80	28	20	17	12	11	9	7	6	8	12	7	29
A <sub>3</sub> (WBM + 1.5g CMC-35)	25	30	19	15	9	7	5	3	3	11	8	2	9
	40	22	14	11	8	5	5	2	2	8	6	2	13
	60	22	14	11	8	6	5	3	3	8	6	2	19
	80	33	23	19	14	11	9	6	5	10	13	4	28

Table 3.5—Rheological parameters of mud samples with 0.5g, 1.0g and 1.5g of CMC-50 at varying temperature conditions.

Sample	T (°C)	Speed and Dial Readings								Plastic Viscosity ( $\mu_p$ )	Yield Point lb/100ft <sup>2</sup>	Gel Strength lb/100ft <sup>2</sup>	
		600 rpm	300 rpm	200 rpm	100 rpm	60 rpm	30 rpm	6 rpm	3 rpm			$\ominus_{600}-\ominus_{300}$	$\ominus_{300}-\mu_p$
B <sub>1</sub> (WBM + 0.5g CMC-50)	25	28	18	14	9	7	5	3	3	10	8	3	13
	40	21	14	11	8	6	5	3	3	7	7	4	13
	60	23	16	13	9	8	6	4	4	7	9	5	20
	80	31	23	19	15	13	11	8	8	8	15	9	30
B <sub>2</sub> (WBM + 1.0g CMC-50)	25	34	22	17	11	9	6	3	3	12	10	4	14
	40	28	18	14	10	8	5	4	4	10	8	4	16
	60	24	17	13	9	8	6	4	4	7	10	6	20
	80	32	24	20	15	13	10	7	6	8	16	7	29
B <sub>3</sub> (WBM + 1.5g CMC-50)	25	38	25	19	13	10	7	4	3	13	12	3	10
	40	26	17	13	9	7	5	3	2	9	8	2	12
	60	29	20	16	11	9	7	4	4	9	11	3	20
	80	30	21	17	12	10	8	5	5	9	12	5	29

Table 3.6—Rheological Parameters of Mud samples of 0.5g, 1.0g and 1.5g of CMC-65 at varying Temperature conditions.

Sample	T (°c)	Speed and Dial Readings								Plastic Viscosity ( $\mu_p$ )	Yield Point lb/100ft <sup>2</sup>	Gel Strength lb/100ft <sup>2</sup>	
		600 rpm	300 rpm	200 rpm	100 rpm	60 rpm	30 rpm	6 rpm	3 rpm			$\ominus_{600}-\ominus_{300}$	$\ominus_{300}-\mu_p$
C <sub>1</sub> (WBM + 0.5g CMC-65)	25	29	18	15	10	8	6	4	4	11	7	4	14
	40	23	15	12	9	7	5	4	3	8	7	4	14
	60	24	17	14	10	8	7	5	4	7	10	7	22
	80	32	24	21	17	15	13	9	9	8	16	10	33
C <sub>2</sub> (WBM + 1.0g CMC-65)	25	38	25	19	13	10	7	4	4	13	12	4	14
	40	30	20	16	11	8	7	4	4	10	10	4	17
	60	25	17	14	10	8	6	4	4	8	9	6	22
	80	33	24	20	14	11	9	6	5	9	15	7	27
C <sub>3</sub> (WBM + 1.5g CMC-65)	25	46	31	24	16	12	9	5	5	15	16	4	12
	40	32	21	17	11	9	7	4	3	11	10	4	14
	60	31	21	17	12	10	8	5	4	10	11	5	21
	80	34	24	20	15	13	10	7	6	10	14	6	28

Table 3.7—Rheological Parameters of Mud samples with 0.5g, 1.0g and 1.5g of CMC-80 at varying temperature conditions

Sample	T (°c)	Speed and Dial Readings								Plastic Viscosity ( $\mu_p$ )	Yield Point lb/100ft <sup>2</sup>	Gel Strength lb/100ft <sup>2</sup>	
		600 rpm	300 rpm	200 rpm	100 rpm	60 rpm	30 rpm	6 rpm	3 rpm			$\ominus_{600}-\ominus_{300}$	$\ominus_{300}-\mu_p$
D <sub>1</sub> (WBM + 0.5g CMC-80)	25	25	16	13	8	7	5	3	3	9	7	3	13
	40	22	14	12	9	7	6	4	4	8	6	5	15
	60	23	16	13	10	8	7	6	6	7	9	8	23
	80	34	25	22	16	14	13	9	9	9	16	12	32
D <sub>2</sub> (WBM + 1.0g CMC-80)	25	30	19	14	9	7	5	3	3	11	7	3	13
	40	24	15	13	8	6	5	3	3	9	6	3	17
	60	24	16	14	10	8	7	5	5	8	8	5	23
	80	30	22	19	14	12	10	7	7	8	14	9	28
D <sub>3</sub> (WBM + 1.5g CMC-80)	25	34	21	17	11	8	6	3	3	13	8	3	10
	40	25	16	12	8	6	5	3	3	9	7	3	14
	60	27	18	14	10	8	6	4	3	9	9	4	20
	80	34	23	19	13	11	8	5	4	11	12	6	30

