SUDAN UNIVERSITY OF SCIENCE AND TECHNOLOGY

College of Petroleum Engineering and Technology

Department of Petroleum Engineering

Project Title:

Effect of Fly Ash on Water Based Drilling Fluids Properties (Garri4 power plant sample)

تأثير الرماد المتطاير على خواص سوائل الحفر ذات الاساس المائي
(عينة محطة قري4 الحرارية)

A Graduation Project Submitted in Partial Fulfillment for the Requirements of the Degree of Bachelor of Science in Petroleum Engineering.

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الإستهلال

يقول الله تعالى في محكم تنزيله :

﴿ اللَّهُ نُورُ السَّمَاوَاتِ وَالْأَرْضِ ۚ مَثَلُ نُورِهِ كَمِشْكَاةٍ فِيهَا مِصْبَاحٌ ۖ الْمِصْبَاحُ فِي زُجَاجَةٍ ۖ الزُّجَاجَةُ كَأَنَّهَا كَوْكَبٌ دُرِّيٌّ يُوقَدُ مِنْ شَجَرَةٍ مُبَارَكَةٍ زَيْتُونَةٍ لَا شَرْقِيَّةٍ وَلَا غَرْبِيَّةٍ يَكَادُ زَيْتُهَا يُضِيءُ وَلَوْ لَمْ تَمْسَسْهُ نَارٌ ۚ نُورٌ عَلَىٰ نُورٍ ۗ يَهْدِي اللَّهُ لِنُورِهِ مَنْ يَشَاءُ ۚ وَيَضْرِبُ اللَّهُ الْأَمْثَالَ لِلنَّاسِ ۗ وَاللَّهُ بِكُلِّ شَيْءٍ عَلِيم ﴾.

صدق الله العظيم

الآية (35): سورة النور.

DEDICATION

Firstly, we would like to praise our God, the creator, the source of inspiration and guidance, who make us able to get such success and honor. Moreover, we would like to dedicate this humble effort to our teachers, colleges, families and friends for their kindness, encouragement and commitment.

Lastly we would like to thank full heartedly everyone makes it possible to achieve this work, and a special thanks to the collage of petroleum Engineering and Technology.

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ABSTRACT

The objective of a drilling operation is to drill, evaluate and complete a well that will produce oil and/or gas efficiently, drilling fluids perform numerous functions that help make this possible so it is very important to design and an adequate drilling mud taking into account all well condition.

This study based on a laboratory experiments and investigates the behavior and properties of drilling mud by adding various concentrations by weight of local Fly Ash that produced in Garri4 power plant, following the API RP13B-1 standard practices, in order to improve the properties of drilling mud and reduce the negative effect of Fly Ash on environment and human health.

The laboratory experiments have been divided into two type of tests, the chemical test and rheological test, in the chemical test the XRD and XRF tests were used to obtain the characteristics of Fly Ash and the results showed that it consists of different types of chemical compounds such as calcium oxide (CaO), silicon dioxide (SiO2) and Ferric oxide (Fe2O3). The other test was to estimate the physical and rheological properties of drilling mud systems that have been developed.

The Analytical results of the laboratory tests indicated that the drilling mud properties were enhanced with the use of fly ash. A comparison between a mud system contains Fly Ash and other mud systems containing Barite, Starch, or PAC-LV, was carried out and the results obtained emphasis that the mud systems containing Fly Ash was good in filtration loss control, gel strength, density, and PH.

التجـــريـــــــــــد

الهدف من اي عملية حفر هو حفر وتقييم واكمال البئر التي ستنتج النفط او الغاز بكفاءة، سوائل الحفر تؤدي العديد من الوظائف التي تمكن من تحقيق تلك الكفاءة ولذلك يجب ان يتم تصميم سائل حفر بصورة ملائمة تراعي ظروف البئر.

هذه الدراسة مبنية على التجارب المعملية وهي تستكشف سلوك وخصائص سائل الحفر عند اضافة تراكيز وزنية مختلفة من الرماد المتطاير المنتج محليا في محطة قري4 لتوليد الطاقة، بناء على التوجيهات القياسية التي يوصي بها معهد البترول الأمريكي (API RP13B-1)، وذلك لتحسين خواص سائل الحفر، و تقليل الأثر السالب للرماد المتطاير على البيئة و صحة الإنسان.

التجارب المعملية التي اجريت قسمت لنوعين من الاختبارات: إختبارات كيميائية و تيارية. الإختبارات الكيميائية اجريت بإستخدام (XRD,XRF) لتعيين التراكيب الكيميائية للرماد المتطاير، و أظهرت النتائج أنه يحتوي على أنواع مختلفة من المركبات الكيميائية، مثل أوكسيد الكالسيوم، ثاني اوكسيد السيليكون و أوكسيد الحديدوزوغيرها.اما الإختبار الآخر فيبين الخواص الفيزيائية و التيارية لنظام سائل الحفر.

أظهرت النتائج التحليلية للإختبارات المعملية أن خواص سائل الحفر تحسنت عند إستخدام الرماد المتطاير, واجريت مقارنة بين نظام سائل حفر يحتوي على الرماد المتطاير مع سوائل حفر أخرى احتوت على بارايت أو(starch) أو (PAC-LV). و كان الاستنتاج من نتائج هذه المقارنة أن سائل الحفر المحتوي على الرماد المتطاير جيد في التحكم في حجم الراشح، و قوة التماسك، و الكثافة، و الأس الهيدروجيني.

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Symbols and abbreviations:

Rpm = revolution per minute.

Ppg = pound per gallon.

θ_{300}= viscosity dial reading at 300 rpm.

θ_{600}= viscosity dial reading at 600 rpm.

θ_{3}@10sec= viscosity dial reading at 3 rpm after 10 second.

θ_{3}@10min viscosity dial reading at 3 rpm after 10 minute.

AV= apparent viscosity.

μ_a= apparent viscosity.

PV=plastic viscosity.

YP= yield point.

K= fluid consistency index.

n= flow behavior index.

ρ_m= the mud density.

M_w = the mass of water.

M_s = the mass of solids.

V_W= the volume of water.

V_s= the volume of solids.

H^+= Hydrogen ions concentration.

API= American petroleum institute.

PAC= Poly anionic Cellulose.

CMC= Sodium Carboxy-Methyle Cellulose.

V_1 = initial volume, bbl.

D_1 = density of weight material.

V_F = Final volume.

D_F = Final density of the mixture.

XRF= X-ray Fluorescence.

XRD= X-ray Diffraction.

SG= specific gravity.

WBM= Water base mud.

SUST= Sudan university of science and technology.

Fig = Figure.

CHAPTER ONE

Chapter one

Introduction

1.1. General

Drilling fluids play a significant role in the petroleum industry all over the world as it's the heart of drilling, it serves many functions such as: controlling formation pressure, removing cuttings from wellbore, sealing permeable formations and also maintaining wellbore stability. So it's very important to make sure that an appropriate selection of drilling fluids must be made due to the variety of formation conditions, drilling fluids also have a high relative cost comparing with the total cost that required to drill a well.

The locally produced fly ash from Garri4 coke-fired power plant poses a real environmental problem, because it is disposed-off in huge piles in the plant's vicinity. The purpose of this research was to study the possibility of the Sudanese fly ash to be used as a chemical additive in the drilling fluids.

1.1. problem statements:

To enhance the efficiency of drilling mud there are many chemicals were used and they are very expensive, for sure it's a good thing to minimize this cost by getting the advantage of using a waste material

Fly ash which is produced from burning of petroleum coke is harmful for human, and makes many maladies, such as cancers, asthma, heart disease, respiratory, stroke, inflammation and immunological reactions. And it is dangerous for animals too. So it's very important to minimize those negative effects on organisms this can be done by reducing its quantity in the air and land, by using it in a useful thing like wells drilling.

1.2. Research objectives:

(1) The main objective is to study the physical and chemical properties of fly ash.

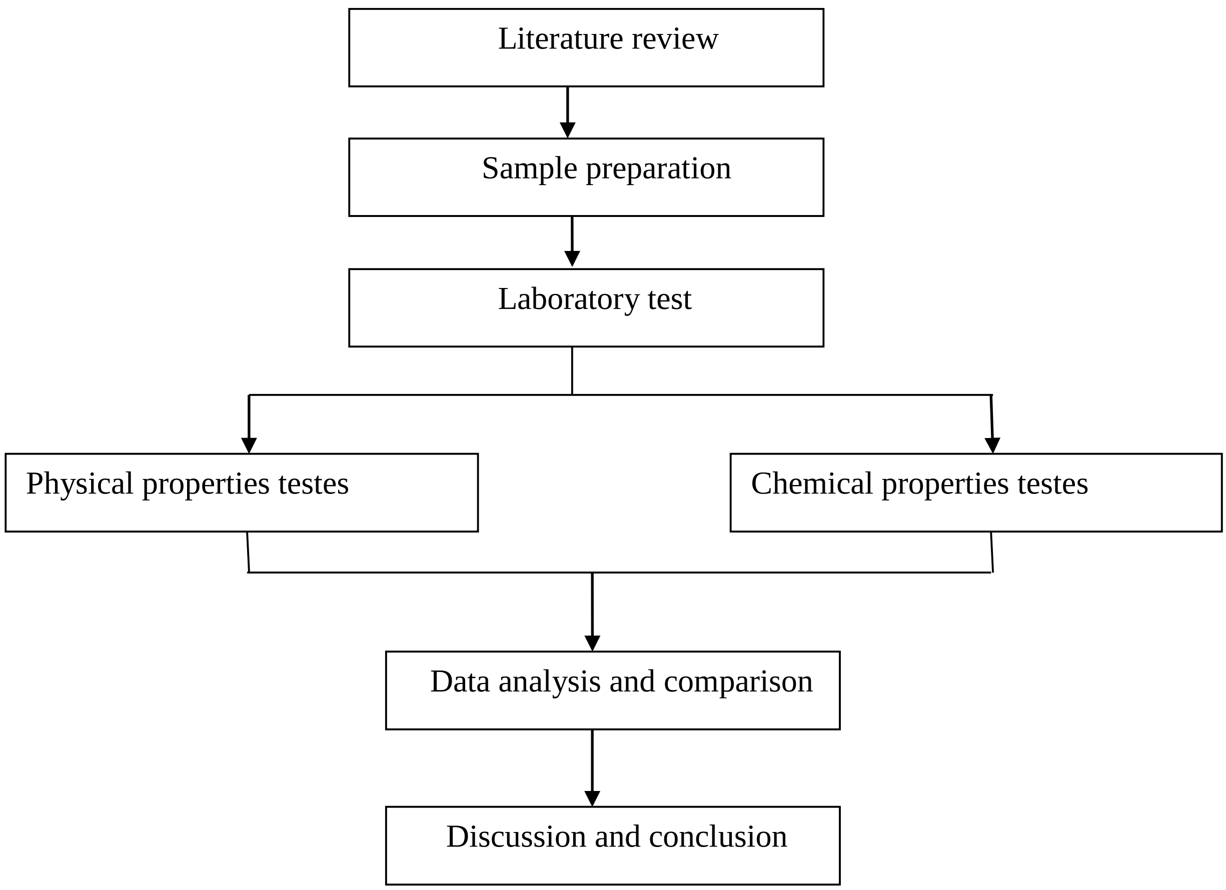
(2) Study the physical properties including (PH, Density, Filtration volume, and rheological properties) of water-based drilling mud mixed with fly ash.

