DEVELOPMENT OF AN ENVIRONMENTALLY-FRIENDLY DRILLING FLUID USING DATE SEEDS AND GRASS

BY

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To my beloved parents, Wahab and Rahmath, my late uncles, Baba and Chichpasha

And my ever encouraging brothers Wali, Wasee and Wafi

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All praise is to ALLAH (S.W.T), the Compassionate, the Merciful, and the Lord of the Worlds and the Hereafter, to Him we belong and to Him shall we return. May ALLAH shower his countless blessings and bounties on Prophet MUHAMMED (S.A.W.S), the Mercy to all Mankind. May ALLAH (S.W.T) send HIS blessings on all the COMPANIONS (R.A) and the AHLUL BAYT (R.A) of Prophet MUHAMMED (S.A.W.S) who are indeed better than us in Emaan and Taqwaa.

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TABLE OF CONTENTS

List Of Tables

LIST OF FIGURES

List Of Abbreviations

WBM: Water Based Mud

OBM: Oil Based Mud

SBM: Synthetic Based Mud

CMC: Carboxy Methyl Cellulose

PSD: Particle Size Distribution

PSA: Particle Size Analyzer

XRF: X-Ray Fluorescence

PV: Plastic Viscosity

YP: Yield Point

GS: Gel Strength

PPB: Pounds per Barrel

ABSTRACT

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The exploration and exploitation of hydrocarbons in this age dictates the use of environmentally-friendly muds and mud additives to protect the existing ecology and habitat of the eco-system. The preservation of the environment on a global level is now important as various organizations have set up initiatives to drive out the usage of toxic chemicals as mud additives. For instance, a set of regulations called the Corporate Regulations for Offshore Drilling Operations in Saudi Arabia established by the Royal Decree No. M/9 of November 18, 1987, stipulates that all drilling fluids that are designated as toxic fluids, and cuttings must be hauled back to an approved onshore disposal site, and that cuttings from such muds should be cleaned using the best practical technology and then be discharged as close as possible to the sea floor. This thesis presents an approach wherein date seeds, grass and grass ash are introduced as environmentally-friendly mud additives which impart no environmental pollution. The research focusses on the rheological as well as the filtration characteristics of simple water-based muds formulated using bentonite, the said natural additives and water.

Particle size distribution test are conducted to determine the particle sizing of the said additives samples. Later, experiments are performed on samples selected

from various particle sizes at various concentrations to study the characteristics and behavior of the newly developed mud at both ambient conditions and high temperatures. A comparison of the proposed additives with a commercially available additive, modified starch is also made to validate the results obtained. It is found out that all three materials with varying particle sizes and concentrations exhibited improved rheology, filtration and pH making the use of date seeds, grass and grass ash suitable for the formulation of a low-cost, environment-friendly and sustainable drilling fluid system.

ملخص الرسالة

الاسم الكامل: وجهي الدين محمد

عنوان الرسالة: تطوير سائل حفر صديق للبيئة باستخدام بذور التمر والعشب

التخصص: هندسة البترول

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استكشاف واستغلال النفط والغاز في هذا العصر يتطلب أن تكون سوائل الحفر والمواد المضافة إلى سوائل الحفر صديقة للبيئة لحماية البيئة وسكان النظام البيئي. الحفاظ على البيئة على المستوى العالمي يعتبر مهم في الوقت الحالي حيث أن منظمات مختلفة حددت مبادرات لمنع استخدام المواد الكيميائية السامة مثل المواد المضافة إلى سائل الحفر.على سبيل المثال، مجموعة من القوانين تسمى لوائح الشركات لعمليات الحفر البحرية في المملكة العربية السعودية اصدرت بموجب المرسوم الملكي رقم م / 9 من 18 نوفمبر 1987، والذي ينص على أن جميع سوائل الحفر التي يتم تصنيفها كسوائل سامة، اضافة إلى فتات الصخور الموجودة في سوائل الحفر يجب أن تنقل إلى موقع التخلص البرية المعتمدة، وكذلك فتات الصخور الموجودة في سوائل الحفر يجب تنظيفها باستخدام أفضل التقنيات العملية ومن ثم يتم تفريغها في أقرب وقت ممكن إلى قاع البحر. تقدم هذه الأطروحة منهاج لتحضير سائل حفر جديد وذلك عن طريق اضافة بذور التمر والعشب ورماد العشب إلى سوائل الحفر كالمضافات صديقة للبيئة والتي لا تلوث البيئة. يركز هذا البحث على الريولوجية إضافة الى خصائص الترشيح لسوائل الحفر المائيه والمتكونة باستخدام البنتونايت والمضافات الطبيعية المذكرة سابقا والماء. تم إجراء اختبار توزيع حجم الجزيئات لتحديد الحجم الجزئي لعينات المواد المضافة. ومن ثم تم إجراء تجارب على عينات مختارة من أحجام الجسيمات المختلفة عند تركيزات مختلفة لدراسة خصائص وسلوك سائل الحفر الجديد عند الظروف المحيطة ودرجات الحرارة المرتفعة. للتحقق من صحة النتائج التي تم الحصول، تم مقارنة المضافات المقترحة مع كلامن المضافات التجارية الموجود حاليا و مضافات النشا المعدلة. من خلال النتائج اتضح ان كل المواد الثلاث ذات احجام الجزيئات والتراكيز المختلفة

أظهرت تحسن في الريولوجيا، والترشيح ودرجة الحموضة مما يجعل استخدام بذور التمر والعشب ورماد العشب مناسب لتكوين سائل حفر رخيص وصديق للبيئة وفعال لعمليات الحفر.

CHAPTER 1

INTRODUCTION

Drilling fluid (also recognized as mud) plays an essential part in the rotary drilling operations. A drilling fluid can be defined as a mixture of clays, water, and chemicals used to drill a borehole into the earth and whose basic functions are to lubricate and cool the drill bit, carry drill cuttings to the surface, and to strengthen the sides of the hole. It can also be defined as a fluid compositions used to assist the generation and removal of cuttings from a borehole in the ground. Most of the problems encountered during the drilling of a well are directly or indirectly related to the mud quality, composition, and its toxicity level. The successful completions of a hydrocarbon well and its cost depend on the properties of the drilling fluid up to some extent.

The oil and gas industry has made tremendous progress in developing techniques, procedures, and less toxic materials for the protection of human health and the environment. In literature, it is well documented that diesel-based/mineral-based drilling fluids have high toxicity levels. The toxicity of drilling fluids and their disposal are strictly controlled to minimize the effects on the subsurface and environment by the government and non-government Environmental Protection Agencies (EPAs). The composition of drilling fluid ranges from a simple clay-water mixture to a complex blend of materials chemically suspended in water or oil. The composition of mud depends on the required functions of mud and the type of mud whether it is water-based, oil-based or synthetic based. The composition muds are mainly water (salt or fresh), mineral oils, barite, and some toxic & nontoxic chemical additives. In general, diesel, kerosene and fuel oils are used as base fluid for oil-base muds (OBMs). The toxicity effects of diesel oils and mineral oils are well documented in the literature. Synthetic-based muds (SBMs) are characterized by the replacement of mineral oil with oil like substance, and are free of inherent contaminants such as radioactive components and toxic heavy metals. Development of SBM as alternatives to conventional OBM in offshore operations was precipitated by toxicity and biodegradability concerns. OBM reduces the effectiveness of some logging tools and remedial treatment for lost circulation which is more difficult. Finally, sometime, detection of gas kicks is more difficult because of gas solubility in diesel oil. The current research trend is in the direction of sustainable petroleum operations where drilling fluid's position is very weak. As a result, minimizing the quantity of oil discharged into the marine environment, use of water-based or synthetic-based mud is encouraged. This scenario leads to the necessity for developing environment friendly natural substitutes which will replace the current practice in the industry. Therefore, it is very important to look for alternative drilling muds to toxic OBMs which are not harmful to humans, the environment and the subsurface formations.

Minimizing the environmental impact of a drilling operation as well as safety considerations both directly affect the choice of drilling fluid additives and drilling fluid systems. Products that have been used in the past may no longer be acceptable. As more environmental laws are enacted and new safety rules applied, the choices of additives and fluid systems must also be reevaluated. To meet the challenge of a changing environment, product knowledge and product testing become essential tools for selecting suitable additives and drilling fluid systems. Environment friendly drilling fluid systems have been developed constantly since the 1990s such as formate drilling fluid system, polyvinyl alcohol (PVA), silicate drilling fluid system, polyolefin drilling fluid system, ether based drilling fluid system, ester based drilling fluid system, etc. All these fluid systems have common features of low toxicity, easy degradation and little effect on environment. However, these fluid systems fail to be widely applied due to high costs or unsatisfactory application effects. There are also some newly developed drilling fluid systems, the additives of which have complex synthetic process, failing to achieve industrialized manufacture. So it is urgent to develop a new environment friendly drilling fluid system which can not only protect environment and reservoir, but also expend low cost, be easy to synthesize, and satisfy needs of drilling engineering.

1.1 Early Development of Drilling Fluid Additives

Water was the first drilling fluid used by the drillers for rotary drilling operations (Brantly, 1961). The Egyptians, far back in the third millennium used water to remove cuttings from holes drilled using hand-driven rotary bits (Brantly, 1971). Around 600 B.C, wells were drilled in China for brine, gas, and water where water was poured into these wells to soften the rock and to help removing the cuttings (Pennington, 1949). Through a patent in 1844, Robert Beart proposed that cuttings from holes being drilled may be removed by water (Beart, 1845). To bring drill cuttings from the borehole to the surface, Fauvelle (1846) pumped water through a hollow boring rod. In 1887, it was proposed in a U.S. patent that a mixture of water and a quantity of plastic material can be used to remove cuttings and also to form an impervious layer along the wall of the borehole.