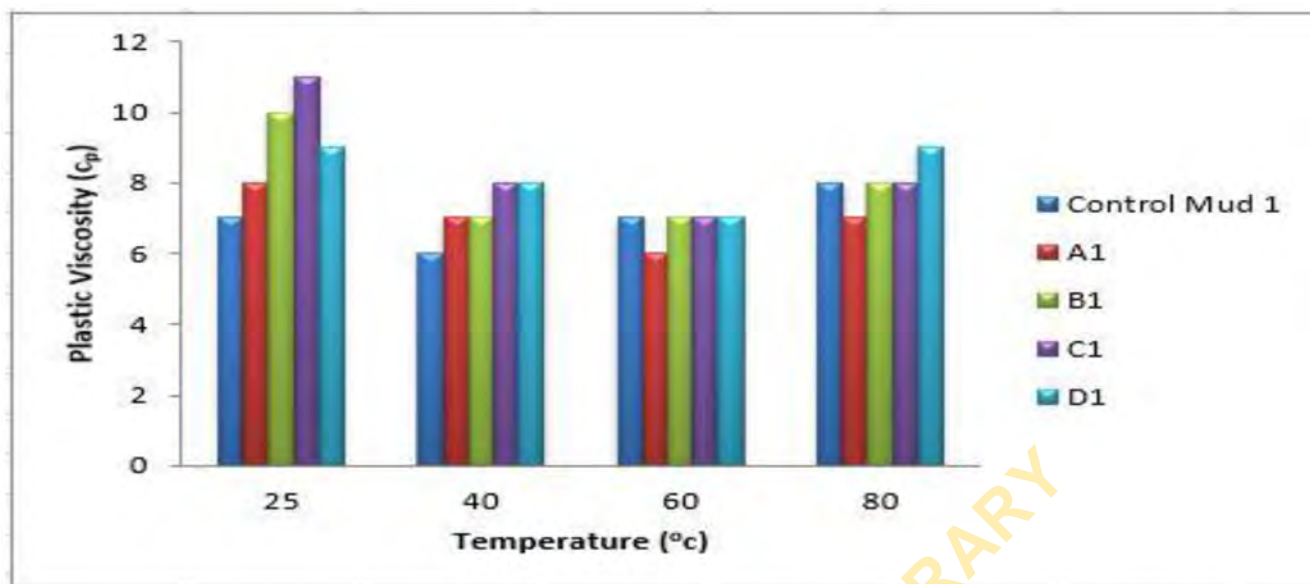


Figure 3.2—Effect of 0.5 g of CMC products on water based mud Plastic Viscosity at varying temperature conditions

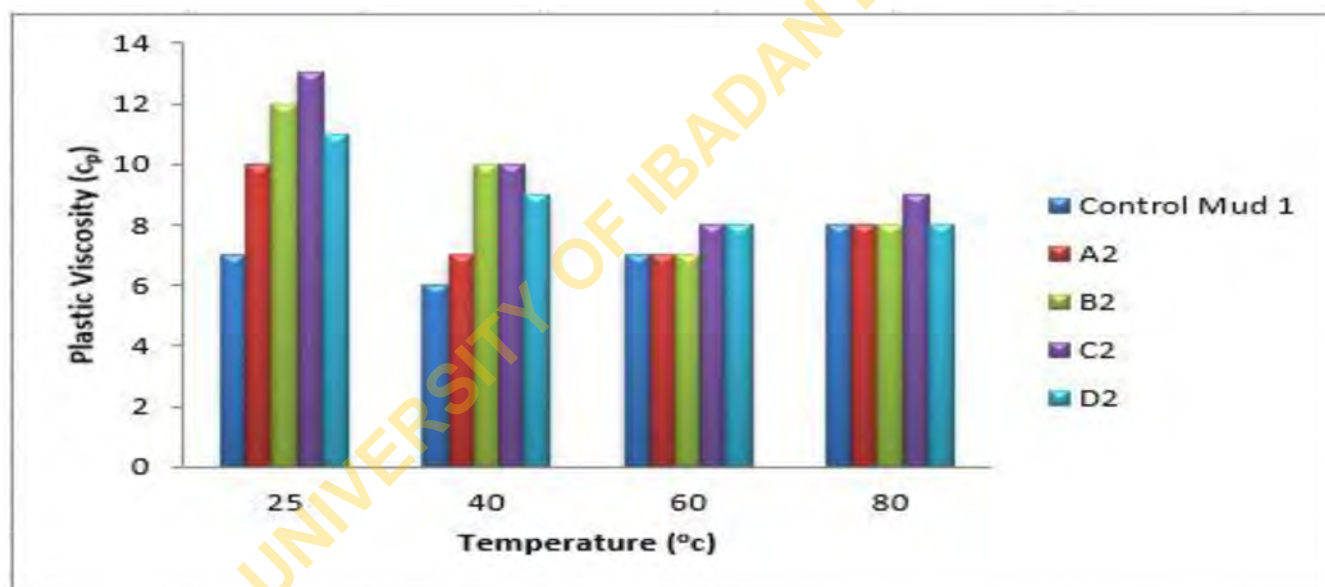


Figure 3.3—Effect of 1.0 g of CMC products on water based mud Plastic Viscosity at varying temperature condition

