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Modifying the filtration properties of water-based Drilling fluids using extracted virgin olive cellule

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أَعُوذُ بِاللهِ مِنَ الشَّيطَانِ الرَّجِيمِ

بِسْمِ اللَّهِ الرَّحْمَانِ الرَّحِيمِ

{ وَٱلنِّينِ وَٱلزَّيْتُونِ ١ وَطُورِ سِينِينَ ٢ وَهَٰذَا ٱلۡبَلَدِ ٱلۡأَمِينِ ٣ لَقَدۡ خَلَقۡنَا ٱلۡإِنسَٰنَ فِيَ أَحۡسَنِ تَقۡوِيم ٤ ثُمَّ رَدَدۡنَٰهُ أَسَفَلَ سَٰفِ ِينَ ٥ إِلَّا ٱلَّذِينَ ءَامَنُو ١ وَعَمِلُو ١ ٱلصُّلِحٰتِ فَلَهُمۡ أَجۡرٌ غَيۡرُ مَمۡنُونِ ٦ فَمَا يُكَذِّبُكَ بَعۡدُ بِٱلدِّينِ ٧ أَلَيۡسَ ٱللَّهُ بِأَحۡكَمِ ٱلۡحَٰكِمِينَ ٨ }

سورة التين

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Abstract

This study explores the potential of cellulose extracted from olive kernels (OKC) and olive pomace (OPC) as a modifier for drilling fluid properties. The cellulose, when integrated into real drilling fluid, exhibited minimal impact on pH levels and density across various concentrations. Plastic viscosity peaked at 0.1% and 0.25%, indicating potential overload points. Yield strength fluctuations correlated with changes in viscosity, impacting system performance.

Increasing cellulose concentration in bentonite resulted in elevated salinity level, thus the optimal filtration efficiency was achieved at 0.025% cellulose concentration from Olive kernels Overall, cellulose from olive in both samples (kernels & pomace) showcased promising potential in enhancing drilling fluid performance, controlling circulation loss, and modifying the mud filtration cake, thus serving as a viable alternative to CMC.

Keywords: drilling fluid, mud cake, Olive kernel's celluloses, Olive pomace cellulose Carboxy methyl cellulose (CMC).

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CHAPTER 1 Introduction

CHAPTER 1 INTRODUCTION

1.1 Introduction:

The liquid specially used in the oil and gas ir led as the drilling fluid or mud. It has several functions that are very essential to the drilling process. A loop is created through which the drilling fluid runs down the drill pipe, through and around the bit to reverse direction back up their surface.

Cooling and Lubrication: Friction between the drill bit and the rock formations creates a lot of heat when holes are being created. Additionally, the drilling fluid also cools and lubricates both the bit and strings to avoid any damage due to torsion. [1]

Formation Stabilization: Different formations that have a variety of instabilities or can collapse under the advancement drill bit. The hydraulic pressure from the drilling fluid holds the wellbore tightly to prevent any formation fluids (like oil, gas or water) from making their way into it. Stability is retained as a result of this process. [1]

Cuttings Removal: The drilling fluid transports the cuttings, debris and also other solids to the surface that enable uninterrupted drills. The viscosity and flow behavior of the fluid assist in carrying the cuttings up through the wellbore, where they can be easily separated at surface via removal. [1]

Pressure Control: Drilling fluid assists in maintaining the formation pressures during the drilling. To keep the hydrostatic pressure at a sufficient level, the fluid prevents the formation fluids to enter wellbore fluids and creates blowout or any other potential disasters. [1]

Wellbore Sealing: Drilling fluid seals the wellbore walls with a thin, and impervious filter cake. This filter cake seals the wellbore and reduces the fluid loss to adjacent formations, preserving reservoir quality and maintaining a good borehole stability. [2]

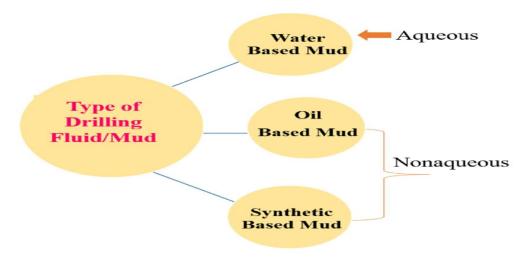


Figure (1.1): Types of drilling

1.2 Benefits of filtration in drilling IIIIa:

Improved Wellbore Stability: Leaching in the drilling fluid helps to remove the reactive clays and formation is unstable thereby reducing the chances of well instability. This can avert such challenges as backlog, varying sticking and also the damage formation.

Enhanced Drilling Fluid Performance: The degradation process assists in improving the performance of the drilling fluids to remove solid particles, colloidal materials and even residual contaminants. This also helps in maintaining the desired rheological properties, fluid density and filtration that allows for effective drilling activities

Minimized Formation Damage: Leaching can minimize the formation damage by eliminating drilling additives, solids and fines from migrating deep into the pore throats thereby reducing permeability. It allows for higher reservoir productivity and also saves on expensive corrective actions.

Reduced Environmental Impact: By leaching, the contaminants and solids are removed from the process that diminishes the environmental impact associated with drilling fluid discharge. It lowers the introduction of many undesirable chemicals into environmental surroundings, preserving aquatic systems and also minimizing the soil contamination [3].