The addition of mud to water as a means of hole stabilization in weak formations commenced in Texas and Louisiana around 1901. The first mud used was called sufficient clay i.e. gumbo, (Hayes and Kennedy, 1903). However, in California, other types of clays from surface deposit were mixed with water using hand shovels by mud crew with little attention paid to the mud properties (Knapp, 1916). Increased drilling activities with time enhances tremendously the demand for mud and also the need to increase mud density for pressure control triggered the commercial sale of heavy mud made by adding heavy minerals to surface clays. The sale of paint-grade barite for oil well used under the brand name Baroid^R by the National Pigments and Chemical Company started in California in 1922 (Stroud, 1925). The George F. Mepham Corporations of St. Louis, Missouri sold iron minerals as mud weighting agent while the California Talc Company, a producer and marketer of clays sold Aquagel^R brand of bentonite as an admixture for cement in 1928 (Stroud, 1926).

However, the problem of the settling of the heavy minerals in some mud became prevalent at that time and thus made a case of the necessity for a suspending agent (thinner). This inclusion allows preventing the heavy minerals from settling down which became inevitable. As a result, the first thinning agent for mud, Stabilite^R, was introduced by T.B. Wayne in 1938 (Parsons, 1932). This product, a mixture of chestnut bark extract and Sodium Aluminate thinned mud without decreasing the density, released entrapped gas, and allowed further increase in mud weight. It is extracted in the form of tannin by hot water from the wood of certain dense hardwood trees which grows in northern Argentina and western Paraguay (Lawton *et al*, 1932). In 1947, leonardite, mined lignin, brown coal, and slack were introduced as partial substitute for quebracho extract and later on, limitations were placed on it due to the World War II (Caraway, 1953).

Oil-based drilling fluids were developed to solve some of the unwanted characteristics of water-base muds. Oil-base drilling fluids originated with the use of crude oil in well completions, but the date of first usage is unknown. Historians believed that a patent application filed by J.C. Swan in 1919 and granted in 1923 marked the beginning of the use of oil to drill the productive zone in shallow, low-pressure wells in many early fields. In 1935, Humble Oil & Refining Company (now ExxonMobil) used an oil mud made from gas oil and spent clay to drill through heaving shale interval in Creek Field, Texas. During the next two years, studies were carried out on cores taken with oil mud in Texas Fields on the connate water content of reservoir sands so as to be able to improve on the formulation of oil-base muds (Schilthuis, 1938). Commercial oil muds became available in 1942, when George L. Miller established the Oil Base Drilling Fluids Company, Los Angeles, California (Miller, 1942). This Company (now Oil Base, Inc.) supplied blown asphalt in the form of Black Magic^{R}, a powder which was mixed with suitable oil at the well site (Miller, 1942).

Use of oil muds for drilling had its drawbacks: Water was a severe contaminant; high risk of fires, low rate of penetration (ROP); very costly; and most importantly, it is not environmentally friendly. Currently, research efforts are directed mainly towards the development of environment friendly drilling fluids that will be a substitute to the oil-base drilling fluids. The current scenario is however different where industry uses the conventional drilling fluid in a massive way. The OBM is the best and the most widely used but very expensive and environmentally unfriendly. Yet the performance of OBM over shadows these limitations. As stricter environmental laws are put in place worldwide as far as oil exploration and production is concerned, its use is becoming difficult and restricted.

1.2 Drilling Fluid Additives

Many substances, both reactive and inert, are added to drilling fluids to perform specialized functions. Table 1-1 list the most commonly used industrial additives for formulating a drilling fluid and the functions of these additives are explained below:

Alkalinity and pH Control: These additives are designed to control the degree of acidity or alkalinity of the drilling fluid. Most common of them are lime, caustic soda and bicarbonate of soda.

Bactericides: Bactericides are used in order to reduce the bacteria count. Paraformaldehyde, caustic soda, lime and starch preservatives are the most common bactericides used in the industry.

Calcium Reducers: These are used to prevent, reduce and overcome the contamination effects of calcium sulfates (anhydrite and gypsum). The most common are caustic soda, soda ash, bicarbonate of soda and certain polyphosphates.

Corrosion Inhibitors: Corrosion Inhibitors are used to control the effects of oxygen and hydrogen sulfide corrosion. Hydrated lime and amine salts are often added to check this type of corrosion. However, oil-based muds have excellent corrosion inhibition properties.

Defoamers: These are used to reduce the foaming action in salt and saturated saltwater mud systems, by reducing the surface tension.

Emulsifiers: Emulsifiers are added to a mud system to create a homogeneous mixture of two liquids (oil and water). The most commonly used emulsifiers are modified lignosulfonates, fatty acids and amine derivatives.

Filtrate Reducers: These are used to reduce the amount of water lost to the formations. The commonly used filtration loss additives are bentonite clays, CMC (sodium carboxy methyl cellulose) and pre-gelatinized starch.

Flocculants: These are used to cause the colloidal particles in suspension to form into bunches, causing solids to settle out. The most common are salt, hydrated lime, gypsum and sodium tetraphosphates.

Foaming Agents: Foaming agents are most commonly used in air drilling operations. They act as surfactants, to foam in the presence of water.

Lost Circulation Materials: These inert solids are used to plug large openings in the formations to prevent the loss of drilling fluid. Nut plugs (nut shells), and mica flakes are commonly used.

Lubricants: These are used to reduce torque at the bit by reducing the coefficient of friction. Certain oils and soaps are commonly used.

Pipe-Freeing Agents: Pipe freeing agents are used as spotting fluids in areas of stuck pipe to reduce friction, increase lubricity and inhibit formation hydration. Oils, detergents, surfactants and soaps are commonly used in the industry as pipe freeing agents.

Shale-Control Inhibitors: These are used to control the hydration, caving and disintegration of clay/shale formations. The commonly used shale inhibitors are gypsum, sodium silicate and calcium lignosulfonates.

Surfactants: These are used to reduce the interfacial tension between contacting surfaces (oil/water, water/solids, water/air, etc.).

Viscosifiers: Viscosifiers are added to impart viscosity to the drilling fluid. Commonly used viscosifiers are bentonite, attapulgite, carboxy methyl cellulose etc.

Weighting Agents: Weighting agents are an important class of additives used to provide a required density to the drilling fluid. Materials such as barite, hematite, calcium carbonate and galena are common names known for this type of additive in the industry.

Table 1- 1: Conventional Drilling Fluid Additives

Function	Additive
Weighing Agents	Galena, Hematite, Magnetite, Iron Oxide, Ilmenite, Barite, Siderite, Celestite, Dolomite, Calcite, Zirconium Oxide, Zinc Oxide, Calcium Carbonate, Manganese Tetraoxide
Thickening Materials (Viscosifiers)	Bentonite, Attapulgite, Sepiolite, Organophilic Clays, Palygorskite, Asbestos, Tamarind gum, Saccharides (sugar), Scleroglucan, Carboxy Methyl Cellulose, Poly Ethylene Glycol, Cellulose Nanofibers, Chitosan, Hydrophobically Modified Hydroxy alkyl Guars (HMHAG)
Filtration Control Materials	Starch, Modified starch, Guar gum, Xanthan gum, Sodium Carboxy Methlycellulose, Hydroxy Ethylcellulose, Acrylic polymer, Alkylene Oxide polymer, Poly glycerols, Poly glycols
Thinners (Conditioning Material)	Tannins, Quebracho, Modified tannins, Polyphosphates, Organic phosphates, Phosphonates, Lignite, Lignosulfonates
Lost Circulation Materials	Cellophane, Cotton seed Hulls, Vermiculite, Mica, Surfactants, Diatomaceous earth, Olive pits, Gilsonite, Bagasse, Perlite, Polyanionic Cellulose, Petroleum Coke, Oat Hulls, Encapsulated Lime, Aqueous Alkali Alumino Silicate, Resins, Pulp residue waste
Shale Inhibitors	Poly oxy alkylene amine (POAM), Potassium Chloride, Sodium Chloride, PHPA, Cationic Starches, Polyacrylamide,

	Polyamine
Lubricants	Carbon black, Fatty acid Esters, Olefins, Phospholipids, Fluoropolymers, Propylene glycol, Gypsum, Modified Ethoxylated Castor Oil derived from Phospho Lipids, Liquid Gilsonite, Terpene, Soybean Oil blend, Triglycerides, Hydrocarbon Emulsions
Bactericides	Hydroxamic acid, Isothiazolinones, Dithiocarbamic acid, Bis sulfate, Dimethyl-tetrahydro-thiadiazine-thione
Surfactants	Alkylpolyglycosides, Amphoteric Surfactants, Acetal ether, Alkanolamine, Alkyl phenol ethoxylates
Corrosion Inhibitors	Alkanol amine solution, Mercaptoalcohols, Polysulfide, Water soluble thiones, Sulfonated alkyl phenol, Polythiether, Thiazolidines

CHAPTER 2

LITERATURE REVIEW

This chapter provides a revision of research on designing environmentally-friendly drilling fluids including the additives which were used to provide the necessary properties to the mud. Also, natural additives used by researchers for the formulation of drilling fluids are summarized and presented.

Environmental considerations have led to increasing interest in the use of water based drilling fluids (WBM) in applications where oil based fluids have previously been preferred. It is to be mentioned that where environmental regulations prohibit the use of oil based mud, high temperature wells are drilled with HPHT water based fluids. Currently, research efforts towards the development of environmentally friendly drilling fluid are from two main stand points:

(1) Using environmentally friendly oils to formulate oil-based muds; (2) Development of water based drilling fluids which simulates the performance of the oil-based drilling fluid, and which are referred to as high performance water-based drilling fluids (HPWBF). In this literature review, a number of previous research works based on the two approaches stated are presented.

Hille *et al.* (1985) developed a HPHT water based fluid system composed of vinlysulfonate and vinlyamide copolymers for improved and sustained good rheological properties even when the electrolytic concentration of the mud increases. The problem with this system is that it rapidly disperses in water and poses minimal degree of environmental effects.

Bailey *et al.* (1986) examined fluid viscosities of muds formulated with a low toxicity mineral oil (LTOBM) and diesel oil with temperature and pressure. The use of mineral oils as replacements for diesel in drilling fluids was rapidly spreading at that time. The authors concluded their findings had a greater impact on the apparent fluid viscosity at high temperatures (77 ^{o}F-212 ^{o}F).