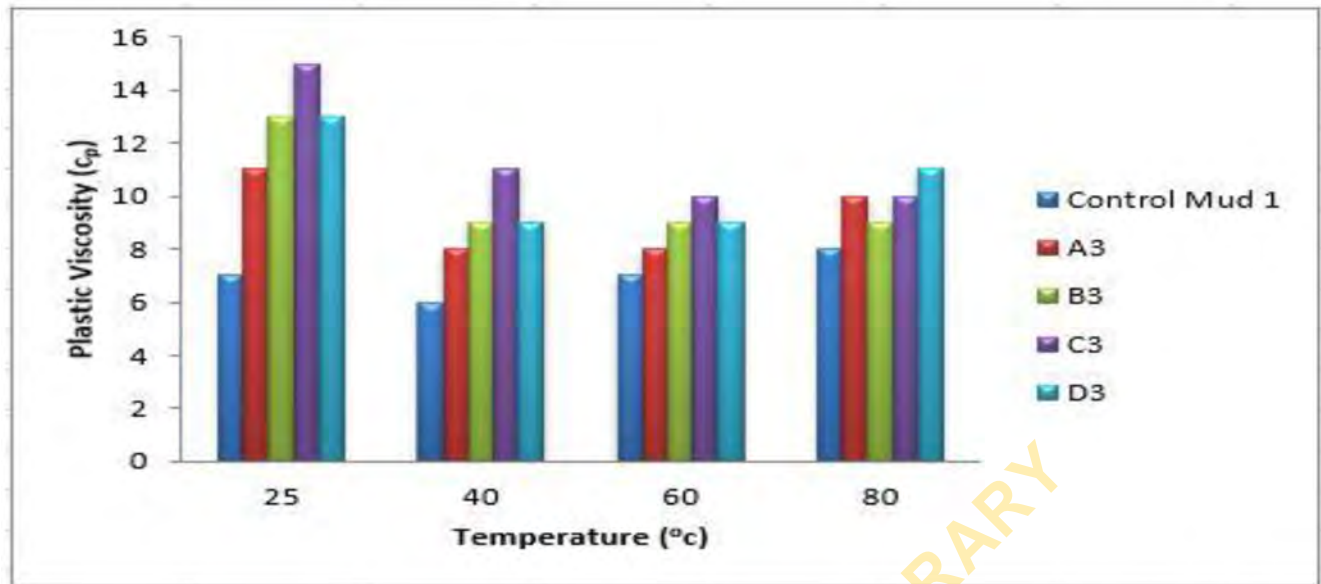


Figure 3.4—Effect of 1.5 g of CMC products on water based mud Plastic Viscosity at varying temperature condition

**Yield Point.** The yield point of an un-weighted mud is usually maintained in the range of 3 to 30 lb/100ft<sup>2</sup> measured at 120°F (49°C) in order to enhance the ability of a mud to carry cuttings to the surface without increasing the frictional pressure drop in the annulus to a point that is enough to cause formation fracture (Bourgoyne et al, 1986). The YP values of WBM samples formulated using the synthesized CMC products fell within the recommended range as shown in fig. 3.5, 3.6 and 3.7. After conducting mud tests between 25°C and 80°C, the range of yield point values obtained were in the range of 5-19, 6-17 and 6-15 lb/100ft<sup>2</sup> for 0.5g, 1.0g and 1.5g of synthesized CMC concentration respectively. It was observed from fig. 3.5, that the addition of 0.5g of the CMC products used in mud test resulted in a decrease in YP values of the mud samples at the range of temperature tested when compared to the values of the control mud. This agrees with Annis and Smith that "at low concentration, CMC deflocculates clay suspensions in much the same manner as lignosulfonate resulting in a reduction of the fluid loss as well as yield point". At 25°C, an increase in the concentration of CMC concentration causes an increase in YP as observed when YP of Mud B, C and D increases from 8-12, 7-16 and 7-10 lb/100ft<sup>2</sup> when the concentration of the respective CMC products was increased from 0.5g to 1.5g respectively. Also, an increase in temperature from 25°C to 40°C causes a reduction in yield point but as temperature rises from 40°C to 80°C, there is an increase in yield point. The addition of CMC caused a reduction in yield point and the tendency to flocculate as temperature increases.

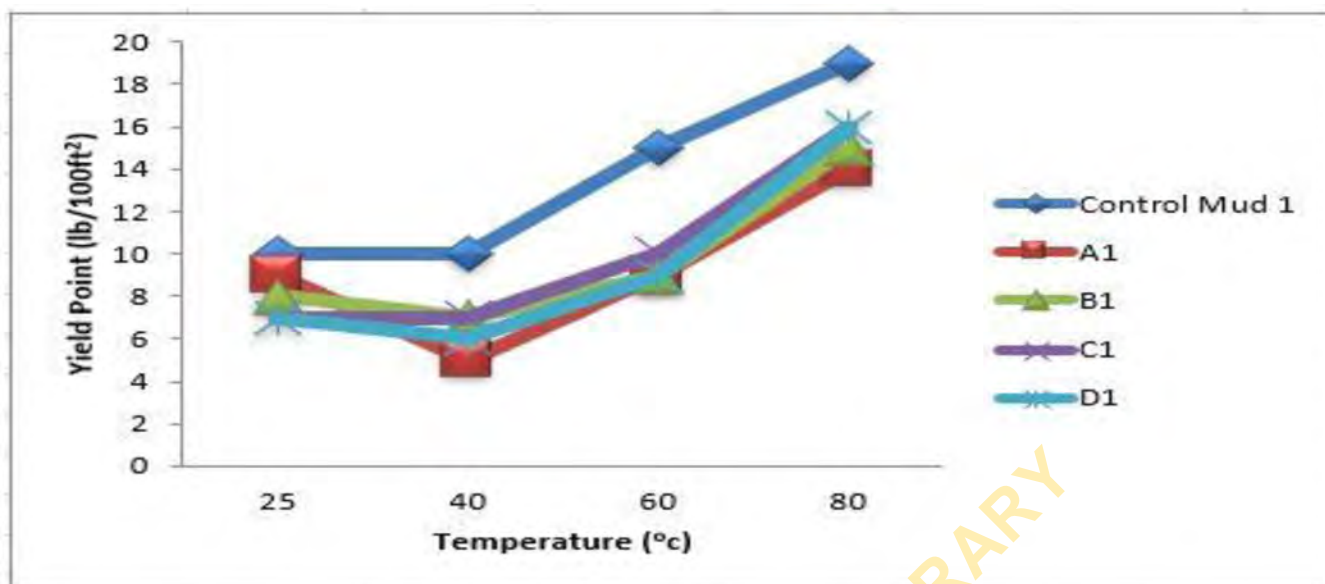


Figure 3.5—Effect of 0.5g of CMC products on WBM Yield Point at varying temperature conditions

