بسم الله الرحمن الرحيم

Sudan University of Science and Technology
College of Graduate Studies

Sodium Carboxymethyle Cellulose Produced from Palm Frond as Drilling Fluids Additive

سليلوز كاربوكسيميثيل الصوديوم المنتج من سعف النخيل كمادة مضافة لسوائل الحفر

A thesis Submitted in Partial Fulfillment for the Degree of M. Sc. in
Petroleum Engineering - Drilling Engineering

M. Sc. Candidate by:

Mohammed Elfatih Ibrahim Ali Ibrahim.

Supervisor:

Cons. Eng. Dr. Ahmed Abdelaziz Ibrahim.

Sep., 2020

بسم الله الرحمن الرحيم

قال تعالى : "رَبِّ أَوْزِعْنِي أَنْ أَشْكُرَ نِعْمَتَكَ الَّتِي أَنْعَمْتَ عَلَيَّ وَعَلَى وَالِدَيَّ وَأَنْ أَعْمَلَ صَالِحًا تَرْضَاهُ وَأَدْخِلْنِي بِرَحْمَتِكَ فِي عِبَادِكَ الصَّالِحِينَ "

صدق الله العظيم

(سورة النمل : الآية 19)

DEDICATION

To my respective parents who have been my constant source of inspiration. They have given me the drive and discipline to tackle any task with enthusiasm and determination. Without their love and support, this research would not have been made possible.

To my dears, all of my family members who were there when i need them and always near….

To my wonderful supervisor Cons. Eng. Dr. Ahmed Abdelaziz Ibrahim donated me his knowledge and never disappoints me or lets me down...

To my best friends and colleagues who were with me step by step, supporting to go forward…

To my school teachers who firstly taught me how to write my first words and I am still following their advices and wish the best for them…

To My dear wife and my children, my pleasure …

I Dedicate This Work …..

ACKNOWLEDGMENTS

The greatest thanks to Allah always before and after.

I would like to express my deep gratitude to everyone who helped me to throughout this work at any step of it…

Firstly, most grateful and appreciation to my supervisor **Cons. Eng. Dr.Ahmed Abdelaziz Ibrahim** for his expertise support and endless valuable advices which guided me throughout work and my engineering career life.

Also, i would like to thank Mr. Ali Mamoun Awab Elrayah and Shazly Sayed Ahmed for their valuable assistance and for their patience, time and effort they spend helping me.

I also would like to thank all of my teachers in college of Petroleum and Mining Engineering.

Disclaimer

Hereon, I am sincerely declaring that the presented MSc dissertation has been completed under the guidance of Cons. Eng. Dr. Ahmed Abdelaziz Ibrahim. The dissertation shows the experimental findings derived from the project “Utilization of Cellulosic Biomasses into Useful Polymers for Oil Field Industry” which is authorized by the Sudan University of Science and Technology by the Project Number: DSR - 1502.

In this dissertation all the Field Data, Graphs, and Other Materials subjected to analysis belong to the Ministry of Petroleum and Mining – Oil Exploration and Production Engineering Authority (OEPA) - Republic of Sudan. The data, graphs and the other materials have been approved by the author and modified in the thesis as well.

The text represents my own original research; design and the emulation analysis are originally from my sole experiments and research, while the theoretical research concerning the principle, guiding inclination detection and corrections that are quoted from others had made out in the way of referencing. Any other external assistance and enlightens has been acknowledged and appreciated.

This research work has not been submitted for any higher degree to any other university or institute except where otherwise acknowledged and got the author and the above mentioned authorities’ permission.

Mohammed Elfatih Ibrahim Ali Ibrahim
Sudan University of Science and Technology – Faculty of Petroleum Engineering and Technology – Petroleum Department
September - 2020

Abstract

This research discusses the use of Sodium Carboxymethyl Cellulose, which is a product of cellulose produced from palm fronds in the laboratories of the Sudan University of Science and Technology and Central Petroleum Laboratories and the Regional Forensic Laboratories Directorate (research project with number **DSR – 1502**). The effect of local product when adding it to the water base drilling fluid system was investigated. A set of tests were performed, including x-ray diffraction testing for commercial sodium carboxymethyl cellulose (Chinese), laboratory produced sodium carboxymethyl cellulose, as well as three different bentonite samples to review physical behavior, composition, comparison, and emulation. The work moved to laboratory experiments on water-based drilling fluid by adding different concentrations of the commercial product as well as the laboratory product of sodium carboxymethyl cellulose in the drilling fluid laboratories of Petroleum Engineering and Technology at the Sudan University of Science and Technology. A comparison was made between the commercial sodium carboxymethyl cellulose and the laboratory sodium carboxymethyl cellulose the results proved the success of the product estimated in comparison to the commercial product (increasing the viscosity of drilling fluid and reducing filtering water). By adding sodium carboxymethyl cellulose in laboratory mud sample and inserting it into the pilot test drilling fluid system used in Block 6 Balela area of PetroEnergy Company for Petroleum Operations, in cooperation with the African Drilling Fluids laboratories, and by comparison with what was used, the addition of sodium carboxymethyl cellulose in the laboratory has proven quite good practical and economical efficacy.

التجريد

هذا البحث يناقش استخدام سليلوز كاربوكسي ميثيل الصوديوم الناتج من السيليلوز المنتج من سعف النخيل معملياً بمعامل جامعة السودان للعلوم والتكنولوجيا ومختبرات النفط المركزية، ومعمل دائرة المختبرات الجنائية الإقليمية (مشروع بحثي بالرقم DSR – 1502) ودراسة تأثير المنتج محليا عند إضافته لسائل الحفر ذو الأساس المائي. تم إجراء مجموعة من الإختبارات متمثلة في اختبار حيود الأشعة السينية لمادة سليلوز كاربوكسي ميثيل الصوديوم التجاري (صيني) ، سليلوز كاربوكسي ميثيل الصوديوم المنتج محليا وكذلك لثلاثة عينات مختلفة من مادة البنتونايت لمراجعة السلوك الفيزيائي ، تركيب العناصر الفيزيائية والمقارنة والمضاهاة. إنتقل العمل إلي التجارب المعملية علي سائل الحفر ذو الأساس المائي بإضافة تراكيز مختلفة من المنتج التجاري وكذلك المنتج المحلي من مادة سليلوز كاربوكسي ميثيل الصوديوم بمعمل كلية النفط جامعة السودان للعلوم والتكنولوجيا. تم اجراء مقارنة بين المنتجين التجاري والمنتج معملياً حيث اثبتت النتائج نجاح المنتج تقديرياً مقارنة للمنتج التجاري وذلك في زيادة لزوجة سائل الحفر وتقليل ماء الرشيح. بأضافة سليلوز كاربوكسي ميثيل الصوديوم المنتج معملياً وإدخاله في نظام سائل الحفر المستخدم بمناطق بليلة مربع 6 التابع لشركة بتروإنرجي لعمليات البترول وبالتعاون مع معامل شركة أفريكان دريلنق فلويدز وبالتحقق مقارنة بما مستخدم فإن إضافة سليلوز كاربوكسي ميثيل الصوديوم المنتج معملياً قد اثبتت فعالية عملية وإقتصادية عالية. فمن النتائج المتحصلة نوصي بزيادة الدراسات لتطوير المنتج سليلوز كاربوكسي ميثيل الصوديوم رفعة لإقتصاديات البلاد.

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CHAPTER ONE

INTRODUCTION

Chapter 1

Introduction

Water based fluids use a fresh water or salt water as a base fluid with various additives such as weighting agents, viscosities and fluid loss control additives to control the properties of the drilling fluid as required in field applications.

This study was clarified the extraction of CMC from local material (Palm fronds) in the laboratories of Sudan University of Science and Technology in two steps firstly cellulose isolation and then extracting CMC.

Mud samples of water-based drilling mud (pure bentonite, bentonite with percentage of commercial CMC and bentonite with percentage of local produced polymer CMC) were prepared according to mud formulations currently used in the wells drilling after that rheological properties and fluid properties were investigated for both commercial and locally produced CMC with bentonite. Also compared local produced CMC to commercial one to know the similarities as well as the differences between the commercial and locally produced CMC and Finally adding the locally produced product to the drilling mud system the same as that is being used in block 6.

1.1. Problem Statement

water-based drilling fluid compositions comprising carboxymethyl cellulose (CMC) are employed in well-drilling operations and perform a variety of functions which influence the drilling rate, cost, efficiency, and safety of the operation. More specifically, drilling fluid compositions prevent the influx of formation fluids into the wellbore, seal exposed permeable formations to prevent leakage of the drilling fluid into the formation, maintain the stability of exposed formulations, cool and lubricate the bit and drill string, hold back pressure, and stabilize the formation. The importance of this research will be in a cost reduction and the ability of producing such material locally that if palm frond extracted CMC matches the API international standard specification.

1.2. Objectives

1) Clarification of extracting CMC from the palm frond.
2) Evaluation of the rheological properties of the commercial CMC and extracted CMC.
3) Evaluation of the flow properties of the commercial CMC and extracted CMC.
4) Compare extracted CMC to commercial CMC.
5) Evaluating the field fluid system by adding the extracted CMC.
6) Economical sensitivity.

1.3. Methodology

1) Collecting the commercial CMC and the extracted CMC.
2) Drilling fluid lap work.
3) Rheological Comparison between commercial and extracted CMC.
4) Flow behavior Comparison.
5) Fluid design according to block 6 properties.
6) Economical sensitivity.

To achieve these objectives of the research a certain methodology has been followed as mentioned above and has helped to get back to previous studies of some papers and researches to be a literature review witch related to water base mud, drilling fluid additives, fluid properties, cellulose recourses, CMC extracting and uses of CMC in drilling fluid.

CHAPTER TWO

LITERATURE REVIEW

CHAPTER 2

Literature Review

To achieve the research objectives a certain methodology has been followed as mentioned in chapter one and has helped to get back to previous studies of some papers and researches to be a literature review including topics related to water base mud , drilling fluid additives , fluid properties , cellulose recourses , CMC extracting , uses of CMC in drilling fluid and other important topics as below.

2.1. Water Base Mud

Water based fluids are drilling fluids which uses fresh water or salt water as its base material. The fluid also consists of various water soluble additives with different functions to improve properties of the fluid to meet the requirements of the borehole. These additives include viscosifiers, weighting agents and fluid loss controllers. Water based drilling fluids have been proven to be more cost effective option both in initial cost as well as drill cutting disposal cost. In addition to that, water based drilling fluids have been proven to be more environmental friendly than other drilling fluids as the base material itself is merely water which pose no threat to the environment (Winson, 2012). However, a down point would be that water based fluids are prone to thermal degradation at temperatures above 121℃ (250°F). Thermal degradation affects the properties of the fluids which makes it less reliable in high temperature-high pressure (Osokogwu, 2014). Water based fluids usually consist of water, barite, clay/polymer and other necessary additives. WBM is predominantly used in the industry due to its environmentally acceptable nature, and also because it is relatively cheap to operate with.

The following designations are normally used to define the classifications of water based drilling fluids (Hussain Rabia, 2001):

Non-Inhibited: the fluid contains no additives to inhibit hole problems.

Inhibited: the fluid contains inhibiting ions such as chloride, potassium or calcium or a polymer which suppresses the breakdown of the clays by charge association and or encapsulation.

Dispersed: thinners have been added to scatter chemically the bentonite (clay) and reactive drilled solids to prevent them from building viscosity.

Non-Dispersed: the clay particles are free to find their own dispersed equilibrium in the water phase

Non-dispersed-non-inhibited fluids: do not contain inhibiting ions such as chloride (Cl-), calcium (Ca2+) or potassium (K+) in the continuous phase and do not utilize chemical thinners or dispersants to affect control of rheological properties.

Spud Gel Mud: Used for top hole drilling, usually in 40 to 50 bbls pills on each connection, and hole volume sweeps and displacement at hole TD. The mud is prepared by pre-hydrating bentonite at 30 ppb (pounds per barrel) for 4-6 hrs prior to use to allow time for the clay to yield (Hussain Rabia, 2001).

CMC Gel Mud: Used as an alternative to the spud mud when the mud system is closed in (Hussain Rabia, 2001).

The CMC added at 1 to 3 ppb, offers some fluid loss control, however, this mud system should only be used in areas of unreactive formations and will be subjected to high levels of dilution (Hussain Rabia, 2001).

Non-dispersed- inhibited fluids: contain inhibiting ions in the continuous phase, however they do not utilize chemical thinners or dispersants.

These mud systems are the most common in drilling problematic formations like reactive clays, sloughing heaving shales and halite salt sections. The mechanisms of inhibition vary according to the type of inhibitive product being used. It is common to utilise two or more products in the same mud system. These mud systems also minimise the reaction with the drilled cuttings and therefore help to avoid the high dilution rates exhibited by other fluid groups (Hussain Rabia, 2001).

Salt Saturated Mud: In this system, the continuous phase, water, is saturated with salt (sodium chloride) usually at 180 mg/L, and mud viscosity is developed with PAC (for filtration) and XC Polymer (for viscosity) and starch is used to control fluid loss. Attapulgite clay can be used for viscosity particle distribution. This system is used for drilling salt sections to balance the formations and avoid wash-outs. This system has a minimum mud weight of 10 ppg (Hussain Rabia, 2001).

It should be noted that the solubility of salt increases with temperature, so the system should be mixed with slightly extra salt to compensate for the increased temperature at downhole conditions. If the drillstring becomes stuck whilst drilling a salt section, spot a fresh water pill across the zone and allow the salt to dissolve.

KCl Polymer Mud: This mud consists of Potassium Chloride (KCl) dissolved in fresh or salt water. Both the potassium and the polymer are used to reduce shale hydration by ion substitution using the potassium ions and encapsulation of the shale by the polymer (Hussain Rabia, 2001).

Potassium chloride is used to inhibit clay hydration. The amount of KCl actually needed for inhibition is difficult to determine. Older formations which contain nonswelling clays, require KCl levels in the 3 to 5 wt% range; whereas, younger shales containing hydratable clays, require KCl levels up to 15 wt%. (Zhang, 2011).

Potassium is a smaller and more highly charged ion than the sodium ion but has a low charge density and is less hydrated than the sodium ion. Hence, the substitution of sodium ions on the shale surface by the potassium ions enable the shale platelets to be closer together and, in addition to this, the potassium ion fits inside the volume of the ion spacing on the clay surface, thereby neutralising the negative charge on the clay surface with a greater strength (Hussain Rabia, 2001).

This results in shale drill cuttings being easier to remove and less contamination in the system. Wellbore stability is also increased by the addition of potassium to mud as a result of creating a non-reactive wellbore ((Hussain Rabia, 2001).

During drilling, the potassium ion is being readily used up on the wellbore and cuttings and further additions of potassium is required to maintain the potassium concentration in the mud system (Hussain Rabia, 2001).

PHPA Muds: PHPA (Partially Hydrolysed Polyacrylamide) is a high molecular weight polymer and is used as a cuttings and wellbore stabiliser. The PHPA molecules bond on clay sites and inhibit the dispersion of solids into the mud system by encapsulating the clay particles. This aides the solids removal process on surface.

The PHPA concentration should be held in excess in the system by 2 to 4 ppb at all times. This system can be used in conjunction with KCl for added inhibition.

Clay inhibition can also be obtained from products like Glycols, Cations and Mixed Metal Hydroxides (Hussain Rabia, 2001).

Dispersed-non-inhibited fluids: do not contain inhibiting ions in the continuous phase, but they do rely on thinners or dispersants such as phosphates, lignosulfonate or lignite to achieve control of the fluids' rheological properties.

Lignite, Lignosulphonate or Phosphate Muds: This is a clay- based fresh water mud which requires high treatment dilution levels while drilling reactive clays. Extra caustic soda needs to be added because of acidic tendencies of system. This is a cheap and easy mud system to maintain, however, it is not common in the oil industry today (Hussain Rabia, 2001).

Inhibited dispersed: contain inhibiting ions such as calcium (Ca2+) or potassium (K+) in the continuous phase and rely on chemical thinners or dispersants to control the fluids rheological properties.

Lime /Gypsum Muds: These muds are built from fresh water but can also be built using seawater. Lime/gypsum muds are often used in areas where shale hydration and swelling result in significant borehole instability. The presence of calcium ions in mud help to stabilise the open shales and prevent sloughing and heaving (Hussain Rabia, 2001).

2.2. Lignite-Lignosulfonate (Deflocculated) Muds

Lignite-Lignosulfonate Muds can be used to drill a variety of formations. They can be weighted up to 18 or 19 lb/gal, provided low-gravity solids (bentonite and drill solids) are in the proper range. As mud density is increased, the bentonite content should be decreased.

The pH range for controlling lignite-lignosulfonate muds is in the 9.5 to 10.5 range. In this range, the magnesium ion is precipitated. Calcium ion should be kept below 200 mg/L. Less than 10,000 mg/L chlorides should not hamper fluid performance, but if chlorides exceed 25,000 mg/L, the mud should be diluted with fresh water.

Lignite-lignosulfonate muds are thermally stable to approximately 325°F. This temperature limit is not well defined and is dependent upon the mud pH, on the type of lignosulfonate used, the length of time exposed to high temperature and the solids content of the mud. Generally, chrome lignosulfonates perform at temperatures higher than lignosulfonates which do not contain chromium. It is significant to note that when lignite-lignosulfonate fluids thermally degrade, carbon dioxide is produced and carbonate ions accumulate in the filtrate (The ChevronTexaco and BP Drilling Fluid Manual, 2002).

2.3. Principal Additives of Lignite/Lignosulfonate (Deflocculated) Muds

Lignite/Lignosulfonate Muds are relatively simple in their makeup, conversion, and maintenance. Table 1 lists additives, functions, and concentrations for a typical mud formulation.

Table 2.1: Additives of Lignite/Lignosulfonate Muds, ChevronTexaco D. F. Manual, 2002).

Principal Additives of Lignite/Lignosulfonate Muds

Additive	Concentration, lb/bbl	Property and Function
Bentonite	10 - 25 Viscosity	Added for viscosity and filtration control
Lignosulfonate	0.25 - 8 Deflocculant	used to control rheology and provide filtration control through deflocculation of the bentonite. improves filtration control by forming a thin, compressible filter cake
Caustic Soda/Caustic Potash (KOH)	for pH 9.5 - 10.5	is added for alkalinity control in a KCl-Polymer Mud rather than caustic soda because it provides pH control without introducing potentially destabilizing sodium ions. Generally, (ZHANG 2011).
Soda Ash (Na2CO3)	0.25 – 1	used to treat out calcium ion hardness and to beneficiate calcium bentonite
Lignite	1 – 4	used as a filtration control agent and as a secondary deflocculant
Barite	as needed for density	used to increase mud density
Low Viscosity PAC/CMC	0.25 - 1.0	Filtration Control
Gilsonite	2 – 8	Filtration Control

2.4. Types of Fluids

There are two basic types of fluids: Newtonian fluids and non-Newtonian fluids .Newtonian fluids are characterized by a constant viscosity at a given temperature and Pressure Common Newtonian fluids include: Water, Diesel, Glycerin and Clear brines (Hussain Rabia , 2001).

Non-Newtonian fluids have viscosities that depend on measured shear rates for a given temperature and pressure. Examples of non-Newtonian fluids include: Most drilling fluids and Cement slurries.

In drilling operations, practically all drilling fluids are non-Newtonian. Even brines which are used as completion fluids are not truly Newtonian fluids, as the dissolved solids in them make them behave in a non-Newtonian manner (Hussain Rabia , 2001).

2.5. Testing Drilling Fluids

Specific physical properties of a drilling fluid must be maintained if the mud is to sufficiently perform its functions. Standard tests are used to evaluate the fluid and determine the condition of the mud. Tests used in this work are described below (Ronald P. Bernhard, 1981).

2.5.1. Density

Of great importance in a drilling operation is the density of mud. It is necessary for controlling the pressure and all pressure control calculations are done based on the weight of the mud column in the hole. This increased density helps to prevent caving and flow into the hole. As density is increased, the buoyancy effect increases carrying capacity for cuttings but decreases settling rate in the mud pit. A mud balance is used to carry out weight measurements on mud samples during drilling operations. During this measurement, precaution must be taken not to include air or entrained gas in the mud sample as this would give a false density measurement, particularly with muds having high yield points or gel strengths (Max R. Annis, 1996).