Perrocine *et al.* (1986) described the properties of high molecular weight vinyl sulfonate copolymers at high temperature, fluid loss control additives in water based drilling fluids. They reported good tolerance to electrolytes and high temperature stability to 350 ^{o}F.

Yassin *et al.* (1991) carried out tests on palm oil derivatives as the continuous phase for oil based drilling fluids and the toxicity effect on plant and aquatic life. The oils used in this case were: Methyl esters of Crude Palm Oil and Methyl esters of Palm Fatty Acid Distilled. Tests were carried out on the physio-chemical properties of these oils such as flash point, pour point, aniline point etc. at varying temperatures and pressures.

Hemphil (1996) carried out studies to predict the rheological properties of ester based drilling fluids under down hole conditions. Rheological tests that simulated field conditions were run in the laboratory on an ester based drilling fluid from the field. The rheological behavior of the fluid was tested under varying ranges of temperature, pressure and ester/water ratios.

E Van Oort *et al.* (1996) formulated an improved water based drilling fluid based on soluble silicates capable of drilling through heaving shale which is environment friendly. However, this is not recommended because silicate has the potential to damage the formation.

Sundermann *et al.* (1996) eliminated drilling problems with high temperature gas wells in northern Germany via the development and use of potassium formate ($KCHO_2$) biopolymer fluid. The formulated drilling fluid allowed a higher mud weight with fewer solids and proved to be very stable requiring only small additions of viscosity and filtration control agents to keep the fluid properties within the desired range.

Brady *et al.* (1998) came up with a polyglycol enriched water based drilling fluid that will provide high level of shale inhibition in fresh water and low salinity water based drilling fluid. However, this formulation has defects on it which are to perform optimally, and electrolytes must be presented.

Nicora *et al.* (1998) developed a new generation dispersant for environmentally friendly drilling fluids based on zirconium citrate. The functions of zirconium citrate are to improve the rheological stability of conventional water based fluids at high temperature.

However, this formulation has a limitation that the concentration of zirconium citrate may be depleted in the drilling fluid due to solids absorption.

Hayet *et al.* (1999**)** developed an additive from the modification of natural polymers hydrophobically for the formulation of non-damaging drilling fluids which are of great importance when drilling through un-cased sections of horizontal wells. Increased hydrophobicity improves viscosity, yield point, and also prevented the sedimentation of suspended solids. However, there is the risk of reduced production induced by reservoir damage when this formulation is used for drilling and well completion.

Sanchez *et al.* (1999) formulated drilling fluids from mineral oil (<0.1% aromatics) and palm tree oil (without aromatics). The work evaluated the toxicity and biodegradability of mineral and palm tree oil based drilling fluids compared to those formulated with diesel. The results indicated that both mineral and palm tree oil to be non-toxic while diesel showed high levels of toxicity.

Skalle *et al.* (1999) suggested the use of microsized spherical monosized polymer beads as a blend to WBDF to improve lubrication.

Thaemlitz *et al.* (1999) formulated a new environmentally friendly and chromium-free drilling fluid for HPHT drilling based on only two polymeric components which make it simple, easy to handle, environmentally friendly, and hence suitable for use in remote areas as compared with traditional HT systems which normally composed of a large number of additives.

Nicora *et al.* (2001) formulated a new low solids oil-base drilling fluid system for HPHT application using cesium formate as the internal phase, and ilmenite as the weighting agent so as to address the problem of stability and rheology reduction due to high solid content of drilling fluids especially, when drilling inclined holes. The limitation of this formulation however, is its environmental unfriendliness.

Sharma *et al.* (2001) developed an environmentally friendly drilling fluid which can effectively replace oil based drilling fluid by using eco-friendly polymers derived from tamarind gum and tragacanth gum. Tamarind gum is derived from tamarind seed while tragacanth gum is from astragalus gummifier. This formulation is also cheaper and has less damaging effect on the formation.

Hector *et al.* (2002) developed a formulation with a void toxicity based on a potassium-silicate system. The advantage of this formulation apart from being environmentally friendly is that cuttings from the use of this drilling fluid can be used as fertilizers.

Durrieu *et al.* (2003) formulated an additive called "booster fluid" which is a mixture of organic nitrogen, phosphorus compounds, and fatty acids that can be added to synthetic oil base fluid system in order to enhance the rate of biodegradation. They observed that synthetic oil based drilling fluid treated with "booster fluid" still demonstrated some level of environmental impact to marine life and hence not totally environmentally friendly.

Warren *et al.* (2003) developed a formulation based on a water-soluble polymer amphoteric cellulose ether, (ACE) which is cheaper, low in solids content, environmentally friendly but with some potential to damage the formation.

Jayne *et al.* (2004) developed a potassium silicate based drilling fluid system which is cheaper, re-useable, can eliminate background gas breakthrough, and eco-friendly as an alternative to sodium silicate based drilling fluid system which can be problematic due to the high sodium loading associated with cuttings generated when it is used to drill.

Davidson *et al.* (2004) developed a drilling fluid system that is environmentally friendly and which will also remove free hydrogen sulphide. It may be encountered while drilling based on ferrous iron complex with a carbohydrate derivative (ferrous gluconate).

Ramirez *et al.* (2005) formulated a biodegradable drilling fluid that maintains hole stability. This mud also enables to drilling through sensitive shale possible based on aluminum hydroxide complex (AHC). This formulation contains some blown asphalt and hence possesses some degree of environmental problem.

Amanullah *et al.* (2006) developed an environmentally friendly thermal degradation inhibitive additive for water-based bentonite mud using raw material from natural sources. This additive which is also able to prevent thickening and flocculation of bentonite, however, becomes ineffective at elevated temperature.

Malloy *et al.* (2007) suggested drilling with compressed air as an alternative to other drilling fluid system. Because compressed air as stressed is very effective in drilling through very hard and dry rock which is very cheap, and environmentally friendly. However, drilling with compressed air has some short comings. It can only be used to drill through hard, non-hydrocarbon, and non-water producing formation. This compressed air fluid is associated with high risk of fire accidents that could occur when air mixes with hydrocarbon during drilling operation.

Sajjad *et al.* (2008) implemented water based glycol muds as an alternative to diesel OBM's. They focused on optimizing mud weight and overall environmental and economic advantages offered by these systems using emulsifying oil and comparing its performance, environmental compatibility and cost with OBM's used in drilling low pressure zones in Iranian oilfields.

Xiaoqing *et al.* (2009) developed an environment acceptable modified natural macromolecule based drilling fluid which composed of shale inhibitor agents, fluid loss control agents, bloomless white asphalt and dry powder of poly alcohols. After a series of rheological tests, performance and environment compatibility tests, formation damage control ability tests, inhibitive property tests, the authors came up that the formulation was suitable for both land and marine drilling activities.

Tehrani *et al.* (2009) formulated a new chrome free, high density HPHT water based fluid system. The new fluid used a combination of clay and synthetic polymers to provide excellent fluid loss control, generate thermally stable rheology, prevented high temperature gelling and improved fluid resistance to drill solids contamination.

Dosunmu *et al.* (2010) developed an oil based drilling fluid based on vegetable oil derived from palm oil and ground nut oil. The fluid did not only satisfy environmental standards, it also improved crop growth when discharged into farm lands. Generally, all these formulation do not have zero environmental impact.

Amanullah *et al.* (2010) proposed the use of waste vegetable oil as an alternative to the use of mineral and diesel oil as the continuous phase in the formulation of high performance drilling fluids for HPHT applications. This formulation is not only eco-friendly, it is also cheap, and will be vastly available because large volumes of waste vegetable oil are generated annually worldwide.

Amin *et al.* (2010) developed an environmentally friendly drilling fluid system based on esters sourced from the Malaysian palm oil bio-diesel production plant which include methyl ester and ethylexyl ester. The short coming of this formulation is that the palm oil bio-diesel market determines the availability of the identified esters (the esters are by-product from the bio-diesel plant which means that increase in demand for bio-diesel, means increase in availability of esters, and vice-versa).

Apaleke *et al.* (2012) formulated a drilling fluid with canola oil as the base oil for an oil based mud system which is environment friendly, sustainable and has zero level of toxicity. The developed canola oil system was found to be stable at room temperature and simulated downhole conditions. Moreover, the canola oil based mud system was formulated without a wetting agent which helped in reduction of the cost of formulation.

Adesina *et al.* (2012) carried out an environmental impact evaluation of three different oil based muds with base fluids as diesel, jatropha oil and canola oil. The results obtained from laboratory tests indicated that jatropha oil pose a great chance of being an environmental viable replacement for the conventional diesel based mud as diesel oil was found to be the most toxic with jatropha oil having the least degree of toxicity.

Burden *et al.* (2013) conducted research on seven samples of drilling fluids to be operated in the African region. The samples included polymer based drilling fluid, amine based drilling fluid, synthetic based mud etc. to which a simple rating method was devised. It was concluded that synthetic based mud was the strongest technically followed by a modified-amine HPWBM system. It was also shown that both of these can be combined with techno-economic feasible treatment and disposal options to minimize environment impact.

Choudhary *et al.* (2013) applied chicory as a corrosion inhibitor for high temperature and strong acidic conditions. Chicory is a perennial bush plant available in many parts of the world. It was studied that chicory can be used to protect corrosion of either organic or inorganic acids up to 250 ^oF. Considering its performance and lack of toxicity issues, chicory was declared to have significant potential for acid corrosion inhibition.

Dias *et al.* (2014) used modified starch as fluid loss additive in invert emulsion drilling fluid. The authors concluded that the systems produced from modified starches presented rheology, filtration properties and electrical stability values within the specifications recommended by the API. Moreover, the formulations developed from starch were able to compete technically with the standard drilling fluid.

Teixeira *et al.* (2014) used hyper branched epoxy resin from glycerol as a non-hazardous environment friendly substrate. The results showed that the hyper branched epoxy resin had a great potential to be used as a loss control and well bore strengthening additive.