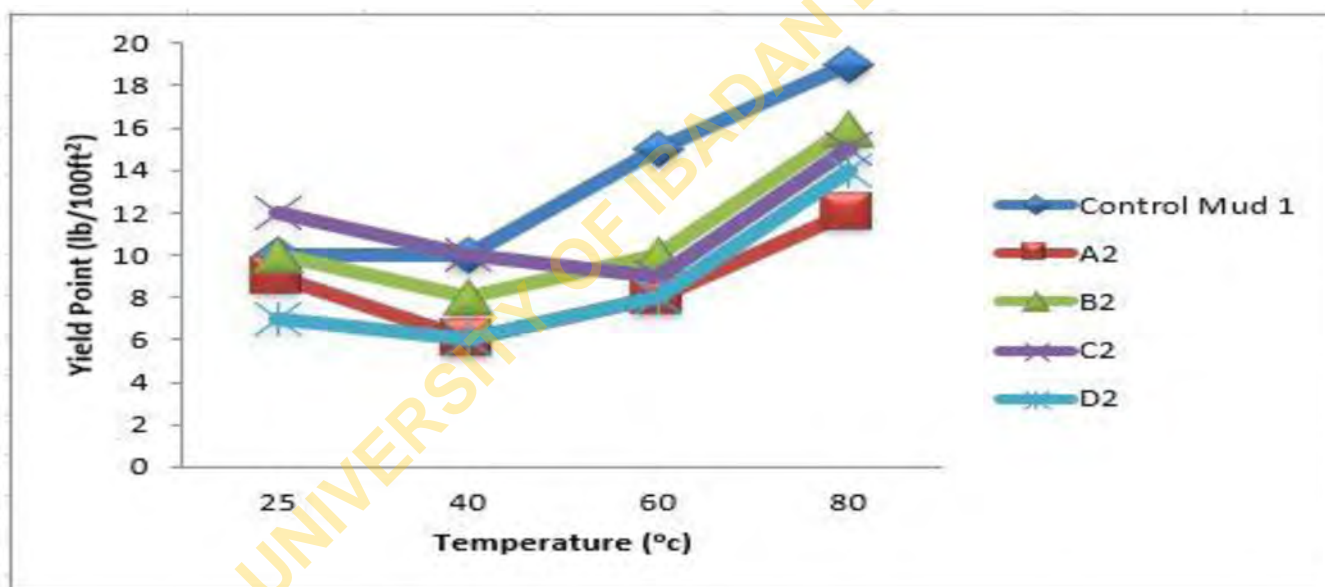


Figure 3.6—Effect of 1.0g of CMC products on WBM Yield Point at varying temperature conditions

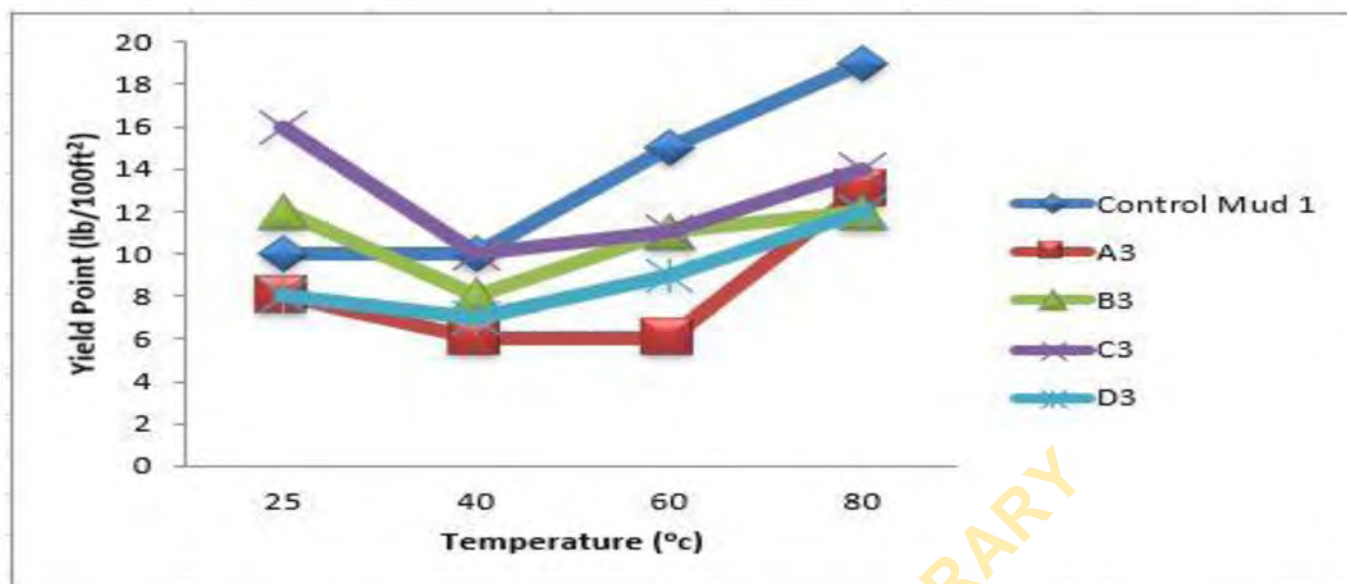


Figure 3.7—Effect of 1.5g of CMC products on WBM Yield Point at varying temperature conditions