Therefore, with a degasser or by stirring at an appropriate speed, the entrained gas can be rid off. Temperature and pressure affect density inversely and proportionally in the sense that increased temperature results in decrease in density while increased pressure causes an increase of density. Down hole conditions do not highly affect density, but the effects of increased temperature and pressure oppose each other and tend to equalize (Max R. Annis, 1996).

Drilling fluid density is required to contain the formation and formation fluids hydrostatic pressure can be calculated as

Hydrostatic pressure (psi) = 0.052 x mud weight (ppg) x depth (ft.)
or
Hydrostatic pressure (kg/cm2) = 0.1 x mud weight (sp.gr) x depth (m)
Mud weight is measured with conventional mud balance which consists of a cup and a graduated arm which is balanced horizontally on a knife edge by moving a counter weight i.e rider. The mud weight can directly be read, in different units, from graduated arm (A. K. Mehra et al. 1994).

Then the study will follow on testing the rheological parameters are used to know the following characteristics of the mud.

(1) Ability to suspend and carry cuttings to the surface.
(2) To analyse the effect of drilled solids contaminants, chemicals and temperature.
(3) To calculate surge and swab pressures.

Generally, viscosity is measured with marsh funnel and provides information about any variation in consistency of the mud. Rheological parameters are measured by using Fann V .G Meter which is a direct reading, concentric cylinders rotary device to determine shear stresses at different shear rates i.e. 600 and 300 rpm (A. K. Mehra et al. 1994).

Rheology is the science of deformation and flow of matter. Rheological parameters define the behavior of fluid flow, which directly influences the calculation of head losses in pipes and the transport velocity of the cuttings. By making certain rheological measurements of the fluid, it is possible to determine how the fluid will flow under varied conditions of temperature, pressure and shear rate (Ana Paula Tertuliano Dantas et al, 2013).

2.5.2 Viscosity - Yield Point

- **Apparent Viscosity (AV)**

Apparent viscosity is the viscosity of a fluid, considered Newtonian, at a determined shear rate. Plastic viscosity, in turn, describes the flow features after it has been initiated. In other words, it is the flow resistance caused by friction between the dispersed particles and between the individual molecules of the dispersant liquid. Finally, yield limit is the viscous parameter determined by the interaction forces between dispersed particles (Ana Paula Tertuliano Dantas et al , 2013).

The shear stress divided by the shear rate (at any given rate of shear) is known as effective or apparent viscosity at any given point. It is measured in centipoises (A. K. Mehra et al. 1994).

$$\mathrm{AV} = \theta_{600}/2 \tag{2.1}$$

Determination of viscosity in the samples tested is evaluated by concentric rotary viscometer. When testing a drilling mud the outer sample cup is rotated, which in turn shears the mud. As the mud shears around the " Bob ", it is caused to rotate (except for slight slippage) until the torque in the spring develops a shear stress at the contact of the mud sample and the " Bob ". This shear stress is more than the shear strength of the fluid, laminar flow begins at the surface of the " Bob " and, with a constant rotation maintained, the flow proceeds away from the "bob" until the complete sample is in laminar flow. With continuous rotation at a constant speed, the torque increases in a linear way after the critical torque is achieved, as shown in Figure 3. The critical torque and the slope of the laminar flow line is dependent on the rheological characteristics of the drilling mud. The viscometer used (FANN 35A) follows the designs of Savins and Roper (1954), which is a direct reading viscometer that enables the plastic viscosity and yield point to be calculated very simply from two dial readings, one at 600 RPM and the other at 300 RPM. By theory, Savins and Roper (1954), calculated the plastic viscosity to be the dial reading at 600 RPM minus the dial reading at 300 RPM. The yield point may be calculated by subtracting the plastic viscosity from the 300 RPM. The equations below give plastic viscosity in centipoises and yield point in pounds per 100 square feet.

The apparent viscosity (shear stress divided by shear rate) may also be calculated when the following information is known:

1 dial unit = 5.11 dynes/cm (shear stress)
1 RPM = 1.7033 reciprocal seconds (shear rate) with 300 centipoises per unit per RPM
so:

$$\textbf{apparent viscosity} = \frac{300 \text{ X Units read RPM}}{\text{RPM}} \tag{2.2}$$

the standard method of measuring apparent viscosity is with rotation of 600 RPM so the equation is simplified to:

$$\textbf{apparent viscosity} = \frac{\textbf{units read 600 RPM}}{\textbf{2}} \tag{2.3}$$

- **Plastic Viscosity (PV)**

It is an important mud property that gives a measure of the internal resistance to flow due to amount, type and size of solids in the mud. Due to collision of solids with one another and with the liquid phase of the mud, mechanical friction is produced deterring movement. The plastic viscosity is essentially a function of the viscosity of the liquid phase and the volume of solids contained in a mud. It describes the expected behavior of mud at the bit. In order to minimize high shear rate viscosity, the plastic viscosity has to be minimized. By decreasing the plastic viscosity, a driller correspondingly reduces the viscosity at the bit giving rise to higher ROP (Nwaoboli Awele, 2014).

Although calculated from measurements at relatively low shear rates, the plastic viscosity is an indicator of high shear rate viscosities. Consequently, it tells us something about the expected behavior of the mud at the bit. One of our design criteria was to minimize the high shear rate viscosity. To accomplish this, it should minimize the plastic viscosity. A decrease in plastic viscosity should signal a corresponding decrease in the viscosity at the bit, resulting in higher penetration rate. Increasing the plastic viscosity is not a desirable means of increasing the hole cleaning ability of a mud. In fact, the increase in pressure drop down the drill string, caused by an increase in Plastic Viscosity, would reduce the available flow rate and tend to offset any increase in lifting ability. In general, high plastic viscosity is never desirable and should be maintained as low as practical. However, time, temperature, and agitation tend to disperse and allow hydration of the individual clay platelets, which results in increased viscosities. In order to combat the tendency of shale particles to disperse and hydrate, the "inhibitive" muds were designed. Materials such as lime, gypsum, lignosulfonate, and polymers are added to inhibit the rate of dispersion and hydration.

Plastic viscosity decreases with increasing temperature, due to thinning of water. If the mud is checked at 130°F, the PV will be about 10 percent lower than at 120°F; if it is checked at 110°F, it will be about 10 percent higher. For this reason, all mud tests should be made at the same temperature, 120°F (Uche Osokogwu, 2014).

It is a measure of flow resistance caused by the interaction of solids in the drilling fluids and expressed in centipoise. (A. K. Mehra et al. 1994).

$$PV = \theta_{600} - \theta_{300} \tag{2.4}$$

- **Yield Point (Yp)**

The yield point, calculated from the Bingham equation, is not the true yield stress necessary to maintain flow, but is a value which is somewhat higher. It is normally close to the value of the shear stress at annular shear rates. Anything that causes changes in the low shear rate viscosities will be reflected in the yield point. For this reason, it is a good indicator of flow behavior in the annulus and compositional changes that affect the flow behavior in the annulus. However, as the shear rate is increased, the particles are electrically attracted to one another, the effect is quite similar. At low shear rates the particles link together, increasing the resistance to flow; at high shear rates the linking bonds are broken and the fluid becomes more like water. These two effects combine to determine the yield point of a mud. The electrical interaction of solids is controlled by chemical treatment, and the mechanical interaction is controlled by adjusting the type and amount of solids or polymer in a mud. High yield points are caused by flocculation of clay solids or high concentrations of colloidal solids. Flocculation may be due to lack of sufficient deflocculent, high temperature, or contaminants such as salt, calcium, carbonates, and bicarbonate. A high solids concentration will aggravate flocculation tendencies from any cause. The yield point is primarily associated with two mud functions: the hole cleaning capability and the pressure control characteristic of a mud. A higher yield point increases the carrying capacity of a mud and increases the circulating pressure drop in the annulus. Associated with increased circulating pressure drop is increased pressure surge and swab from pipe movement. (Uche Osokogwu, 2014).

It is a measure of internal resistance caused by electrical environment of the particles and is expressed in Ibs/100 ft2 (A. K. Mehra et al. 1994).

$$Y_P = \theta_{300} - PV \tag{2.5}$$

A graphic translation of the calculations for yield point, plastic viscosity and apparent viscosity is shown in Figure ((2.1) and (2.2)).

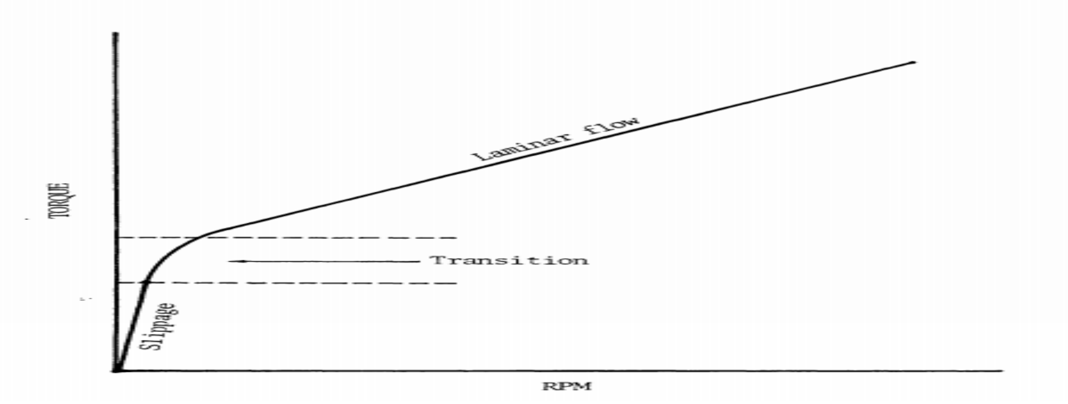


Figure 2.1: Consistency curve in a direct indicating viscometer (Ronald P. Bernhard, 1981).

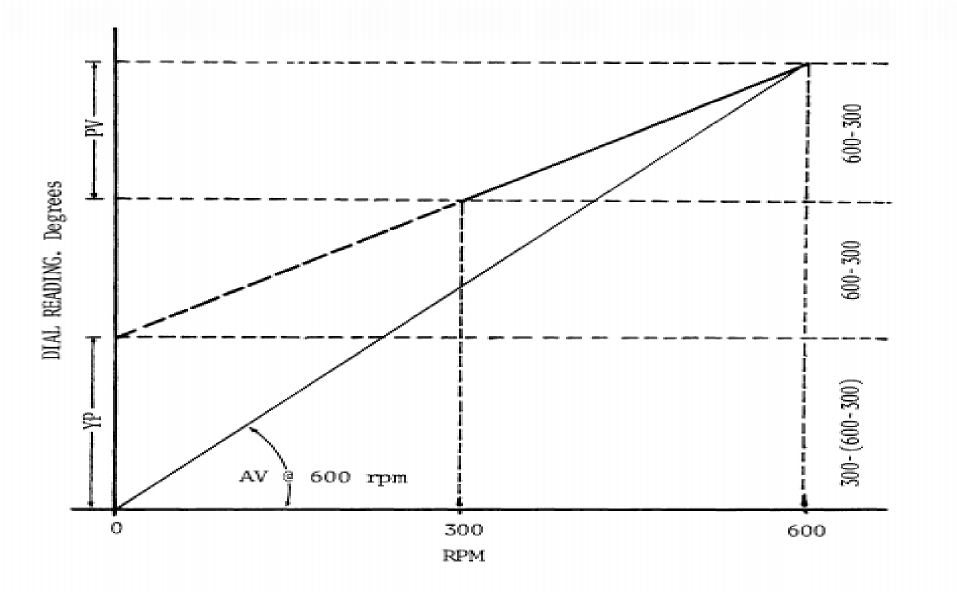


Figure 2.2: Graphic interpretation of determination of flow parameters in a two speed direct indicating viscometer (Ronald P. Bernhard, 1981).

2.5.3 Gel Strength

Gel strength is the measuring of thixotropic properties of a drilling fluid under non-flow conditions, while the yield point measures these properties under flow conditions. However, both the yield point and gel strength deal with the attractive forces between solid particles in the mud (Ronald P. Bernhard, 1981).

Generally, gel strengths are of two types, a weak-fragile gel strength or a strong-progressive gel strength. A weak gel strength seems to be associated with a thin mud and initially has a high gel strength which is very easily broken and will increase only slightly with increase of stagnation time. A strong gel strength is associated with thicker muds and on initial rotation is hard to break. The longer the stagnation time the greater the gel strength becomes (Ronald P. Bernhard, 1981).

Both yield point and gel strength result from flocculation forces and are related to thixotropic properties, so as the yield point decreases the gel strength generally decreases. Gel strengths are determined by the FANN 35A viscometer at 3 RPM. Mud is allowed to stagnate for 10 seconds, then the outer cup is rotated at 3 RPM - by observing the dial on the viscometer the maximum deflection (before the gel breaks), is recorded. The same procedure is repeated after allowing the mud to stand for 10 minutes, the gel strengths are reported in pounds per 100 square feet (Ronald P. Bernhard, 1981).

2.5.4 Filtration

The filtration properties of drilling muds are a measure of the ability of the solid phase of a fluid to form a thin, low-permeability cake of filtered solids. The less permeability the cake has, the thinner the cake will form. This property is dependent on the size, type and volume of colloidal material in the fluid. The loss of fluid from the mud is dependent on permeability of the filter cake, permeability of the formation being drilled and pressures at the bore hole-formation contact. When minimum water loss is maintained, a thinner filter cake forms and drilling problems are minimized. If a thick filter cake develops then the effective size of the bore hole is reduced and various problems are created, such as an increase of torque on the rotating pipe, excessive drag when the pipe is pulled and adherence of the pipe to the wall. Also formation damage may occur due to filtrate and filter cake invasion. In the evaluation of filtrate properties both the low temperature low pressure (LTLP) test and the high temperature high pressure (HTHP) tests are used, because in deep drilling low pressure low temperature tests are misleading (Ronald P. Bernhard, 1981).

2.5.5 Hydrogen Ion Concentration

The hydrogen ion concentration is the reciprocal of the hydrogen ion concentration in grams' moles per liter. To measure pH a glass electrode meter was used. The meter consists of: 1) a glass electrode made of a thin-walled bulb of special glass; 2) a reference electrode consisting of a saturated calonel cell; 3) an amplifier, for amplifying the potential difference between the mud sample and the glass electrode; 4) a meter reading in pH units; and 5) a standard buffer solution for instrument calibration. The effect of pH on muds is influential on clay dispersion because of its effects on base exchange equilibrium, but the electro-chemical conditions in a system vary from fluid to fluid, so the effect of changing pH also will vary. Systems with high pH filtrate may dissolve formation cements such as amorphous silica, releasing fine particles which may block pores, eventually causing impermeability (Ronald P. Bernhard, 1981).

2.5.6. Sand Content

The amount of sand particles larger than 200 mesh size are measured through standard API sand apparatus. In this test, a fixed volume of mud is diluted and passed through 200 mesh sieve. The residue left gives the measure of sand content in volume percentage of sand in mud (A. K. Mehra et al. 1994).

2.6. Carboxyl-methyl Cellulose (Na) (CMC)

Carboxy methyle cellulose is one of the number of Free hydroxyle groups. most important cellulose derivatives which obtained by a chemical modification of natural cellulose (Bono, et al ,2009). CMC is a polymer derived from cellulose which is insoluble in water but becomes soluble when modified to the form of a polyelectrolyte (Ana Paula Tertuliano Dantas et al , 2013).

CMC is generally prepared by the reaction of Alkali cellulose with mono choloro acetic acid, by reacting the free hydroxyl group in the Anhydro glucose units (AGU) with various chemical substitution groups, the introduction of substituent disturb the inter molecular and intra molecular hydrogen bonds in cellulose ,which leads to liberation of hydrophilic character of numerous hydroxyle groups and restriction of the chains to closely associate (Togrual and Arslan, 2003), Howver substitution with Alkyl groups reduces the 3, HO-6) in the Anhdro glucose unit can be substituted and the maximum degree of substitution (DS) is being 3 (salami et al,1994).

Carboxymethylation of poly saccharide is a widely studied conversation since it is simple and leads to products with a variety of promising properties. In general, the poly saccharide is activated with aqueous alkali hydroxide mostly sodium hydroxide and converted with mono choloro acetic acid or it is sodium salt.

It is important to note that, the carboxy methyl cellulose has an acidic function, meaning that (CMC) is an anionic poly electrolyte, CMC has many interesting properties when dissolved in aqueous solution, but this will depend on the CMC grade and the solution condition.

2.6.1. CMC in drilling fluid and chemical structure

The CMC is used in water based drilling fluids as a filtrate reducer, to reduce losses by filtration and produce very thin filter cakes that are capable of preventing fluid flowing through the geological formations, also used as viscosifier in drilling fluid mud. The filter loss decreased with CMC increase (Abusabah E. Elemam et al, 2014).

The effect of increasing bentonite percentage (solid phase) on rheological properties and filtration loss for water base drilling fluid prepared from local bentonite (Umm Ali area, SUDAN). Chemical tests were carried out to assess quality of local bentonite. In that study Carboxymethyle Cellulose CMC is used to increase viscosity and decrease filtration loss. The major finding showed that when adding 7% of CMC and 7% of local bentonite, the rheological properties, filter loss and yield point to plastic viscosity ratio were within the acceptable range of American Petroleum Institute (API) specifications (Rashid A. M. Hussein et al, 2014).

Hydroxy propyl methylcellulose (HPMC) polymer as a viscosifying agent in cement slurry. The HPMC possesses a strong thermal thickening and solid suspension ability which means low viscosity at ambient temperature and viscosity increase with respect to temperature. Thus, a series of experiments was conducted on HPMC polymer as a viscosifying agent in cement slurry. Different concentrated solutions were prepared (i.e. 1.5 to 2.5 wt%), in order to characterize their viscosity with respect to shear rate and increasing temperature up to 100°C. Then, cement slurries were prepared with HPMC as the primary viscosifier, to determine the rheological properties at high temperatures. It was observed that HPMC polymer has increased viscosity from 90°C to 100°C. The improved rheology of cement slurry, in terms of plastic viscosity and yield point at different concentrations, revealed HPMC as a capable viscosifying agent at high temperature. Moreover, increased gel strength with increasing concentration was also observed, which may cause a higher pumping rate on the surface. It was concluded that HPMC polymer acts as a viscosifying agent at high temperature. HPMC-based slurries showed high rheology (plastic viscosity and yield point) with HPMC polymer. Thus, the use of this additional additive should be considered in the design of appropriate cement slurry (Ghulam Abbas et al, 2013).

The polymer is a cellulose derivative obtained by chemical reaction of alkali cellulose with Sodium Mono choloro acetate under controlled condition. The chain length of a typical CMC used in drilling fluid is normally between 500-5000 glucose units. The properties of CMC solutions are thickening, emulsifying stabilizing, membrane shaping and dispersing regarding to other solutions (Reza Mirzai, 2015). The salt tolerance is affected by DS (degree of substitution) of the polymer. The affectivity of the CMC based drilling fluid decreases when the concentration of salt exceeds 50000 ppm (part per million). The more the DS factor of the polymer, the more tolerance it has for salts and Ca2+. For solubility in water the DS factor must be higher than 0.45. The range of DS factor of CMC's is normally 0.4-0.8 making them water soluble. The viscosity provided by CMSs depends on the length of their chain and also on DP (degree of polymerization). The longer the chain the more viscous property it provides for the mud system.

Molecular Formula: [C6H7O2(OH)2CH2COONa]n

Structural Formula is shown in Figure 2.3.

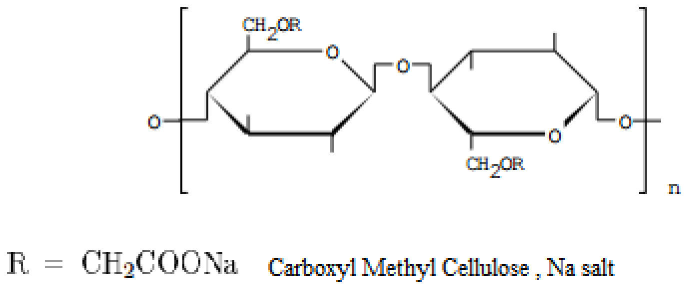


Figure 2.3: Chemical Structure of Sodium Carboxyl Methyl Cellulose (Reza Mirzai, 2015).