(3) Identify weather fly ash can improve the efficiency of drilling mud or not.

1.3. Scope:

The scope of this research is covering the physical and rheological properties of drilling mud includes, density, plastic viscosity, apparent viscosity, yield point, gel strength, PH and filtration.

1.4. Research flow chart:



CHAPTER TWO

Chapter 2

Literature Review and Theoretical Background

2.1. Literature review:

Vikas Mahto and Rajat Jain (2013) studied the effect of fly ash on the rheological and filtration properties of water based drilling fluids with the objective of the development of environmentally acceptable non-damaging and inhibitive drilling fluid system to drill sensitive formations. Initially, different drilling fluids combinations were prepared using carboxy methyl cellulose (low viscosity grade), polyanionic cellulose, xanthan gum, and potassium chloride. The rheological properties as well as filtration properties of these drilling fluids were measured by API recommended methods. These drilling fluids show very good rheological behavior but poor filtration loss characteristics. When fly ash was added in these drilling fluid combinations, a nanoparticles fluid system was established which has better control on filtration properties without affecting the rheological properties and has good potential for the drilling of sensitive formation.

Pornthip Korsinwattana(2014) investigate the physical and chemical properties of fly ash and drilling mud mixed with fly ash. She mixed drilling mud with concentration of various fly ashes in 3,5 and 7 % w/v that measured at 30,60and 90 C, represent that the drilling mud mixed with %3w/v of fly ash at 30 C testing is a high potential additive for enhancement rheological properties of water base drilling mud specially in the increasing of apparent viscosity, yield point ,pH and high efficiency of resistivity reduction however the high concentration of fly ash effects to the increasing of filter loss ,mud cake thickness and sand content.

Eman salah El din Mohamed(2016) studied the possibility of using fly ash that is produced from petroleum coke burning in garri4 power station plant north of Khartoum, she found that garri4 fly ash could be used successfully for replacement of sodium sulfide in the main soaking, beside using it to replace the hydrate lime in the unhairing and reliming processes.

2.2. Drilling fluid background:

Drilling fluid (or drilling mud) is a mixture of water/oil, clay and various chemicals; drilling fluid is the most important element in any drilling operation and play important role and should be designed carefully to provide its desired function. In addition to serving these functions, the drilling fluid should not (I) have properties detrimental to the use of planned formation evaluation techniques, (2) cause any adverse effects upon the formation penetrated, or (3) cause any corrosion of the drilling equipment and subsurface tubulars. (Adam T.Bourgoyne. et.al -1986)

2.2.1. Drilling Fluid Functions:

Drilling fluid functions describe tasks which the drilling fluid is capable of performing, although some may not be essential on every well. Removing cuttings from the well and controlling formation pressures are of primary importance on every well. Though the order of importance is determined by well conditions and current operations, the most common drilling fluid functions are:

1. Remove cuttings from the well.
2. Control formation pressures.
3. Suspend and release cuttings.
4. Seal permeable formations.
5. Maintain wellbore stability.
6. Minimize reservoir damage.
7. Cool, lubricate, and support the bit and drilling assembly.
8. Transmit hydraulic energy to tools and bit.
9. Ensure adequate formation evaluation.
10. Control corrosion.
11. Facilitate cementing and completion.
12. Minimize impact on the environment.

(MI-SWACO -2006)

2.2.2 Drilling fluid selection criteria:

Drilling fluids are selected on the basis of one or more of the following criteria:

- Cost
- Application and Performance
- Production Concerns

• Logistics

• Exploration Concerns

• Environmental Impact and Safety.

(Chevron Texaco and BP -2002)

2.2.3. Drilling fluid classification:

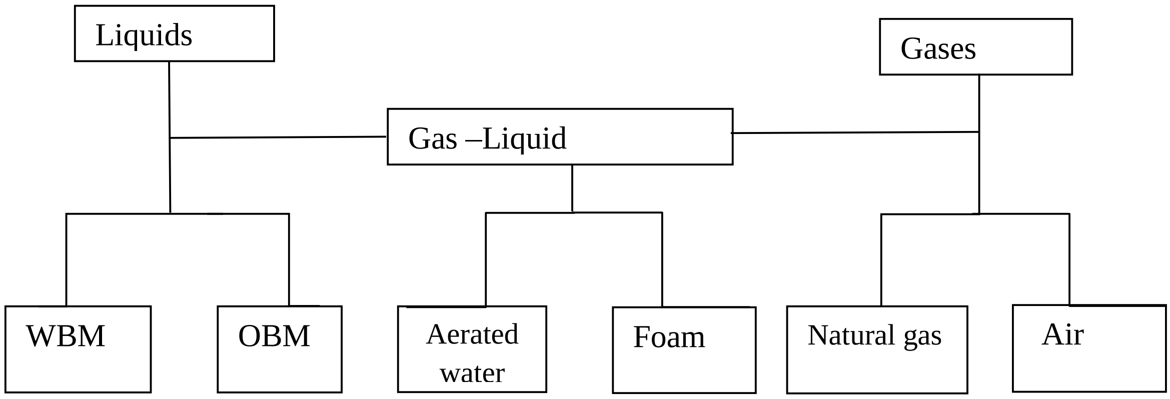


Fig. (2.1): classification of drilling mud

These are fluids where water is the continuous phase. The water may be fresh, brackish or seawater. Whichever is most convenient and suitable to the system or is available. The following designations are normally used to define the classifications of water based drilling fluids:

1. Non-dispersed-Non – inhibited.
2. Non-dispersed – Inhibited.
3. Dispersed - Non-inhibited.
4. Dispersed – Inhibited.

(Rabia, H -2001)

2.3. Drilling fluid properties:

The physical and chemical properties allow the function of the fluids to be fulfilled. The most important fluid properties include:

2.3.1. Density:

Mud density is defined as the mass of a given sample of mud divided by its volume. Mud weight is dependent upon the quantity of solids in the liquid phase, either in solution or suspended by the particles of the liquid phase.

$$\rho_m = \frac{M_w + M_s}{V_W + V_s} \qquad (2.1)$$

Where,

ρ_m= the mud density

M_w = the mass of water

M_s= the mass of solids

V_W= the volume of water

V_s= the volume of solids

Mud weight is measured in the field by using mud balance and it is expressed in pounds per U.S gallon (ppg), or pound per cubic foot.

2.3.2. Rheological Properties:

The rheological parameters describe the flow behavior of drilling fluid, as mentioned in chapter 3.

2.3.3. Filtrate & Filter Cake:

The fluid lost to the rock is described as "filtrate", and the layer of solids deposited on the rock surface is described as "filter cake". The volume of filtrate and thickness of filter cake of a mud sample can be determined by using a "Filter press".

2.3.4. PH value:

The PH of a solution is the logarithm of the reciprocal of the (H+) concentration in gram moles per litter, expresses as:

$$pH = - \log(H^{+}) \qquad (2.2)$$

where,

H^{+}= Hydrogen ions concentration

The PH value is important because its affect the solubility of the organic thinners and the dispersion of clays presents in the mud.

2.4. Drilling fluid additives:

There are many drilling fluid additives which are used to develop the key properties of the mud. The variety of fluid additives reflects the complexity of mud systems currently in use. The complexity is also increasing daily as more difficult and challenging drilling conditions are encountered. The most common types of additives used in water-based and oil- based muds. These are:

2.4.1. Weighting Material:

Weighting materials or densifers are solids material which when suspended or dissolved in water will increase the mud weight. Most weighting materials are insoluble and require viscosifers to enable them to be suspended in a fluid. clay is the most common viscosifier.

Mud weights higher than water (8.3 ppg) are required to control formation pressures.

Table (2.1): material used as densifiers

Material	**Principle component**	**Specific gravity**	**%Acid solubility**
Galena	PbS	7.4-7.7	0
Haematite	Fe_2O_3	4.9-5.3	50+
Magnetite	Fe_3O_4	5-5.2	0
Illmenite	$FeO.TiO_3$	4.5-5.1	20
Barite	$BaSO_4$	4.2-4.6	0
Siderite	$FeCO_3$	3.7-3.9	95+
Celestite	$SrSO_4$	3.7-3.9	0
Domomite	$CaCO_3.MgCO_3$	2.8-2.9	99
Calcium carbonate	$CaCO_3$	2.6-2.8	99

Barite (or barites)is barium sulphate,$BaSO_4$and it is the most commonly used weighting material in the drilling industry.

2.3.2. Viscosifiers:

The ability of drilling mud to suspend drill cuttings and weighting materials depends entirely on its viscosity. Without viscosity, all the weighting material and drill cuttings would settle to the bottom of the hole as soon as circulation is stopped.