### Gel Strength.

#### 10 seconds Gel Strength

The 10s gel strength of drilling mud samples tested at room temperature varied from 2 lb/100ft<sup>2</sup> to 4 lb/100ft<sup>2</sup> when the synthesized CMC products were used in mud formulation between 0.5g and 1.5g concentration as shown in fig. 3.8, 3.9 & 3.10. This GS values represent a range of gel strength reduction between 42.86% and 71.43% when compared with a 7 lb/100ft<sup>2</sup> of the control sample. This GS values satisfies the minimum requirement for the suspension of barite and mud cuttings. In order to suspend barite, gel strength of 2 to 4 lb/100ft<sup>2</sup> is necessary (Annis and Smith, 1996). It was observed from the control mud that as temperature increases, there is an increase in gel strength. This increase in gel strength is due to the effect of flocculation of clay particles. 0.5g of the synthesized CMC product reduces mud gel strength from 7 lb/100ft<sup>2</sup> to 2-4 lb/100ft<sup>2</sup> at 25°C, 9 lb/100ft<sup>2</sup> to 3-5 lb/100ft<sup>2</sup> at 40°C, 12 lb/100ft<sup>2</sup> to 4-8 lb/100ft<sup>2</sup> at 60°C and 20 lb/100ft<sup>2</sup> to 8-12 lb/100ft<sup>2</sup> at 80°C. When the concentration is increased above 0.5, there is a further reduction in mud gel strength as shown in fig. 4.13 and 4.14 below.

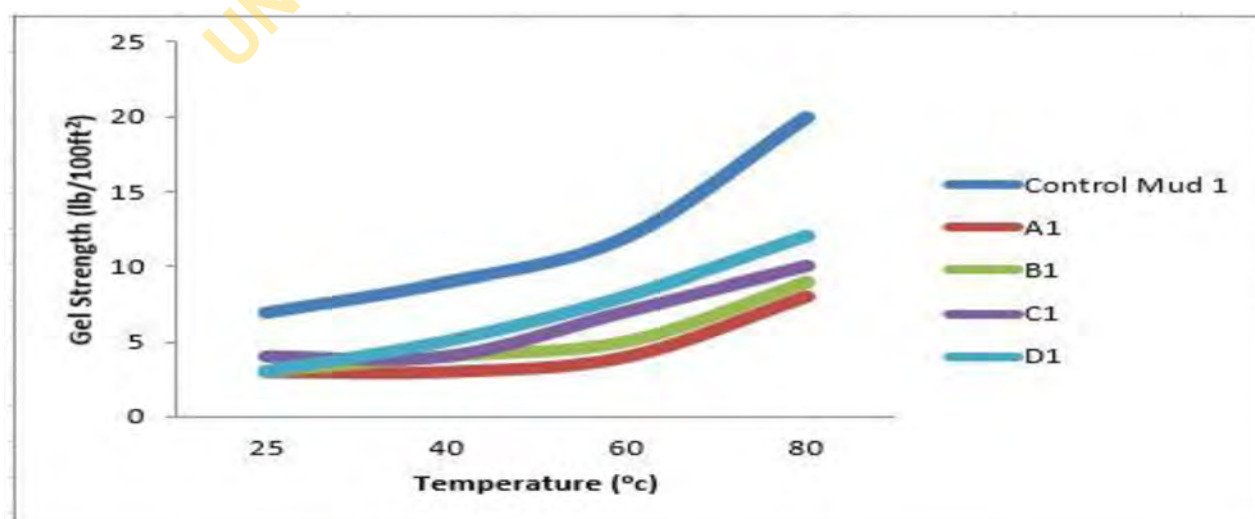


Figure 3.8—Effect of 0.5g of CMC products on 10 seconds Gel strength at varying temperature condition

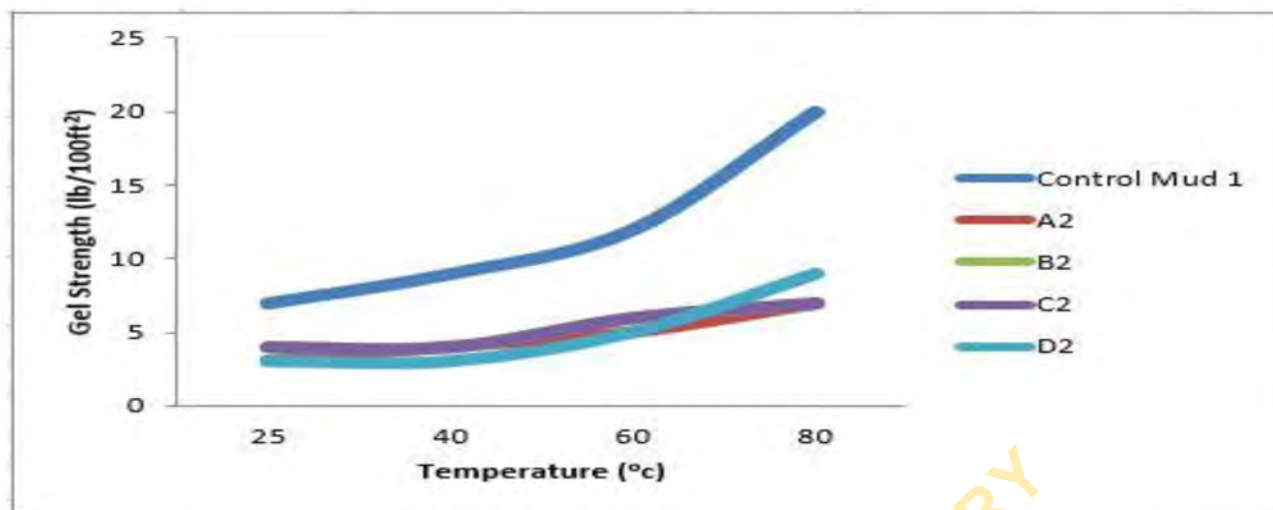


Figure 3.9—Effect of 1.0g of CMC products on WBM 10 seconds Gel strength at varying temperature conditions