Extended Equipment Life: Leaching helps to prevent the accumulation of solids and also deposits in the drilling equipment such as mud pumps, storage tanks or fluid recirculation systems. This increases the lifespan of this equipment, therefore reducing the overall maintenance costs as well and enhancing the general drilling efficiency. [4]

1.3 The role of CMC in filtration:

Filtration Control: CMC is widely used as a filtration control additive in the drilling fluids. It assists in the limitation of the fluid loss and maintaining a desirable rheology characteristic during filtration. A thin filter cake is formed by CMC adsorption on the wellbore wall, thus reducing the excessive fluid loss to the formation. [5]

Cake Formation: CMC assists in the development of a solid and also impermeable filter cake. When CMC is included in the drilling mud, it adsorbs onto the particles of filter cake making them stable and decreasing their compressibility. This leads to a better filtration control and minimizing of the intrusion of drilling fluids in the formation.

Suspension of Solids: CMC serves as a suspending medium for the solid particles in the drilling fluid. It allows the keeping of solids in suspension and rheological properties for the system to prevent settling. This becomes very crucial in preventing the solids build-up within the wellbore and ensuring a smooth drilling operations.

Filtrate Quality: CMC enhances the quality of the filtrate. It helps to lower the filtrate viscosity and also restricts solid formation that may cause problems with formations. Through the regulation of filtration rate and the characteristics of the filtrate, CMC helps to limit such harmful effects on the formation development and reservoir productive.

Compatibility with Other Additives: Compatibility with various secondary additives usually added in the drilling muds is achieved by CMC. It can be easily blended with many other polymers, viscosifiers and also fluid-loss control agents without any complications. This enables the development of very specific drilling fluid systems which are based on the identified well conditions. [6]

1.4 The problems of CMC in terms of cost:

Higher Cost Compared to Alternatives: CMC can be more expensive than the other filtration control additives or fluid-loss cont. The Purity, grade and sourcing influence the cost of CMC. This also additionally higher cost adds to the overall costs of formulating drilling fluids, particularly for high volume or long duration operations

Dosage Requirement: Compared to the other additives, CMC often requires a higher dosage in order to obtain the right filtration control and rheological characteristics. It can cause a greater cost due to the need for a larger quantity of CMC added during the drilling. This may be a considerable factor while formulating the fluid for high density or temperature drilling

Potential Waste and Disposal Costs: As CMC is a non-biodegradable additive, its disposal hence may incur many additional costs and environmental implications.

There is a lot of need for proper handling and also disposal of the drilling fluids containing CMC, including compliance with all regulations. The disposal costs may lead to a rise in the total cost of drilling. [7]

1.5 Carboxy methyl Cellulose (CMC)-Oil Drilling:

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 [21]

Γ	LV-CMC MV-CMC HV-CMC				C			
	Appe	arance	White or light yellow powder, freely flowable					
	Wat	ter,%	10 max					
	CM	IC,%	80.0 min	85.0 min		95.0 min		
	Substitution degree,%		0.80 min	0.65 min		0.8 min		
	P	РН	7.0-9.0	7.0-9.0		6.5-8.0		
Туре	Item		Typical measured values				s	
			Distilled w	vater	Brine	Satura	ted brine	
Base mud	Filtration loss, ml			60±10)	90±10	100±10	
	d Apparent viscosity, mPa•s			6 max		6 max	10 max	
	РН			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	Addition	rate, g/l			7.0 max	10.0 max	
	10ml Apparent viscositymPa•s				4.0 max	6.0 max		
MV-CMC	Addition rate,g/l		6.0		14.0	3.5		
	C Apparent viscosity,mPa•s			15 min		15 min	15 min	
	Filtration loss,ml			9 max		9 max	9 1	max

 Table (1.1): Physical Specification of CMC for oil drilling liquids [22]

1.6Cellulose in the olive kernel and olive pomace:

Olive kernels, a byproduct of olive oil production, are a rich source of cellulose They are a type of lignocellulosic material, with hemicellulose, cellulose, and lignin as the main compounds The high carbon content of olive kernels makes them suitable for the synthesis of porous carbon materials Cellulose extraction from olive kernels involves a multi-step chemical process to remove lipids, hemicellulose, and ligni [8]



Figure 0: Olive kernels

1.7 Quantity and Information About the Presence of Cellulose from Olive Kernels:

Cellulose is a complex carbohydrate and the main structural component of plant cell walls. It is found in varying quantities in different plant materials, including olive kernels.

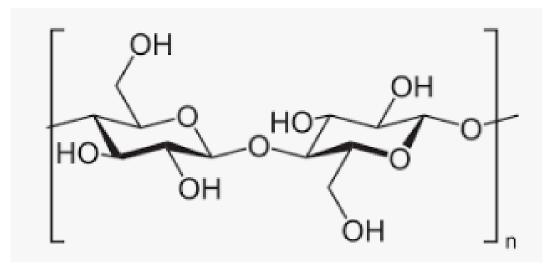


Figure (1.3): structural component of Cellulose

The quantity of cellulose in olive kernels can vary depending on factors such as the variety of olives, maturity stage, and processing methods. However, it is generally estimated that olive kernels contain around 30-40% cellulose.

Cellulose is insoluble in water and has a high resistance to digestion by human enzymes. This makes it an important dietary fiber that provides bulk to the diet and aids in maintaining regular bowel movements. It also offers various health benefits, such as promoting satiety, reducing cholesterol levels, and supporting healthy blood sugar levels.

In addition to cellulose, olive kernels also contain other components like lignin, hemicellulose, and pectin. These compounds contribute to the overall fiber content of olive kernels and have their own unique properties and health benefits.

It's worth noting that while cellulose is present in olive kernels, its extraction for commercial purposes may not be economically viable due to the relatively low quantity compared to other sources of cellulose-rich materials like wood or cotton.