Li *et al.* (2014) used natural vegetable gum in drilling fluids for high temperature resistance and environmental protection. The temperature resistance of the drilling fluid was increased from 100^oC to 140^oC. The salt-resistant ability as well as the shale roll recovery also improved. In addition, the developed drilling fluid induced less formation damage and could effectively protect the formation.

Zhang *et al.* (2014) worked on nitration-oxidation lignosulfonate as an environment friendly drilling fluid additive. The results showed that NOLS could improve viscosity, reduce filtration loss at high temperatures, inhibit swelling of clay and displayed good temperature resistance. Moreover, NOLS benefited to the growth of wheat seedling and could be used as a fertilizer in agriculture after waste drilling disposal.

CHAPTER 3

RESEARCH CHALLENGES AND OBJECTIVES

This chapter states the problem of using toxic additives for the formulation of drilling fluids and its effect on marine life and the environment. The need to design an environmentally friendly drilling fluid is expressed and a summary of natural additives used in the oil and gas industry is presented. The chapter also addresses the objectives and the methodology of this study.

3.1 Knowledge Gap

Drilling fluid's position is in a challenging environment if its status is analyzed based on sustainability though there is a tremendous advancement in this technology. This is due to the complex formulation of the mud system which is needed to meet the different desired properties for smooth functioning while drilling. Saving our planet in a sustainable fashion is one of the major challenges for the researchers, industries, government and non-governmental agencies. Undoubtedly, the petroleum industry is one of the hazardous and unsustainable trades that call for an important and timely initiative to find out a gateway for greening the industry. This study is aimed toward this destiny.

3.2 Need for the research

The oil and gas industry has made tremendous progress in developing techniques, procedures, and less toxic materials for the protection of human health and the environment. In literature, it is well documented that diesel-based/mineral-based fluids have high toxicity levels (Rana, (2008); Duchemin *et al.* (2008); Dosunmu *et al.* (2010); Ammanullah, (2010); Hossain *et al.* (2010); Hossain, (2011)). Toxicity of drilling fluids to a large extent also depends on the type of additive, which means that even water based drilling fluid systems can also be environmentally unfriendly if the right additives are not used for its formulation. Toxicity levels of additives is influenced directly by the quantity of the drilling fluid used, concentration of the additive in the drilling fluid, and the rate at which the sump drilling fluid disperses when discharged into the environment. Almost every day toxic materials are disposed to the environment. There is no specific worldwide statistical data for this.

Becket et al. (1976) conducted acute toxicity test on 34 drilling fluid components using Rainbow Trout. They observed that organic polymer additives are extremely viscous, and at high viscosities, fish could not circulate the materials past the gills resulting in their deaths due to suffocation. Miller et al.'s (1980) experimental observation shows that additives such as asbestos, asphalt, vinyl acetate, and a host of others caused slight reduction in plant yield at low concentrations, increased reduction in plant yield at higher concentrations. Finally, they concluded that diesel oil, and potassium chloride (KCl) causes the most severe damage to plant yield. Younkin et al. (1980) reported that waste drilling fluid and/or sump fluid discharged into the terrestrial environment cause green plants to become variegated (loose chlorophyll) which results stunted in growth, and finally leads to the death of the plants. Murphy et al. (1984) studied the contamination of shallow ground water by oil and gas well drilling fluids in Western Dakota, U.S.A. Candler et al. (1992) reported that drilling fluid's heavy metals such as cadmium (Cd), and mercury (Hg) discharged into the environment through sump/drain may be picked up by fishes and other living entities in the sea. Ultimately, these discharged heavy metals are being consumed by human beings through those living entities. The toxic heavy metals then get passed on to humans via consumption of such contaminated seafood resulting in food poisoning and a number of other health problems. According to Ameille et al. (1995) and Greaves et al. (1997), the most observed symptoms in workers exposed to not-environment friendly drilling fluid additive such as aerosols are cough and phlegm. They also reported that workers exposed to mist and vapor from mineral oils (major continuous phase of oil based drilling fluids) showed increased prevalence of pulmonary fibrosis. Jonathan et al. (2002) also reported the toxic effects of drilling fluid additives on the physiology, fertility, and growth of fish egg and fry. They concluded that at high concentrations of additives, fish fry, and even mature fish will die. The authors of this article also gathered that drillers became chronically asthmatic due to prolonged exposure to toxic, and not-user friendly drilling fluids particularly the oil-based (diesel and mineral oil based) drilling fluids. The medical report shows that they were not asthmatic before joining to the company as a driller (from unpublished and undisclosed documents).

As a result, nowadays the toxicity of drilling fluids and their disposal are tightly controlled to minimize the effects on the subsurface and environment by the government and non-government EPAs. Yet, it is a challenge to tackle and reduce the level of health hazard and environmental disaster coming from drilling fluids. It is also a challenge to find out the solution of these challenges. Moreover, the existence of current drilling fluids depends on the greening process of the mud. Different government and non-government environmental agencies are also active in this regard which also is trying to solve the future challenges for the drilling fluid industry. Hence, the proposed research points out in brief the challenges needed to be addressed by the researchers.

Several researchers worldwide have come up with natural substitutes which function up to or better than their toxic counterparts and have become vital ingredients of the drilling fluid. Some of these natural additives used as additives in the drilling fluid are listed here:

Starch: - Starch is manufactured from either corn or potatoes and is supplied as a water soluble powder which can be treated with a preservative. It is either non-ionic or slightly anionic and is used as a fluid loss additive for all types of muds. It is particularly useful in a salt water system and requires a bactericide to prevent rapid degradation.

Biopolymers: - Biopolymers are polysaccharides manufactured from bacterial fermentation. They have extremely complex structures with high molecular weights (> 1 to 2 million) and are slightly anionic. Examples include xanthan gum, such as Kelzan XC, Zanvis, Xanvis, XC Polymer, Flodril S and Flopro; Wellan gum, such as Biozan; Scleroglucan gum, such as Shellflo-S. Biopolymers are primarily used as rheology control agents as they develop high, low shear- rate viscosities which are useful for suspension and carrying capacity.

Guar Gum: - Guar gum is a polysaccharide manufactured from the endosperm of the seed of the guar plant and is used as a viscosifier in drilling fluids. It has a complex structure with a high molecular weight (Chilingar and Croushorn, 1964). Examples include the regular guar gum, a natural material containing impurities, and hydroxypropylguar, a guar gum modified for purity and consistency.

Peach seeds: - Grounded peach seeds were used as a lost circulating material thereby decreasing the loss of drilling fluid into the formation. It was observed that the grounded peach seeds with a coating reduced the gel strength and viscosity of the drilling fluid which resulted in low working loads to pump the fluid down into the borehole.

Tree Bark: - Correctly defined sizes of bark fractions coupled with carboxy methyl cellulose were used as additives in a drilling fluid which reduced the loss of water (filtration loss) into the formation.

Nut Shells: - Finely grounded nutshells, nutshell flour and waterproofed sugarcane fibers were used as lost circulation materials to seal off fractures and inter granular permeability.

Cocoa Bean Shells: - A lost circulation controller for use in drilling fluids formed from cocoa bean shells with a specific particle size distribution was patented in the year 1984.

Corn Cob Outers: - An additive to reduce fluid loss from drilling fluids was developed using corn cobs and rice products. Polymers were also added to further reduce the fluid loss and the frictional resistance of the pipe movements.

Rice Fractions: - Comminuted rice fractions with other plant products were used as lost circulation materials to decrease the amount of fluid loss to the underground formations. Rice fractions are available in the form of rice hulls, rice tips, rice straws and rice bran. These different parts of the rice plant are separated commercially and are widely available in rice mills. The rice fraction is a common by-product when finished rice is brought to the market.

Cotton Seed Hulls: - Cotton seed hulls are fibrous, biodegradable and are excellent bridging agents when large particle size materials are needed. They are used in water based systems and are to be avoided for use in oil based muds.

Tamarind Gum: - An ecological friendly water based drilling fluid was developed by studying the rheological behavior of tamarind gum and polyanionic cellulose on bentonite water suspensions. Tamarind gum is a low viscosity modifier with almost the same viscosity of guar gum and extracted from the tamarind tree in India. The formulated drilling fluid exhibited minimum formation damage on sandstone cores.

Sugar Cane Ash: - Sugar cane ash along with cellulose like material and an oleaginous fluid were used as a filtration control agent in an environment friendly drilling fluid. The advantage of using such a composition ensured that the viscosity of the drilling fluid does not increase by more than 10% unlike that of CMC.

3.3 Replacement of Toxic Additives with Natural Substitutes Proposed in this Research

As stated, the Environmental Protection Agency (EPA) considers some earth metals such as zinc, chromium, lead, mercury, cadmium, nickel, asbestos and various phenol compounds as hazardous and toxic ingredients in the drilling mud. It is high time to emphasize the use of naturally occurring substances as additives to improve the work environment around people who are daily involved in this business. Henceforth, for this study, crushed date seeds, grass ash and dried powdered grass are proposed to be used as additives in the drilling fluid. The various advantages of natural substances in drilling fluids give a clear picture that these additives will tend to decrease the environmental pollution without compromising the basic function of the drilling fluid.

Date Seeds: - The fruit of the date palm tree is an important crop in the Middle Eastern countries (Biglari *et. al*, 2009) and is composed of a fleshy pericarp and seed. The seed constitutes about 10 to 15 % of the date fruit weight (Hussein *et. al*, 1998). The date seed is often considered as a byproduct of dates processing plants which produce pitted dates, date syrups and date confectioneries (Al Farsi and Lee, 2008). The production of date fruits in this world is estimated to be 6.9 million tons of which 863 thousand tons of date seeds are extracted (FAO, 2007). About 18 % of the world's total production of date fruits is contributed by Saudi Arabia (Research and Agricultural Development Affairs, 2006). At present, date seeds are used mainly for cattle feeds such as camel, sheep and even the poultry industry. Proximate analysis of Saudi Arabian date seeds indicated that these contain high amounts of protein, crude fat and fibers. It is also a proven fact that date seeds serve as a natural source of phenolic compounds and an antioxidant (Ammar *et. al*, 2009). Umoren *et.al* from King Fahd University of Petroleum and Minerals found that date palm seed extracts inhibited the corrosion of mild carbon steel in steel pipelines and performed better when corroded with hydrochloric acid than sulfuric acid.