Table (2.2): viscosifer materials

Material	Principal Component
Bentonie	Sodium/Calcium Aluminosilicate
CMC	Sodium Carboxy-Methyle Cellulose
PAC	Poly anionic Cellulose
Xanthan Gum	Extracellullar Microbial Polysaccharide
HEC	Hyroxy-ethyl Cellulose
Guar Gum	Hydrophilic Polysaccharide
Resins	Hydrocarbon co-polymer
Silicates	Mixed Metal Silicates

2.4.3. Filtration control material:

Filtration control materials are compounds which reduce the amount of fluid that will be lost from the drilling fluid into a subsurface formation caused by the differential pressure between the hydrostatic pressure of the fluid and the formation pressure. Bentonite, polymers, starches and thinners or deflocculants all function as filtration control agents.

2.4.4. Rheological control materials:

When efficient control of viscosity and gel development cannot be achieved by control of viscosifier concentration. Material is called “thinners”, “dispersants”, and/or “deflocculates” are added. These materials cause a change in the physical and chemical interactions between solids and/or dissolved salts such that the viscous and structure forming properties of the drilling fluid are reduced. Thinners are also used to reduce filtration and cake thickness.

2.4.5. Alkalinity and PH control:

The PH affects several mud properties including:

1. Detection and treatment of contaminant such as cement and carbonate.

2. Solubility of many thinner and divalent metal ions such as calcium and magnesium.

Alkalinity and pH control additives include: NaOH , KOH, Ca(OH)2, NaHC03 and Mg(OH)2.

2.4.6. Lost circulation material:

Lost circulation material is added to a mud to control loss of mud into highly permeable sandstones, natural fractures, cavernous formation, and induced fractures. Before a mud filter cake can be deposited, lost circulation additives must bridge across the large opening sand provide a base upon which the mud cake be built. Note that the largest fracture or opening that may be sealed is 0. 25in.larger openings must be sealed using special techniques.

2.4.7. Lubricating material:

Lubricating materials are used mainly to reduce friction between the wellbore and the drill string. Lubricating materials include: oil (diesel. mineral, animal, or vegetable oils), surfactants graphite, asphalt, gilsonite, polymer and glass beads.

2.4.8. Shale stabilization materials:

Shale stabilization is achieved by the prevention of water contacting the open shale section. Shale stabilizers include: high molecular weight polymers, hydrocarbons, potassium and calcium salts (e.g. KCl) and glycols.

(Rabia, H -2001)

2.5. Fly ash background:

Fly ash is a fine grey powder consisting of spherical particles that are generated in combustion of pulverized coal in a thermal power plant, it’s also known as flue-ash. Ash that doesn’t rise is called bottom ash. fly is generally captured by electrostatic precipitators or other particle filtration equipment before the flow gases reach the chimneys of coal-fired power plants and together with bottom ash removed from the bottom of the furnace is in this case jointly known as coal ash.

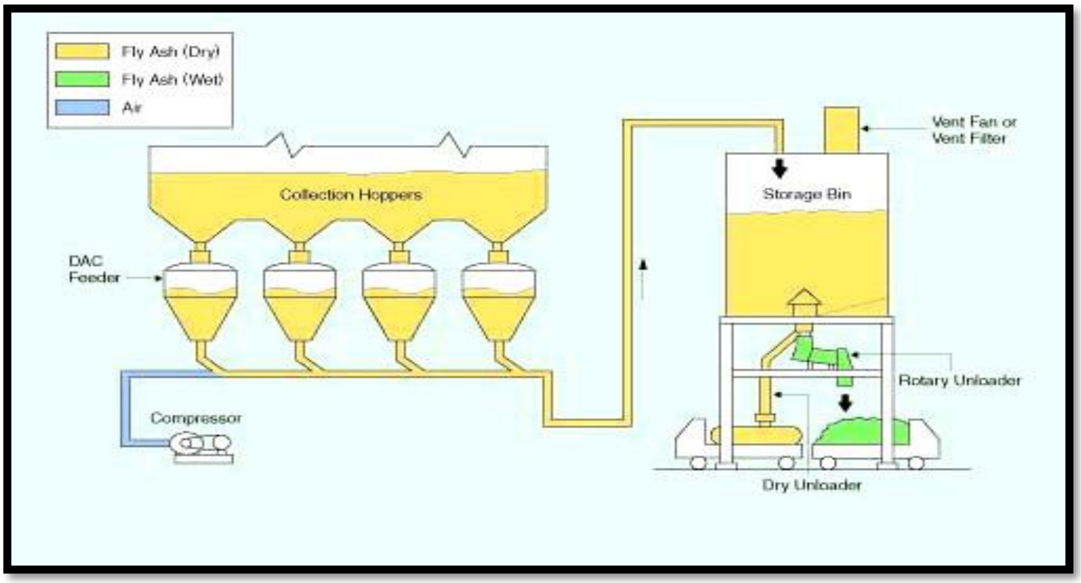


Fig. (2.2): Schematic of fly ash production process

2.5.1. Classification of fly ash:

There are two classes of fly ash defined by ASTM C618: Class F fly ash and Class C fly ash. The main differences between these classes are the amount of calcium, silica, alumina, and iron content in the ash.

2.5.1.1. Class F fly ash

It's produced from burning of harder, older anthracite and bituminous coal. this type is pozzolanic in nature, and contains less than 20% lime (CaO). Possessing pozzolanic properties, the glassy silica and alumina of class F fly ash requires a cementing agent such as Portland cement quicklime, or hydrated lime –mixed with water to react and produce cementations compounds. Alternatively adding a chemical activator such as sodium silicate (water class) to a class F can form a geopolymer.

2.5.1.2. Class C fly ash

Fly ash produced from the burning of younger lignite or sub-bituminous coal, in addition to having pozzolanic properties, also has some self-cementing properties in the presence of water, class C fly ash hardens and gets strangers overtime. Class c fly ash generally contains more than 20% lime (Cao). un like class F self-cementing class C fly ash does not require an activator.

Table 2.3:sample oxide analysis of fly ash class F and fly ash class C

COMPOUNDS	FLY ASH CLASS F %	FLY ASH CLASS C %
SIO2	20-60	40-60
AL2O3	5-35	20-30
Fe2O3	10-40	4-10
CaO	1-12	10-30
MgO	0-5	1-6
SO3	0-4	0-2

The chief difference between class F and class C fly ash is in the amount of calcium and the silica, alumina, and iron content in the ash. In class F fly ash, total calcium typically ranges from 1 to 12 percent.

2.5.2. Properties of fly ash:

2.5.2.1. Physical Properties:

Fly ash consists of fine, powdery particles that are predominantly spherical in shape, either solid or hollow, and mostly glassy (amorphous) in nature. The carbonaceous material in fly ash is composed of angular particles. Although sub bituminous coal fly ashes are also silt –sized, they are generally coarser than bituminous coal fly ashes.

The color of fly ash can vary from tan to grey to black depending on the amount of unburned carbon on the ash. The lighter the color, the lower carbon content. Lignite or sub bituminous fly ashes are usually light tan to buff in color, indicating relatively low amount of carbon as well as presence of some lime or calcium. Bituminous fly ashes are usually some shade of grey, with the lighter shade of gray generally indicating higher quality of ash.

2.5.2.2. Chemical properties:

Depending upon the source and make-up of the coal being burned, the components of fly ash vary considerably but all fly ashes includes:

- Silicon Dioxide (SiO_2).
- Calcium Oxide (CaO) also known as Lime.
- Iron (III) Oxide (FeO_2).
- Aluminum Oxide (Al_2O_3).

2.5.3. Uses of fly ash

Generally, there is no governmental registration or labeling of fly ash utilization in the different sectors of the economy, industry, infrastructures and agriculture. But there are many existing uses of Fly ash such as:

- Waste stabilization and solidification.
- Mine reclamation stabilization of soft solid.
- Road sub base construction.
- Cement clinkers production.
- As agreement substitute material (e.g. for brick production).
- Mineral filler in asphaltic concrete.
- Agricultural uses: soil amendment, fertilizer, cattle feeders, soil stabilization in stock feed yards, and agricultural stakes.
- Loose application on rivers to melt ice.
- Loose application on roads and parking lots for ice control.

(Eman Salah El Din Mohamed -2016)

CHAPTER THREE

Chapter 3

Research Methodology

3.1. Introduction:

The main objective of the study is to evaluate the effect of fly ash on the drilling fluid properties, by estimating it's physical and rheological properties, drilling mud is tested according to the API Standard practices (API RP 13B-1) for determining the following characteristics of water Based drilling fluids:

I. Drilling fluid density (mud weight).
II. Viscosity and Gel strength.
III. Filtration and PH.

3.2. Fly ash collection:

Fly ash sample was collected directly from the stack in Garri4 power plant, and the other additives were also prepared. The tests had been carried out at Sudan university of science and technology drilling fluid research lab. The sample of fly ash was sieved manually for less than75-micronby mesh sieve at room temperature following API recommended practices. A 99.5 % of the gross sample were collected through the mesh sieve. The chemical properties of Fly Ash were listed in table (4.1).

Fig (3.1): Mesh Sieve Less Than 75-Micron

3.3. Material balance:

Material balance concepts are useful to the mud engineer for solving many field problems that can be represented as simple mathematical relationship. Applications for the material balance method are:

a. Weight up.
b. Dilution.
c. Mixing two fluids.
d. System building.
e. Solids analysis.

The general material balance equation is written as follows:

$$\boldsymbol{V_1 D_1 + V_2 D_2 + V_3 D_3 + \text{etc.} = V_F D_F} \qquad \textbf{(3.1)}$$

Where:

V_1 = initial volume, bbl

D_1 = density of weight material, lb/gal

V_F = Final volume, bbl

D_F= Final density of the mixure, lb/gal

3.4. Materials Description:

There are many materials used to improve the drilling fluid properties those includes:

1. Bentonite:

This is the most widely used additive in the oil industry. The Drilling grade bentonite is a naturally occurring clay containing the clay mineral smectite and accessory minerals such as quartz, mica, feldspar and calcite. This material increases the viscosity of mud and thus increase its ability to suspend solid materials.

Fig. (3.2): Bentonite Sample (SUST lab)

2. Barite:

Barite is barium sulphate, $BaSO_4$ and it's the most commonly used weighting material in the drilling industry, it has a specific gravity in the range of (4.20 –

4.60). It is normally supplied to a specification where the specific gravity is about 4.2.