2.6.2. Carboxy methyl Cellulose Sodium (CMC)-Oil Drilling Grade

Molecular Formula: $[C_6H_7O_2(OH)_2CH_2COONa]n$

Structural Formula:

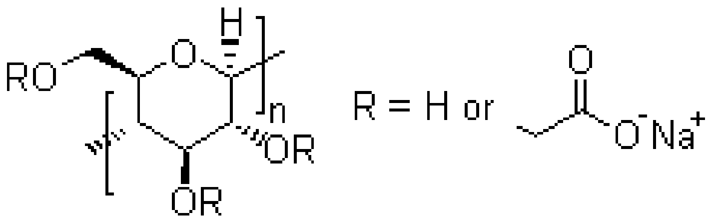


Figure 2.4: Structural Formula for CMC -Oil Drilling Grade (Trustin Chemical, 2019)

Specification:

Table ((2.2) and (2.3)) shown the Physical Specification and Technical Specification of **CMC** for Oil drilling liquids .

Table 2.2: Physical Specification of CMC for oil drilling liquids (Trustin Chemical, 2019)

	LV-CMC	MV-CMC	HV-CMC
Appearance	White or light yellow powder, freely flowable		
Water,%	10 max		
CMC,%	80.0 min	85.0 min	95.0 min
Substitution degree,%	0.80 min	0.65 min	0.8 min
PH	7.0-9.0	7.0-9.0	6.5-8.0

Type	Item		Typical measured values		
			Distilled water	Brine	Saturated brine
Base mud	Filtration loss, ml		60±10	90±10	100±10
	Apparent viscosity, mPa•s		6 max	6 max	10 max
	PH		8.0±1.0	8.0±1.0	7.5±1.0
HV-CMC	Mud generating capacity, m3 /t		200 min	150 min	160 min
LV-CMC	Filtration loss = 10ml	Addition rate, g/l	--	7.0 max	10.0 max
		Apparent viscositymPa•s	--	4.0 max	6.0 max
MV-CMC	Addition rate,g/l		6.0	14.0	3.5
	Apparent viscosity,mPa•s		15 min	15 min	15 min
	Filtration loss,ml		9 max	9 max	9 max

Table 2.3: Technical Specification of CMC for oil drilling liquids (Trustin Chemical, 2019)

Type	Specification		
CMC-HVT	Viscometer 600r/min	Distilled water	30.0 min
		40g/dm3 brine	30.0 min
		Saturated brine	30.0 min
	Filtration Loss (ml)		10.0 max
CMC-LVT	Viscometer 600r/min		90.0 max
	Filtration Loss (ml)		10.0 max

2.6.3. CMC Types

2.6.3.1 High-Viscosity CMC (CMC-HVT)

High-viscosity carboxymethylcellulose (CMC HVT) is an alkali metal salt of carboxymethylcellulose. Table (2.4) shows physical specification of CMCHVT according to API.

CMC-HVT shall be free from any starch or starch derivatives. Therefore, a qualitative starch determination shall be performed before proceeding with the CMC-HVT performance testing. If starch is found, no further testing should be performed and the sample shall be rejected.

Table 2.4 : CMC-HVT Physical Specifications (API Specification 13A 18th Edition, August 2010)

Requirement	Standard
Starch or starch derivates presence	No
Solution properties:	
Viscometer dial reading at 600 r/min	
- in deionized water	Minimum 30
- in 40 g/l salt solution	Minimum 30
- in saturated salt water	Minimum 30
Filtrate volume, millilitres	Maximum 10,0

2.6.3.2 Low-Viscosity CMC (CMC-LVT)

Technical-grade low-viscosity carboxymethylcellulose (CMCLVT) is an alkali metal salt of carboxymethylcellulose.

CMC-LVT shall be free of any starch or starch derivatives. Therefore, a qualitative starch determination shall be performed before proceeding with the CMC-LVT performance testing. If starch is found, no further testing should be performed and the sample shall be rejected. Table (2.5) shows physical specification of CMCHVT according to API.

Table 2.5: CMC-LVT Physical Specifications (API Specification 13A 18th Edition, August 2010)

Requirement	**Standard**
Starch or starch derivates presence	No
Solution properties	
Viscometer dial reading at 600 r/min	Maximum 90
Filtrate volume, millilitres	Maximum 10

2.6.4. Rheology of CMC suspension

Many studies have been done on CMC rheological properties (Hafez Balavi, 2017). It was reported that Newtonian behavior at the lowest concentration and pseudo plastic, thixotropic behavior at the higher-end concentration were observed in the concentration range 1-5%. By increasing CMC concentration at a constant shear rate, the apparent viscosity increases. This is because of the increase in the intermolecular interactions between CMC molecules. The rheological behaviour of CMC suspensions can be expressed by Cross model as follows (Hafez Balavi, 2017) :

$$(\mu - \mu_\infty)/(\mu_0 - \mu_\infty) = 1/(1 + (\lambda_c.\gamma)_n) \qquad (2.6)$$

where μ is the viscosity at any shear rate γ, $\mu 0$ and $\mu\infty$ are viscosities at zero and infinite shear rates, respectively, λc is a time constant with the dimensions of time and n is the degree of dependence of viscosity on shear rate (flow behavior index). As “n” increases, CMC suspension flow behavior tends to more shear thinning manner.

CMC suspensions are strongly time-dependent materials, so they are strongly thixotropic. This dependency is due to the changes occurring in the inner structure of the fluid caused by particle interaction forces such as van der Waals forces. These forces act in microscopic scale and lead to the formation of a rigid continuous particle network which can resist the flow (Hafez Balavi, 2017).

2.6.5. Rheology of bentonite-CMC suspension

The addition of cellulose-based additives to clay suspensions has been taken into account significantly due to their colloidal and rheology modifying capacity. Their applications in cosmetics, chemical paints, food products, and specifically in water-based drilling fluids make them interesting materials to work with (Hafez Balavi, 2017). They enable clay-based drilling mud to fulfill its functions such as stabilizing the borehole (cake formation), cleaning the hole (evacuating the cuttings), and cooling and lubricating the string and the bit. Among many cellulose-based additives, CMC is a great material for increasing the viscosity, controlling the mud fluid loss, and maintaining enough flow properties at high temperature-high pressure (HPHT) condition.

The effect of CMC addition on the rheological properties of bentonite clay suspensions has been studied and experimented thoroughly (K. Benyounes et al, 2010 and A. Benchabane et al, 2006). An increase in apparent viscosity is observed by increase in CMC concentration. This is because of more polymer chain entanglements due to concentration increment (Hafez Balavi, 2017).

This chapter covered a number of topics related to research subject of the study and after reviewing the previous studies of a number of scientific papers and references, the research methodology was developed and will be discussed in detail in chapter three which including methodology steps.

CHAPTER THREE

METHODOLOGY

CHAPTER 3

Methodology

This chapter will introduce the methodology that was followed to achieve the current work. The objectives of this work were achieved through the research methodology by collecting commercial and extracted local CMC and then measuring the rheological properties at drilling fluid laboratory, conduct XRD (x-ray diffraction) test and study the flow behavior for both commercial and local CMC using Modified power low model. Finally, study is it economy to producing CMC locally.

3.1 CMC Collection

3.1.1 Commercial CMC

AS a result of the lack of commercial product in the local market, the process of collecting the commercial product was made by contacting China, and then a one kilo gram were purchased to carry out the laboratory experiments and compare it with a local product. The transfer of the commercial product from China to Sudan was coordinated through air flight with the cooperation of some friends in China (Figure (3.1)).

Figure 3.1: Commercial CMC- HV

3.1.2 Local CMC Extraction

The palm frond fibers raw materials collected from Khartoum state (Sudan), washed and air-dried then ground used Electrical grinder. For Extraction of cellulose from palm frond fibers The sample was soaked with suitable quantity of water in 1L beaker for about 20 minutes then decanted after that. The palm frond fibers were cooked in 1 M (4%NaOH), (500 mL) for 4 hours at 80ºC several times and washed with distilled water. The resulting product bleached with white Clorox solution (contains1.7%NaOCl) and (Sodium hydroxide 2.5% and acetic acid 75ml) for 4 hours at 80ºC four times. And washed with distilled water several times until the order of hypochlorite could no longer be detected and dried at room temperature.

A quantity of 2 grams of extracted cellulose was suspended in 40ml of (iso-propanol, n-Butanol, Ethanol, Methanol) and stirred continuously. 10mL of aqueous NaOH solution (30%) were added drop wise over a period of 30min. The mixture was stirred for one hour at room temperature. 6.00g.Of mono chloro acetic acid were dissolved in 10mL of solvent (iso propanol, Ethanol), and were added to the alkalized cellulose, the temperature was a raised to 55ºC. The reaction was allowed to continue for (4hours) then neutralized with 90% acetic acid and filtered. The obtained CMC solid was washed by 70% ethanol five times to remove undesirable by products then dried at 60ºC in an oven.

For purification, the dried CMC was dispersed on 60mL of 95% ethanol and stirred for 5minutes. Then 10 mL of 2M nitric acid were added and the mixture was agitated for 2min. The mixture was then heated to boiling for 5min and agitated further for 15min and left to settle. After the solution had settled, the supernatant liquid was filtered and discarded. The precipitate was washed with 80Ml of 95% ethanol and further wash was applied with hot 80% ethanol at 60ºC, until the acid and salts were removed. The precipitate was washed with methanol and transferred to a beaker and heated until the alcohol was removed. The beaker with the precipitate was dried in the oven at 105ºC for three hours, same as Figure (3.2).

Figure 3.2: Local CMC Extraction

3.2. Materials and Methods

Three different types of bentonite were tested in drilling fluid laboratory using YM-2 mud balance for mud weight measurement, PH papers for PH measurements, ZNN-D6 Six speed rotation viscometer to quantify the rheological properties of the drilling fluids at room temperature and atmospheric pressure, ZNS-4 Inflator air supply water loss meter to investigate filtrate volume and mud cake of drilling fluids under a pressure of 0.69Mpa and room temperature for 30 minutes' period. Also XRD test was carried out for both commercial, local produced CMC and three types of bentonite after that select Pure Indian bentonite and used it.

Commercial CMC-HV was added with a different percentage (from o.5% to 8%) to 22.5g bentonite in 350ml deionized water.

For local produced CMC was added with a different percentage (from 5% to 25%) to 22.5g bentonite in 350ml deionized water.

Filtrate volume, mud cake and rheological properties measurements were carried out under constant pressure (0.69MPa) and different temperature conditions (25ºC to 90°C) each temperature 30 minutes using GGS-71 HTHP water loss meter.

3.3. Laboratory Samples Preparation

- For pure bentonite without additives (Table (3.1) and Appendix (A)): 350 Milliliters of deionized water was measured and poured into the mixing cup with 22.5grammes of bentonite and was rehydrated for 5minutes under stirring condition using D90-Mixing mud machine.
- For pure CMC (Table (3.2)):350Milliliters of deionized water was measured and poured into the mixing cup with 10.5grammes of CMC and was rehydrated for 20 minutes under stirring condition using D90-Mixing mud machine.
- For the mixture of bentonite – CMC:350 Milliliters of deionized water was measured and poured into the mixing cup,5% of CMC was added to 22.5 grams of bentonite and then was rehydrated for 20 minutes under stirring condition using D90-Mixing mud machine. This process was repeated with different concentration of CMC as shown in table (3.1).

The first sample (22.5g bentonite, 350ml distilled water and 0.1g CMC) was put in a mixer and mixed well for five minutes. After that, a quantity of a well-mixed sample was taken to determine its density using the Mud balance. Then the sample was put in the HPHT device at a starting temperature of 50° Centigrade, at a pressure of 0.69MPa; for thirty minutes. And determining the volume of filtration loss for thirty minutes. After that the filtration paper was taken to measure the mud cake thickness. After that, the speeds were measured (Ө300, Ө600, Ө3, Ө6, Ө100, Ө200, Ө3@10sec, Ө3@10min) using ZNN-D6 Six speed rotation viscometer. Next, the pH of the sample was measured with the pH paper.

The speeds were used to calculate the apparent viscosity (AV), the plastic viscosity (PV), the yield point (YP) and the gel strength using the following equations:

$AV = \theta600/2$ (3.1)

$PV = \theta600 - \theta300$ (3.2)

$Yp = \theta300 - PV$ (3.3) Gel Strength = $\theta3\ 10sec / \theta3\ 10min$ (3.4)

The above procedures were repeated for other quantities of the samples (from sample No.2 to No.5) and at different temperatures (25ºC, 50ºC, 75ºC, 90ºC, 100ºC and 110°C) as below table (3.1).

Table 3.1: Two Types of CMC Samples Weighting.

	Deionized water (ml)	**Bentonite (g)**	**Commercial CMC (g)**	**Laboratory CMC (g)**
Sample 1	350	22.5	0.1	1.1
Sample 2	350	22.5	0.5	2.3
Sample 3	350	22.5	1	3.4
Sample 4	350	22.5	1.5	4.5
Sample 5	350	22.5	2	5.6

All these procedures were followed for both commercial and local extracted CMC.

3.4.X-ray Diffraction Test

3.4.1. Identification of Clays Using XRD

Basal reflections give d-spacing of the basal layer which represent the thickness of the silicate layers and the unit cell often contains multiple layers. Clay mineral peaks are generally being distinguished by the width halfway up the peak (i.e. the full width at half maximum, FWHM). Well-defined crystalline minerals have sharp peaks while clays, which range from crystalline to non-crystalline, produce broad peaks with noticeable width on both sides. These broad peaks make it easy to pick out which peaks are contributed by clays. These peaks can be compared to known diffraction patterns for better identification but if some peaks are broader than others, it is likely that multiple clays are present. The Clay Mineral Society maintains a collection clays for the purpose of comparison to unknown clays. Because the majority of the clays available from the Clay Mineral Society are naturally formed, they can contain minerals other than the desired clay. Diffraction patterns calculated using theoretical methods do not generally match with experimental diffraction patterns, so using diffraction patterns from known samples to help identify a clay is preferable to calculation. Some minerals can be eliminated from identification using background information or prior analysis.

Well-crystallized and pure samples are ideal for x-ray diffraction, but this is rarely the case for clay. Clay minerals are almost always mixed with very small amounts of non-clay minerals which can produce intense peaks, even when there is very little of the sample is not a clay. If additional minerals are known to be presents, attempts should be made to separate clays from non-clays otherwise additional peaks should be expected. Some common minerals associated with naturally occurring clays are; quartz, feldspars, zeolites, and carbonates organic matter is sometimes present. Synthesis of clays can reduce the presence of some of these associated materials but does not guarantee pure samples as quartz or other associated materials are still commonly produced alongside synthetic clays.

3.4.2. Preparation for Clay Mineral X-ray Diffraction

Clays should be separated from the non-clay minerals to reduce interference of peaks. Non clay minerals can usually be separated by sieving samples at a small enough mesh. Samples should be lightly crushed but not pulverized because non clay minerals will be reduced to clay sizes and become impossible to separate from the sample. Lightly crushing breaks a part the soft clays while keeping harder non clays intact for easier removal.

Samples should be as homogeneous as possible, both in grain size and composition before mounting them for X-ray diffraction and long, flat, and thick samples are ideal. glass slide method used for sample preparation, A glass microscope slide is covered with a suspension of sample in water then placed in and left to dry at room temperature.

3.4.3. Preparation for Bulk Samples X-ray Diffraction

Used Smear Method it is a quick method that is good identifying bulk sample constituents. The sample is crushed with a mortar and pestle until the powder is able to be brushed onto a glass slide. The powder is then mixed with a few drops of a dispersant solution, usually ethanol but others are available, and spread evenly over the slide. Both large and small grain size fractions can utilize this method.

3.4.4. Philips X'Pert 1 X-ray Diffractometer

It is a versatile device to characterise crystalline materials. It is capable of performing various types of measurements and analyses. The diffractometer has a stationary, centrally placed, X-ray tube with (para focusing) line focus and (parallel beam) point focus employing two goniometers for each type of focus. The line focus is mainly used for general diffraction work and phase analysis. Furthermore, it is used for determination of stresses in (sub) surface layers and line profile analysis. The point focus is mainly used for texture measurements. All measurements are done at room temperature. An oven is available to perform measurements at elevated temperatures under gas protection if required.

3.5. Rheological Models

The six-speed viscometer consisting of 3,6,100,200,300 and 600rpm is normally used for higher quality rheology control. To determine rheology constants. This study was used The Modified Power Law Model (Herschel Buckley model).

This model is as a result of the combined theoretical and practical aspects of both the Bingham Plastic and the Power Law models. It is also known as the Yield Power Law or Herschel-Buckley Model. The values of “n and K” are similar to those derived using the Power Law model. Assumptions made by this model are that a certain amount of stress must be applied to fluids to initiate movement and for these fluids having a yield stress, the calculated values of “n and K” will be different.

For Modified Power Law fluids, the relationship exists

$\tau = \tau_0 + k(\gamma)^n$ (3.5)

Where

K= consistency index

n =flow behavior index

τ_0 = the fluid´s yield point at zero shear rate. In theory, this value is similar to the Bingham Plastic yield point, though it is calculated value is different.

With n = 1, the Bingham Plastic Model is formed.

With τ_0 = 0, the Power Law Model is derived.

The Modified Power Law Model is very suitable for both water and oil based drilling muds because both exhibit shear thinning behavior and have a shear stress at zero shear rate. It is however, very complex to find the n, K and τ_0 values.

In this research, the above-mentioned methodology was followed, as well as the tools that will be mentioned later in Appendix A were used, to reach the results that will be enumerated in the next Chapter Four results and discussion.

CHAPTER FOUR

RESULTS AND DISCUSSION

CHAPTER 4

Results and Discussion

4.1. Bentonite Rheological Tests

physical and rheological tests were conducted for three types of bentonite (A, B and C) and selected bentonite type B to be used in the drilling fluid that to study the effect of adding CMC to drilling fluid to improve the properties of drilling fluid, Table (4.1) is including the results of rheological tests for the three samples.

By adding 22.5g Pure Bentonite (P.bentonite) to 350ML (deionized water) and mixed it for 5 mints then took Viscometer dial reading (**ϴ** @ 3, 6, 100, 200, 300 and 600rpm). PV using equation (3.2) and YP using equation (3.3), were calculated. then measured mud weight and PH. Filtration volume for 7.5mints and took mud cake thickness. Depending on American Petroleum Institute (API) standards all tests were carried out.

Table 4.1: Pure Bentonite Samples (A, B and C) Rheological Properties.