Grass Ash: - Grass Ash is the product formed when dried grass is burned. The principal component of ash is silicon. Research done on a particular grass ash for a civil engineering project indicated that grass ash could be used as a substitute for cement as its production requires neither high technology nor sophisticated hardware, with the process being simple, economical and well suited for rural areas in developing countries.

Grass: - The term 'Grass' needs no formal introduction as it is the principal fodder for cattle across the globe and its use is known to humankind for centuries. The preamble of this research is to introduce grass as an environment friendly additive in the drilling fluid.

3.4 Objectives

1. To investigate the particle size distribution of the proposed additives which are date seeds, grass and grass ash.
2. To formulate an environmentally friendly mud system using the above natural substitutes as additives.
3. To investigate the characteristics and behavior of the newly formulated drilling mud system using the proposed additives.

CHAPTER 4

RESEARCH METHODOLOGY

This chapter presents the laboratory equipments used in this research. Also included are the formulae which are used to determine various parameters such as weight of the material after conducting particle size distribution, rheological parameters such as apparent viscosity, plastic viscosity and yield point.

4.1 Equipments used for Experimentation

This study is an applied research where series of experiment have been conducted and the following equipments were used:

Mud Balance – A mud balance is used to determine the mud density after mixing all the drilling fluid additives. Normally, the required mud weight is calculated before mixing as determined by so many factors such as bottom-hole pressure, the section of the hole to be drilled, etc. Figure 4-1 shows conventional mud balance equipment.

Figure 4- 1: Mud Balance

Rotational Viscometer – A Fann Model 35 viscometer was used to measure the rheological properties (i.e. plastic viscosity, yield point, and gel strength) of the fluid samples. The Fann Model 35 viscometer is a Couette type, coaxial cylinder rotational viscometer, used to determine single or multi-point viscosities. Fig. 4-2 shows a rotational viscometer.

Figure 4- 2: Fann Model 35 Viscometer

Weigh balance: A weigh balance is used for measuring the amount of additives to be added into the mud. It must be ensured that the surface of the balance is wiped clean otherwise, measured weights will be inaccurate. Figure 4-3 shows a digital weighing balance.

Figure 4- 3: Weigh Balance

Multicell API filtration loss tester: This apparatus is used basically for testing water based mud filtration loss. Test pressure is set at 100 psi and room temperature as per API guidelines. Figure 4-4 shows a multicell API filtration loss tester.

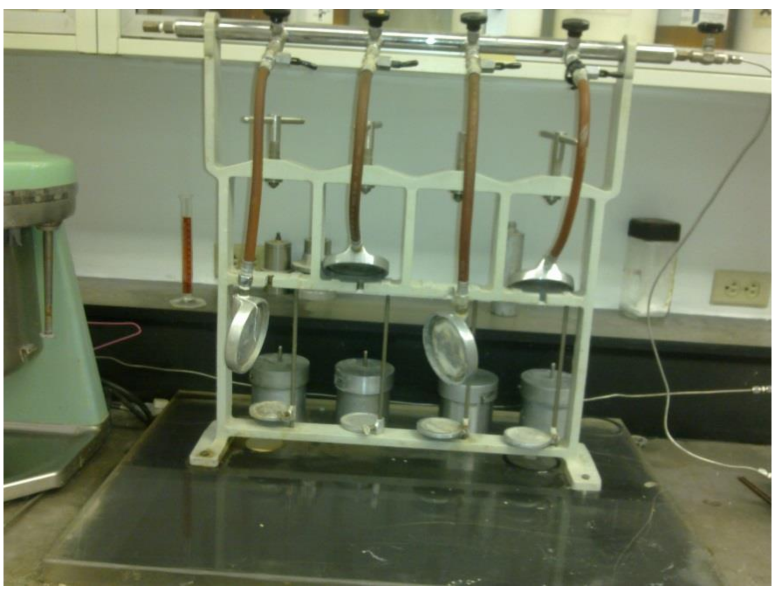

Figure 4- 4: Filtration Loss tester

Hamilton Beach Mixer: This is used for mixing the mud. It has three speeds: high, medium and low. The mud should be sheared long enough for each additive to be dispersed in the fluid phase of the mud system. Figure 4-5 shows a Hamilton Beach Mixer.

Figure 4- 5: Hamilton Beach Mixer

Heating Oven: This is used for heating mud samples at a particular temperature of interest to simulate down-hole conditions. Samples are placed in the oven long enough so that the desired temperature of the drilling fluid is attained. Figure 4-6 shows an oven.

Figure 4- 6: Heating Oven

Thermocup: A thermocup is used to maintain a temperature so that the drilling fluid remains hot and actual simulated downhole conditions prevail during measurements. Fig. 4-7 shows a thermocup used in this study.

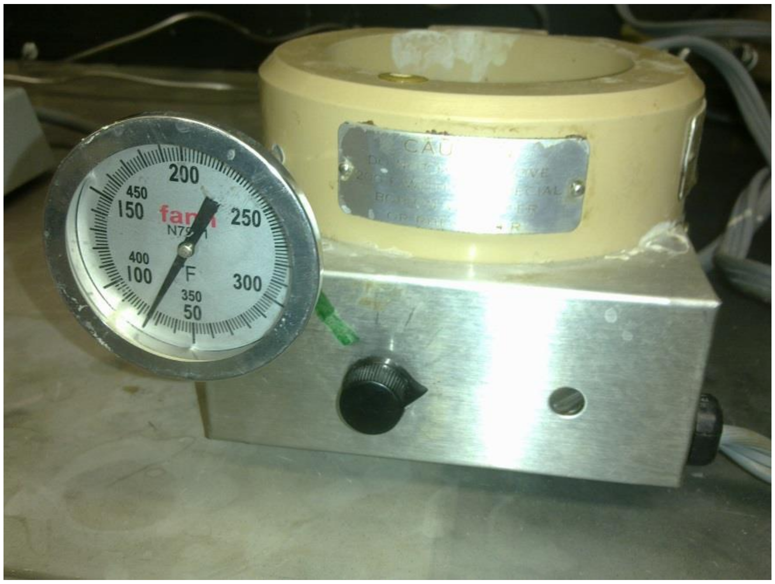

Figure 4- 7: Thermocup

Resistivity meter: A Fann model 88C resistivity meter is used to determine the resistivity of the filtrate collected after the low pressure room temperature filtration loss experiment. The equipment offers ranges from 2 to 200 ohm-meter. Fig. 4-8 shows a resistivity meter.

Figure 4- 8: Resistivity meter

pH meter: A pH meter determines the hydrogen ion concentration of a solution. Fig. 4-9 shows a pH meter used for this research.

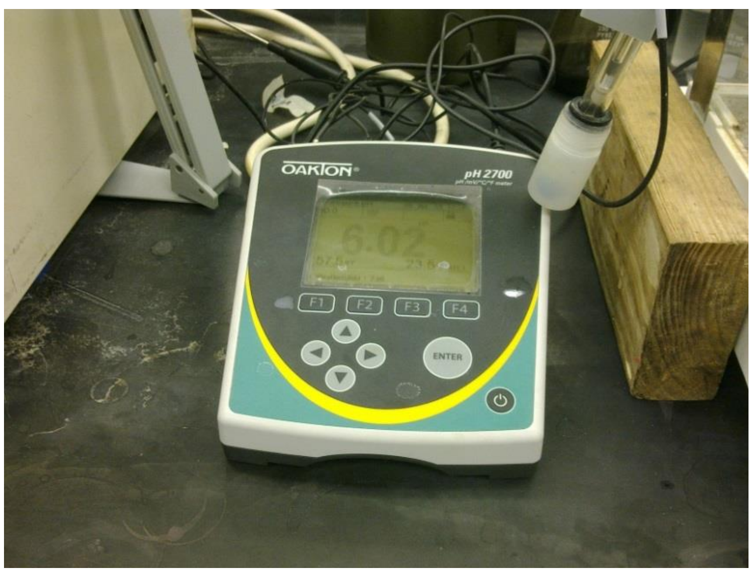

Figure 4- 9: pH meter

4.2 Preparation of Samples and Formulae used

Particle Size Distribution: Samples of date seeds, grass ash, and grass were collected form the eastern province of Saudi Arabia. These materials were then dried in a sunny area for about a week and then crushed in a grinding machine. Part of dried grass was then burnt to get grass ash. The sample materials prepared for the test were then placed in a sieve shaker with openings in the decreasing order of sieve size (viz. increasing order of sieve number). Table 4-1 shows the mesh numbers and their corresponding sizes. Granular particles get accumulated on different sieves which give the particle size distribution of the substance. It is assumed that no losses were incurred during the whole run of the experiment i.e. the sum of the weights of the aggregates accumulated on individual sieves give the total weight of the sample taken. A Fritsch Laser Particle Size Analyzer was used for estimating the particle size of finer fractions. This PSA is equipped with state-of-the-art computer software which records readings directly to a computer.