Fig. (3.3): Barite Sample (SUST lab)

3. Soda Ash:

It refers to sodium carbonate (Na_2CO_3), used to treat the hardness materials such as calcium.

Fig. (3.4): Soda Ash Sample (SUST lab)

4. Caustic soda:

Caustic soda is potassium hydroxide (KOH) to control mud PH.

Fig. (3.5): Caustic Soda (SUST lab)

5. PAC - LV:

Low Viscosity Polyanionic Cellulose (PAC-LV), is a semi-synthetic polymer which has been modified to increase its tolerance to salts (up to saturation) and calcium. Used for filtration control, viscosity modification and shale stabilization.

Fig. (3.6): PAC-LV Sample (SUST lab)

6. Starch:

Starch is natural polymer used in drilling primarily to reduce filtrate loss and to provide viscosity, it disperses in water to form a swollen particle that physically blocks the bore space.

Fig. (3.7): Starch Sample (SUST lab)

7. Fly Ash:

Fly Ash is a by-product created during the combustion of coke and coal in coke or coal –fired power plants. It has a fine particles rise with the flue gasses and are collected with filter bags or (ESP)electrostatic precipitators. It has a specific gravity range from 1.9 to 2.6.

Fig. (3.8): Fly Ash Sample (SUST lab)

3.5. Laboratory tests:

The test procedures divided into two groups physical and chemical properties tests. Following the relevant API standard practices.

3.5.1. Chemical properties:

The objective of chemical properties is being to measure the composition and elements of the additive (Fly Ash) by using X-ray Diffractometer (XRD) and X-ray Fluorescence spectrometer (XRF), respectively:

3.5.1.1. X-ray Fluorescence:

Sample is prepared to use 0.5 to1.0 gram, its compacted and spread out to the holder. Sample holder is analyzed by X-ray fluorescence spectrometer(XRF), EDX-8000 (Figure 3.8) and spent time to 200 seconds. A typical X-ray generator passes an electric current through a filament, which cases an electron to be emitted. These electrons are then accelerated by high voltage (usually somewhere between 20 and 100 kV) towards an anode (target). Results are analyzed in the spectrum, including Rayleigh and Compton scattered characteristic line from the X-ray generator, peak caused by X-ray diffraction, and sum/escape peak. A quantitative technique, the peak height of any element is directly related to the concentration of that element within the sampling volume. The XRF results are presented as the percentage of major elements.
(pornthip korsinwattana 2014)

Fig.(3.9): Shimadzu (EDX-8000) X-ray Fluorescence

(<u>Forensic laboratories</u>)

3.5.1.2. X-ray Diffraction:

Amount of 1.0 to 1.5 grams of samples are compacted and spread out to holder. Sample holder is analyzed by X-ray diffractometer (XRD), XRD-7000 X-ray Diffractometer (Figure 3.9) and spent time 15 minutes per sample. XRD performed on polycrystalline material the incident X-ray beam is diffracted by innumerous crystallites in specific 2 Theta directions. Data is recorded the exact 2 Theta positions a narrow slit in front of a point detector is required. Conditions of analysis include a Cu standard ceramic sealed tube (0.4x12 mm), X-ray generation (30 kV, 10mA), angular range analysis (2Θ, 5^{o} to 80^{o}) and accuracy ($\pm 0.02^{o}$ throughout the entire measuring range) Results are calculated relative intensity, divide the absolute intensity of every peak by the absolute intensity of the most intense peak, and then convert to a percentage. (pornthip korsinwattana 2014)

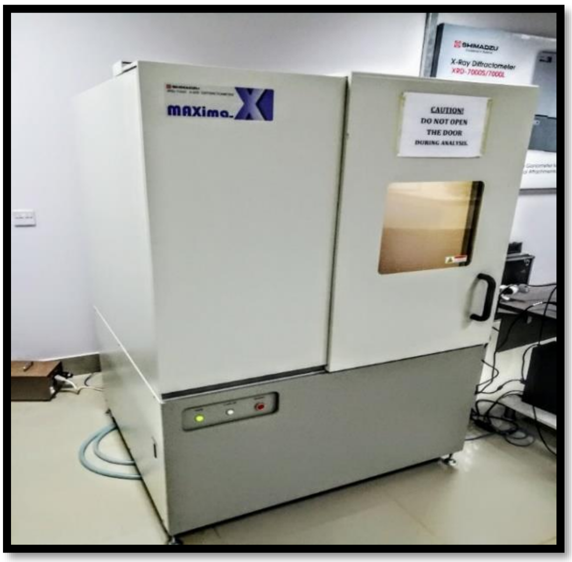


Fig. (3.10): Shimadzu, Maxima XRD-2000 X-ray diffractometer
(Forensic laboratories)

3.5.2. Physical and rheological properties:

The physical properties consist of density, rheology, filtration and hydrogen ion, as listed below:

3.5.2.1 Density: pounds/gallon (lb/gal)

The density (commonly referred to as mud weight) is measured with a mud balance of sufficient accuracy. For all practical purposes, density means weight per unit volume and is measured by weighing the mud. The weight of mud may be expressed as a hydrostatic pressure gradient in $lb/in2$ per 1,000 ft of vertical depth (psi/1,000 ft), as a density in lb/gal, $lb/ft3$ or Specific Gravity (SG).

3.5.2.2 Apparent viscosity(μ_a)

Is a rheological property calculated from rheometer readings. It measures the shear rate of drilling fluid specified by API. Apparent viscosity is expressed in centipoises (cP); it indicates the amount of force required to move one layer of f0luid in relation to another. The apparent viscosity can calculate from the following equation:

$$\mu_a = \Theta_{600}/2 \qquad (3.2)$$

Where:

Θ_{600} = viscosity dial reading at 600 rpm.

3.5.2.3. Plastic viscosity(PV): centipoise (cps)

Plastic viscosity is the shearing stress in excess of yield point that will induce a unit rate of shear. It is that part of flow resistance caused by mechanical friction, which occurs: (1) between the solids in the mud, (2) between the solids and the liquid that surrounds them, and (3) with the shear of the liquid itself. Therefore, all practical viscosities can be calculated from equation:

$$\text{PV}=\Theta_{600} - \Theta_{300} \qquad (3.3)$$

Where:

Θ_{300}=viscosity dial reading at 300rpm.

3.5.2.4. Yield point(γ_p): lbs/100 ft

Yield point is the second component of resistance to flow in drilling fluid. It is a measurement of electro-chemical or attractive forces in a fluid underflow condition. These forces are a result of negative charges located on or near the particle surfaces and are dependent on: (1) the surface properties of mud solids, (2) volume concentration of solids, and (3) the electro-chemical environment of ions. The yield point could be regulated by the use of chemical additives. Therefore, it dictates the nature and degree of treatment necessary to maintain a desirable fluid viscosity. The yield point value can be calculated from equation:

$$\gamma_p = \Theta_{300} - PV \qquad (3.4)$$

3.5.2.5. Gel strength: lbs/100 ft^2(10 sec/10 min)

Gel strength is a measurement of the thixotropic properties of drilling fluid under static condition. Similar to the yield point, gel strength is a measure of the electro-chemical attractive forces between solid particles. Yield point and gel strength are the result of the flocculation forces of a thixotropic fluid. Gel strength is measured by rotational speed of 3 rpm. The drilling fluid is allowed to stand undisturbed for 10 seconds and 10 minutes that are referred to initial gel strengths and 10 minutes gel strength respectively, at which time of an outer cup is rotated at 3 rpm and the maximum deflection of the dial is recorded. The gel strength results are reported in lb/100ft2.

3.6. Power law model:

The power law model's parameters in the term of behavior index (n) and consistency (k) are calculated from viscometer reading using following equations:

$$n = 3.32 \times \log(\Theta_{600}/\Theta_{300}) \qquad (3.5)$$

$$k = 510 \times \Theta_{300}/511^{n} \qquad (3.6)$$

where:

n = no flow behavior index (Non-Newtonian).

k = fluid consistency index.

3.7. Equipment and Tests Procedures Description:

There are many equipments that used to estimate the drilling fluid properties include:

3.7.1. Digital balance:

A digital device used to measure the weight of solid materials in gram unit, as shown in fig (3.10):

Fig (3.11): Digital balance (SUST lab)

3.7.2. Mud Mixer:

An instrument used for mixing the mud components, its mainly consist of motor, cup and a shaft with fan to agitate the mixture. Fig (3.11) shows mud mixer:

Fig (3.12): Mud Mixer (SUST lab)

3.7.3. Mud Balance:

This device used to estimate drilling mud density after it being prepared by mud mixer.   As shown in fig (3.12) bellow:

Fig (3.13): Mud Balance (SUST lab)

Mud density test Procedures:

1) Remove the lid from the cup, and completely fill the cup with the mud to be tested.

2) Replace the lid and rotate until firmly seated, making sure some mud is expelled through the

3) Wash the mud from the outside of the cup, and dry it.

4) Place the balance arm on the base, with the knife edge resting on the fulcrum.

5) Move the rider until the graduated arm is level, as indicated by the level vial on the beam.

6) At the edge of the rider closest to the cup, read the density or weight of the mud.

7) Report the result to the nearest scale division, either in lb/gal, lb/ft3,
psi/1,000 ft of depth or Specific Gravity (SG).

8) For balances not showing the desired scale, the equations shown in Table 1 may be used. (MI-SWACO -2006)

3.7.4. PH indicator:

The PH indicator sticks see fig (3.14) are coated with indicators of such nature that the color is dependent on the PH of the fluid in which the sticks is placed. Standard color charts are supplied for comparison with the test stick, allowing estimation of PH to 0.5 PH units over the entire PH range.

Fig. (3.14): PH indicator

PH test procedures:

1) Place an indicator stick in the mud and allow it to remain until the color has stabilized, usually less than a minute. Rinse the stick off with deionized water but do not wipe.

2) Compare the colors of the stick with the color standard provided and estimate the pH of the mud.