1.8 Problem Statement:

In water-based drilling fluids, CMC compositions of the type are widely used. In well-drilling activities and play multiple roles that significantly affects the drill rate, cost of operations. The effectiveness, efficacy and security of its work. In a more precise sense, drilling fluid compositions retard the influx Seal permeating formations to prevent contamination of formation fluids into the wellbore. Drilling mud into the formation, keep formulations exposed stability immobilized and cooling its

lubricating Drill out of formation, hold back pressure and stabilize the formations. The significance of this research will be measured based on its importance. Be not only in a cost reduction for concrete production through olive kernel extracted component but also on this material local producing capability. The CMC specification complies with the international standard for specifications of API

CHAPTER 2 LITERATURE REVIEWS

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2.1 Drilling fluids "muds":

Drilling fluid, sometimes referred to as drilling mud, is a specifically prepared fluid that is used during the drilling of gas and oil wells. It has several advantages and functions that make it an essential part of the drilling process.

Drilling mud is primarily used to facilitate the removal of drill cuttings from the wellbore, which helps with the drilling process. It lessens friction and heat produced while drilling by cooling and lubricating the drill bit. Additionally, by stabilizing the wellbore walls, the mud helps to avoid cave-ins and collapses.

In addition to these basic functions, drilling mud serves several other important purposes:

Pressure control: Drilling mud exerts hydrostatic pressure on the wellbore walls, which helps to prevent formation fluids (such as oil or gas) from flowing into the wellbore. This pressure control is crucial for maintaining well integrity and preventing blowouts.

Formation evaluation: Drilling mud carries cuttings to the surface, allowing geologists and engineers to analyze them for information about the subsurface formations being drilled. This data helps in determining potential hydrocarbon reservoirs and making informed decisions about further drilling operations.

Well control: Drilling mud can be adjusted with additives to control well pressure during various stages of drilling, completion, or production. These additives can help prevent formation damage or improve well productivity.

Hole cleaning: The circulation of drilling mud removes drill cuttings from the wellbore, ensuring that they do not hinder further drilling progress or cause equipment damage.

Drilling mud is typically composed of a base fluid (water-based or oil-based), along with various additives such as clays (e.g., bentonite), polymers, weighting agents (e.g., barite), viscosities (e.g., xanthan gum), and lubricants. The specific composition depends on factors such as well conditions formation characteristics, and environmental regulations.

Overall, by performing essential tasks like cooling, lubrication, pressure management, hole cleaning, and formation evaluation, drilling mud contributes significantly to safe and effective drilling operations.

2.2 Drilling Fluid Additives:

Additives to drilling fluids are compounds that are incorporated into the fluid to improve its performance and solve particular problems that may arise during drilling operations. These additives accomplish a number of tasks, including boosting wellbore stability, lowering friction, preventing formation damage, managing viscosity, and improving drilling fluid stability.

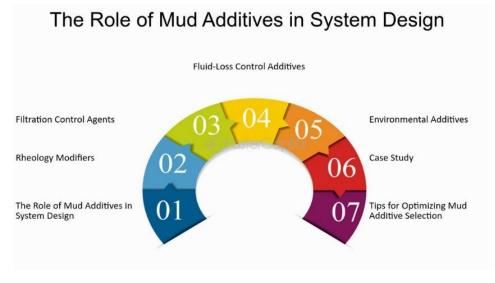


Figure (2.1): Drilling Fluid Additives role

Here Are Some Common Types of Drilling Fluid Additives:

Viscosities: These additives increase the viscosity of the drilling fluid to carry cuttings out of the wellbore and maintain hole stability. Examples include bentonite clay and polymers.

Weighting agents: Used to increase the density of the drilling fluid, weighting agents help control formation pressure and prevent blowouts. Barite is a commonly used weighting agent.

Fluid loss control agents: These additives reduce fluid loss into permeable formations by forming a filter cake on the wellbore walls. They help maintain wellbore stability and prevent formation damage. Examples include cellulose derivatives and synthetic polymers.

Lubricants: Lubricating additives reduce friction between the drill string and wellbore walls, minimizing torque and drag during drilling operations. Graphite or oil-based lubricants are commonly used.

Shale inhibitors: These additives help prevent shale swelling or disintegration by stabilizing shale formations during drilling operations. Potassium chloride or certain polymers can be used as shale inhibitors.

Corrosion inhibitors: Used to protect metal equipment from corrosion caused by corrosive elements present in drilling fluids, corrosion inhibitors extend equipment life and reduce maintenance costs.

Surfactants: Surface-active agents or surfactants alter interfacial tension between different phases in the drilling fluid system, aiding in emulsification or dispersion of solids or hydrocarbons.

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 [16]. 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 [16]

2.3. 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 [17].

• 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 [18]. 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 [18].

Drilling fluid density is required to contain the formation and formation fluids hydrostatic pressure can be calculated as Hydrostatic pressure (psi) = 0.052 x mudweight (ppg) x depth (ft.) or Hydrostatic pressure $(kg/cm^2) = 0.1$ 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 [19]. 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 analyze 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 [19]. 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 [20].

• Apparent Viscosity (AV)

Apparent viscosity refers to the degree of apparent with Newtonian fluid being set at a given shear speed. Subsequently, plastic viscosity means the flow properties once it has been set in. In other words, we can say that it is the basis for. resistance to flow brought about by friction between dispersed particles and the interaction of individual molecules. of the dispersant liquid. Lastly, yield limit is the relaxation time that was predicted by functional measuring method. forces of particles at the distance from each other (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 ([19]). (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

• 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 [21]. 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.

• 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 deflocculant, 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. [22].

• 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 ([17]).