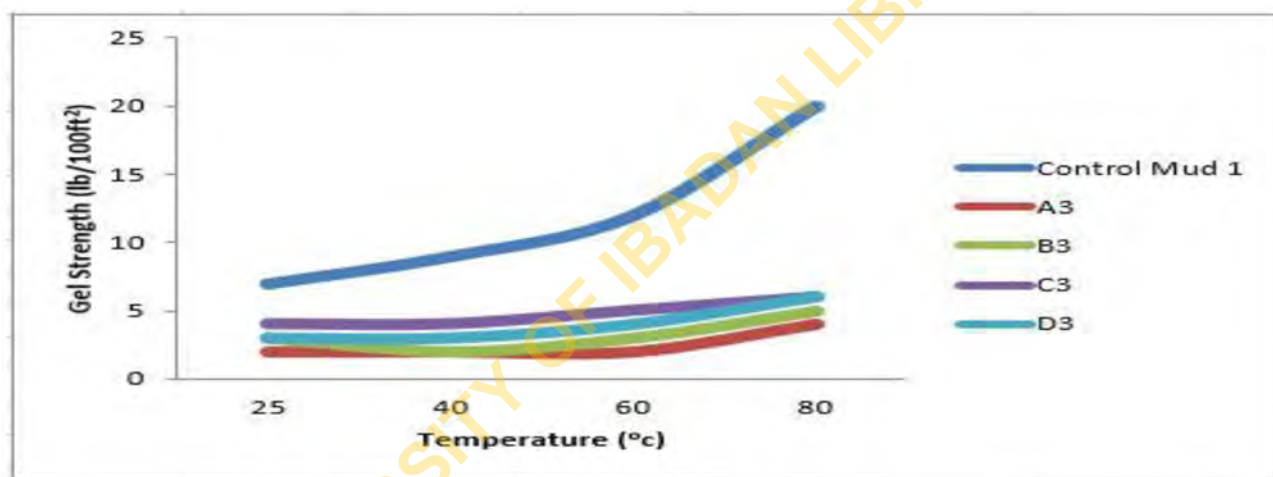


Figure 3.10—Effect of 1.5g of CMC products on WBM 10 seconds Gel strength at varying temperature conditions.

### Effect of Synthesized CMC Products on Fluid Loss

The results from the fluid loss experiments conducted at low temperature and pressure conditions for each mud samples formulated at concentrations of 0.5g, 1.0g and 1.5g of the synthesized CMC products are shown in tables 3.8, 3.9 and 3.10 respectively. The tables show the fluid loss taken at 2 minutes interval for 30 minutes. It was observed that when 0.5g of the synthesized CMC products were used in WBM formulation per laboratory barrel of mud as shown in table 3.8 and fig 3.11, fluid loss reduced from 15.8ml of control mud 1 without CMC to between 13.2ml and 12.0ml of mud with delonix CMC products which represents a 16.46% to 24.05% reduction. Control Mud 2A with the foreign product gave a 20.25% reduction in fluid loss while Mud C<sub>1</sub> formulated with 0.5g of CMC-65 gave the highest reduction in fluid loss. At higher CMC concentration of 1.0g and 1.5g, CMC-65 gave 32.91% and 30.38% reduction in fluid loss while the foreign CMC product gave 29.11% and 30.38% reduction in fluid loss respectively. Although other synthesized products gave a significant reduction in fluid loss, they were not a more effective substitute to the foreign product as compared to CMC-65 that gave the highest reduction in fluid loss at all concentration. Therefore, CMC products from this work can serve as a substitute to the foreign product.

**Table 3.8—Fluid loss test results of Mud samples formulated with 0.5g of the synthesized CMC Products**

Samples/ time (mins)	2	4	6	8	10	12	14	16	18	20	22	24	26	28	30
Control Mud 1	3.6	5.2	6.8	8.0	9.0	9.8	11	11.6	12.4	13.2	13.6	14.3	14.6	15.4	15.8
Control Mud 2A	2.4	4.0	5.0	6.0	6.6	7.4	8.0	8.6	9.2	9.8	11.0	11.8	12.0	12.2	12.6
A <sub>1</sub>	3.0	4.2	5.4	6.4	7.1	7.8	8.4	9.0	9.6	10.2	11.0	11.4	12.0	12.4	12.8
B <sub>1</sub>	3.2	4.4	5.3	6.2	6.9	7.6	8.2	8.8	9.3	9.6	10.4	11.0	11.4	11.8	12.2
C <sub>1</sub>	3.2	4.3	5.3	6.1	6.8	7.5	8.1	8.7	9.2	9.6	10.2	10.8	11.2	11.6	12.0
D <sub>1</sub>	3.4	4.6	5.8	6.7	7.4	8.2	8.8	9.4	10.2	10.6	11.2	11.8	12.4	12.8	13.2