3) Report the pH of the mud to the nearest 0.5 pH units. (MI-SWACO -2006)

3.7.5. Six-speed Viscometer:

Drilling mud is tested for the rheological properties, Rheology testing is carried out by a Viscometer Fig (3.15) and measured by using six rotational speeds (3, 6, 100, 200, 300 and 600 rpm) for the viscosity, yield point and gel strength relate to flowing properties of drilling mud The apparent viscosity, plastic viscosity and yield point are calculated from 300 and 600 rpm reading following formulas from API standard.

Fig (3.15): Six-speed Viscometer (SUST lab)

Rheological properties test procedures:

- Procedures for apparent viscosity, plastic viscosity and yield point determination:

  1) Place recently agitated sample in a cup and adjust surface of mud to scribed line on the rotor sleeve.

  2) Start the motor by placing the switch in the high-speed position with the gear shift all the way down. Wait for a steady indicator dial value, and record the 600 RPM reading. Change gears only when motor is running.

  3) Change switch to the 300-RPM speed. Wait for a steady value and record 300-RPM reading.

  4) Plastic viscosity in centipoise = 600 reading minus 300 reading.

5) Yield Point in lb/100 ft2 = 300 reading minus plastic viscosity in centipoise.

6) Apparent viscosity in centipoise = 600 reading divided by 2.

- Gel strength determination:

1) Stir sample at 600 RPM for approximately 15 secs and slowly lift the gear assembly to the neutral position.

2) Shut motor off and wait 10 sec.

3) Flip switch to the low-speed position and record maximum deflection units in lb/100 ft2 as initial gel. If the dial indicator does not return to zero with motor off, do not reposition.

4) Repeat 1 and 2, but allow 10 min, then place switch in the low-speed position and read maximum deflection units as the 10-min gel. (MI-SWACO -2006)

3.7.6. API Filter Press:

Filtration is tested by using Fann filter press (Figure 3.16) which determines the API filtrate loss through standard filter paper and the filter cake thickness under static conditions. It consists of fluid cup support by a frame, a filtering medium and a pressurized nitrogen gas cylinder and regulator. A graduated cylinder is used to measure the discharged filtrate. The 100 psig is applied to a column of fluid for the 30 minutes' period, which filtrate volume and filter cake thickness are measured and recorded.

FIG (3.16): API Filter Press (SUST lab)

API fluid loss test procedures:

1) Have air or gas pressure of 100 psi available.

2) Remove the lid from the bottom of the clean and dry cell. Place the O-ring in an undamaged groove, then invert to fill. Any mechanical damage could prevent it from sealing. Seal the inlet with a finger.

3) Fill the cell with mud to within 1 ⁄4 inch of O-ring groove. Place filter paper (Whatman No. 50 or equivalent) on top of O-ring. Place the lid on the filter paper with the flanges of the lid between the flanges of the cell, and turn clockwise until hand tight. Turn the cell over and insert the male cell coupling into the female filter press coupling and turn either direction to engage.

4) Place a suitable graduated cylinder under the filtrate opening to receive the filtrate.

5) Open the inlet valve applying pressure to the cell. (A rapid fluctuation downward of the needle can be seen as pressure fills the cell).

6) The normal API test period is 30 min. At the end of the test, close the valve. Pressure will be shut off at the source, and the pressure will bleed off automatically. Remove the cell.

7) Report the fluid loss in milliliters unless otherwise specified.

8) Disassemble the cell, discard the mud and use extreme care to save filter paper with a minimum of disturbance of the cake. Wash the cake gently to remove excess mud. Measure the thickness of the filter cake and report in 32nds of an inch. (MI-SWACO - 2006)

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CHAPTER FOUR

Chapter 4

Results and Discussion

4.1. Introduction

This chapter describes results of the laboratory experiments and their analysis, the results consist of the chemical properties of Fly ash, the rheological properties of mud systems, PH and Filtration.

4.2. Chemical properties

The chemical properties are determined by estimating the elements and compounds of the fly Ash composition by using XRD and XRF analysis.

Table (4.1) Analytical results of fly ash

Elements	Result (%)	Compounds	Result (%)
Ca	66.186	CaO	55.976
Si	13.012	SiO2	21.315
Fe	6.414	SO3	13.281
S	5.852	Fe2O3	4.471
Ba	3.267	BaO	1.360
Ni	1.711	NiO	1.055
Sr	1.494	SrO	0.850
Mn	1.050	MnO	0.669
Y	0.373	K2O	0.414
K	0.301	TiO2	0.359
Ce	0.291	Y2O3	0.229
Zn	0.050	ZnO	0.022

4.3. Determination of rheological properties, density, and filtration volume:

To study the effect of fly ash on the mud properties, many tests were done, such as mud weight test, PH, rheological, and filtration test. And many equations were used to identify the flow type, and calculate plastic viscosity, apparent viscosity, yield point and gel strength. All procedures of testing following the API recommended practices of pilot test, the composition of mud systems is illustrated in the table below:

Table (4.2): Basic Mud system components

Water Volume/ Ml	Solid materials / gram		
	Soda ash	Bentonite	Caustic soda
350	0.01	25.5	0.1

Table (4.2) illustrates the main components of each mud system.

Table (4.2): Basic mud system properties

properties	Units	Value
Mud Weight	ppg	8.63
ϴ600	rpm	35
ϴ300	rpm	28
ϴ3 @10sec	rpm	12
ϴ3@ 10min	rpm	46
Apparent Viscosity (AV)	cp	17.5
Plastic Viscosity(PV)	cp	7
Yield Point (YP)	$Ib/100ft^2$	21
Gel strength	$Ib/100ft^2$	12/46
Non Newtonian Index (n)	_	0.32
Consistency	_	3.8

Index (K)		
PH	_	11
Fluid loss volume/1min	ml	4.4
Fluid loss volume/7.5min	ml	10
Fluid loss volume/30min	ml	20
Yp/PV	_	3

Table (4.3) illustrates the result of laboratory experiments which have been done for the basic mud system.

Table (4.4): Properties of basic mud system consist of different concentrations of barite

Properties	**Units**	**Percentage of Barite by weight (%)**			
		1%	3%	**5%**	**7%**
Mud weight	Ppg	8.66	8.67	8.7	8.74
θ600	Rpm	25	23	27	28
θ300	Rpm	20	18	21	23
θ3 @10sec	Rpm	8	9	9	9
θ3@ 10min	Rpm	49	43	52	53
Apparent Viscosity (AV)	Cp	12.5	11.5	13.5	14
Plastic Viscosity (PV)	Cp	5	5	6	5
Yield Point(YP)	$Ib/100ft^2$	15	13	15	18
Gel strength	$Ib/100ft^2$	8/49	9/43	9/52	9/53

Non-Newtonian Index (n)	_	0.32	0.35	0.36	0.28
Consistency Index (K)	_	2.7	2.02	2.2	4
PH	_	**11**	**11**	**11**	**11**
Fluid loss volume/1min	ml	4.2	4.1	4	3.8
Fluid loss volume/7.5min	ml	9.6	9.4	9	9
Fluid loss volume/30min	ml	19.2	18.8	18	18
YP/PV	_	3	2.6	2.5	3.6

Table (4.4) illustrates the results obtained from testing basic mud system consist of different concentrations of barite (1%, 3%, 5%, and 7%).

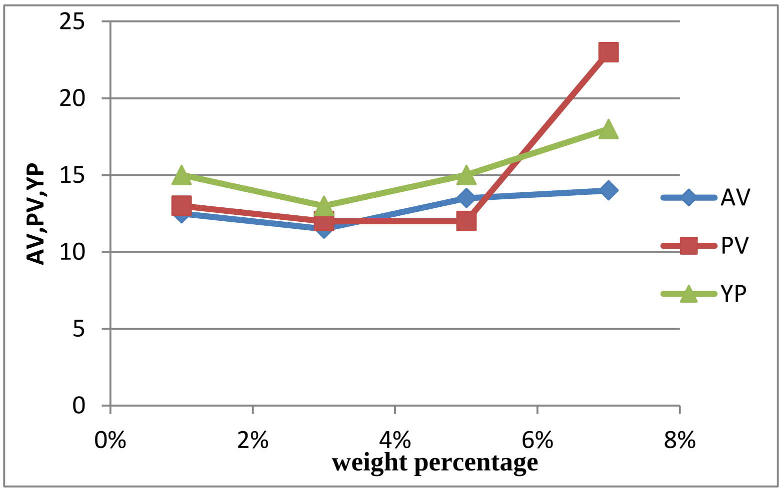

Fig. (4.1): AP, PV, YP vs Concentration of Barite

Figure (4.1) shows that, the PV of barite increases with concentration increment by a rate faster than the increment of AV rate, and also show the behavior of them and YP.

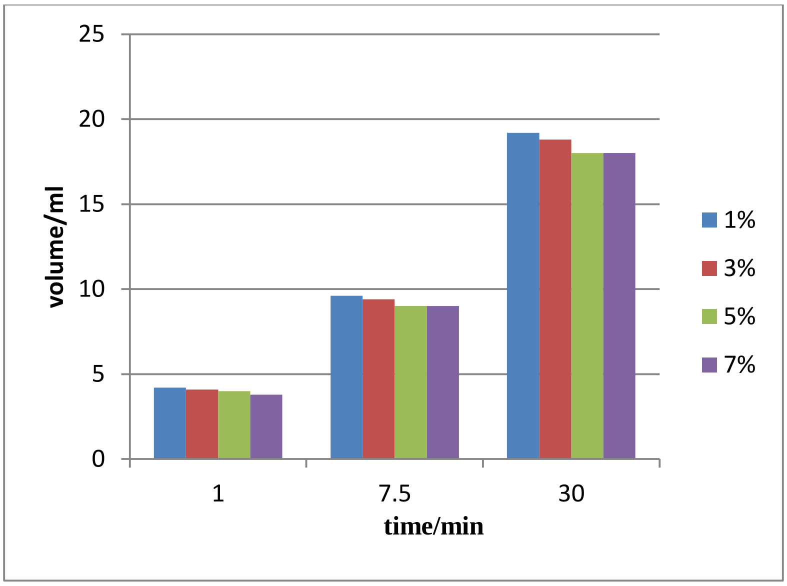

Fig. (4.2): filtrate volume of mud containing Barite vs Time

Figure (4.2) illustrates the filtrate volume of mud containing barite with concentrations of 1%, 3%, 5%, and 7%.