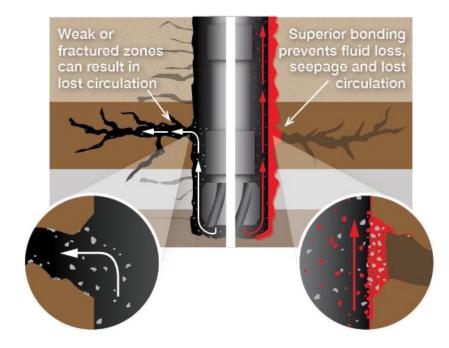


Figure (2.2): Superior bonding prevents fluid loss, seepage and lost circulation

CHAPTER 3 EQUIPMENT & MATERIALS

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3.1 Equipment:

• **Oven:** It is used to heat the liquid sample and dry the solid materials. (Sheldon Manufacturing)



Figure (3.1): Oven

• **Mixer with Container**: A mixer with a container is a versatile tool used for blending and mixing various ingredients or substances. It typically consists of a motorized base and a detachable container or bowl where the mixing takes place. (Shale Tech Solution)



Figure (3.2): mixer with container

• **PH meter:** A pH meter is a scientific instrument used to measure the acidity or alkalinity of water-based solutions. (MRC lab)



Figure (3.3): PH meter

• **FTIR:** Fourier Transform Infrared Spectroscopy. It is an analytical technique used to identify organic, polymeric, and in some cases, inorganic materials (LPD



lab services)

Figure (3.4): FTIR

• **Mud balance:** A mud balance is a device used to measure the weight of drilling fluid, cement, or any type of liquid or slurry. (FANN Model 140)



Figure (3.5): Mud balance

Rotary Viscometer: A viscometer, is an instrument used to measure the viscosity of a fluid, Viscosity refers to the resistance of a fluid to flow. (MXBAOHENG ZNN-



D6)

Figure (3.6): viscometer divise

• **API Filter Press:** is a type of industrial filtration equipment commonly used in the oil and gas industry. It is designed to separate solids from liquids by passing the mixture through a series of filter plates with filter media. (Qingdao Haitongda Special Instrument Co.,Ltd.)



Figure (3.7): API Filter Press

3.2 Materials:

• Dried sample of **olive pomace** after several operations such as (grinding and shaking)

Collection date: November 27th 2022

Press Date: December 28th 2023

Collection place: Musalatah Olive Press



Figure (3.8): olive pomace

• Dried sample of **olive kernel** after several operations such as (grinding and shaking)

Collected from: Sabha's farms

Date of collecting: April 4/23

*Stored from 2022 season



Figure (3.9): olive kernel

• API Test Calibration Bentonite: API Test Calibration Bentonite is a specific type of bentonite used for calibration and reference purposes in API (American Petroleum Institute) testing. According to PRC, it is designed to meet the standards set by the API for testing drilling fluids and it was packaged on November 18th 2023.

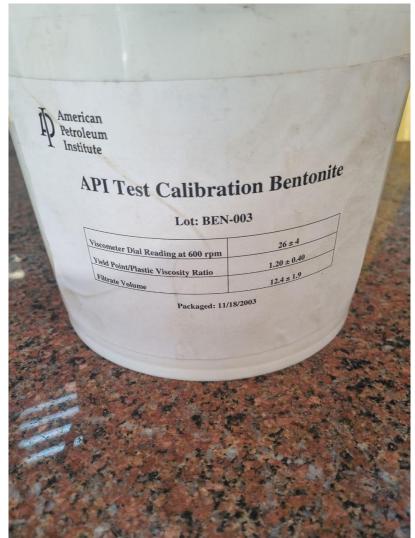


Figure (3.10): API Test Calibration Bentonite

3.3 The chemical part that used in the extraction part:

- Ethanol
- Methanol
- Acetone
- sodium hydroxide
- Hydrogen peroxide
- Acetic acid

CHAPTER 4 EXPERIMENT&PROCEDURES

CHAPTER 4 EXPERIMENT & PROCEDURES

4.1 Procedures

There are three main parts in this process from preparation to the mixing point:

4.1.1 Firstly, preparation of the sample:

1-Collection and preparation: Collecting olive kernel fresh can be generated from the residuals left after extraction of oil out. To rid the kernel off of any large impurities or debris, yields Pick up the olive rind, and gather fresh olive kernel. Ensure that the impurities or debris are large chunks. In case such need arises, eliminate all the stem, seeds and other non-cellulose materials others. Avoid using a dull knife: either use the sharpest one or opt for a food processor to cut the olive into tiny pieces smaller than 3 mm. This also assists in supporting the secondary size reduction and processing.

2-Washing: The olive pit is washed thoroughly with water to remove dirt, oils and other contaminants. This step helps in obtaining clean cellulose fibers.

3-Drying: After washing, the kernels were dried to remove excess moisture. The kernels can be air dried until completely dry.

4. Grinding or milling: After cutting, the size of the olive pit was reduced by grinding. This step aims to break up the kernel into smaller particles, which increases the surface area for cellulose extraction, using a mechanical grinder or mill



Figure (4.1): Ground olive kernel

4.1.2 Secondly, The Chemical treatment and extraction of cellulose:

1-Solvent extraction: The procedure of solvent extraction or washing with acetone and ethanol involves the use of organic means like acetone and also ethanol which are included during chemical reactions to extract desired substances.

The procedure involves 20 g of the sample, and the addition to themof100 mL acetone solution (CH3)2CO / solventand10 ml ethanol C2H5OH is performed for three hours under a temperature of 85 Deg C.



Figure (4.2): Solvent extraction

2-Alkaline treatment: After the completion of the solvent extraction process and drying the sample for 24 hours, the alkaline treatment phase is initiated.