	Bentonite (C)	Bentonite (B)	Bentonite A
ϴ3	5	7	5
ϴ6	7	10	10
ϴ100	10	13	16
ϴ200	12	16	21
ϴ300	15	18	27
ϴ600	24	25	37
PV	9	7	10
YP	6	11	17
Gel Strength, 10sec / 10min	5/34	9/35	5/37
Density	8.7	8.65	8.7
Viscosity-marsh funnel	36 sec	42 sec	47 sec
PH	8	8	8
Filtration volume	14	18.8	15

4.2. XRD Tests for Bentonite Types and CMC Types

4.2.1. XRD Tests for Bentonite Types

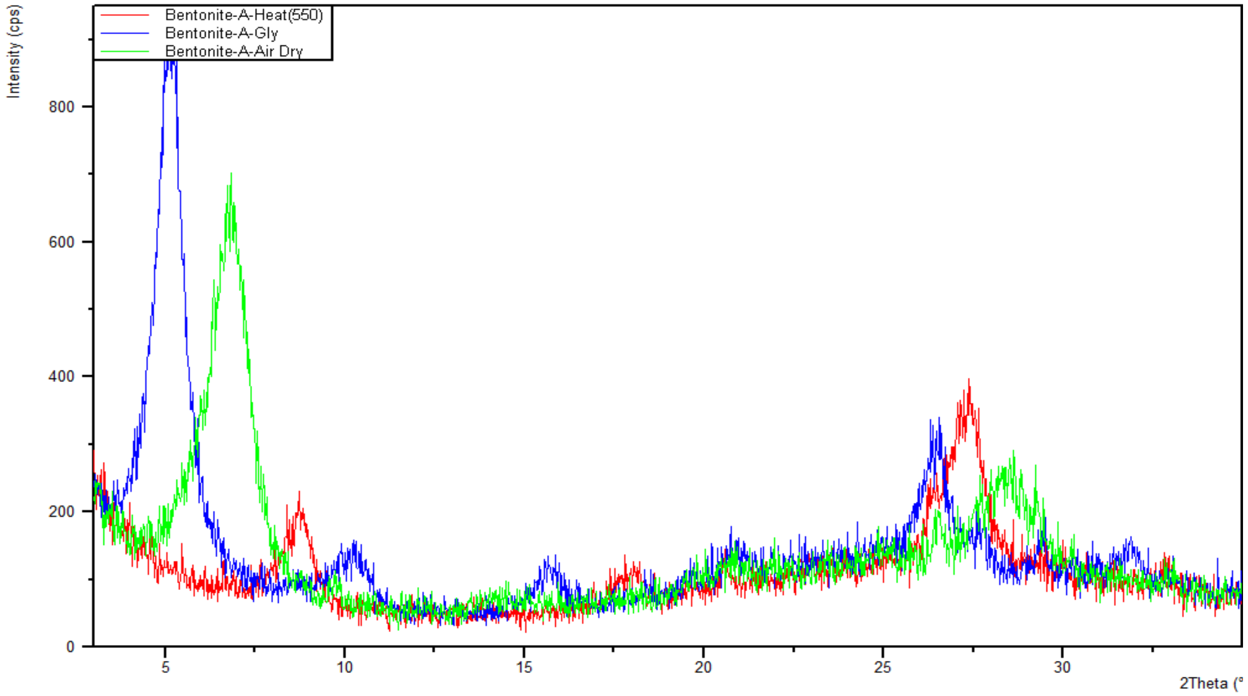


Figure 4.1: Bentonite Type A XRD Patten.

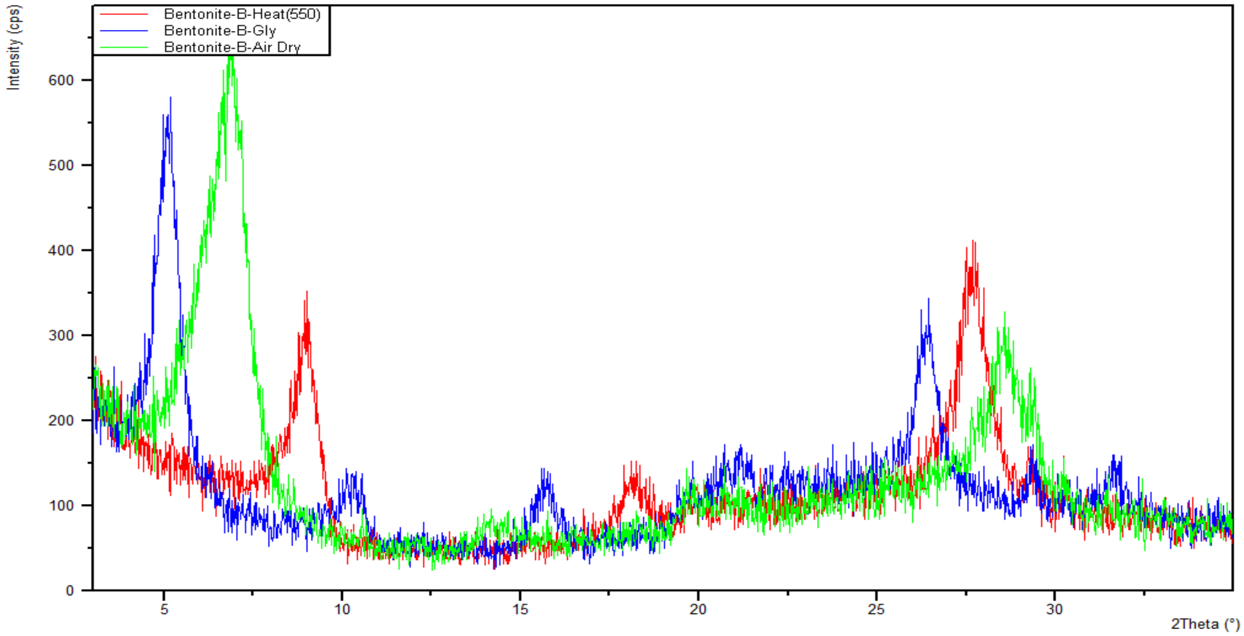


Figure 4.2: Bentonite Type B XRD Patten.

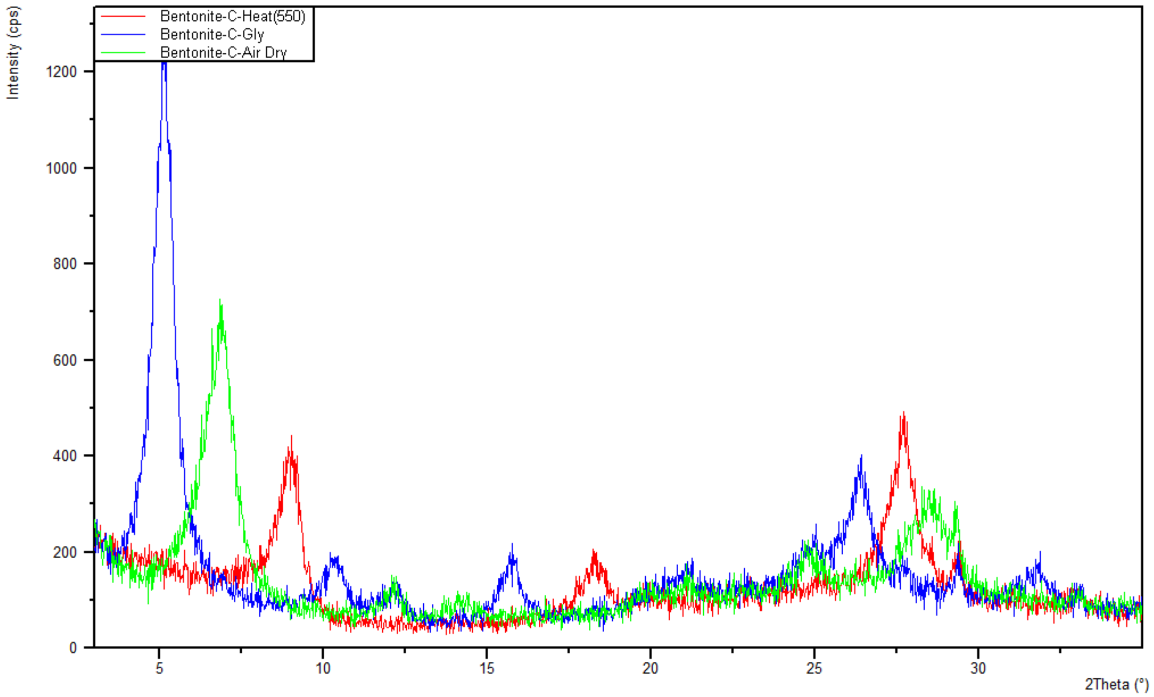


Figure 4.3: Bentonite Type C XRD Patten.

Figures ((4.1), (4.2) and (4.3)) showed XRD test results for the three samples of bentonite, and the results of XRD test and report (Appendix (B)) proved that three types of samples are bentonite and followed the same behavior as montmorillonite family.

4.2.2. XRD Tests for Commercial and Laboratory Produced CMC

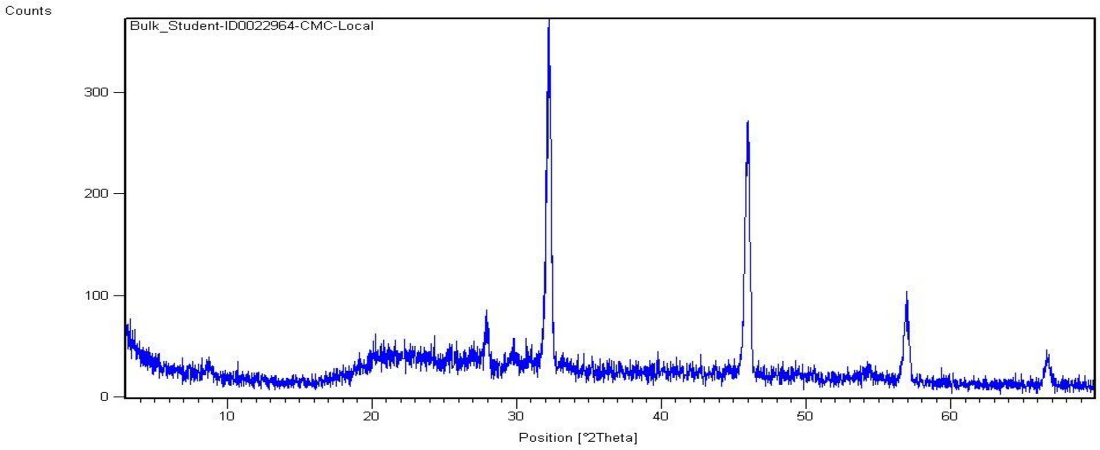


Figure 4.4: Laboratory Produced (Local product) CMC XRD Patten.

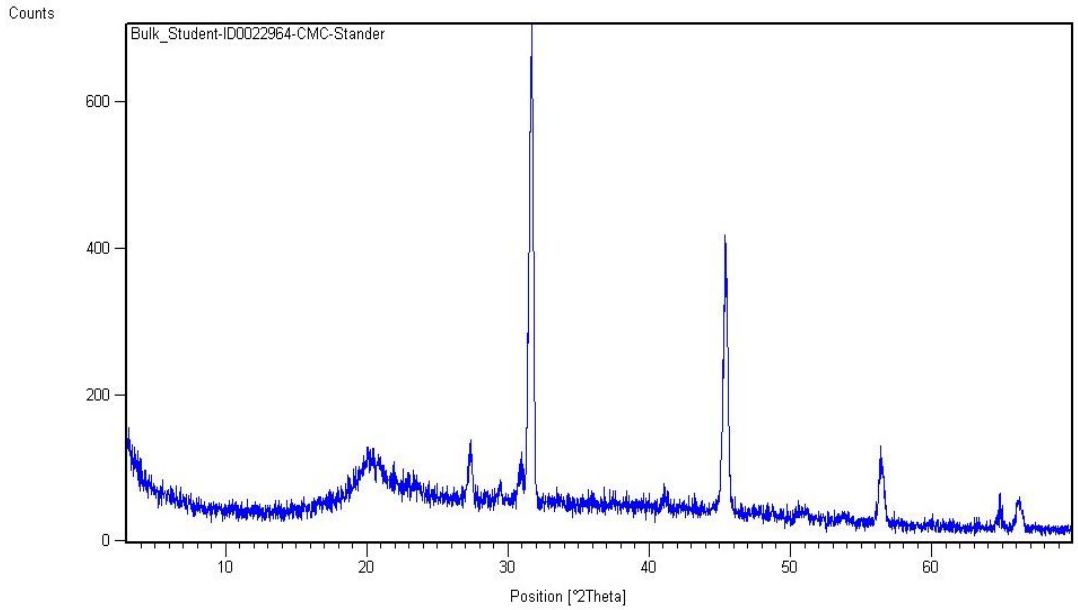
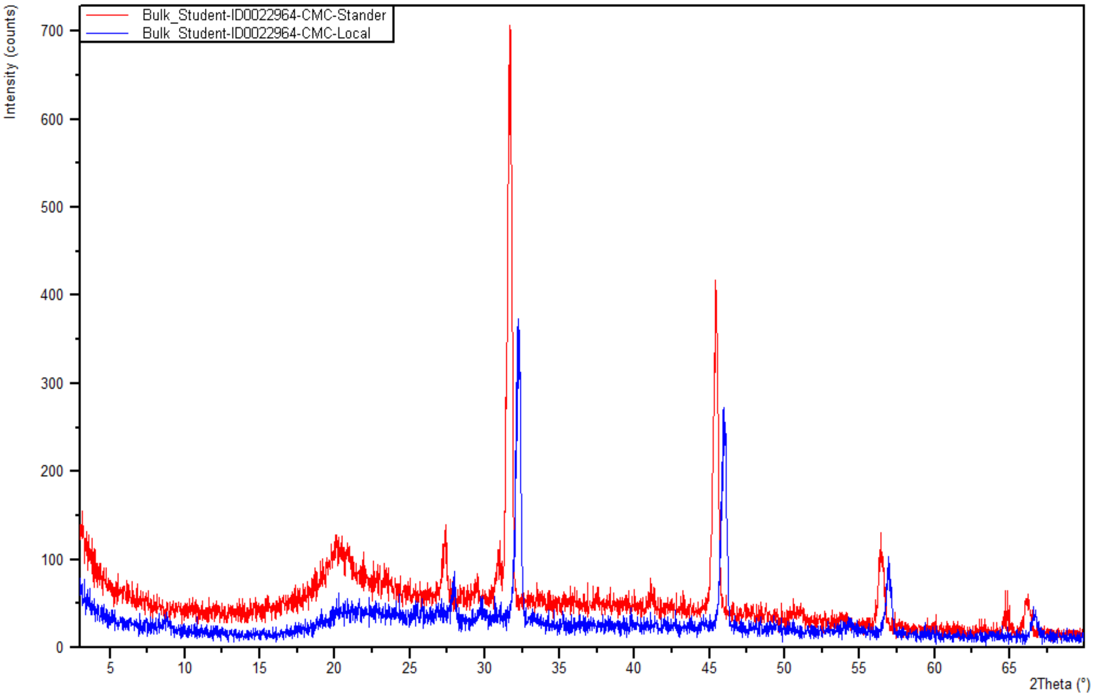


Figure 4.5: Stander Produced CMC XRD Patten.



4.6: Laboratory Produced and Stander CMC XRD Patten.

Figures: ((4.4) and (4.5)) and appendix (B) showed the XRD tests result for commercial (C.CMC) and laboratory produced (L.CMC). Figure (4.6) contained the both types of CMC and as per result of tests for both samples of commercial and laboratory CMC it is clear that laboratory produced CMC Patten as same as commercial CMC.

4.3. Laboratory Experiments

4.3.1. Rheological Tests for Commercial CMC

4.3.1.1. Pure Commercial CMC (C.CMC)

Added 2.2g of pure commercial CMC to 350ML (deionized water) and mixed for 20 min (as per API specification 13A/ISO 13500) then took Viscometer dial reading at 600 rpm (ϴ600) and that to determine the type of CMC. CMC Viscometer dial reading at 600 rpm in the range of high viscosity CMC (H.V) as per test result which it was mentioned in table (4.2).

Table 4.2: Six speed viscometer, ϴ600 reading for pure commercial CMC.

	Commercial	CMC H.V API Specs
	CMC	
ϴ600	37	Minimum 30

4.3.1.2. Effect of Adding Commercial CMC to Water Base Mud

Water Base Mud Formulation:

Mud type: Gel Mud - Bentonite: 22.5 g - Water: 350 ML

Add weight of CMC (0.1g (0.44%), 0.5g (2.22%), 1.0g (4.44%), 1.5g (6.67%) and 2.0g (8.89%)) to 22.5g pure Bentonite in 350ML (deionized water) and mixing for 20min then Viscometer dial reading at 3, 6, 100, 200, 300 and 600rpm after that mud weight, PH, filtration volume and mud cake thickness weere measured.

Table 4.3: P.bentonite and Bentonite with C.CMC Concentration Properties @25℃.

CMC %	Viscometer dial reading at 600 rpm	Filtration Volume (ml)	Plastic Viscosity (PV)	Yield Point (YP)
0	25	19	7	11
0.44	50	16.8	12	34
2.22	73	13.2	18	37
4.44	115	11.2	23	69
6.67	162	9.6	27	108
8.89	197	8.8	28	141

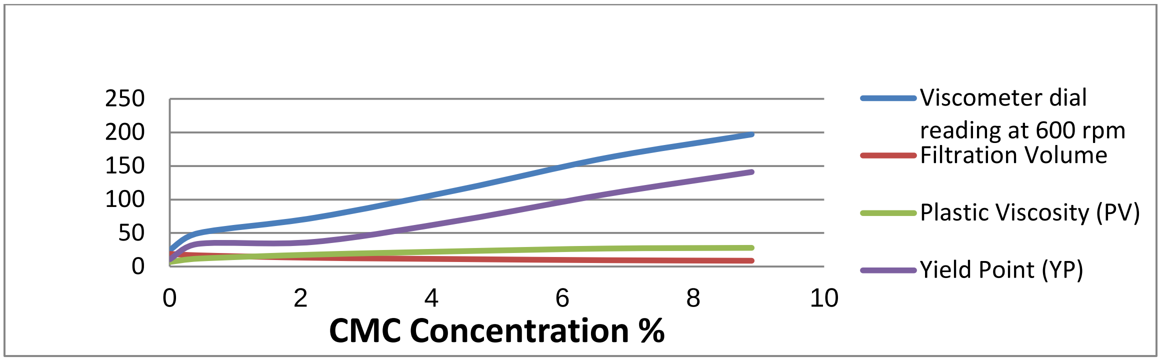


Figure 4.7 : **C.CMC Concentration VS Ө600, Filtration Volume, PV and YP @ 25℃.**

Table (4.3) and figure(4.7) showed that Viscometer dial reading at 600rpm, Plastic Viscosity (PV) and Yield Point (YP) increased by the increased of CMC Concentration, Filtration Volume Decreased by the increased of CMC Concentration, all tests carried out at **25**℃.

Table 4.4: P.bentonite and Bentonite with C.CMC Concentration Properties @50℃.

CMC %	Viscometer dial reading at 600 rpm	Filtration Volume (ml)	Plastic Viscosity (PV)	Yield Point (YP)
0.44	86	12	14	58
2.22	98	10	14	70
4.44	132	7	13	106
6.67	175	6	20	135
8.89	227	6	17	193

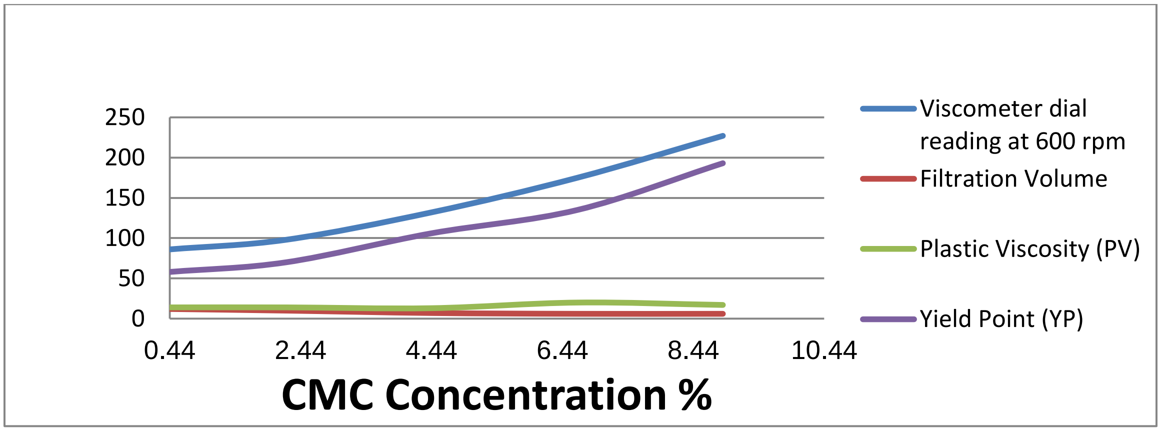


Figure 4.8 : **C.CMC Concentration VS Ө600, Filtration Volume, PV and YP @ 50℃.**

Table (4.4) and figure (4.8) showed the Viscometer dial reading at 600rpm and yield point increased by increased of CMC Concentration, Filtration Volume also still decreased by increased CMC Concentration at **50**℃,temperature affected in Plastic Viscosity (PV) became unstable some by increased CMC Concentration.

Table 4.5: P.bentonite and Bentonite with C.CMC Concentration Properties @75℃.