**Table 3.9—Fluid loss test results of Mud samples formulated with 1.0g of the synthesized CMC Products.**

Samples/ time(mins)	2	4	6	8	10	12	14	16	18	20	22	24	26	28	30
Control Mud 1	3.6	5.2	6.8	8.0	9.0	9.8	11	11.6	12.4	13.2	13.6	14.3	14.6	15.4	15.8
Control Mud 2B	2.0	3.4	4.2	5.0	5.8	6.2	7.0	7.6	8.0	8.6	9.0	9.4	9.8	10.6	11.2
A <sub>2</sub>	4.2	5.6	6.8	7.6	8.4	9.0	9.6	10.4	11.2	11.8	12.2	12.8	13.0	13.6	14.0
B <sub>2</sub>	3.6	4.8	5.8	6.6	7.4	7.8	8.4	9.0	9.5	9.8	10.5	11.2	11.6	11.8	12.2
C <sub>2</sub>	2.2	3.6	4.2	5.3	5.8	6.4	7.0	7.4	8.0	8.6	9.0	9.4	9.8	10.4	10.6
D <sub>2</sub>	2.8	4.0	5.2	6.0	6.8	7.2	7.8	8.4	9.2	9.6	10.0	10.6	11.0	11.4	11.8

**Table 3.10—Fluid loss test results of Mud samples formulated with 1.5g of the synthesized CMC Products.**

Samples/ time(mins)	2	4	6	8	10	12	14	16	18	20	22	24	26	28	30
Control Mud 1	3.6	5.2	6.8	8.0	9.0	9.8	11	11.6	12.4	13.2	13.6	14.3	14.6	15.4	15.8
Control Mud 2C	1.6	3.0	4.0	4.8	5.6	6.2	6.8	7.4	8.0	8.4	9.0	9.4	9.8	10.4	11.0
A <sub>3</sub>	3.2	4.6	5.6	6.4	7.0	7.6	8.2	8.8	9.4	9.8	10.4	11.0	11.2	11.6	12.2
B <sub>3</sub>	3.8	5.0	5.8	6.6	7.2	7.6	8.0	8.4	9.0	9.4	9.8	10.4	11.0	11.2	11.6
C <sub>3</sub>	3.4	4.6	5.4	6.0	6.8	7.2	7.6	8.0	8.6	9.0	9.2	9.6	10.2	10.6	11.0
D <sub>3</sub>	3.2	4.3	5.2	5.8	6.4	7.0	7.5	8.0	8.4	8.8	9.4	9.8	10.2	10.8	11.2

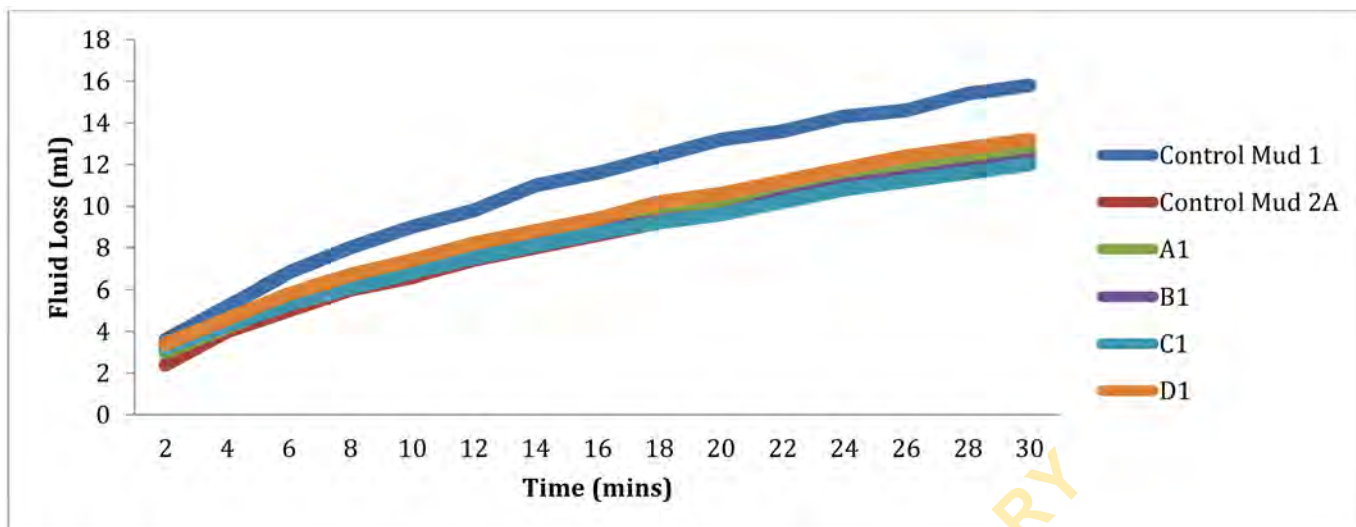


Figure 3.11—Effect of 0.5g of CMC products on Mud samples Fluid Loss in comparison to the control Mud.