Table (4.5): Properties of basic mud system containing Fly ash

Properties	Units	Percentage of Fly Ash by weight (%)				
		1%	**3%**	**5%**	**7%**	**9%**
Mud weight	ppg	8.66	8.68	8.7	8.72	8.72
Ө600	rpm	23	24	25	31	80
Ө300	rpm	17	18	19	27	65
Ө3 @10sec	rpm	8	9	9	31	80
Ө3@ 10min	rpm	41	43	50	90	115
Apparent Viscosity (AV)	cp	11.5	12	12.5	15.5	40
Plastic Viscosity (PV)	cp	5	6	6	4	25
Yield Point (YP)	Ib/100ft^2	12	12	13	23	40
Gel strength	/100ft^2	8/41	9/43	9/50	31/90	80/115
Non-Newtonian	_	0.43	0.41	0.39	0.2	0.3

Index (n)						
Consistency Index (K)	_	1.1	1.4	1.6	7.7	10
Ph	_	**11**	**11**	**12**	**12**	**12**
Fluid loss volume/1min	ml	4	3.6	3.4	3.4	_
Fluid loss volume/7.5min	ml	9.2	9	8.6	8.4	_
Fluid loss volume/30min	ml	18.4	18	17.2	17.4	_
YP/PV	_	2.4	2	2.1	5.7	1.6

Table (4.5) illustrates the results of laboratory experiments of Fly Ash mud system with concentrations of 1%, 3%, 5%, and 7%.

Hint:

From table (4.5) it's obvious that after the concentration of 7% the solution was saturated with fly ash, thus there was a sharp change in the mud system behavior when adding more fly ash to the solution.

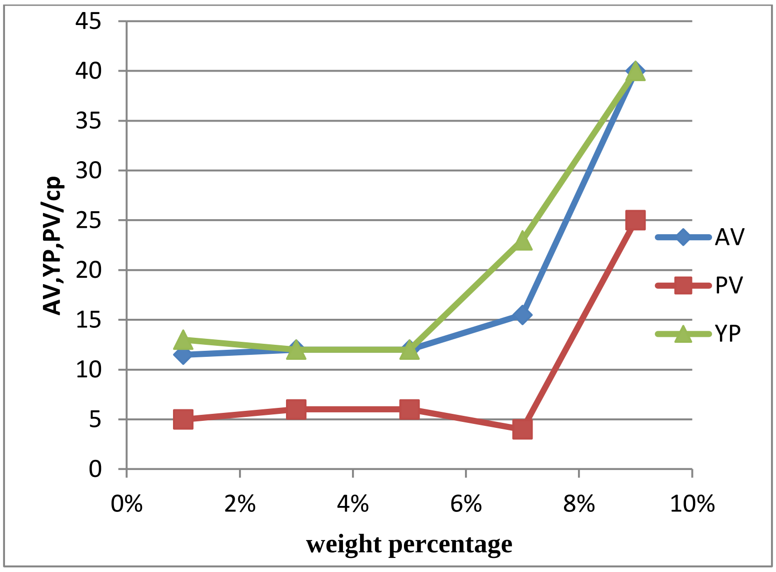

Fig (4.3): AP, PV, YP vs concentration of fly ash

Figure (4.3) shows that, the PV of Fly Ash increases with the concentration increment by rate slower than the increment of AV rate. YP and AV after concentration 5% increase sharply, and the figure also shows the behavior of them.

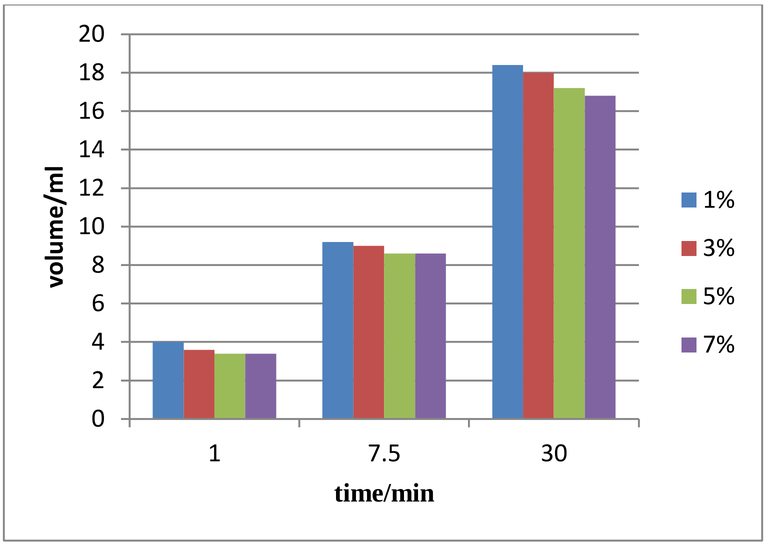

Fig. (4.4): Filtrate volume of mud system containing Fly Ash vs time

Figure (4.4) illustrates filtration volume of mud containing Fly Ash with concentration of 1%, 3%, 5%, and 7%.

Table (4.6): Properties of basic mud system containing PAC-LV

Properties	Units	Percentage of PAC-LV by weight (%)			
		1%	3%	**5%**	**7%**
Mud weight	ppg	8.69	8.69	8.7	8.7
$\theta 600$	rpm	28	43	56	68
θ300	rpm	21	32	44	54
θ3 @10sec	rpm	11	15	20	25
θ3@ 10min	rpm	46	52	60	66
Apparent Viscosity (AV)	cp	14	16	22	27
Plastic Viscosity (PV)	cp	7	11	12	14
Yield Point (YP)	$Ib/100ft^2$	14	21	22	40
Gel strength	$Ib/100ft^2$	11/46	15/52	20/60	25/65
Non-Newtonian Index (n)	_	0.41	0.42	0.34	0.33
Consistency	_	1.6	2.3	5.2	6.9

Index (K)					
Ph	_	**11**	**11**	**11**	**11**
Fluid loss volume/1min	ml	4.4	3.2	3	2.8
Fluid loss volume/7.5min	ml	9.2	7.2	6.2	6
Fluid loss volume/30min	ml	18.4	14.4	12.4	12
Yp/pv	_	2	1.9	1.8	2.85

Table (4.6) illustrates the results of the laboratory experiments which have been done for PAV-LV mud system with concentrations of 1%, 3%, 5%, and 7%.

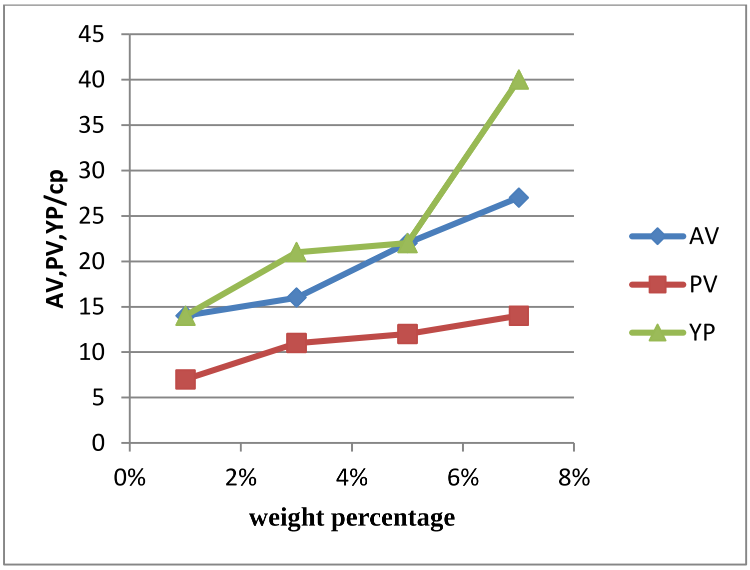

Fig (4.5):AP, PV, YP vs PAC-LV concentration

Figure (4.5) shows that, the PV of barite increases with the concentration increment by rate slower than the increment of AV rate, the YP increases sharply at concentration 5%, and the figure also shows the behavior of them.

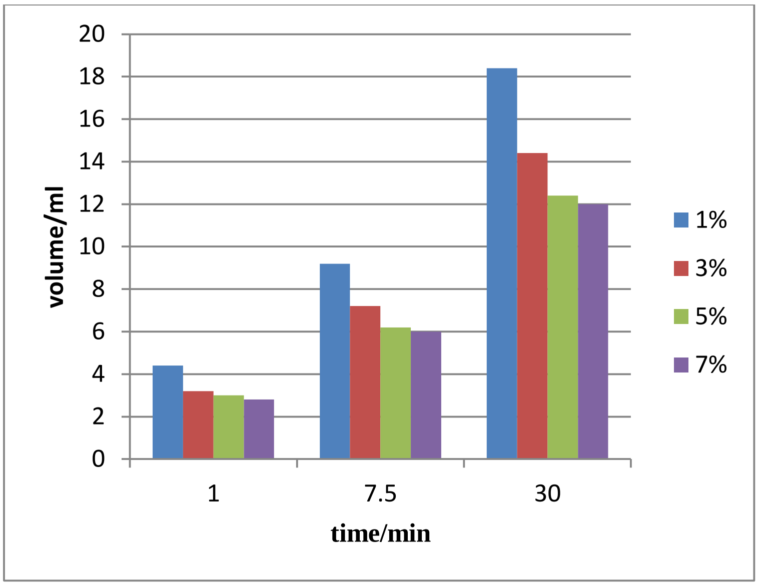

Fig. (4.6): Filtrate volume of mud system containing Barite vs Time

Figure (4.6) illustrates the filtration volume of mud containing PAC-LV with concentration 1%, 3%, 5%, and 7%.