Table 4- 1: Mesh Numbers with corresponding Sieve sizes

Mesh Number	Sieve Size or Screen Opening (microns)
30	600
50	300
80	180
100	150
120	125
140	106
170	90
200	75

Eq. 4.1 is used in order to gain consistency in the units for better comparison.

$$\Phi = -\frac{\log_{10} d}{\log_{10} 2} \quad 4.1$$

Where,

Φ = called Φ unit, dimensionless

d = diameter of the sieve opening, mm

The weight of the material on the sieve can be calculated using Eq. 4.2

$$W_m = W_{ms} - W_s \quad 4.2$$

Where,

W_m = the weight of material on the sieve, gm

W_{ms} = the weight of sieve and material, gm

W_s = the weight of the sieve, gm

The percent weight retained on each sieve can also be given by Eq. 4.3

$$W_{rs} = \frac{W_{mr}}{W_{tm}} \quad 4.3$$

Where,

W_{rs} = percent weight retained on each sieve, %

W_{mr} = weight of material retained, gm

W_m = total weight of material, gm

Rheological Characterization: The viscometer is designed to facilitate the use of the Bingham plastic model in conjunction with drilling mud. Bingham plastic model relates shear stress and shear rate by the following equation as (Darley et al., 1988):

$$\tau = \tau_0 + \mu_0 \gamma \quad 4.4$$

Where

τ = shear stress, ${lb_f}/{100\ ft^2}$

τ_0 = yield point, ${lb_f}/{100\ ft^2}$

μ_0 = plastic viscosity, centipoise

γ = shear rate, sec^{-1}

The viscometer has a torsion spring-loaded bob that gives a dial reading proportional to torque, which is analogous to the shear stress. The rotational speed is designed to follow the Bingham plastic model (American Society of Mechanical Engineers, 2005) as:

$$\theta = YP + PV({\omega}/{300}) \qquad 4.5$$

Where

θ = dial reading,

YP = yield point, ${lb_f}/{100\ ft^2}$

PV = plastic viscosity, centipoise

ω = rotation speed, rpm.

The determination of PV and YP is obtained from the dial readings at 600 rpm and 300 rpm as follows:

$$PV = \theta_{600} - \theta_{300} \text{ and } YP = \theta_{300} - PV \qquad 4.6$$

Where

θ_{600} = 600 rpm dial reading, dimensionless

θ_{300} = 300 rpm dial reading, dimensionless

CHAPTER 5

RESULTS AND DISCUSSION

In this chapter, the characterizations of the proposed additives are discussed. XRF study is conducted to know better the composition of the proposed additives. Particle size analysis is conducted to evaluate the mud rheology at different particle sizes. Moreover, mud formulations and the measured mud properties are discussed comprehensively.

5.1 Particle Size Distribution and Compositional Analysis

This section presents the results of particle sizing and XRF analysis for the three proposed natural additives which are date seeds, grass and grass ash. Particle-size distribution (PSD) is an important tool to evaluate the potential use of samples and influences how well aggregates function in an engineering project. Appropriate amount of fine particles in a drilling fluid indicates a firm filter cake which retards invasion of the drilling fluid into the formation and helps in maintaining the borehole stability. A XRF study is conducted to know better the composition of the proposed additives which will aid in the development of a sustainable drilling fluid being both environmentally-friendly and cost effective.

5.1.1 Particle Size Distribution

Particle Size Distribution is extensively used by geologists in geomorphological studies to evaluate sedimentation and alluvial processes and by civil engineers to evaluate materials used for foundations, road fills and other construction purposes. In the oil and gas industry, PSD analysis finds its application in determining the filtration loss properties and the amount of solids content retained in the drilling fluid after the fluid is pumped into the system. A drilling fluid containing particles of sizes ranging up to the requisite maximum should be able to effectively bridge the formation and form a filter cake (in the case of a water-based mud). Above 10 Darcys or in fractures, larger particles are required, and most likely the amounts needed to minimize spurt losses increase with the size of the opening. In general, with the increasing concentration of bridging particles, bridging occurs faster and spurt loss declines (Growcock and Harvey, 2004). Filtrate invasion into the formation can substantially reduce the permeability of the near wellbore region either by particle plugging, clay swelling or water blocking. Permeability of the filter cake is dependent on the particle size distribution as increasing particle size decreases the permeability due to the fact that colloidal particles get packed very tightly. For non-reservoir applications, enough particles of the required size range are usually present in most drilling fluids after cutting just a few feet of rock. These particles impact the choice of various drilling equipment (i.e. shale shakers, desanders, desilters etc.) at surface and thus can be effectively designed by having a beforehand knowledge of the particle sizing in the drilling mud.

5.1.2 Elemental Analysis using XRF

X-Ray Fluorescence or simply XRF is a process whereby electrons are displaced from their atomic orbital positions, releasing a burst of energy that is characteristic of a specific element. This release of energy is then registered by the detector in the XRF instrument, which in turn categorizes the energies by element. Here is a detailed breakdown of the process (also refer Fig. 5-1):

1. An x-ray beam with enough energy to affect the electrons in the inner shells of the atoms in a sample is created by an x-ray tube inside the handheld analyzer. The x-ray beam is then emitted from the front end of the XRF analyzer.
2. The x-ray beam then interacts with the atoms in the sample by displacing electrons from the inner orbital shells of the atom. This displacement occurs as a result of the difference in energy between the primary x-ray beam emitted from the analyzer and the binding energy that holds electrons in their proper orbits; the displacement happens when the x-ray beam energy is higher than the binding energy of the electrons with which it interacts. Electrons are fixed at specific energies in their positions in an atom, and this determines their orbits. Additionally, the spacing between the orbital shells of an atom is unique to the atoms of each element, so an atom of potassium (K) has different spacing between its electron shells than an atom of gold (Au), or silver (Ag), etc.

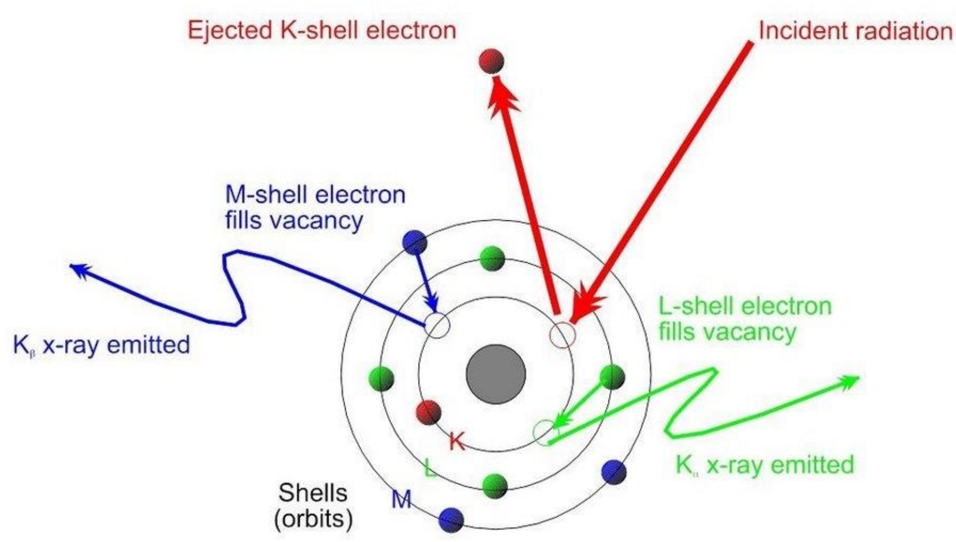

Figure 5- 1: Working of XRF

3. When electrons are knocked out of their orbit, they leave behind vacancies, making the atom unstable. The atom must immediately correct the instability by

filling the vacancies that the displaced electrons left behind. Those vacancies can be filled from higher orbits that move down to a lower orbit where a vacancy exits. For example, if an electron is displaced from the innermost shell of the atom (the one closest to the nucleus), an electron from the next shell up can move down to fill the vacancy. This is fluorescence.

4. Electrons have higher binding energies the further they are from the nucleus of the atom. Therefore, an electron loses some energy when it drops from a higher electron shell to an electron shell closer to the nucleus. The amount of energy lost is equivalent to the difference in energy between the two electron shells, which is determined by the distance between them. The distance between the two orbital shells is unique to each element, as mentioned above.
5. The energy lost can be used to identify the element from which it emanates, because the amount of energy lost in the fluorescence process is unique to each element. The individual fluorescent energies detected are specific to the elements that are present in the sample. In order to determine the quantity of each element present, the proportion in which the individual energies appear can be calculated by the instrument or by other software.

The entire fluorescence process occurs in small factions of a second. A measurement using this process can be made in a matter of seconds. The actual time required for a measurement will depend on the nature of the sample and the levels of interest. High percentage levels take a few seconds while part-per-million levels take a few minutes.

Use of XRF in the petroleum industry: Commercial clays such as bentonite or other chemically treated clays are added to the drilling fluid for control of rheological and filtration properties. The total of commercial clays and drilled solids is called as “low gravity solids” (LGS). Weighting materials (barite or barium sulfate) are used to bring the fluid to the required density, necessary to contain underground formation fluids by hydrostatic pressure exerted by the mud column in the annulus. The concentration of these weighting materials is known as “high gravity solids” (HGS). It is important for effective control of the properties of the fluid to know the individual concentrations of all types of solids (i.e. LGS and HGS). These entities are either measured directly or calculated from the density and solids volume fraction of the drilling fluid both of which can be measured but is laborious. Traditionally, the LGS-HGS volume ratio is measure using a retort, a technique that requires good operator skills, takes atleast 45 minutes and has an error margin of 15% (Bloys *et al., 1994*).

XRF, introduced in the oil and gas industry for the analysis of core samples, is now deployed to monitor the concentrations and differentiate various solids type (LGS and HGS) in the drilling fluids (Houwen *et al., 1996*). XRF has the advantage of more frequent measurement, greater precision and less dependence on operator skills. It is extensively used for the characterization of bentonite and other clay types for different clay applications. The authors after cogitation, remark the application of XRF to determine the elemental composition of additives to limit the usage of toxic chemicals in environmentally sensitive areas. For this purpose and due to the unavailability of the elemental composition of date seeds, grass and grass ash in the literature, the authors have taken the initiative to conduct XRF studies on the three said specimens.

5.2 Date Seeds: Elemental Analysis and Particle Size Distribution

Table 5-1 is the distribution of elements in the date seeds sample. The date seeds sample contained potassium, calcium, iron, chlorine, silicon, sulfur, phosphorous and manganese with potassium and calcium as the major contributors by net weight percent. Small traces (below 10% net weight) of sulfur, phosphorous and manganese were recorded. Fig. 5-2 shows the spectra exhibited by the date seeds sample as revealed by XRF. It should be noted that the intensity of the peaks in the XRF is not a quantitative measure of the elemental concentration.