CMC %	Viscometer dial reading at 600 rpm	Filtration Volume (ml)	Plastic Viscosity (PV)	Yield Point (YP)
0.44	107	10	11	85
2.22	142	9.6	26	90
4.44	119	8.6	11	97
6.67	160	7.6	15	130
8.89	192	7.4	16	160

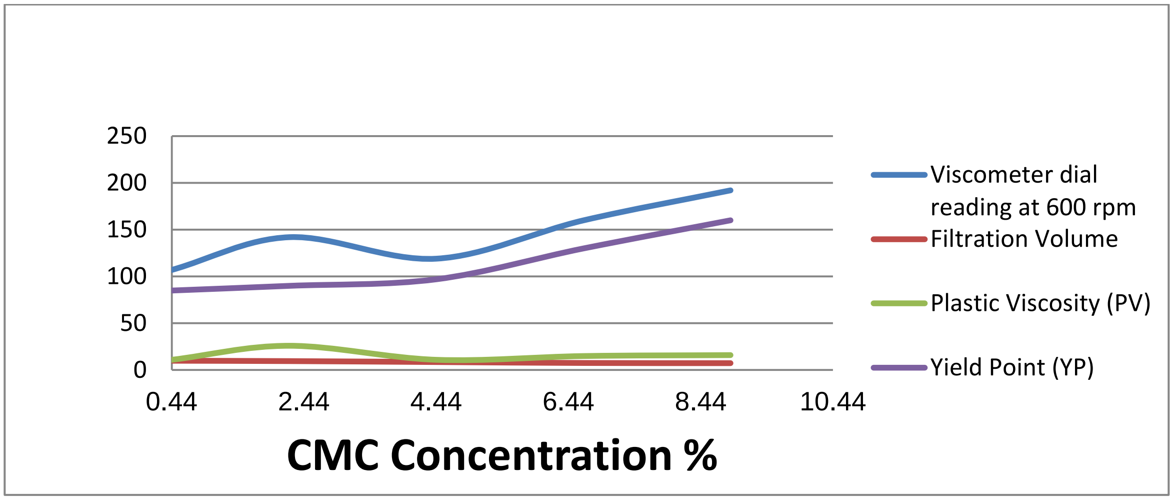


Figure 4.9 : C.CMC Concentration VS ϴ600, Filtration Volume, PV and YP @ 75℃.

Table (4.5)and figure (4.9) showed that the increase of temperature affected in two properties which are viscometer dial reading at 600rpm and Plastic Viscosity (PV) became unstable, that means the CMC affected at **75**℃ .

Table 4.6: P.bentonite and Bentonite with C.CMC Concentration Properties @90℃.

CMC %	Viscometer dial reading at 600 rpm	Filtration Volume (ml)	Plastic Viscosity (PV)	Yield Point (YP)
0.44	125	11	8	109
2.22	146	11	11	124
4.44	138	8.6	13	112
6.67	180	9	22	136
8.89	195	8	23	149

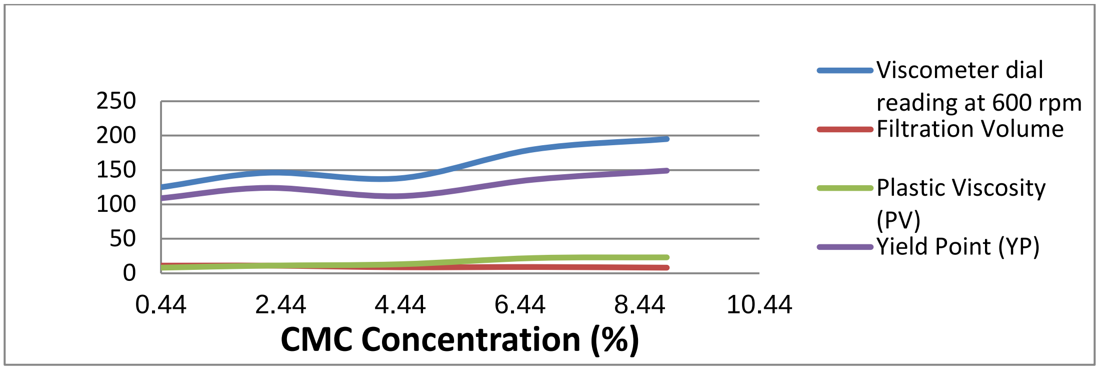


Figure 4.10 : **C.CMC Concentration VS ϴ600, Filtration Volume, PV and YP @ 90 ℃.**

Table (4.6) and figure (4.10) showed that the effect of CMC by the increased of temperature became larger than the effect at 50° and 75° centigrade in the properties of Viscometer dial reading at 600rpm, Filtration Volume and Plastic Viscosity (PV) became unstable by increasing of temperature.

Table 4.7: Properties of Bentonite with 6.67% C.CMC at Different Temperatures.

Temperature	Viscometer dial reading at 600 rpm	Filtration Volume (ml)	Plastic Viscosity (PV)	Yield Point (YP)
25	162	9.6	27	108
50	175	6	20	135
75	160	7.6	15	130
90	180	9	22	136

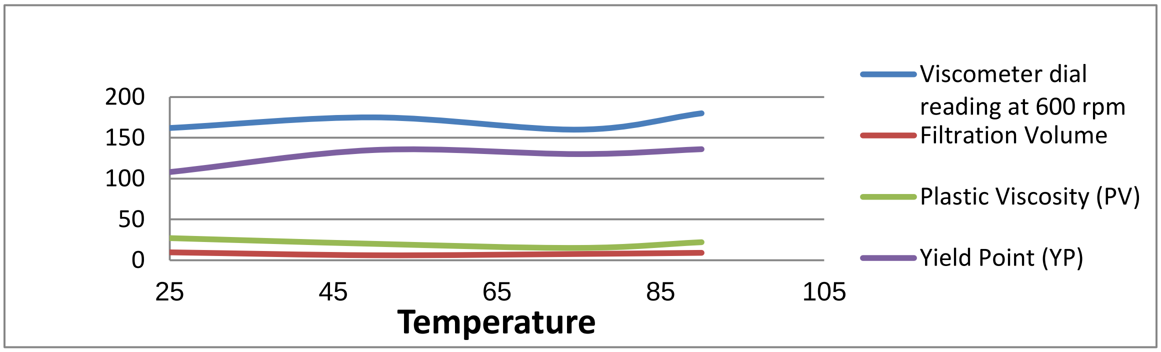


Figure 4.11: Properties of Bentonite with 1.5g of C.CMC at Different Temperatures.

Table (4.7) and figure (4.11) showed the effect of temperature in the properties of Viscometer dial reading at 600rpm, Filtration Volume, Plastic Viscosity (PV) and Yield Point with constant concentration of CMC (6.67% or 1.5g). As a result of these sets of test the CMC stabled till temperature 50° centigrade and became unstable after that temperature.

4.3.2. Rheological Tests for Laboratory Produced CMC

4.3.2.1. Pure Laboratory Produced CMC (L.CMC)

Add 10.5g pure CMC to 350ML (deionized water) and mixing for 20min (as per API specification 13A/ISO 13500) then took Viscometer dial reading at 600rpm (ϴ600) and that to determine the type of CMC.

Table 4.8: Six Speed Viscometer Dial Reading for Pure Laboratory Produced CMC.

	Extracted CMC	**API Specs CMC L.V**
ϴ600	30	Maximum 90

Table (4.8) showed that the laboratory produced CMC in a range of low viscosity CMC (CMC-L.C).

4.3.2.2. Effect of Adding Laboratory Produced CMC to Water Base Mud:

Water Base Mud Formulation:

Mud type: Gel Mud - Bentonite: 22.5 g - Water: 350 ML

Add weight of CMC (1.1g (5%), 2.3g (10%), 3.4g (15%), 4.5g (20%) and 5.6g (25%)) to 22.5g pure Bentonite in 350ML (deionized water) and mixing for 20min then take Viscometer dial reading at 3, 6, 100, 200, 300 and 600rpm after that measure mud weight, PH. filtration volume for 30min and take mud cake thickness.

Table 4.9: P. bentonite and Bentonite with L.CMC Concentration Properties @25℃.

CMC %	Viscometer dial reading at 600 rpm	Filtration Volume (ml)	Plastic Viscosity (PV)	Yield Point (YP)
0	25	19	7	11
5	28	15.6	10	8
10	31	13.6	11.5	8
15	34	12.4	13	8
20	36	10.8	14	8
25	38	10	15	8

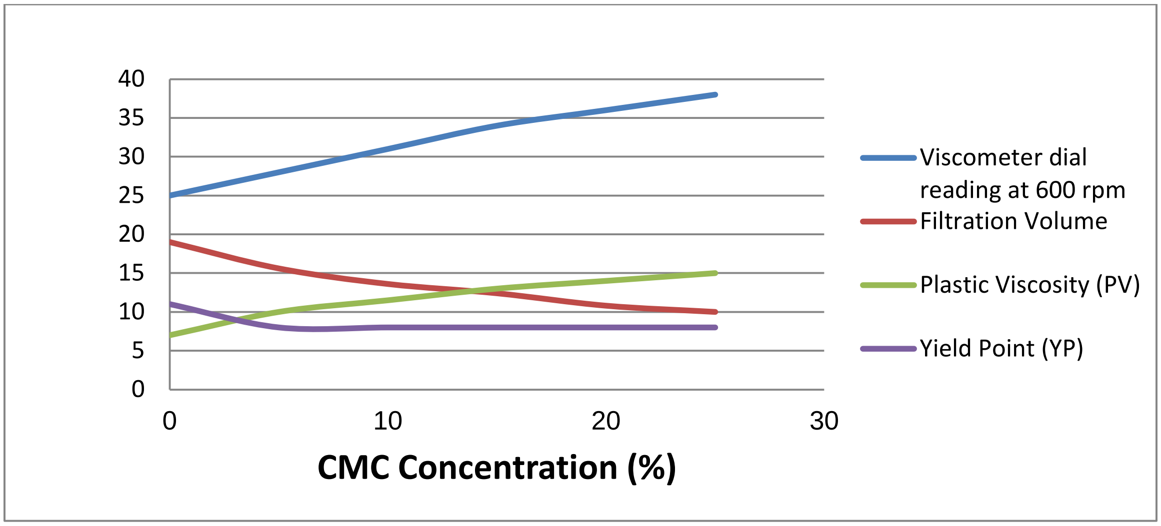


Figure 4.12 : L.CMC Concentration VS ϴ600, Filtration Volume, PV and YP @ 25℃.

Table (4.9) and figure (4.12) showed that Viscometer dial reading at 600rpm and Plastic Viscosity (PV) increased by the increased of CMC Concentration, Yield Point(YP) decreased to 8 and became stable, Filtration Volume Decreased by the increased of CMC Concentration, all tests carried out at **25**℃.

Table 4.10: P.bentonite and Bentonite with L.CMC Concentration Properties @50℃.

CMC %	Viscometer dial reading at 600 rpm	Filtration Volume	Plastic Viscosity (PV)	Yield Point (YP)
5	47	8	12	23
10	50	7	14	19
15	68	6.2	23	22
20	70	5.8	23	24
25	72	5.8	18	17

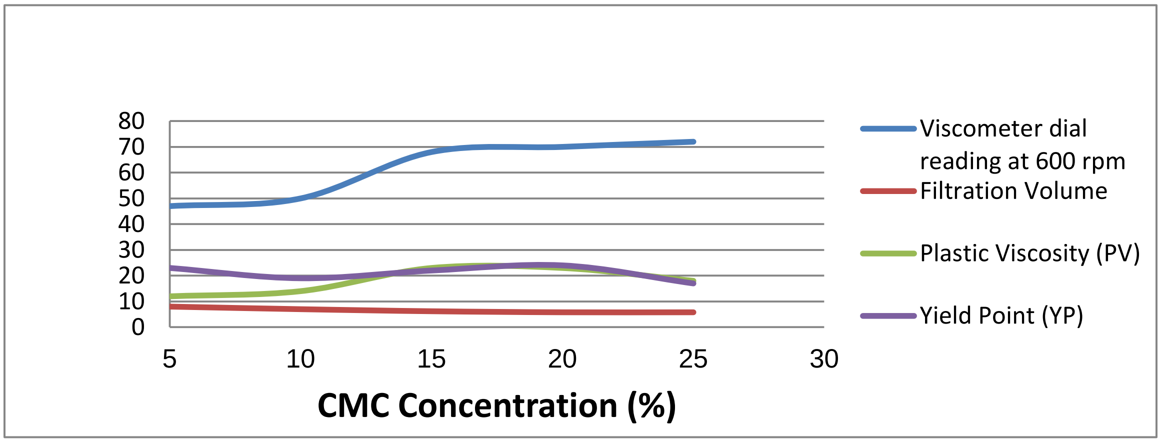


Figure 4.13 : L.CMC Concentration VS ϴ600, Filtration Volume, PV and YP @ 50℃.

Table(4.10) and figure(4.13) showed the Viscometer dial reading at 600rpm and yield point increased by increased of CMC Concentration. Filtration Volume also still decreased by increased CMC Concentration at **50**℃ , temperature affected in Plastic Viscosity (PV) became unstable some by increased CMC Concentration.

Table 4.11: P.bentonite and Bentonite with L.CMC Concentration Properties @75℃.

CMC %	Viscometer dial reading at 600 rpm	Filtration Volume	Plastic Viscosity (PV)	Yield Point (YP)
5	60	8.6	12	36
10	57	8.6	13	31
15	59	7.6	11	37
20	72	6.8	18	36
25	61	7.4	20	21

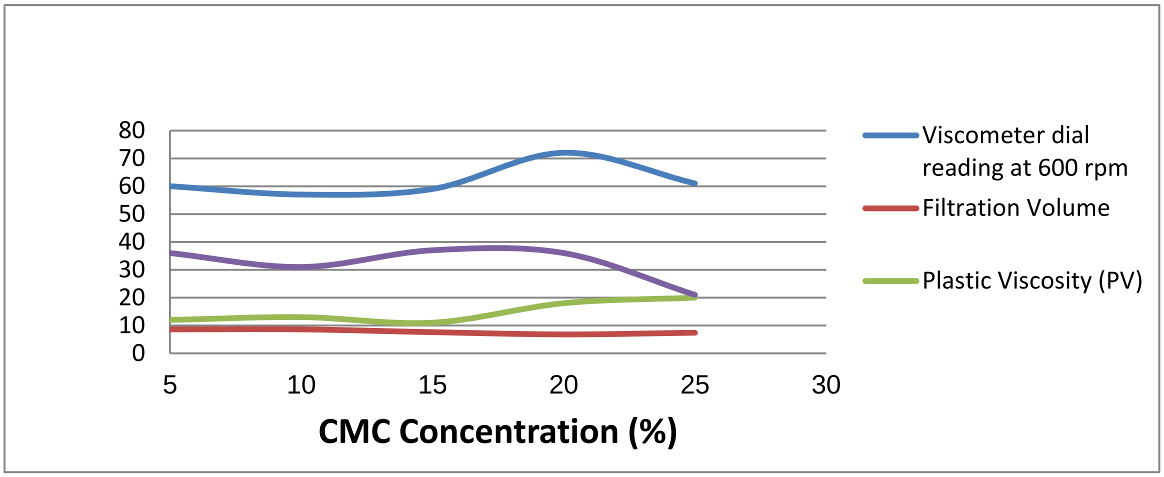


Figure 4.14 : L.CMC Concentration VS ϴ600, Filtration Volume, PV and YP @ 75℃

Table(4.11) and figure(4.14) showed that the increase of temperature had smooth effect in yield point but still the viscometer dial reading at 600rpm and Plastic Viscosity (PV) are stable increased by the increased of CMC concentration and filtration volume decrease by the increased of CMC concentration , that means the CMC stable till **75**℃ .

Table4.12: P.bentonite and Bentonite with L.CMC Concentration Properties @90℃.

CMC %	Viscometer dial reading at 600 rpm	Filtration Volume (ml)	Plastic Viscosity (PV)	Yield Point (YP)
5	55	10	8	39
10	62	9	11	40
15	56	8.2	15	26
20	62	8.2	17	28
25	55	8.6	17	21

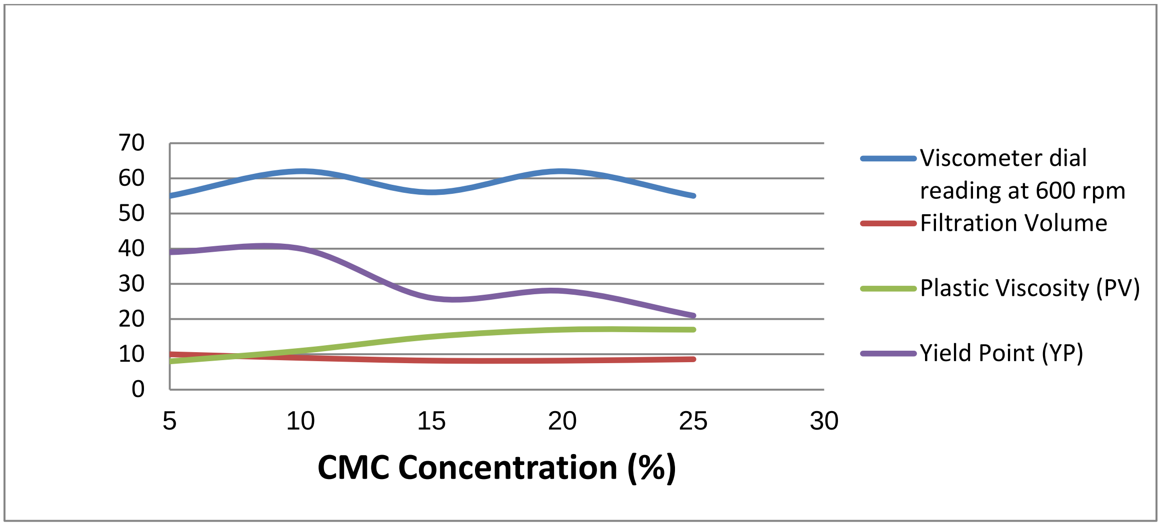


Figure 4.15 : L.CMC Concentration VS ϴ600, Filtration Volume, PV and YP @ 90 ℃.

Table (4.12) and figure (4.15) showed that the effect of CMC by the increased of temperature became larger than the effect at 50° and 75° centigrade in the properties of Viscometer dial reading at 600rpm and Plastic Viscosity (PV) became unstable by increasing of temperature. Filtration volume still decreased by the increased of CMC concentration.

Table 4.13: Properties of Bentonite with 20% L.CMC at Different Temperatures.

Temperature	Viscometer dial reading at 600 rpm	Filtration Volume	Plastic Viscosity (PV)	Yield Point (YP)
25	36	10.8	14	8
50	70	5.8	23	24
75	72	6.8	18	36
90	62	8.2	17	28

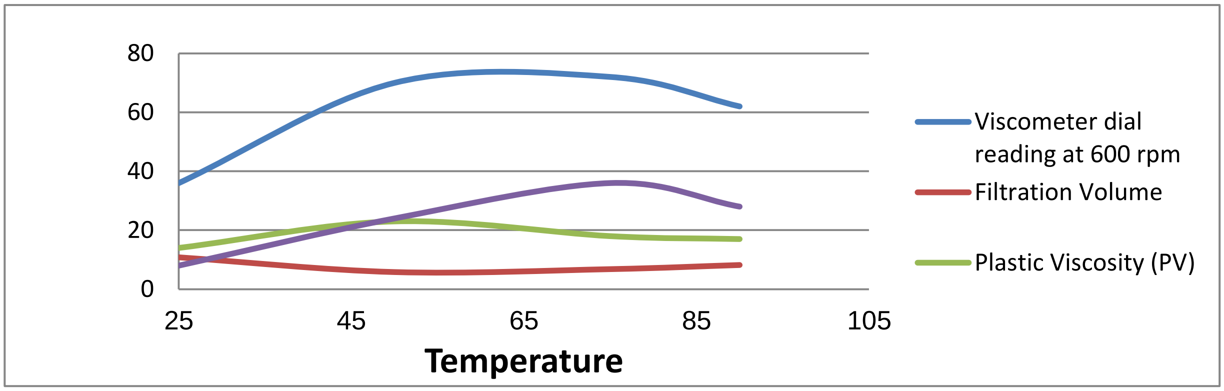


Figure 4.16 : The Properties of Bentonite with 4.5g of L.CMC at Different Temperatures

Table (4.13) and figure (4.15) showed the effect of temperature in the properties of Viscometer dial reading at 600rpm, Filtration Volume, Plastic Viscosity (PV) and Yield Point with constant concentration of CMC (20% or 4.5g). As a result of these sets of test the CMC stabled till temperature 50° centigrade and became unstable after that temperature.