Table. (4.7): Properties of mud system containing starch

Properties	Units	Percentage of Starch by weight (%)			
		1%	3%	5%	7%
Mud weight	Ppg	8.70	8.67	8.7	8.7
ϴ600	Rpm	20	33	37	40
ϴ300	Rpm	15	24	28	35
ϴ3 @10sec	Rpm	9	15	18	24
ϴ3@ 10min	Rpm	30	40	42	50
Apparent Viscosity (AV)	Cp	12.5	11.5	13.5	14
Plastic Viscosity (PV)	Cp	5	5	6	5
Yield Point (YP)	Ib/100ft^2	15	13	15	18
Gel strength	Ib/100ft^2	9/30	15/40	18/42	24/50
Non-Newtonian Index (n)	_	0.41	0.46	0.4	0.2

Consistency Index (K)	_	1.1	1.3	2.3	10
Ph	_	**11**	**11**	**11**	**11**
Fluid loss volume/1min	ml	4.2	4.1	4	3.8
Fluid loss volume/7.5min	ml	9.6	9.4	9	9
Fluid loss volume/30min	ml	19.2	18.8	18	18
YP/PV	_	3	2.6	2.5	3.6

Table (4.7) illustrates the results of laboratory experiments conducted on a basic mud system with Starch in concentrations of 1%, 3%, 5%, and 7%.

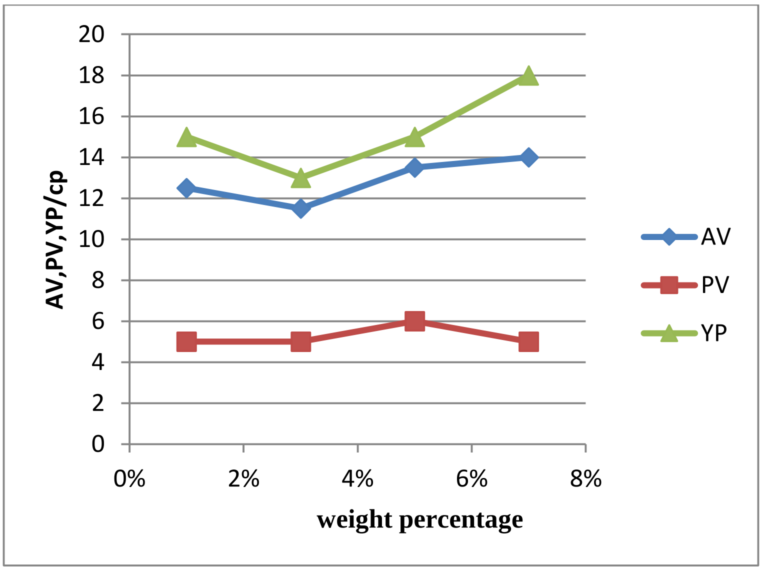

Fig. (4.7): AP, PV, YP vs starch concentration

Figure (4.7) shows that, the increment of PV, AV and YP is slightly, and also shows the behavior of them.

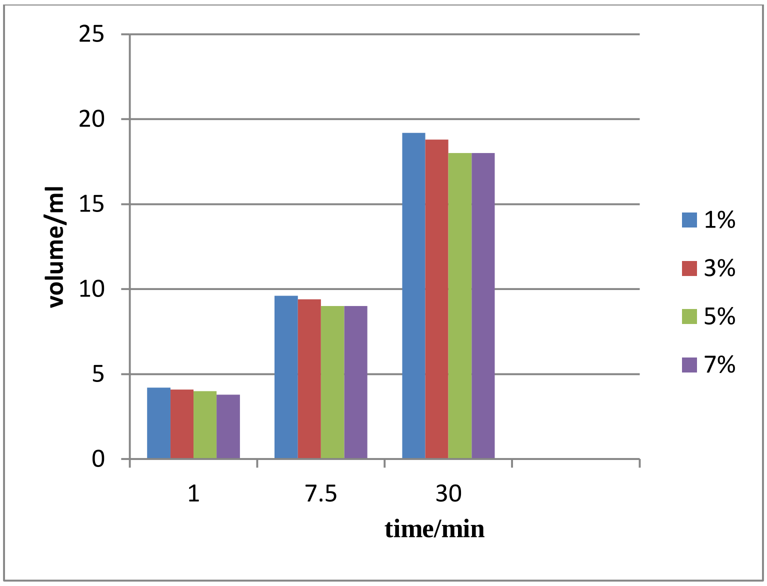

Fig. (4.8): Filtrate volume of mud system containing Starch vs Time

Figure (4.8) illustrates filtration volume of mud containing Starch with concentration of 1%, 3%, 5%, and 7%.

4.4. Discussion:

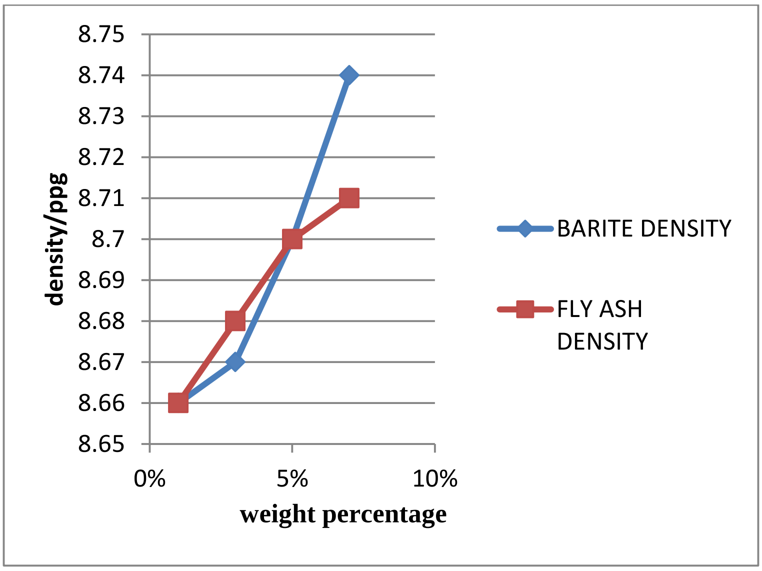

Fig. (4.9): density vs weight percentage barite and fly ash

When adding fly Ash, the density increases by a constant rate, until reaches point then the rate of increasing will decrease. In the other hand when adding barite, the increment is sharp.

Hint:

The specific gravity of Fly Ash (SG)=2.56

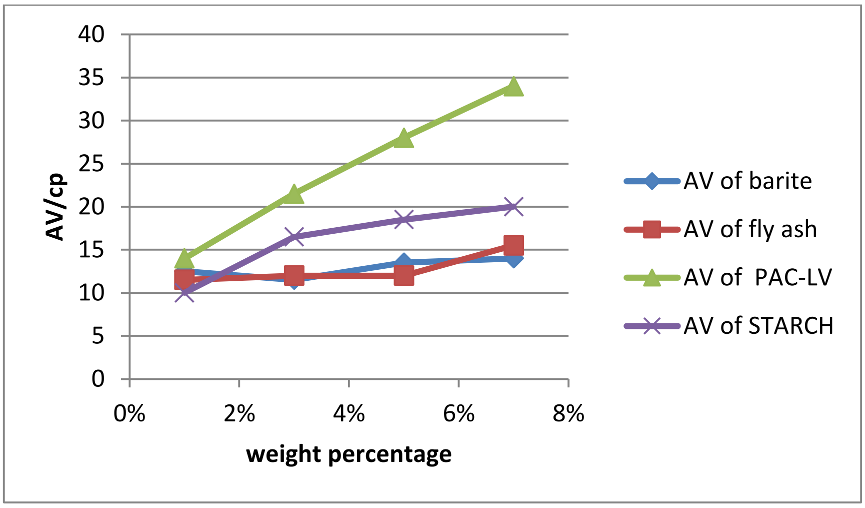

Fig. (4.10): AV vs weight percentage

From fig. (4.10) it's obvious that AV of fly ash system increases slightly with the increment of weight percentage like barite mud system.

The AV of starch mud system is higher than AV of fly ash mud system. And it increases with high rate of increment, till reaches 3%, then the rate decreases. The AV of PAC-LV system increases by a sharp rate of increment.

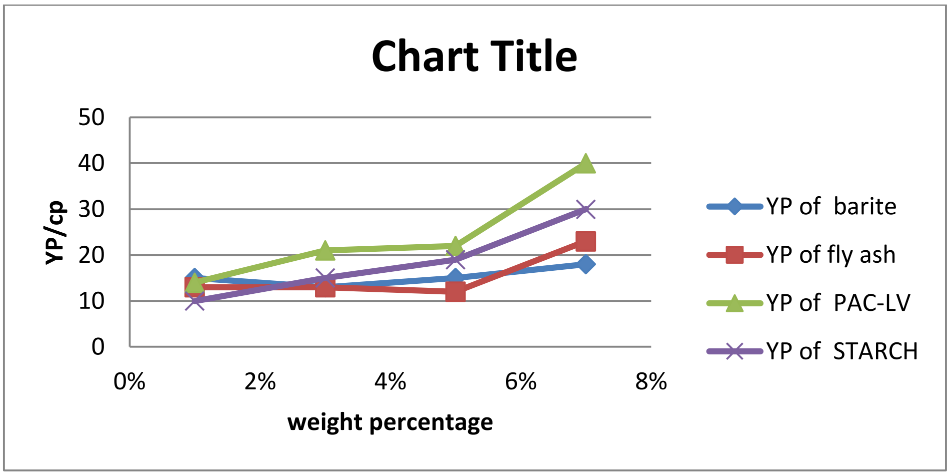

Fig. (4.11): YP vs weight percentage

From fig. (4.11) it's obvious that the YP of Fly Ash system starts with a constant value, then increases significantly after 5% of Fly Ash.