Alkaline Solution: solution of [sodium hydroxide (6g) (1-5% W/v) with 100 ml of H₂O] with 10 g of sample.

Heating: The mixture of olive kernel powder and alkaline solution was heated to 90°C for 3 hours. Heating increases the reaction rate and helps decompose lignin and hemicellulose.



Figure (4.3): Heating

Stirring: The processing mixture is stirred or agitated continuously during alkaline treatment. Stirring ensures the homogeneous scattering of an alkaline liquid, improves contact between a solvent and olives particles, which facilitates its admission into plant material.

Filtration and washing: After the required time has elapsed, this mixture is usually filtered to separate the cellulose-rich residue from the liquid phase containing dissolved lignin and hemicellulose. After that, the cellulose residue is washed with water or any traces of alkali and side impurities are detected by Litmus paper.



Figure (4.4): Filtration and washing

3-Bleaching: After completing the alkaline treatment stage and washing the sample with water to achieve a neutral state, the acid treatment stage was carried out using of 3g of sample with acetic acid solution CH_3COOH (5%) and H_2O_2 solution (5.7%) to further remove impurities and improve whiteness and obtain purified cellulose .The experiment was conducted at temperature of 80°C for a duration of 2 hours.

The sample is washed with water to achieve a neutral pH state, and its pH is determined using litmus paper



Figure (4.5): Bleaching

4-Cellulose fibers were air dried: Once the cellulose fibers were thoroughly rinsed, they were carefully transferred from the filter paper or mesh to a clean, flat surface or drying racks. The fibers are arranged in a thin layer to facilitate the drying process. The cellulose fibers are left to air dry in a well-ventilated area away from direct sunlight. It may take several days for the cellulose to dry completely.



Figure (4.6): Drying

5-Store the dried cellulose: Once the cellulose fibers are completely dry, store them in a clean, airtight container to protect them from moisture and contamination. Store the container in a cool, dry place away from direct sunlight



Figure (4.7): The final product at this stage (the extracted cellulose from Olive kernel & pomace)

Yield of cellulose (efficacy)= (Oven dry weight of produced cellulose) / (Total sample) *100%

The amount of cellulose in the sample is found to be = 83.3%

- 4.1.3 The Mixing stage:
- **1.** Run IR test for the olive kernel and pomace in the order to investigate the new material.
- 2. Preparation of different concentrations of olive kernel and pomace:

Туре	concentrations	sample weight	Bentonite Weight	water weight	Preparation time	Resting time
OKC	0.025%	0.5625g	22.5 g	350 ml	15 m	none
OKC	0.05%	1.125 g	22.5 g	350 ml	15 m	none
OKC	0.1%	2.25 g	22.5 g	350 ml	15 m	none
OKC	0.25%	5.625 g	22.5 g	350 ml	15 m	none
OKC	0.4%	9 g	22.5 g	350 ml	15 m	none
OPC	0.025%	0.5625g	22.5 g	350 ml	15 m	24 hr
OPC	0.05%	1.125 g	22.5 g	350 ml	15 m	24 hr
OPC	0.1%	2.25 g	22.5 g	350 ml	15 m	24 hr
OPC	0.2%	4.5 g	22.5 g	350 ml	15 m	24 hr
OPC	0.3%	6.75 g	22.5 g	350 ml	15 m	24 hr
OPC	0.4%	9 g	22.5 g	350 ml	15 m	24 hr

Table(4.1). Preparing different concentrations with bentonie

3. Slices from the olive kernel and pomace sample preparation:

I. The olive kernel and pomace sample should be weighted to mix it with the right proportion of bentonite.



Figure (4.8): Sample weighing process

II. The specific weight percentages should be mixed with bentonite via a blender.



Figure (4.9): Sample mixing process

III. The mixed samples should be left to rest for 24 hours.



Figure (4.10): Samples after rest

After the 24-hour period, the following tests are initiated:

1- The samples are subjected to a pH test to measure their pH level.



Figure (4.11): Samples PH measuring process

2- The density of each sample is measured using a mud balance.



Figure (4.12): density measuring process

3- The viscometer is used to measure the viscosity of the samples.



Figure (4.13): viscosity measuring process

4- at this point the filtration test is conducted (which is the main test in this research):



Figure (4.14): filtration measuring process

5-the salinity test is run with the filtrate that comes out from the previous test.



Figure (4.15): salinity measuring process

6- calculate the gel strength before and after 10mintues. (By using the viscometer)

7- calculate both the yield point strength & ratio.

4.2 Experiment:

4.2.1 PH test:

- A. A Calibrate the pH Device
- B. Prepare the Sample
- C. Immerse the pH Electrode
- D. Read and Record pH Value

4.2.2 Density test:

- A. Prepare the Measurement Device
- B. Collect the Sample
- C. Place the Sample in the Measurement Device
- D. Record the Measurement
- E. Account for Temperature

4.2.3 viscosity test:

- A. Prepare the Measurement Device
- B. Collect the Sample
- C. Place the Sample in the Measurement Device
- D. Measure the Ø600 and Ø300

Now calculate the PV & AV

 $(PV) = \emptyset 600 - \emptyset 300$

 $(AV) = \emptyset 600/2$

Where: PV is the Plastic viscosity AV is the Apparent viscosity

4.2.4 Gel Strength:

From the previous viscometer test :-

Take the measurement of the $\emptyset 600$ exactly after (10seconds) and then take a second measurement after (10 minutes) from finishing the spinning part of the viscometer.

where:

The Ø600 after (10seconds) is the gel strength of the mixture after (10seconds) The Ø600 after (10 minutes) is the gel strength of the mixture after (10 minutes)

4.2.5 yield point strength calculations:

(Yield strength) = $\emptyset 300 - (P V)$

CHAPTER 5 Result and Discussion

CHAPTER 5 Result and Discussion

5.1.1 FTIR Result Olive Kernel (OKC):

The FT-IR spectra of cellulose showed characteristic bands at 3351.36, 2910.16, 1043.64, 1602.95, 1302.16 cm⁻¹, related to v O-H stretching, v CH₂ stretching, v C-O-C stretching, v OH bending from absorbed water, CH bending.