4.4. Comparison Between Commercial and Laboratory CMC

4.4.1. Rheological Tests for Commercial and Laboratory CMC at 25℃

Table 4.14: Dial Reading at 600rpm at Different C.CMC Concentration.

Commercial CMC %	Viscometer dial reading at 600 rpm
0	25
0.44	50
2.22	73
4.44	115
6.67	162
8.89	197

Table 4.15: Dial Reading at 600 rpm at Different L.CMC Concentration.

Laboratory produced CMC %	Viscometer dial reading at 600 rpm
0	25
5	28
10	31
15	34
20	36
25	38

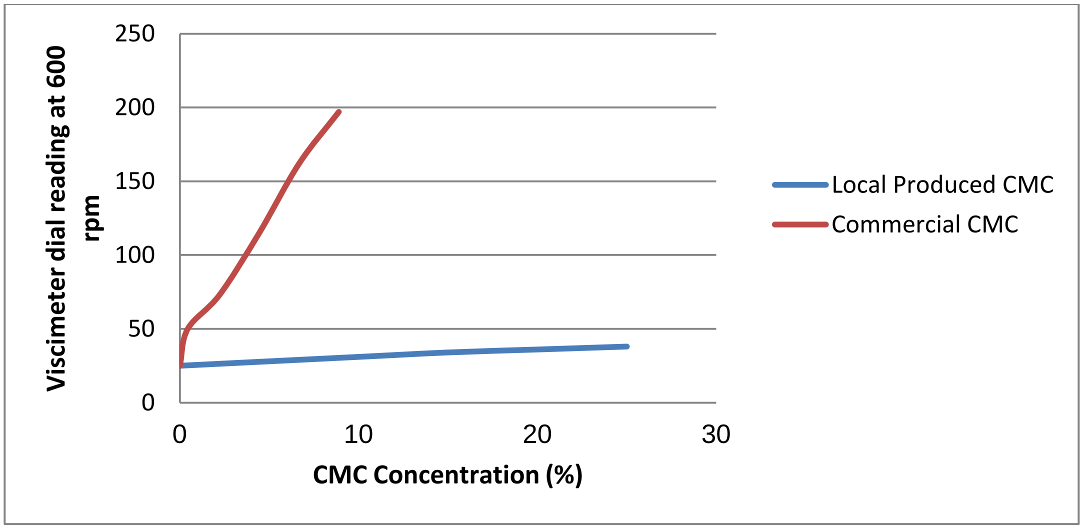


Figure 4.17: **CMC Concentration VS Viscometer Dial Reading at 600 rpm.**

Table 4.16: Filtration Volume at Different Concentration of Commercial CMC.

Commercial CMC %	Filtration Volume
0	19
0.44	16.8
2.22	13.2
4.44	11.2
6.67	9.6
8.89	8.8

Table 4.17: Filtration Volume at Different Concentration of Laboratory CMC.

Laboratory produced CMC %	Filtration Volume
0	19
5	15.6
10	13.6
15	12.4
20	10.8
25	10

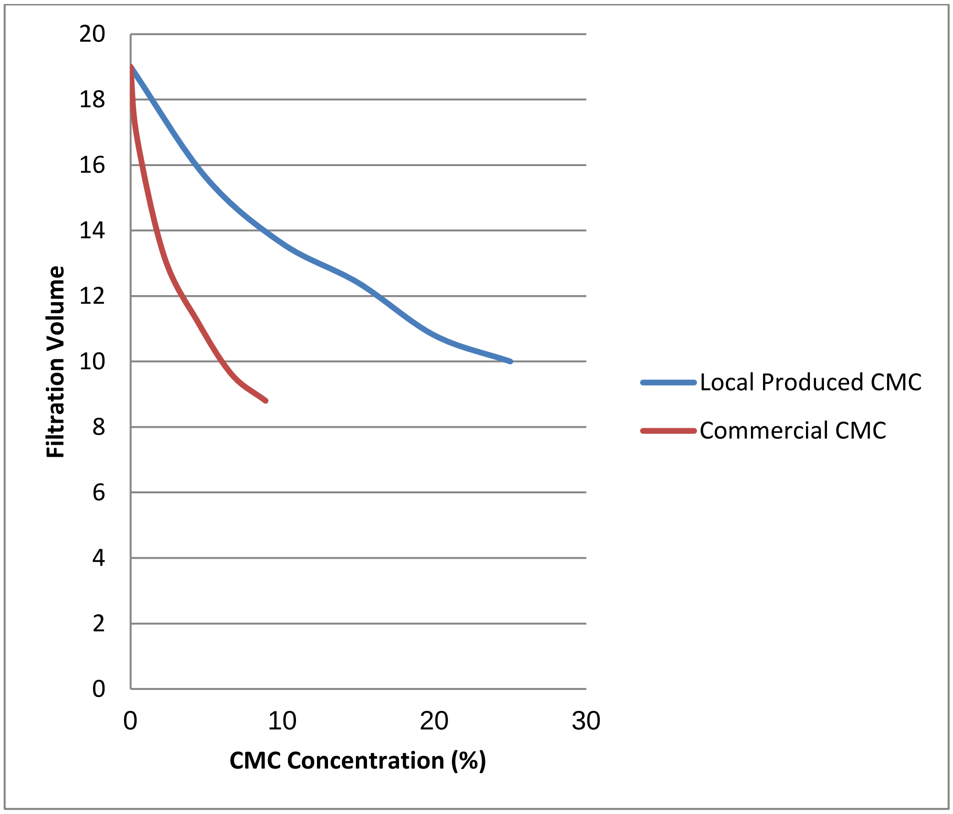


Figure 4.18: Filtration Volume at Different Concentration of CMC.

Table 4.18: Plastic Viscosity at Different Concentration of Commercial CMC.

commercial CMC %	Plastic Viscosity (PV)
0	7
0.44	12
2.22	18
4.44	23
6.67	27
8.89	28

Table 4.19: Plastic Viscosity at Different Concentration of Laboratory CMC.

Laboratory produced CMC %	Plastic Viscosity (PV)
0	7
5	10
10	11.5
15	13
20	14
25	15

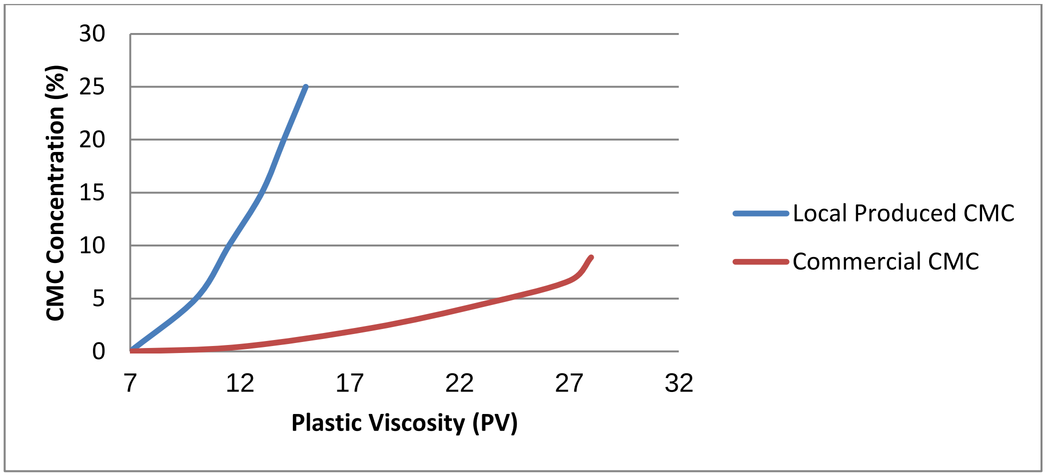


Figure 4.19: Plastic Viscosity (PV) at Different Concentration of CMC.

Table 4.20: Yield Point (YP) at Different Concentration of Commercial CMC.

commercial CMC %	Yield Point (YP)
0	11
0.44	25
2.22	37
4.44	69
6.67	108
8.89	141

Table 4.21: Yield Point (YP) at Different Concentration of Laboratory CMC.

laboratory produced CMC %	Yield Point (YP)
0	11
5	8
10	8
15	8
20	8
25	8

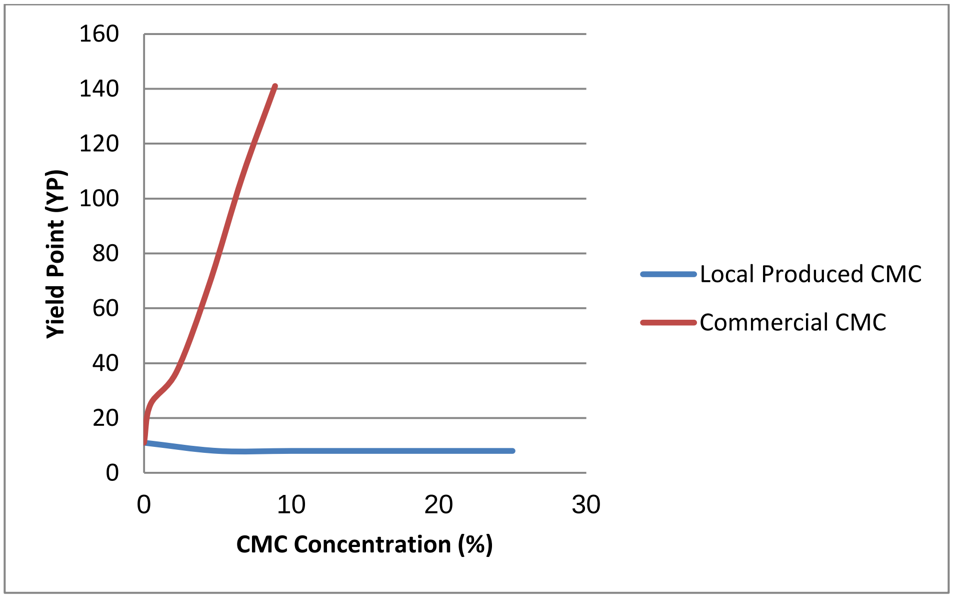


Figure 4.20: Yield Point (YP) at Different Concentration of CMC.

Table ((4.14) and (4.15)) and figure (4.17) showed Viscometer dial reading at 600rpm increased by the increasing of CMC Concentration in both types of CMC. Also Table ((4.16) and (4.17)) and figure (4.18) showed that the Filtration Volume Decreased by increasing of CMC Concentration in both types of CMC. Table (4.18 and 4.20) and figure (4.19) showed that the Plastic Viscosity (PV) increase by increasing CMC Concentration. Table ((4.20) and (4.21)) and figure (4.20) showed that yield point increased by increasing of CMC concentration in commercial CMC and more stable in laboratory produced CMC.

4.4.2. Rheological Tests for Commercial and Laboratory CMC at 50℃

Table 4.22: Viscometer Dial Reading at 600rpm at Different Concentration.

Commercial CMC %	Viscometer dial reading at 600 rpm
0.44	86
2.22	98
4.44	132
6.67	175
8.89	227

Table 4.23: Viscometer Dial Reading at 600rpm at Different Concentration

Laboratory produced CMC %	Viscometer dial reading at 600 rpm
5	47
10	50
15	68
20	70
25	72

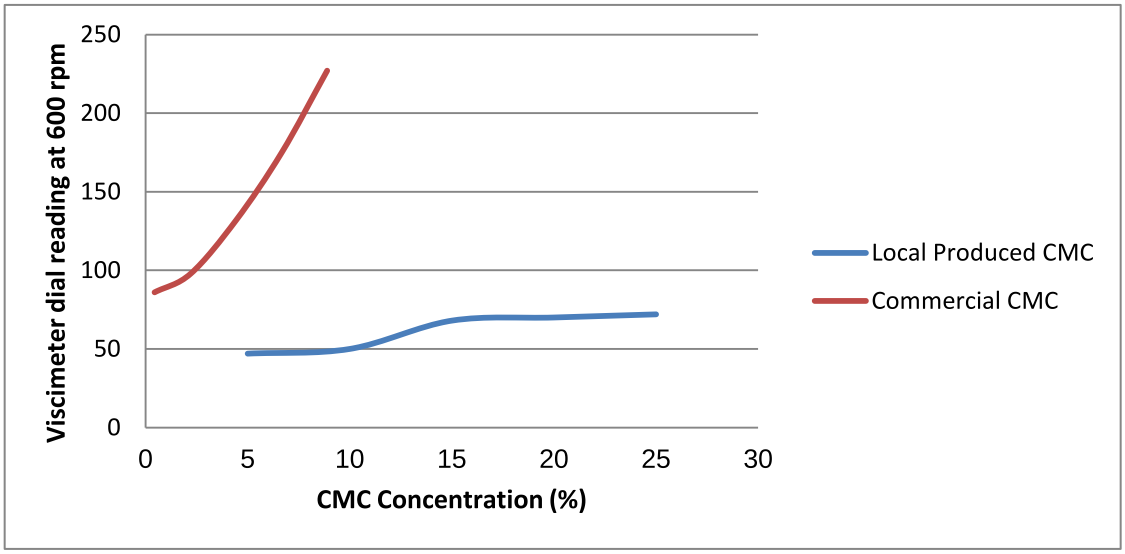


Figure 4.21: CMC Concentration VS Viscometer Dial Reading at 600 rpm.

Table 4.24: Filtration Volume at Different Concentration of Commercial CMC.

Commercial CMC %	Filtration Volume
0.44	12
2.22	10
4.44	7
6.67	6
8.89	6

Table 4.25: Filtration Volume at Different Concentration of Laboratory CMC.

laboratory produced CMC %	Filtration Volume
5	8
10	7
15	6.2
20	5.8
25	5.8

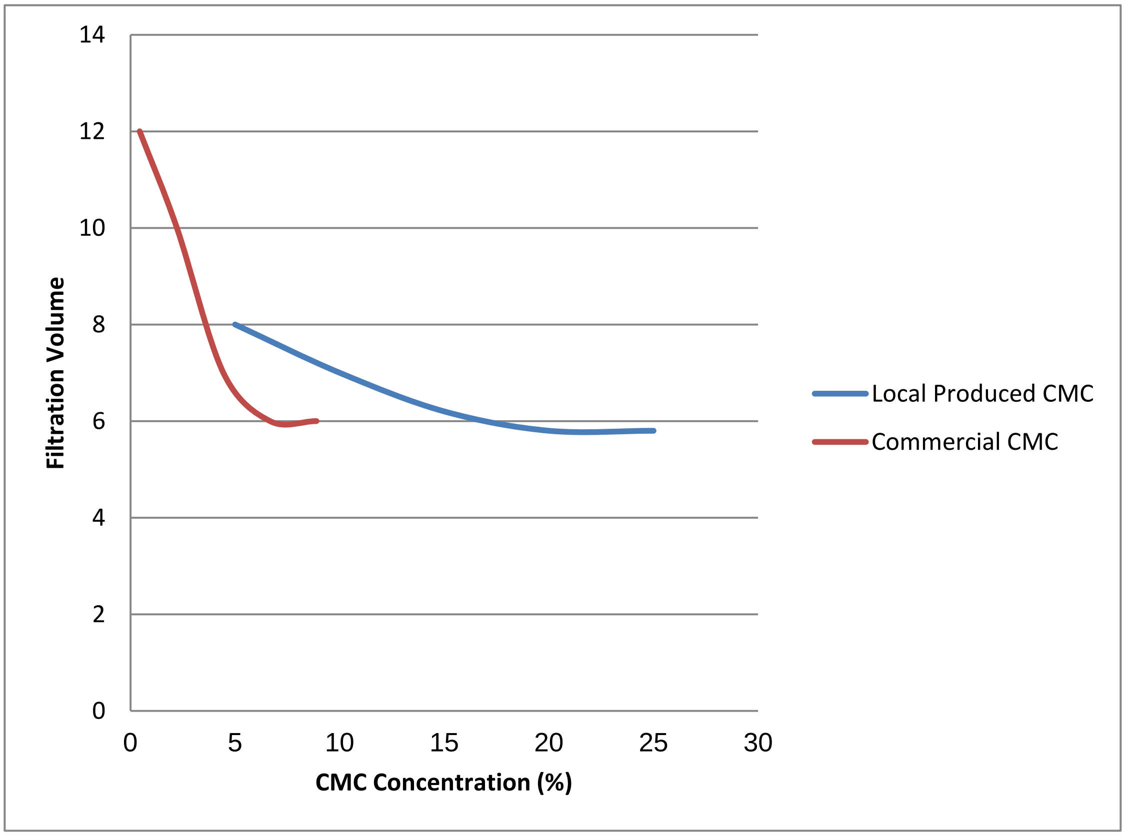


Figure 4.22: Filtration Volume at Different Concentration of CMC.

Table 4.26: Plastic Viscosity (PV) at Different Concentration of Commercial CMC.

Commercial CMC %	Plastic Viscosity (PV)
0.44	14
2.22	14
4.44	13
6.67	20
8.89	17

Table 4.27: Plastic Viscosity (PV) at Different Concentration of Laboratory CMC.

Laboratory produced CMC %	Plastic Viscosity (PV)
5	12
10	14
15	23
20	23
25	18

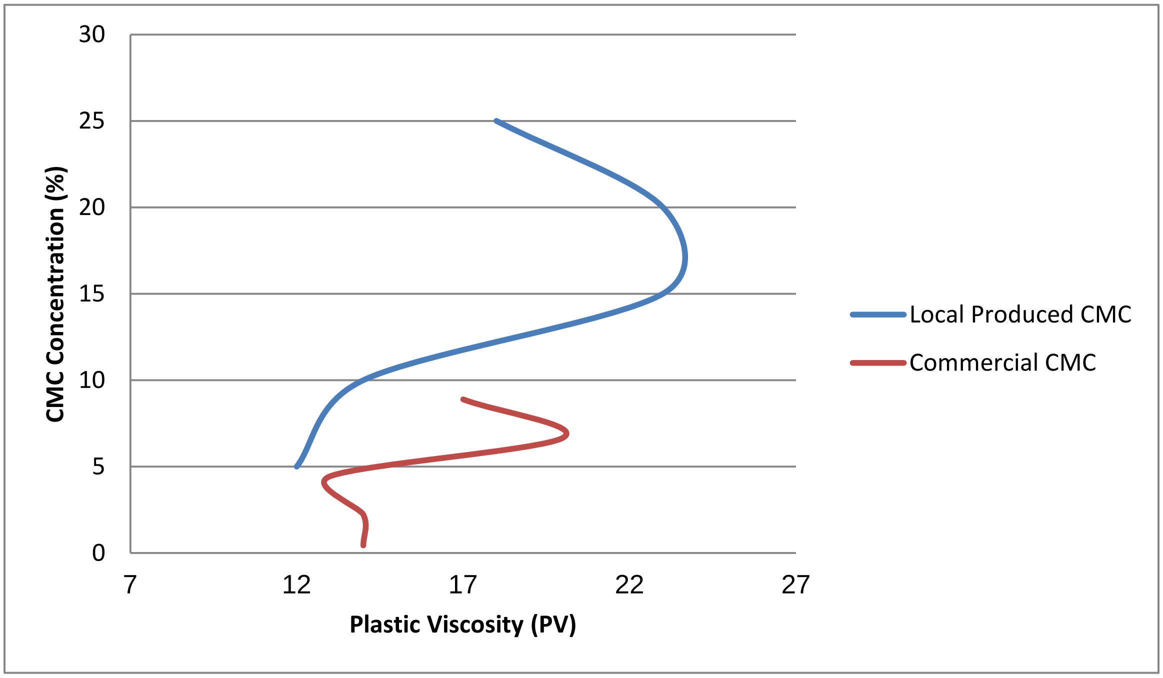


Figure 4.23: Plastic Viscosity (PV) at Different Concentration of CMC.

Table 4.28: Yield Point (YP) at Different Concentration of Commercial CMC.