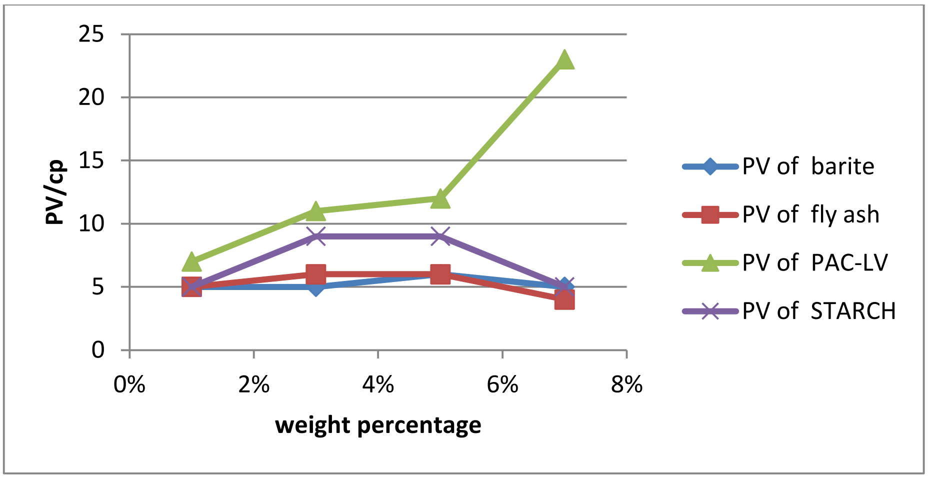

Fig. (4.12): PV vs Weight Percentage

From Fig. (4.12) it's obvious that the PV of fly ash system is increased slightly when comparing it with the YP of drilling mud contains PAC-LV and STARCH.

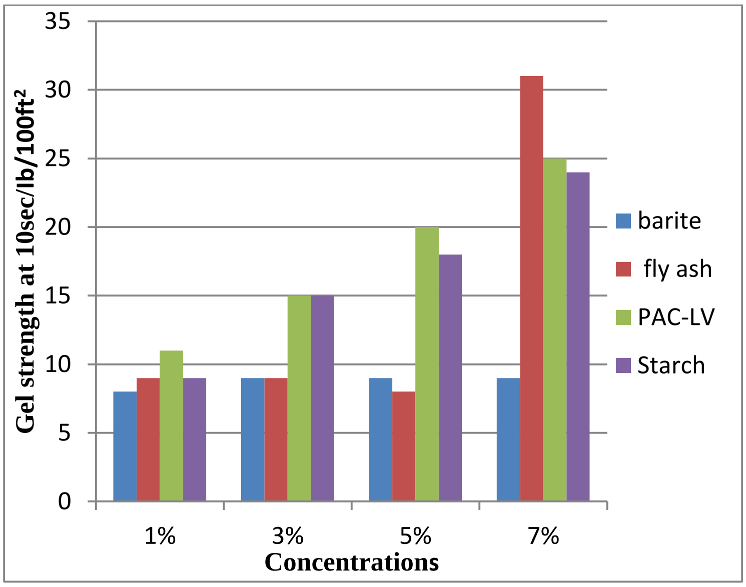

Fig. (4.13): Gel strength at 10-seconds vs Concentration

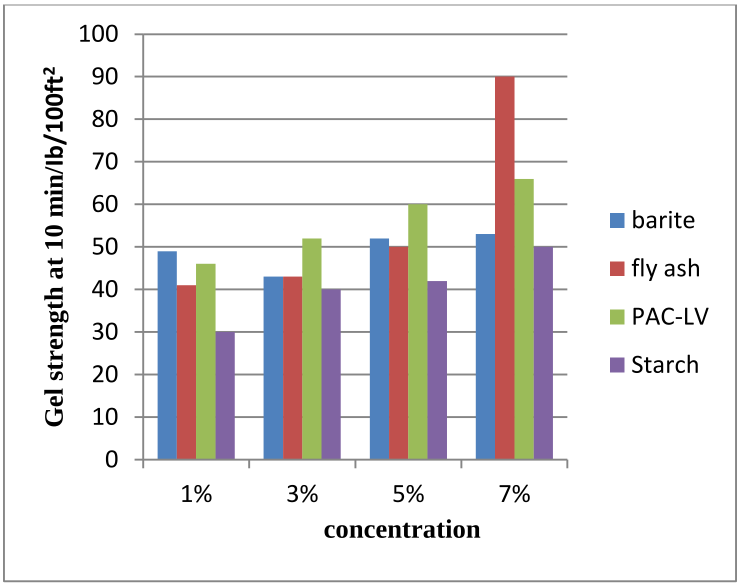

Fig (4.14): Gel strength at 10-minutevs Concentration

The gel strength of fly ash system increased slightly, till reaching a concentration of 5%, then started to increase significantly, because the solution was beginning to be saturated.

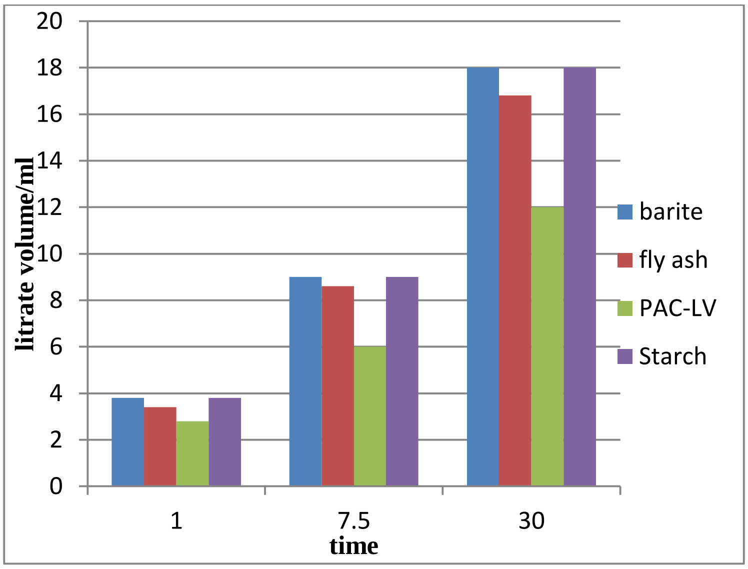

Fig. (4.15): Filtrate Volume vs Time for different mud systems

When adding fly ash to the mud system, the small particles and the chemical reaction increased the viscosity and closed the porous so the volume of filtration reduced.

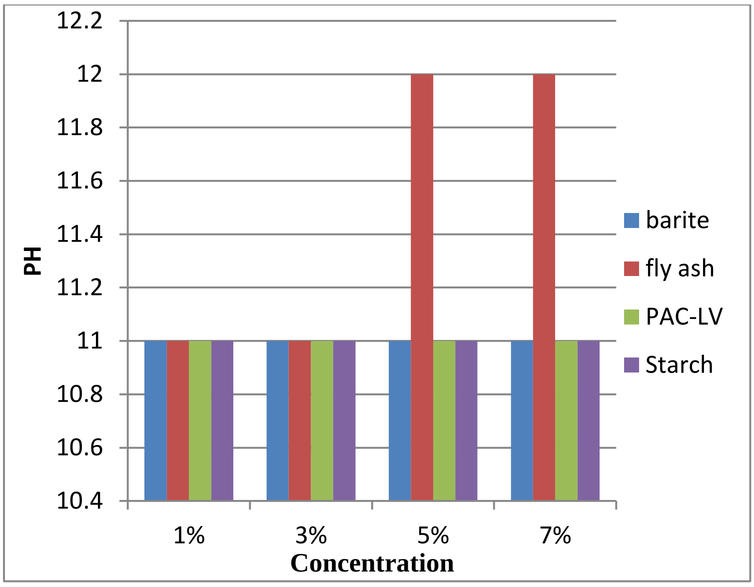

Fig. (4.16): PH vs concentration for different mud system

The PH of the fly ash mud system increased with the increment of weight due to chemical compounds which is present in Fly Ash.

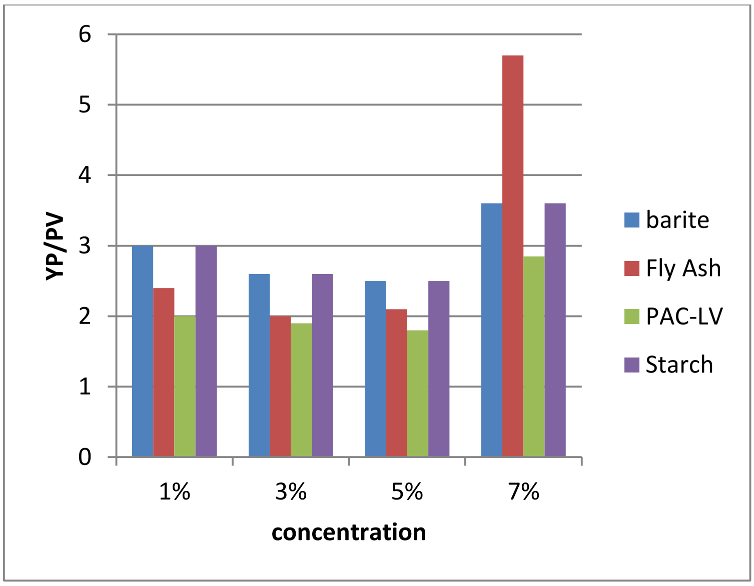

Fig (4.16): YP/PV vs Concentration

The rate of YP/PV change depend on the reading from 6-speed of viscometer (ϴ300 and ϴ600). And figure (4.17) illustrates the ratio YP/PV for each mud system.

CHAPTER FIVE

Chapter 5

Conclusion and Recommendations

5.1. Conclusions:

Based on the result of the tests performed, the following conclusions were obtained:

- The dominant elements in the sample of fly ash are Ca, Si and Fe.
- The rheological properties when adding 7% concentration of fly ash to the basic mud system meets the API specification of bentonite.
- The density of mud system increased with the addition of fly ash to the system, and the rate of increment decreased when the concentration of fly ash reached 5%.
- Addition of Fly ash to the mud system increased the PH, decreased the filtration volume, increased both PV and AV slightly and increased the gel strength.
- The addition of chemicals must be carried out carefully with respect to the precedence. From practice when adding Fly Ash before Bentonite, there was no significant change in the value AV, but when adding fly Ash after Bentonite, the minerals in Fly Ash react and increase the AV.

5.2. Recommendations:

From the data collected and the research investigations the results lead to recommend that:

- To Study the effect of temperature on Fly Ash mud system.
- To conduct other tests such as swelling test, sand content, friction factor resistivity test for water based mud with in the presence of Fly Ash in the drilling mud system.
- To conduct the laboratory tests under high pressure and high temperature conditions.
- To Study the effect of fly ash addition to oil based muds.
- To conduct a cost estimation study.

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