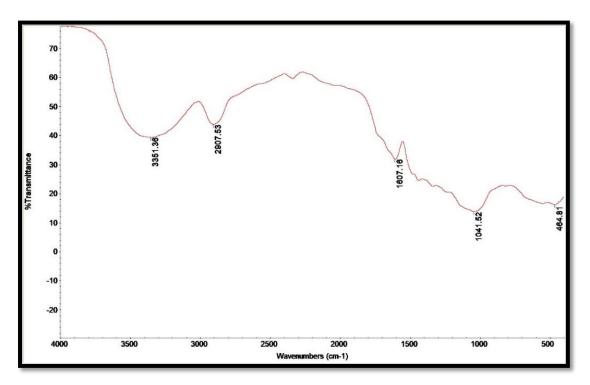


Figure (5.1): FTIR Result Olive Kernel (OKC):

5.1.2 FTIR Result Olive Pomace (OPC):

The FT-IR spectra of cellulose showed characteristic bands at 3511.36, 2907.53, 1041.52, 1607.16, 1310.22 cm⁻¹, related to v O-H stretching, v CH₂ stretching, v C-O-C stretching, v OH bending from absorbed water, v CH bending

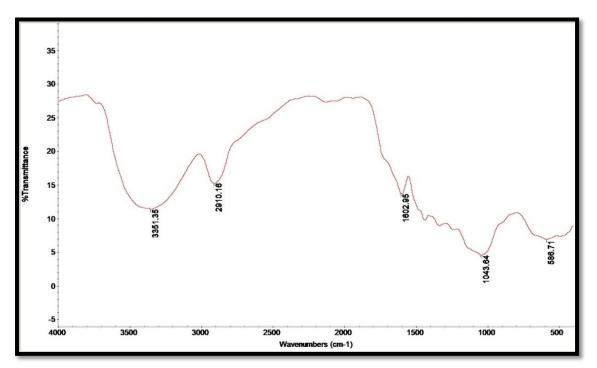


Figure (5.2): FTIR Result Olive Pomace (OPC)

5.2 PH test Results:

Concentrations	PURE	0.025%	0.05%	0.1%	0.25%	0.4%
ОКС	9.2	8.5	8.52	8.6	8.65	8.7
OPC	9.2	8.6	8.67	8.68	8.7	8.72

Table (5.1): PH result Table.

The experiment involved adding natural cellulose extracted from OKC to drilling fluid at different percentages. The effects of this addition were evaluated by measuring the pH values of two samples: OKC and OPC. The results indicated slight variations in pH values with different cellulose concentrations For the OKC sample, the pH values ranged from 8.5 to 8.7 as the cellulose percentage increased from 0.025% to 0.4%. Similarly, for the OPC sample, the pH values ranged from 8.6 to 8.72 as the cellulose percentage increased

The observed differences in pH values between the cellulose concentrations were not significant, suggesting that the addition of cellulose from OKC or OPC had minimal impact on the overall pH level of the drilling fluid. It is worth noting that the pH values obtained were within a narrow range, indicating that the drilling fluid remained relatively stable despite the cellulose addition.

These findings imply that the addition of OKC or OPC cellulose to drilling fluid is unlikely to cause major pH disruptions.

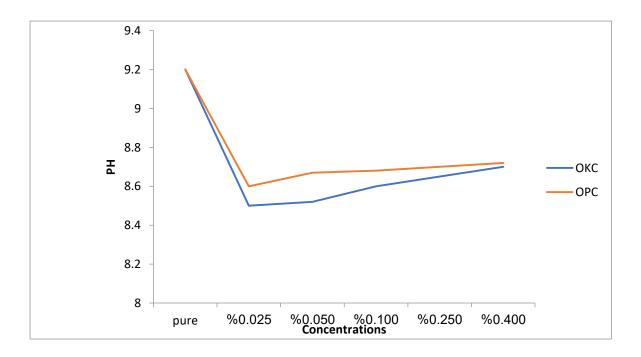


figure (5.3): PH resultes for the OKC and OPC mixed with bentonite.

5.3 Density test Results:

Concentrations	PURE	0.025%	0.05%	0.1%	0.25%	0.4%
ОКС	8.33	8.65	8.65	8.65	8.65	8.7
OPC	8.33	8.6	8.6	8.60	8.6	8.7

 Table (5.2): Density resultes Table.

At the same concentrations, it can be observed that both samples yield similar density values. It can be inferred that the cellulose extracted from OKC and OPC have similar density properties at the specified concentrations.

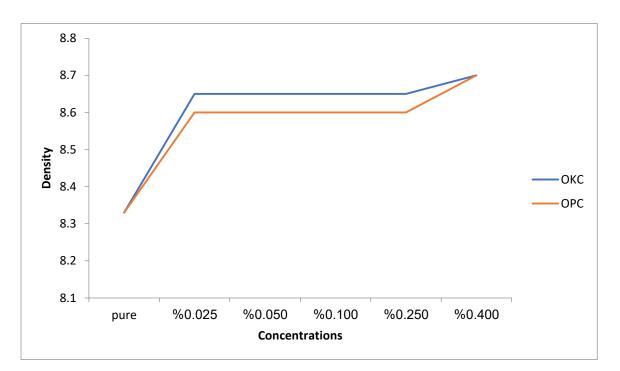


figure (5.4): Density resultes for the OKC and OPC mixed with bentonite.