Table 5- 1: XRF analysis of Date Seeds

Element	Atomic Number	Net Normal weight %
Potassium (K)	19	37.34
Calcium (Ca)	20	29.69
Iron (Fe)	26	10.36
Chlorine (Cl)	17	7.80
Silicon (Si)	14	6.21
Sulfur (S)	16	4.41
Phosphorous (P)	15	3.79
Manganese (Mn)	25	0.41

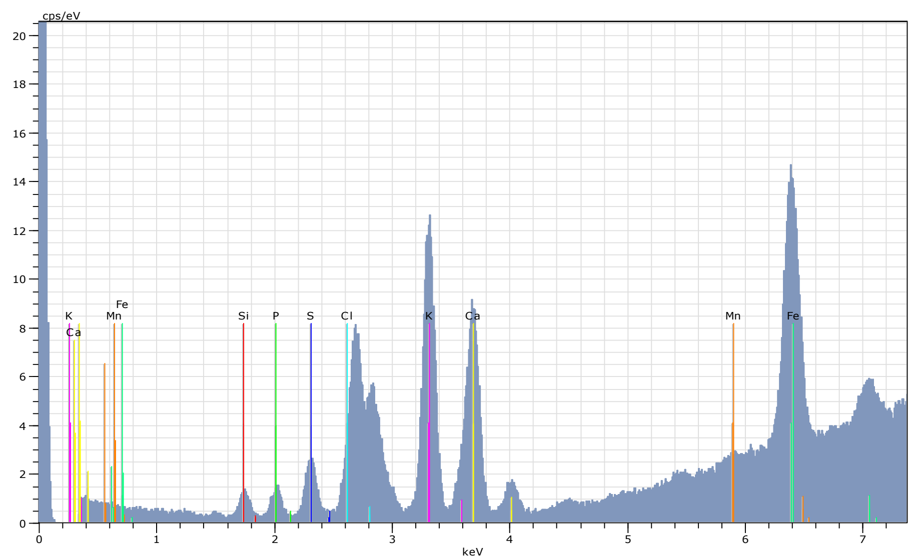

Figure 5- 2: Spectra of the Date Seeds sample

Table 5-2 shows the sieve analysis of date seeds sample data used for the construction of the two curves. Figure 5-3 shows the normal or probability distribution curve of the date seeds sample. Sieves of sizes 600, 300, 150, 125, 75 microns and a no sieve pan were used to sample the date seeds powder. It can be inferred from the bar diagram that majority of the date seeds material is retained on the 300 size sieve and as per the API 13C bulletin, particles of this size are classified as intermediate ones.

Table 5- 2: Sieve Analysis of Date seeds sample

Weight of Date seeds: 643.4 gms						
Sieve Size (μ)	Φ unit (dimensionless)	Sieve weight (gm)	Sieve + Date Seeds (gm)	Weight of Date Seeds Retained (gm)	Percent Weight Retained (%)	Total Percent Weight Retained (%)
600	0.74	417	543.9	126.9	19.72	19.72
300	1.74	372.6	774.1	401.5	62.40	82.12
150	2.74	346.9	448.2	101.3	15.74	97.87
125	3	346.7	359.3	12.6	1.96	99.83
75	3.74	339.6	340.7	1.1	0.17	100
No sieve	4.32	254.4	254.4	0	0	100

Figure 5-4 shows the frequency distribution curve of the date seed sample. An observation of the graph shows that at and above 50% cumulative weight, the relative percentage of finer particles (reading on the X - axis to the right of the curve) is more in this sample. This implies that the date seed sample has good tendency to get suspended in the drilling mud, prevent fluid loss and form a filter cake to avoid unnecessary fluid loss to the formation.

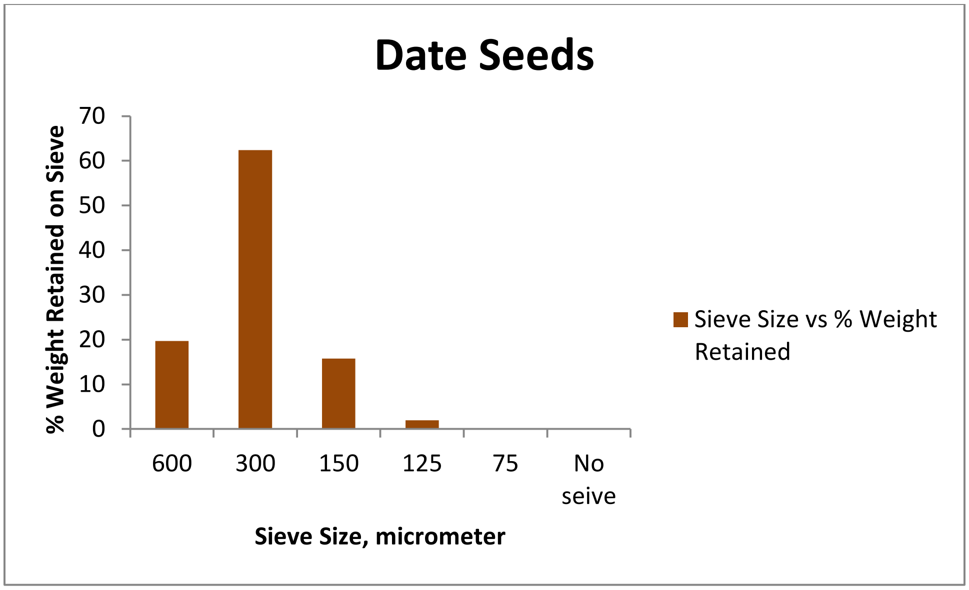

Figure 5- 3: Normal Distribution of Date Seeds on various sieves

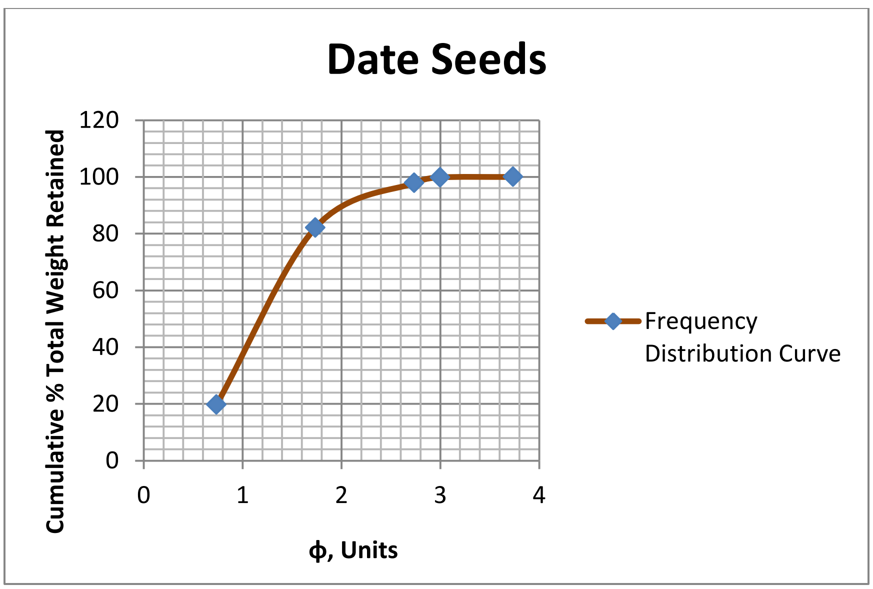

Figure 5- 4: Frequency Distribution Curve of Date Seeds

5.3 Grass Ash: Compositional Analysis and Particle Size Distribution

XRF conducted on the grass ash sample indicated the presence of silicon, calcium and potassium and chlorine as the highest contributors by net normal weight percentage. Small percentages of magnesium, sulfur, iron, phosphorous, aluminium, titanium and manganese are also found in this specimen. Table 5-3 illustrates all the elements present in the grass ash sample. Figure 5-5 is the spectra exhibited by this sample.

Table 5- 3: XRF analysis of Grass Ash

Element	Atomic Number	Net Normal weight %
Silicon (Si)	14	27.56
Calcium (Ca)	20	23.46
Potassium (K)	19	21.82
Chlorine (Cl)	17	15.51
Magnesium (Mg)	12	3.40
Sulfur (S)	16	2.99
Iron (Fe)	26	2.00
Phosphorous (P)	15	1.70
Aluminium (Al)	13	1.21
Titanium (Ti)	22	0.24
Manganese (Mn)	25	0.09

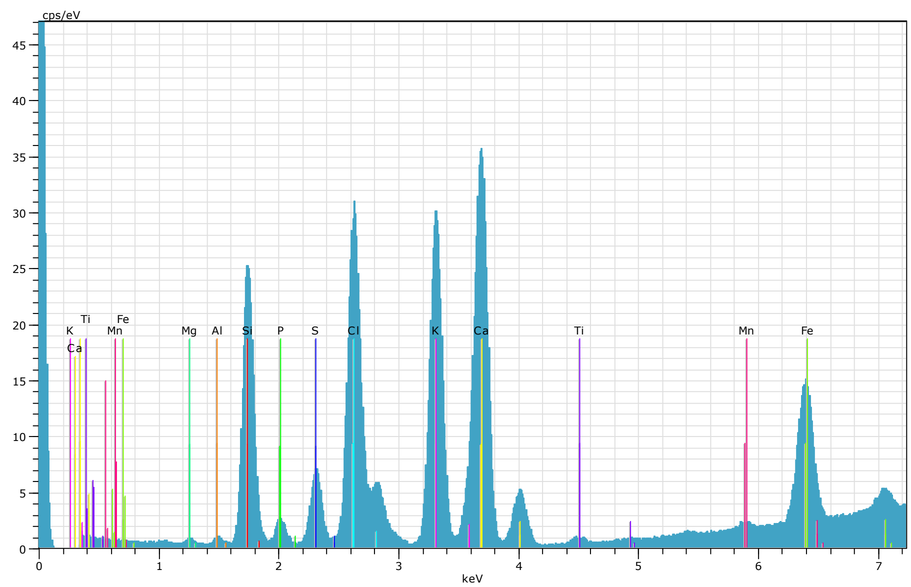

Figure 5- 5: Spectra exhibited by the Grass Ash sample

Table 5-4 shows the sieve analysis of grass ash data used in the construction of the two curves. Figure 5-6 represents the normal distribution curve of the grass ash sample. The grass ash material is sieved on sizes 300, 150, 106, 90, 75 microns and a no sieve pan. The percentage of aggregate accumulated on the no sieve pan is the highest which indicates that the sample consists of fine particles.