Commercial CMC %	Yield Point (YP)
0.44	58
2.22	70
4.44	106
6.67	135
8.89	193

Table 4.29: Yield Point (YP) at Different Concentration of Laboratory CMC.

laboratory produced CMC %	Yield Point (YP)
5	23
10	19
15	22
20	24
25	17

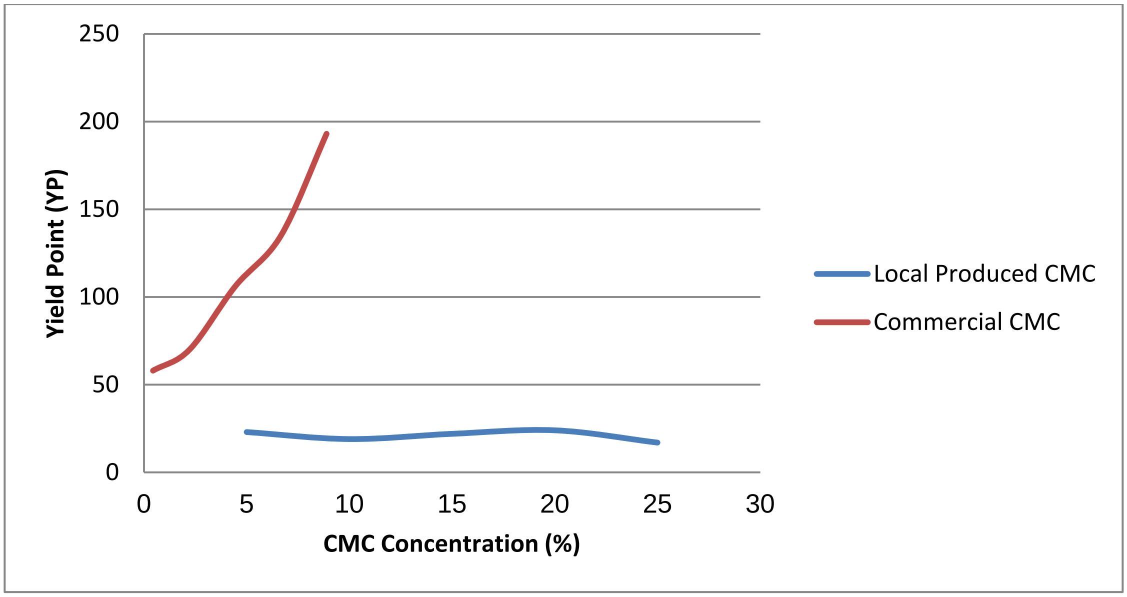


Figure 4.24: Yield Point (YP) at Different Concentration of CMC.

Table ((4.22) and (4.23)) and figure (4.20) showed Viscometer dial reading at 600rpm increased by the increasing of CMC Concentration in both types of CMC. Also Table ((4.24) and (4.25)) and figure (4.21) showed that the Filtration Volume Decreased by increasing of CMC Concentration in both types of CMC. Table ((4.26) and (4.27)) and figure (4.22) showed that the Plastic Viscosity (PV) affected by temperature and became unstable in commercial CMC meanwhile its stable in laboratory CMC. Table ((4.28) and (4.29)) and figure (4.23) showed that yield point increased by increasing of CMC concentration in commercial CMC and laboratory produced CMC.

4.5. Flow Behavior Comparison

4.5.1. Modified Power Law

$$\tau = \tau_0 + k(\gamma)^n \qquad (4.1)$$

Where

$$K = \frac{\theta_{300}}{512^n} \qquad (4.2)$$

K= consistency index

$$n = 3.32\log\left(\frac{\theta_{600}}{\theta_{300}}\right) \qquad (4.3)$$

n =flow behavior index

τ_0 = the fluid´s yield point at zero shear rate.

Using modified power low to study flow behavior for commercial CMC and laboratory produced CMC25, using the result of tests at 25℃.

Table 4.30: Shear Stress and Shear Rate for Commercial CMC.

shear rate	shear stress
78	190.3638224
81	191.1850409
105	197.0580397
125	201.2349462
135	203.1404539
162	207.8114863

Table 4.31: Shear Stress and shear Rate for Laboratory Produced CMC.

shear rate	shear stress
3	8.572821345
8	9.149453737
11	9.441111604
17	9.963101961
22	10.35750866
36	11.34445451

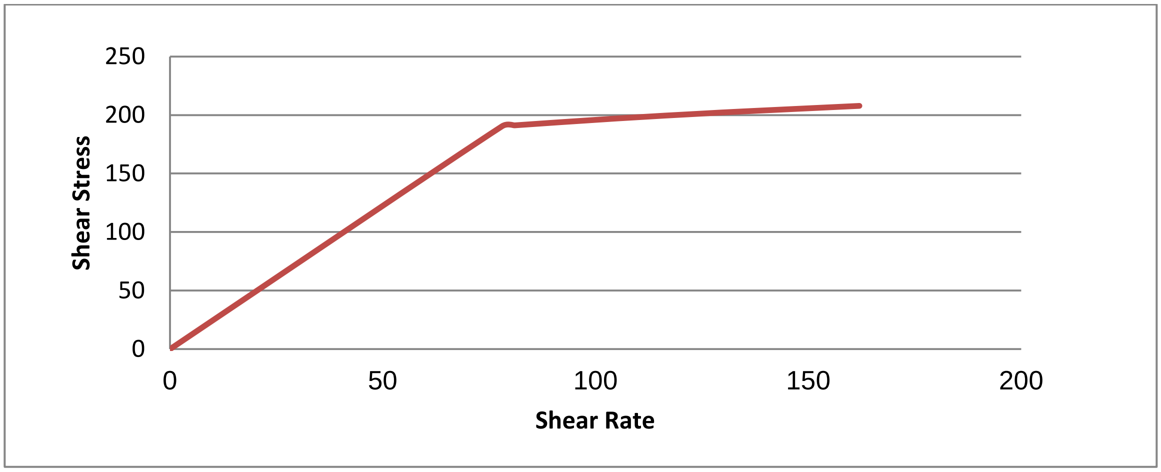


Figure 4.25 : Shear stress VS Shear Rate for Commercial CMC

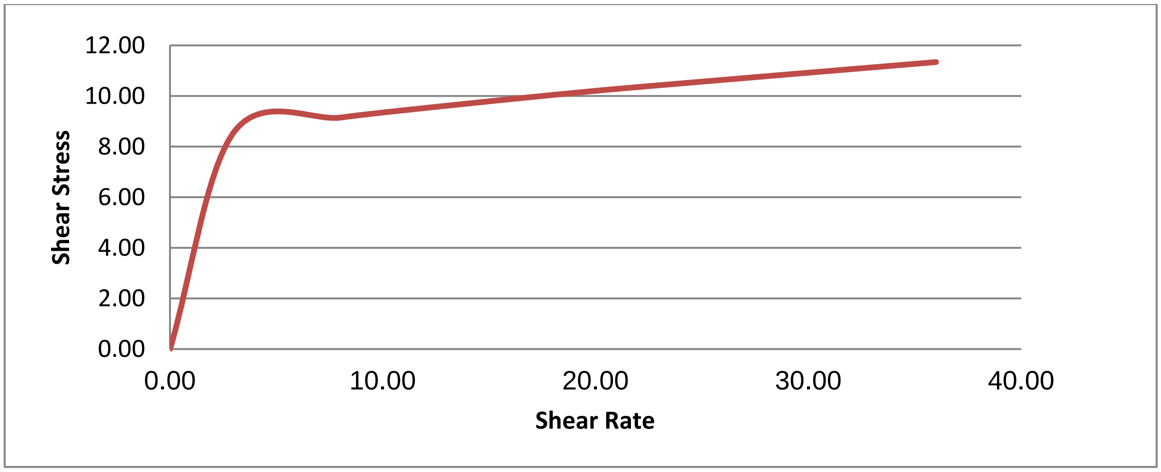


Figure 4.26 : Shear Stress VS Shear rate for Laboratory Produced CMC.

4.6. Drilling Fluid Design According to Block 6 Properties

Formulation 1 for Water Base Mud as block 6 systems:

Table 4.32: Formulation (1) for Water Base Mud.

No.	Additive	Quantity
1	water	350 ml
2	Caustic Soda	0.5 g
3	Soda Ash	0.5 g
4	Flow ZAN	0.5 g
5	PAL FLC	2 g
6	PAC LV	4 g
7	KCL	15 g

Table 4.33: Formulation (1) for Water Base Mud Tests Result.

No.	Rheological properties@	Result
1	600rpm	36
2	300rpm	24
3	200rpm	18
4	100rpm	13
5	6rpm	7
6	3rpm	4
7	PV(Cp)	12
8	YP (Ib/100ft2)	12
9	Fluid Loss @30 mins	10.2
10	PH	8.5

Formulation (2) for Water Base Mud without PAC LV

Table 4.34: Formulation 2 for Water Base Mud.

No.	Additive	Quantity
1	water	350 ml
2	Caustic Soda	0.5 g
3	Soda Ash	0.5 g
4	Flow ZAN	0.5 g
5	PAL FLC	2 g
6	KCL	15 g

Table 4.35: Formulation (2) for Water Base Mud Tests Result.

No.	Rheological properties@	Result
1	600rpm	10
2	300rpm	6
3	200rpm	5
4	100rpm	4
5	6rpm	3
6	3rpm	2
7	PV(Cp)	4
8	YP (Ib/100ft$_2$)	2
9	Fluid Loss @30 mins	17.2
10	PH	8.5

Formulation (3) for Water Base Mud with L.CMC

Table 4.36: Formulation 3 for Water Base Mud.

No.	Additive	Quantity
1	water	350 ml
2	Caustic Soda	0.5 g
3	Soda Ash	0.5 g
4	flozane	0.5 g
5	PAL FLC	2 g
6	KCL	15 g
7	CMC	4g

Table 4.37: Formulation 3 for Water Base Mud Result.

No.	Rheological properties@	Result
1	600rpm	14
2	300rpm	10
3	200rpm	8
4	100rpm	6
5	6rpm	4
6	3rpm	3
7	PV(Cp)	4
8	YP (Ib/100ft$_2$)	6
9	Fluid Loss @30 mins	15.6
10	PH	9

Tables ((4.32) and (4.33)) showed the drilling fluid system and the rheological properties it cleared in table (4.44), in tables ((4.34) and (4.35)) used same system but this time without water loss material (without PAC LV) and that to find the effect of adding CMC, after added CMC to the system in tables ((4.36) and (4.37)) there was an increase in fluid viscosity and the filtration volume decreased from 16.4 to 15.6ml, it was not like PAC LV efficiency which it had filtration volume 10.2ml but still its good performance in improving the fluid properties by increasing viscosity and decreasing filtration volume.

4.7. Economical Sensitivity of CMC

The process of producing CMC laboratory will undoubtedly provide the country with a large amount of hard currency that is spent annually to import large quantities of CMC, which is used in many purposes, including drilling fluids. In this study, CMC is produced laboratory and extracted from palm fronds, which is a laboratory available material.

Refer to the Customs Authority to find out the quantities imported annually from CMC as well as their cost in hard currency during the years 2014, 2015, 2016, 2017 and 2018 as per bellow table:

Table 4.38: CMC Economical Sensitivity for Years from 2014 to 2018

Year	Net Mass - KG	Custvall - USD
2014	1469404.00	2,490,417.90
2015	1914442.00	3,906,194.74
2016	854912.00	1,544,618.66
2017	1323332.00	2,130,923.13
2018	1394537.00	1,572,685.36

Depend on the results of a set of rheological tests which were carried out in this chapter and economical sensitivity, some on conclusions and recommendations were found and it will have mentioned in chapter five (conclusions and recommendations).

CHAPTER FIVE

CONCLUSION AND RECOMMENDATION

Chapter 5

Conclusion and Recommendation

5.1. Conclusions

The rheological properties were tested, and the XRD test was done in Central oil laboratories for three types of bentonite, and then the type C was chosen for its use and experiments were conducted on it because it has a large filtration volume, so the local produced CMC can be used to improve its properties. Both commercial and locally produced CMC were used and added to chosen types bentonite in different proportions and then measured the rheological properties of the drilling fluid.

The following conclusion can be the highlight of this research:

1. Commercial CMC six speed Viscometer dial reading at 600rpm in the range of H.V viscosity CMC and Locally produced CMC six speed Viscometer dial reading at 600rpm in the range of LV viscosity CMC.
2. Both commercial and locally produced SMC were added to bentonite in different proportions and quantities, and the effect of each on the bentonite appeared and the rheological properties of drilling fluid improved, when adding both commercial and locally produced products the viscosity of drilling fluid increased and the volume of filtered water decreased.
3. It was observed that when adding a little weight from the commercial CMC, the viscosity value increases significantly, so the viscosity value is affected more than the filtration volume, but when adding the locally produced CMC, both the viscosity and filtration volume are changed regularly.
4. The rheological properties of the drilling fluid to which the commercial CMC was added, as well as the local product were tested under different temperatures, from 25° C to 90° C. It was observed that the commercial product showed temperature instability effect starting from 50° C, while the local product showed better stability effect up to temperature of 60° C, meaning that the local product is more stable under high temperatures, and both of them lose their properties after the temperature of 75°C.

5. When conducting a number of experiments for the commercial product and the local product, an increase in the plastic viscosity of both commercial product and locally product observed with an increase in the concentration.
6. Yield point increases as increase of CMC concentration for commercial product and its stable in the local produced CMC.
7. Follow behavior for both commercial and locally produces CMC has been tested used modified power law and the result found the behavior of the same scheme of modified power law model.
8. Locally produced CMC has been used with KCL mud and it was given a good result of increasing viscosity but some foams found that affect in increasing filtration volume and was solved by adding antifoaming agent.

5.2. Recommendations

In this research, rheological tests were performed under different temperatures on the local product, and the results obtained proved the feasibility and effectiveness of the product, especially when used with bentonite, which led to improving the properties of bentonite.

The followings actions are recommended for more studies and to improving CMC locally produced from palm leaf:

1. Recommended to use pure palm leaf after powdering and added to drilling fluid to study the effect of additive in filtration volume reading.
2. It is recommended to improve the produced CMC using nano Particles techniques.
3. It is recommended to do more tests for produced CMC with other types of drilling fluid and other additives and that to study the effect of CMC by other additives.
4. When adding the product to the KCL drilling fluid, foam was formed during mixing process, it is recommended to add anti foam.
5. It is recommended to produce a large quantities of CMC and used in rig site to drill a real well for the beneficial of economies of the country.

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APPENDIX A

APPARATUSES
LABORATORY
EXPERIMENTS
PHOTOS

Apparatuses and Laboratory experiments photos

Apparatuses

1. Digital Weighing Scale (g)

Used for Material Weight Measurement.

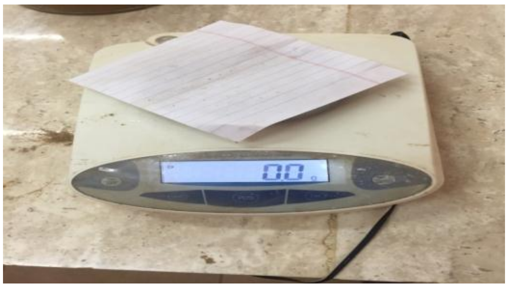


Figure A.1: Digital weighing scale

2. Mixing Mud Machine

Used For mixing drilling fluid.

Figure A.2: D90-Mixing mud machine

3. Mud Balance (g/cm3, Lb/gal)

Used for density measurement.

Figure A.3: YM-2 mud balance.

4. PH Papers

Used for PH Measurement.

Figure A.4: PH Papers

5. ZNN-D6 Six Speed Rotation Viscometer (rpm)

Used for dial speed measuring.

Figure A.5: ZNN-D6 Six speed rotation viscometer

6. Digital Vernier Inch Metric (mm)

Used for filter cake thickness.

Figure A.6: Digital Vernier Inch Metric

7. ZNS-4 Inflator Air Supply Water Loss Meter (MPA-time)

Used for fluid filtration Measurement.

Figure A.7: ZNS-4 Inflator air supply water Loss meter.

8. GGS-71 HTHP water loss meter (MPa, °C,)

Used for determine fluid volume filtration

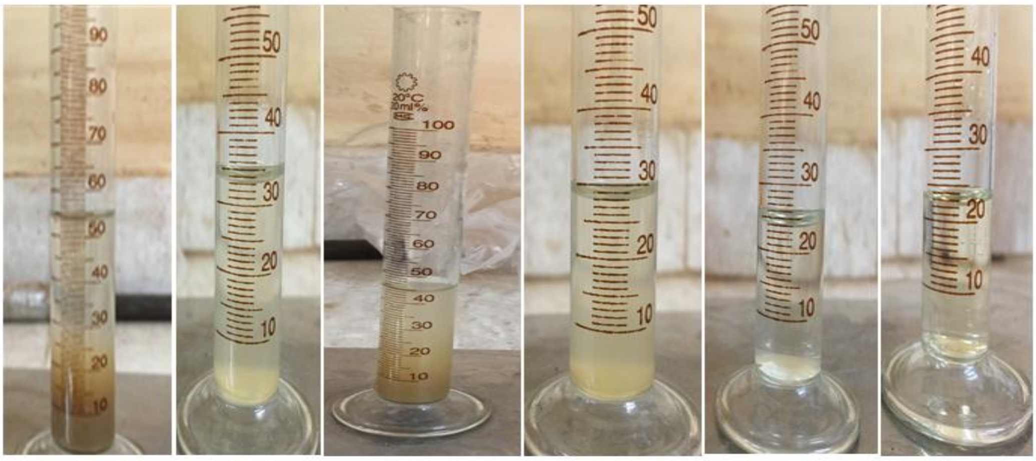
Figure A.8: GGS-71 HTHP water Loss meter.

9. Philips X’ Pert 1 X-ray Diffract meter

Figure A.9 : Philips X’ Pert 1 X-ray Diffract meter

Laboratory experiments photos

Figure A.10: Materials Preparation.



Pure Bentonite

22.5 g Benttonite 0.1 g C.CMC

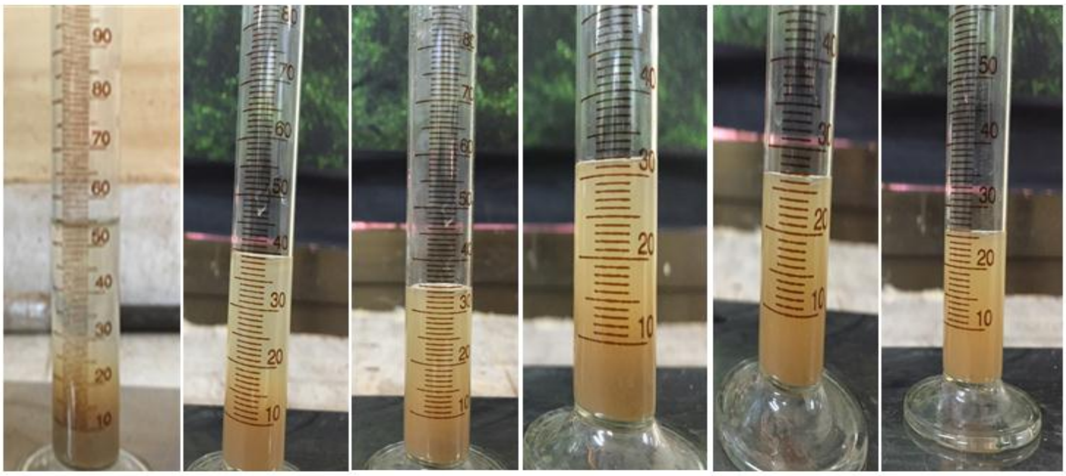
22.5 g Benttonite 0.5 g C.CMC

22.5 g Benttonite 1 g C.CMC

22.5 g Benttonite 1.5 g C.CMC

22.5 g Benttonite 2.0 g C.CMC

Figure A.11: Filtration Volume at Different Concentration of Commercial CMC.