5.4.1 Plastic viscosity (P.V) test Results:

Concentrations	PURE	0.025%	0.05%	0.1%	0.25%	0.4%
ОКС	7	8	9	10	9.5	9.4
OPC	7	8.5	9.2	10.4	9.5	9.5

Table (5.3): Plastic viscosity resultes for the OKC and OPC mixed with bentonite.

Based on the provided data, we can analyze the results of the two samples, OKC and OPC, in terms of Plastic viscosity (P.V) at different concentrations.

For both samples, as the concentration decreases, the Plastic viscosity tends to decrease as well. This indicates that higher concentrations of the samples result in higher viscosity.

Comparing the two samples, we can see that the values of Plastic viscosity are generally similar between OKC and OPC at the same concentrations. However, there are slight variations in some cases.

At a concentration of 0.25% and 0.4%, both samples display the same Plastic viscosity values of 9.5. This suggests that the two samples have similar flow properties at these concentrations.

At a concentration of 0.1%, the OKC sample has a Plastic viscosity of 10, while the OPC sample has a slightly higher value of 10.4. This indicates that the OPC sample may have a slightly higher resistance to flow compared to the OKC sample at this concentration.

At lower concentrations of 0.025% and 0.05%, the OPC sample continues to exhibit higher Plastic viscosity values compared to the OKC sample. The values for the OPC sample are 8.5 and 9.2, respectively, while the OKC sample has values of 8 and 9.

There was a breakthrough between concentrations of 0.1 % & 0.25 and the reason is likely that the internal structure of the sample have reached the maximum plastic degree therefore, it might be a point where the system becomes overloaded with cellulose particles. This overload could lead to the entanglement or aggregation of cellulose molecules, causing a decrease in the effectiveness of cellulose as a thickening agent. Later on, direct decline in the results was noticeable

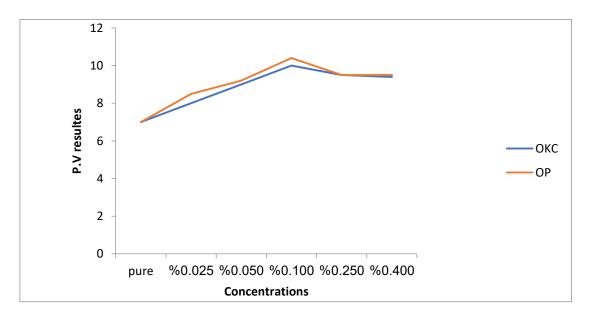


figure (5.5): P.V resultes for the the OKC and OPC mixed with bentonite.

5.4.2 Apparent viscosity (A.V) test Results:

Concentrations	PURE	0.025%	0.05%	0.1%	0.25%	0.4%
ОКС	13	12	21	16.5	12.5	14
OPC	13	16.5	12.5	15	15.5	14.5

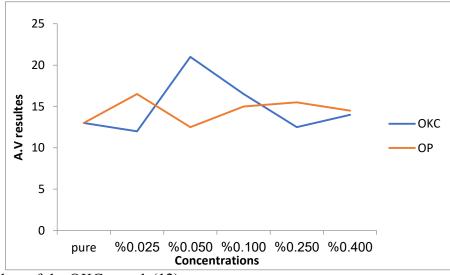
Table (5.4): A.V	resultes for	the OKC mixed	with bentonite.
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At the 0.4% concentration, the apparent viscosity of the OPC sample (14.5) is slightly higher than that of the OKC sample(14)

At the 0.25% concentration, the apparent viscosity of the OPC sample (15.5) is higher than that of the OKC sample(12.5)

The apparent viscosity of the O.K sample was at its highest level (16.5) when using concentration of 0.1% while in O.P sample was (15) when using the same concentration , this is explained by the highest degree , because some materials exhibit shear-thinning behavior, where their viscosity decreases with increasing shear rate. As the concentration of cellulose increases, the shear rate experienced by the fluid might also increase, causing the apparent viscosity to decrease despite the increase in cellulose concentration. And thus, it's a point of research in the future

At the 0.025% concentration, the apparent viscosity of the OPC sample (16.5) is



higher than of the OKC sample(12)

figure (5.6): A.V resultes for the the OKC and OPC mixed with bentonite.

5.5 Yield strength Results:

Concentrations	PURE	0.025%	0.05%	0.1%	0.25%	0.4%
ОКС	10	8	24	13	11	10
OPC	10	13	9	20	13	7

Table (5.5): Yield strength resultes for the OKC mixed with bentonite.

Enhanced Yield Strength: The addition of natural cellulose extracted from OKC and OPC may have contributed to an increase in yield strength. Cellulose is a strong and fibrous material, and its presence in the drilling fluid may have provided additional structural support, resulting in higher yield strength. The ability of cellulose particles to hydrate and disperse uniformly in the drilling fluid can influence its rheological properties. At higher concentrations, cellulose particles might have difficulty hydrating or dispersing effectively, leading to a decrease in yield strength.

Optimal Concentration: The results indicate that the yield strength varied depending on the concentration of the cellulose additive. At certain concentrations (0.1% for OKC and 0.1% and 0.25% for OPC), the yield strength was higher compared to other concentrations. This suggests that there may be an optimal concentration range for achieving maximum yield strength enhancement.