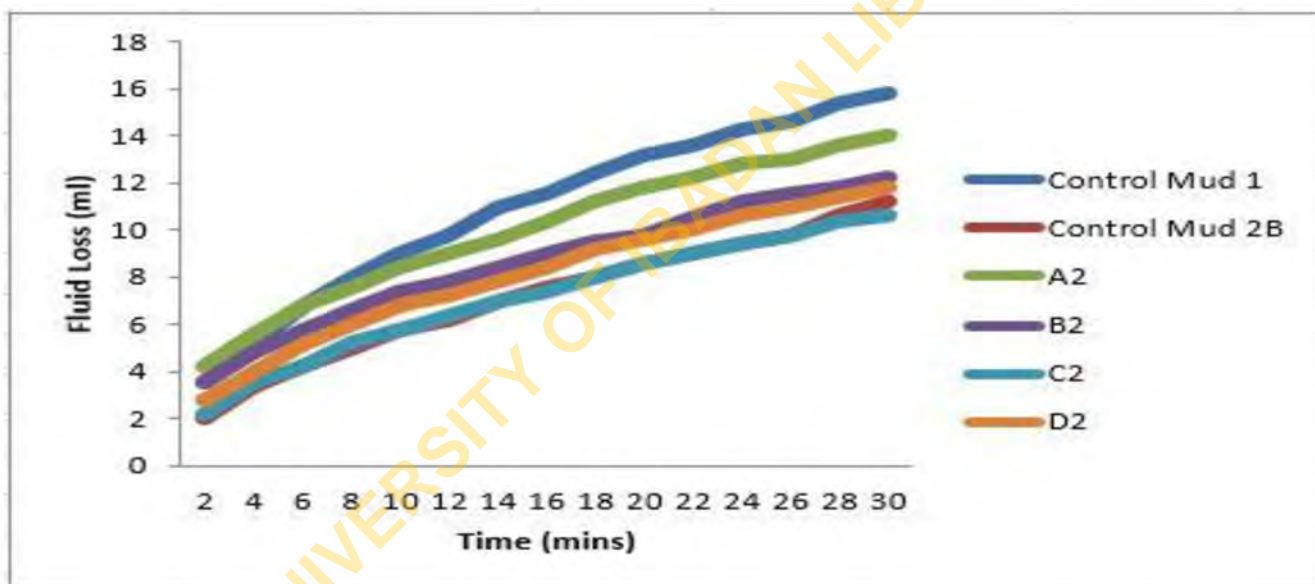


Figure 3.12—Effect of 1.0g of CMC products on Mud samples Fluid Loss in comparison to the control Mud.

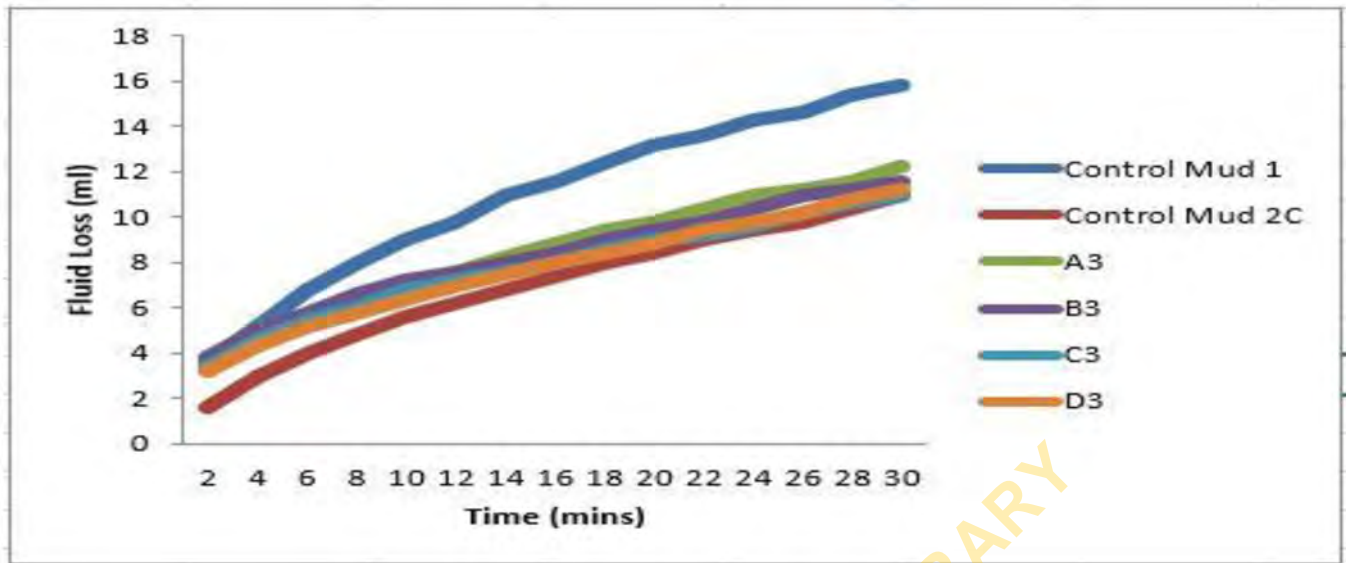


Figure 3.13—Effect of 1.5g of CMC products on Mud samples Fluid Loss in comparison to the control Mud.

## Conclusions

There exist a vast amount of bio-based solid wastes generated across several industries that can serve as useful feedstock for the production of oilfield chemical additives. These bio-based wastes such as sawdust are recyclable, renewable, bio-degradable and cheap. In this study, it was discovered that;

1. Sawdust of delonix regia wood is a versatile feedstock for the production of sodium carboxymethyl cellulose.
2. The properties of the synthesized CMC products are dependent on the reaction parameters such as the quality of cellulose feedstock and its particle size, NaOH concentration, reaction time and temperature, Monochloroacetic acid (MCA) concentration and solvent used.
3. Increasing the MCA concentration to the optimum amount increases the solubility and viscosity-building ability of CMC and increasing the MCA concentration above the optimum concentration reduces its quality.
4. The synthesized carboxymethyl cellulose products yielded good rheological properties suitable for drilling fluid applications.
5. The use of low concentrations of about 0.5g to 1.0g the synthesized products per laboratory barrel of mud could reduce filtration to between 11.4% and 32.9% when measured at low temperature and pressure conditions. The synthesized products from this work can be used as substitute for foreign product.

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