Pure Bentonite

22.5 g Benttonite 1.1 g L.CMC

22.5 g Benttonite 2.3 g L.CMC

22.5 g Benttonite 3.4 g L.CMC

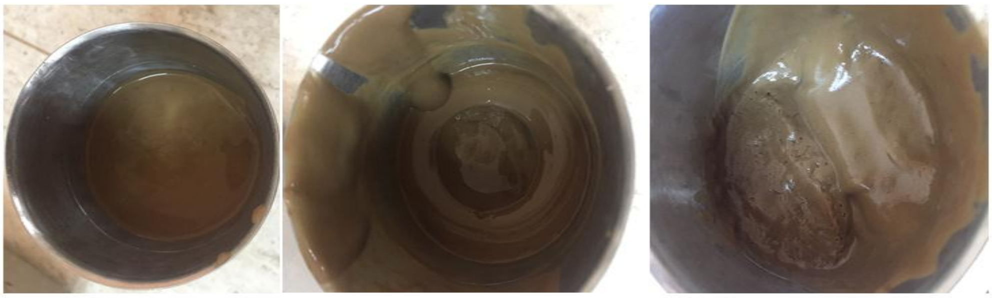
22.5 g Benttonite 4.5 g L.CMC

22.5 g Benttonite 5.6 g L.CMC

Figure A.12: Filtration Volume at Different Concentration of Laboratory Produced.

Drilling Fluid @50°C. Drilling Fluid @75°C. Drilling Fluid @90°C. Drilling Fluid @110°C.

Figure A.13: The effect of temperature on bentonite and commercial CMC mixture.



Drilling Fluid @50°C. Drilling Fluid @75°C. Drilling Fluid @90°C.

Figure A.14: The effect of temperature on bentonite and Laboratory Produced CMC mixture.

Figure A.15: Forming foam during the material mixing process for KCL mud.

KCL mud without CMC KCL mud with CMC

Figure A.16: Filtration Volume for KCL mud.

Figure A.17: The product is not completely soluble.

Figure A.18: The researcher was exposed to a face fire during laboratory experiments.

APPENDIX B

LABORATORY CMC PHASE ANALYSIS REPORT

Match! Phase Analysis Report

Sample: Laboratory CMC

Sample Data

File name	202019.RAW
File path	H:/402019
Data collected	14:44:41 2019, 23 أبريل
Data range	9.980º 79.993º
Number of points	4897
Step size	0.014
Rietveld refinement converged	No
Alpha2 subtracted	No
Background subtr.	No
Data smoothed	No
2theta correction	0.02º
Radiation	Xrays
Wavelength	1.540600 Å

Table B.1: Matched Phases.

Index	*Amount (%)*	*Name*	*Formula sum*
A	84.4		Cl Na
B	12.3		Al2 O6 Sr3
C	3.3		Fe0.91 Si0.09
	19.0	*Unidentified peak area*	

A: Cl Na (84.4 %)

Formula sum	Cl Na
Entry number	964300181
FigureofMerit (FoM)	0.838152
Total number of peaks	18
Peaks in range	8
Peaks matched	7
Intensity scale factor	0.80
Space group	F m 3 m
Crystal system	cubic
Unit cell	a= 5.6393 Å
I/Ic	4.71
Calc. density	2.164 g/cm³
Reference	Barbara J?rgens, Elisabeth Irran, Julius Schneider, Wolfgang Schnick, "Trimerization of NaC2N3 to Na3C6N9 in the Solid: Ab Initio CrystalStructure Determination of Two Polymorphs of NaC2N3 and

of Na3C6N9 from X rayPowder Diffractometry", Inorganic Chemistry **39**, 665670 (2000)

B: Al2 O6 Sr3 (12.3 %)

Formula sum	Al2 O6 Sr3
Entry number	969015879
FigureofMerit (FoM)	0.820234
Total number of peaks	152
Peaks in range	139
Peaks matched	46
Intensity scale factor	0.20
Space group	P a 3
Crystal system	cubic
Unit cell	a= 15.8476 Å
I/Ic	8.23
Calc. density	4.134 g/cm³
Reference	Alonso J., Rasines I., Soubeyroux J., "Tristrontium dialuminium hexaoxide: An intricate superstructure of perovskite_cod_database_code 1008450", Inorganic Chemistry **29**, 47684771 (1990)

C: Fe0.91 Si0.09 (3.3 %)

Formula sum	Fe0.91 Si0.09
Entry number	969006627
FigureofMerit (FoM)	0.850250
Total number of peaks	3
Peaks in range	2
Peaks matched	2
Intensity scale factor	0.08
Space group	I m 3 m
Crystal system	cubic
Unit cell	a= 2.8105 Å
I/Ic	12.47
Calc. density	7.980 g/cm³
Reference	Zhang J., Guyot F., "Thermal equation of iron and Fe0.91Si0.09Sample: P = 8.9 GPa, T = 773 K", Physics and Chemistry of Minerals **26**, 206211 (1999)

Table B.2: Candidates.

Name	***Formula***	***Entry No.***	***FoM***
Porphyrazinealuminiumchloride	C16 Al Cl N16 S4	964309965	0.7313
rubidium magnesium leucite	Mg O12 Rb2 Si5	962104396	0.7110
	C4 N5 S3	964116295	0.7064
CaZrO3	Ca O3 Zr	965910141	0.7049
Dilead magnesiotellurate(VI) (Elpasolite)	Mg O6 Pb2 Te	961008899	0.7007
(Ba0.994 Sr0.006) (Ti0.937 Ce0.063) O3	Ba0.994 Ce0.063 O3 Sr0.006 Ti0.937	961522093	0.6976

Dilead magnesiotellurate(VI) (Elpasolite)	Mg O6 Pb2 Te	961008900	0.6973
Helium	He	969011632	0.6968
(Ba0.1 La0.9) (Mg0.633 Nb0.367) O3	Ba0.1 La0.9 Mg0.633 Nb0.367 O3	961533966	0.6960
(Ni Zn)	Ni Zn	961523189	0.6959
Sr1.4La0.6MgTaO5.8	La0.6 Mg O5.8 Sr1.4 Ta	961545081	0.6958
La (Mg0.667 Nb0.333) O3	La Mg0.667 Nb0.333 O3	961533970	0.6957
	N O P	969006974	0.6954
SrLaMgTaO6	La Mg O6 Sr Ta	961545080	0.6950
Wulfenite	Mo O4 Pb	969008106	0.6948
	B Cl	969008996	0.6939
	Mo O4 Sr	964001307	0.6924
Quartz	O2 Si	969000780	0.6919
Wulfenite	Mo4.24 O16 Pb3.76	962018108	0.6917
Sr1.6La0.4MgTaO5.7	La0.4 Mg O5.7 Sr1.6 Ta	961545083	0.6910
Stolzite	O4 Pb W	969009815	0.6897
Dilead magnesium tungstate I	Mg O6 Pb2 W	961001661	0.6887
	Mg O6 Pb1.999 W	969014620	0.6887
K La (W O4)2	K La O8 W2	961531513	0.6878
(N H4)4 (Pt2 Cl (P2 O5 H2)4)	Cl H24 N4 O20 P8 Pt2	962020264	0.6876
Potassium Barium Bismuth oxide	Ba4 Bi3 K O	962011039	0.6874
Wulfenite	Mo O4 Pb	969009405	0.6873
Rb2 Se	Rb2 Se	961535078	0.6869
	Fe0.63 Li2.37 N	968101636	0.6867
	Cl H56 Mo8 N12 O56 P9	967000306	0.6853
	Cl H56 Mo8 N12 O56 P9	967030617	0.6853
Te6 (As F6)4 (S O2)2	As4 F24 O4 S2 Te6	964343746	0.6852
Tl F	F Tl	961542072	0.6850
Pb1.02 (W O4) O0.13	O4.13 Pb1.02 W	961529180	0.6849
(Pb0.99 La0.01) (W O4)	La0.01 O4 Pb0.99 W	961521518	0.6833
Halite	Cl Na	969006378	0.6833
Calciolangbeinite	Ca1.325 K2 Mg0.67 O12 S3	969016688	0.6833
	Ba3.97 Eu0.13 Lu4 O48 P12 Sr7.92	967032535	0.6832
Vanadium Oxide	O2 V	964336392	0.6824

Nd Rh0.79 Ge0.54	Ge0.54 Nd Rh0.79	961526438	0.6822
dirubidium dimanganese trisulfate	Mn2 O12 Rb2 S3	962209088	0.6816
Iron	Fe	969014114	0.6815
Iron	Fe	969014447	0.6815
(La0.1 Ba0.9) (Mg0.05 Ti0.95) O3	Ba0.9 La0.1 Mg0.05 O3 Ti0.95	961531091	0.6813
Ba Ti O3	Ba O3 Ti	961542141	0.6810
(Bi0.3 Pb0.7) (Fe0.3 Ti0.7) O3	Bi0.3 Fe0.3 O3 Pb0.7 Ti0.7	961541977	0.6808
Te	Te	962020223	0.6807
Te	Te	961531231	0.6805
Tb Rh1.1 Sn3.4	Rh1.1 Sn3.4 Tb	961525421	0.6801
Argon	Ar	969011636	0.6794
Hydrogen	H	969012909	0.6790
(Pb0.94 La0.067) (W O4) O0.18	La0.067 O4.18 Pb0.94 W	961529185	0.6782
and 150 others...			

Search Match

Settings

Reference database used	COD-Inorg REV198327 2017.07.03
Automatic zeropoint adaptation	Yes
Minimum figureofmerit (FoM)	0.60
2thea window for peak corr.	0.30 deg.
Minimum rel. int. for peak corr.	1
Parameter/influence 2theta	0.50
Parameter/influence intensities	0.50
Parameter multiple/single phase(s)	0.50

Table B.3: Peak List.

No.	***2theta [º]***	***d [Å]***	***I/I0***	***FWHM***	***Matched***
1	10.33	8.5582	23.47	0.2002	
2	10.45	8.4550	33.34	0.2002	
3	10.67	8.2826	28.57	0.2002	
4	10.85	8.1442	52.78	0.2002	
5	10.98	8.0507	21.16	0.2002	

6	11.14	7.9355	34.90	0.2002	
7	11.24	7.8638	44.03	0.2002	
8	11.35	7.7870	30.26	0.2002	
9	11.51	7.6791	44.35	0.2002	
10	11.66	7.5862	34.20	0.2002	
11	11.83	7.4736	42.97	0.2002	
12	12.01	7.3626	51.03	0.2002	
13	12.13	7.2936	31.70	0.2002	
14	12.30	7.1926	38.44	0.2002	
15	12.44	7.1118	28.26	0.2002	B
16	12.61	7.0135	31.82	0.2002	
17	12.74	6.9429	19.29	0.2002	
18	12.85	6.8850	22.16	0.2002	
19	13.91	6.3596	18.29	0.2002	B
20	17.49	5.0657	21.55	0.2002	
21	17.89	4.9532	19.63	0.2002	
22	18.02	4.9193	24.14	0.2002	
23	18.15	4.8834	25.01	0.2002	
24	18.26	4.8552	47.86	0.2002	
25	18.51	4.7905	25.35	0.2002	B
26	18.63	4.7583	29.62	0.2002	
27	18.92	4.6878	25.98	0.2002	
28	19.07	4.6490	19.72	0.2002	
29	19.21	4.6174	20.47	0.2002	
30	19.41	4.5688	32.67	0.2002	
31	19.62	4.5201	37.57	0.2002	
32	19.79	4.4823	18.20	0.2002	
33	20.04	4.4277	45.48	0.2002	
34	20.27	4.3766	25.07	0.2002	B

35	20.58	4.3131	41.84	0.2002	
36	20.73	4.2818	22.58	0.2002	
37	20.86	4.2547	27.34	0.2002	
38	20.99	4.2281	22.58	0.2002	B
39	21.10	4.2078	22.95	0.2002	
40	21.24	4.1806	19.05	0.2002	
41	21.39	4.1514	28.05	0.2002	
42	21.53	4.1232	18.49	0.2002	
43	21.85	4.0646	32.99	0.2002	
44	22.00	4.0368	19.97	0.2002	
45	22.15	4.0100	19.94	0.2002	
46	22.40	3.9665	26.14	0.2002	B
47	22.57	3.9361	25.38	0.2002	
48	22.79	3.8981	38.34	0.2002	
49	23.00	3.8640	63.56	0.2002	B
50	23.59	3.7690	22.30	0.2002	
51	23.76	3.7411	19.51	0.2002	
52	24.51	3.6293	21.80	0.2002	B
53	25.34	3.5120	21.96	0.2002	
54	26.79	3.3255	21.22	0.2002	
55	27.28	3.2660	32.89	0.2002	A
56	27.52	3.2382	136.10	0.2002	B
57	27.84	3.2017	20.20	0.2002	
58	28.21	3.1611	51.73	0.2002	B
59	29.27	3.0483	36.67	0.2002	B
60	29.46	3.0293	24.06	0.2002	
61	29.77	2.9991	19.37	0.2002	
62	30.41	2.9372	18.28	0.2002	B
63	30.75	2.9049	20.20	0.2002	B

64	31.29	2.8565	34.26	0.2002	
65	31.46	2.8415	54.52	0.2002	
66	31.88	2.8052	1000.00	0.2002	A,B
67	32.79	2.7292	22.95	0.2002	
68	33.07	2.7064	20.98	0.2002	
69	33.70	2.6572	39.02	0.2002	B
70	33.96	2.6375	21.82	0.2002	
71	34.11	2.6265	44.58	0.2002	
72	34.37	2.6070	18.19	0.2002	
73	36.24	2.4770	25.02	0.2002	B
74	36.89	2.4347	19.20	0.2002	B
75	37.18	2.4166	17.23	0.2002	B
76	37.86	2.3742	15.83	0.2002	
77	38.14	2.3574	18.11	0.2002	B
78	38.32	2.3471	16.00	0.2002	B
79	39.13	2.3003	17.20	0.2002	
80	39.35	2.2876	19.96	0.2002	B
81	39.46	2.2815	26.21	0.2002	
82	40.05	2.2497	16.78	0.2002	B
83	41.55	2.1719	17.44	0.2002	B
84	42.51	2.1247	20.18	0.2002	B
85	43.36	2.0849	20.06	0.2002	
86	43.51	2.0783	23.01	0.2002	
87	44.69	2.0259	17.83	0.2002	B
88	44.96	2.0145	23.37	0.2002	B
89	45.19	2.0048	25.33	0.2002	
90	45.63	1.9866	550.49	0.2002	A,C
91	45.76	1.9813	40.94	0.2002	B
92	46.58	1.9484	22.09	0.2002	

93	46.73	1.9422	20.01	0.2002	
94	47.02	1.9312	18.06	0.2002	B
95	47.62	1.9082	16.66	0.2002	B
96	48.15	1.8884	17.19	0.2002	B
97	50.96	1.7905	17.61	0.2002	B
98	52.76	1.7335	16.47	0.2002	B
99	54.00	1.6969	38.74	0.2002	A
100	54.12	1.6931	17.00	0.2002	
101	54.32	1.6875	17.37	0.2002	B
102	54.95	1.6697	16.95	0.2002	B
103	55.19	1.6629	16.05	0.2002	
104	55.68	1.6494	19.16	0.2002	
105	56.10	1.6381	16.88	0.2002	B
106	56.29	1.6331	33.88	0.2002	B
107	56.63	1.6241	190.81	0.2002	A
108	56.81	1.6192	54.71	0.2002	B
109	57.79	1.5942	16.55	0.2002	B
110	58.01	1.5885	17.22	0.2002	
111	58.27	1.5821	18.98	0.2002	B
112	66.14	1.4117	22.95	0.2002	B
113	66.32	1.4082	55.71	0.2002	A
114	66.47	1.4056	23.43	0.2002	B,C
115	75.05	1.2647	21.66	0.2002	B
116	75.15	1.2632	27.35	0.2002	
117	75.42	1.2594	107.82	0.2002	A,B
118	75.53	1.2578	35.44	0.2002	
119	75.65	1.2561	29.58	0.2002	B
120	77.22	1.2344	16.38	0.2002	B

Rietveld Refinement using FullProf

Calculation was not run or did not converge.

Crystallite Size Estimation using Scherrer Formula

Calculation
was not run.

Table B.4: Integrated Profile Areas.

Based on calculated profile ***Profile area***	***Counts***	***Amount***
Overall diffraction profile	302034	100.00%
Background radiation	220372	72.96%
Diffraction peaks	81662	27.04%
Peak area belonging to selected phases	31455	10.41%
Peak area of phase A (Cl Na)	*34236*	*11.34%*
Peak area of phase B (Al2 O6 Sr3)	*27849*	*9.22%*
Peak area of phase C (Fe0.91 Si0.09)	*3182*	*1.05%*
Unidentified peak area	57436	19.02%

Table B.5: Peak Residuals.

Peak data	***Counts***	***Amount***
Overall peak intensity	1062	100.00%
Peak intensity belonging to selected phases	431	40.60%
Unidentified peak intensity	631	59.40%

Diffraction Pattern
Graphics

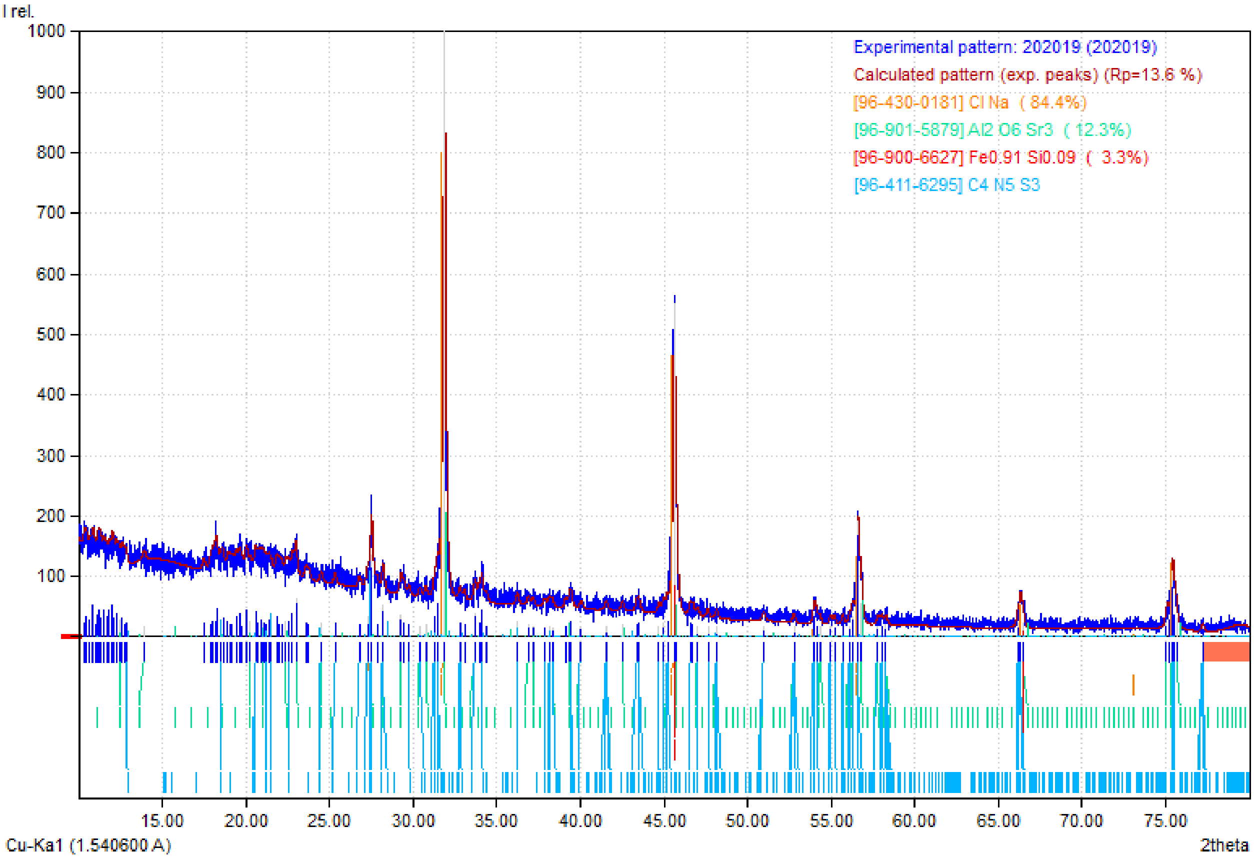


Figure B.1: Match! Copyright © 2003-2017 CRYSTAL Germany