Different Cellulose Properties: The two types of cellulose additives, extracted from OKC and OPC, may have different properties than affect their performance in the drilling fluid. This could explain the variation in yield strength between the two additives at different concentrations.

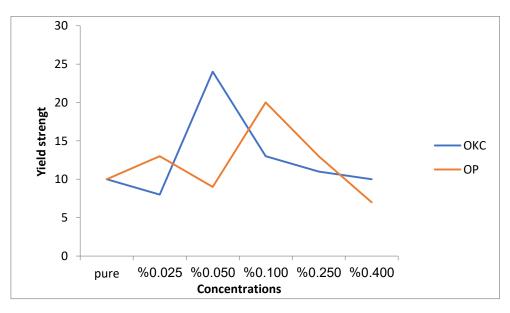


figure (5.7): Yield strength resultes for the the OKC and OPC mixed with bentonite.

5.6 Salinity Results:

Concentrations	0.025%	0.05%	0.1%	0.25%	0.4%
ОКС	185	192	203	217	234
OPC	158	173	196	233	262

Table (5.6): Salinity resultes for the the OKC and OPC mixed with bentonite.

Table 5.6 shows the results of the Salinity test for different concentrations of OKC, mixed in Bentonite. While, the figure 5.6 shows the results of the same Salinity test of different concentrations of same material, mixed with bentonite.

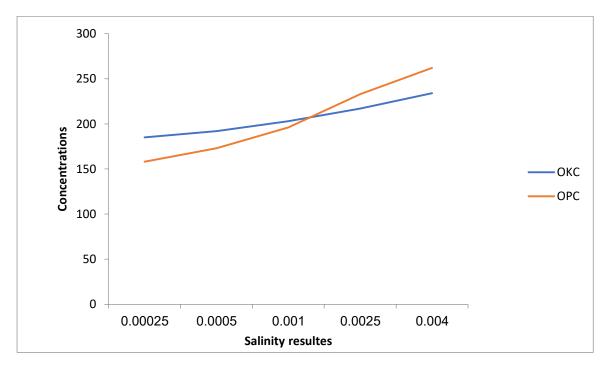


figure (5.8): Salinity resultes for the the OKC and OPC mixed with bentonite.

5.7 filtration Results:

Concentrations	PURE	0.025%	0.05%	0.1%	0.25%	0.4%
ОКС	12.5	10	10.5	11	11.5	11.5
OPC	12.5	11.5	11.5	11.5	11.5	12.5

Table (5.7): filtration resultes for the the OKC and OPC mixed with bentonite.

Effect of Cellulose Addition: The addition of natural cellulose extracted from OKC had an impact on the filtration results in both the OKC and OPC samples. The filtration results varied depending on the percentage of cellulose

Optimal Cellulose Percentage: In the OKC sample, the highest filtration result of 11.5 was obtained when 0.4% and 0.25% of cellulose by weight of the drilling fluid were added. This suggests that a higher cellulose percentage may have a negative effect on filtration.

Consistent Filtration Results: In the OPC sample, the filtration results remained consistent at 11.5 for all the cellulose percentages tested (0.4%, 0.25%, 0.1%, 0.05%, 0.025%). This indicates that the addition of cellulose from OPC did not significantly affect the filtration performance of the drilling fluid.

Potential Applications: The results suggest that the addition of cellulose extracted from OKC can potentially improve the filtration efficiency of drilling fluids. This can be beneficial in drilling operations where controlling fluid loss and maintaining drilling fluid properties are crucial.

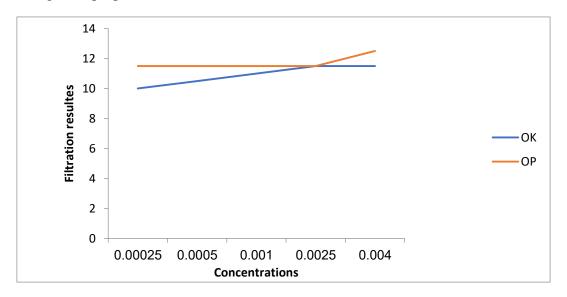


figure (5.9): Filtration resultes for the the OKC and OPC mixed with bentonite.

CHAPTER 6 CONCLUSION& RECOMMENDATIONS

CHAPTER 6 CONCLUSION & RECOMMENDATIONS

6.1 Conclusion:

- > The material has almost no effect on PH levels.
- > The density almost constant at various concentration.
- ➤ The plastic viscosity peaks at 0.1 % & 0.25 and it is likely to be the point where the system becomes overloaded with cellulose particles.
- The apparent viscosity peaks in O.K sample at (16.5) when using concentration of 0.1% while in O.P sample was (15) when using the same concentration because some materials exhibit shear-thinning behavior, where their viscosity decreases with increasing shear rate.
- > the severe yield strength changes are due changing in viscosity, and viscosity affects yield strength directly via this relation [Yield strength = \emptyset 300 (P.V)]
- Increasing the concentration of the extracted cellulose from OKC and OPC in bentonite leads to increasing of salinity.
- > The best filtration value is 10 at 0.025% of extracted cellulose from OKC.
- Increasing the concentration of the substance does not mean obtaining better results.
- Based on the results of the tests it was confirmed that the extracted cellulose from OKC and OPC has a pretty good feature in enhanced Drilling fluid & control circulation loss.

6.2 **Recommendation:**

- The change of viscosity with the difference in temperature must be taken into consideration during the preparation of concentrates.
- ➤ Based on the results, we strongly recommended to use different concentrations, materials and compare them with current results.
- there may be an optimal concentration range for achieving maximum yield strength enhancement.
- Also, the best-case scenario might be pretty feasibly if it has been found, since the better results were at the lowest concentrations.

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