Table 5- 4: Sieve Analysis of Grass Ash sample

Weight of Grass Ash: 199.2 gms						
Sieve Size (μ)	Φ unit (dimensionless)	Sieve weight (gm)	Sieve + Date Seeds (gm)	Weight of Date Seeds Retained (gm)	Percent Weight Retained (%)	Total Percent Weight Retained (%)
300	1.74	371.7	389.7	18	9.04	9.04
150	2.74	347	389.2	42.2	21.18	30.22
106	3.24	343.3	372.2	28.9	14.50	44.73
90	3.48	338.4	360.6	22.2	11.14	55.87
75	3.74	339.4	379.2	39.8	19.98	75.85
No sieve	4.32	254.3	302.5	48.2	24.20	100

From the frequency distribution curve of grass ash (Fig. 5-7), at and above 50% cumulative weight, relative percentage of finer particles is the highest. As the highest percentage (by weight) of this sample settled on the no sieve pan, a Fritsch Laser Particle Size Analyzer was used to determine the particle sizing of the finer fraction. Three attempts were conducted to obtain better results and these are plotted in Fig. 5-8. Particle Size is read on the X-axis of Fig. 5-8 while the normal and frequency distributions are read on the Y-axis (right and left of the Y-axis respectively). Inference drawn from this plot indicates that the average particle size of the finer fraction at 50% net weight is 26 microns (frequency distribution curve). Thus, it is inferred that the use of grass ash is suitable for drilling fluid application.

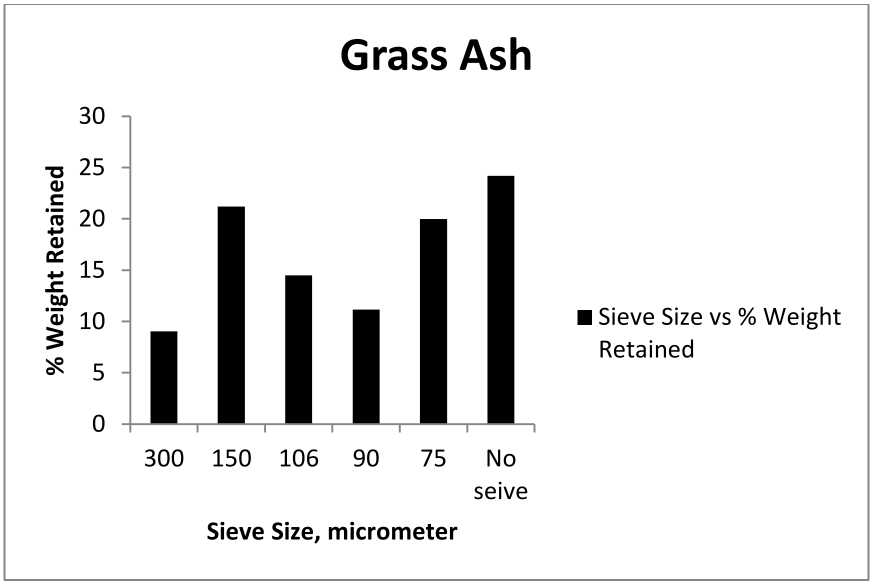

Figure 5- 6: Normal Distribution of Grass Ash on various sieves

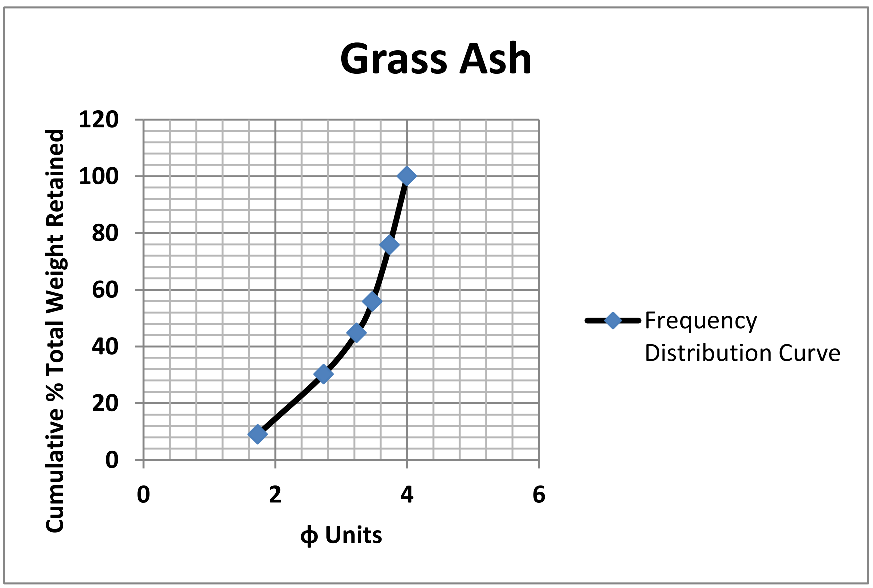

Figure 5- 7: Frequency Distribution Curve of Grass Ash

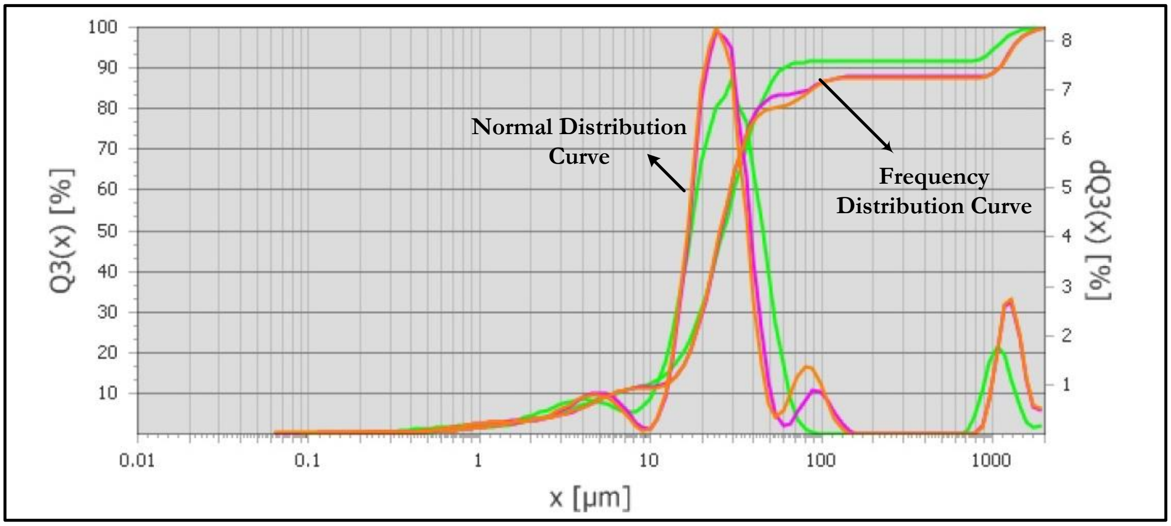

Figure 5- 8: Particle size distribution of finer particles of Grass Ash using a Particle Size Analyzer

5.4 Grass: Elemental Analysis and Particle Size Distribution

XRF analysis of the grass sample pointed calcium, potassium and chlorine as the highest contributors by net normal weight percentage. Sulfur, Silicon, Iron, Phosphorous and Manganese are also found in this specimen as small traces. Table 5-5 illustrates the elements present in the grass sample. Figure 5-9 is the spectra exhibited by this sample.

Table 5- 5: XRF analysis of Grass

Element	Atomic Number	Net Normal weight %
Calcium (Ca)	20	53.80
Potassium (K)	19	19.83
Chlorine (Cl)	17	15.54
Sulfur (S)	16	3.89
Silicon (Si)	14	3.13
Iron (Fe)	26	2.46
Phosphorous (P)	15	1.24
Manganese (Mn)	25	0.12

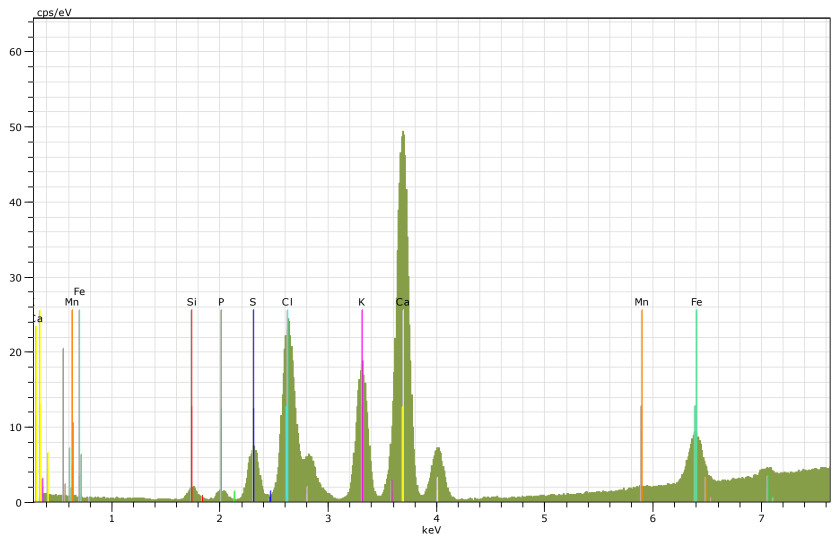

Figure 5- 9: Spectra exhibited by the Grass Sample

Table 5-6 is the sieve analysis of dried powdered grass data used in the construction of the two curves. Figure 5-10 is the normal distribution curve of the grass sample. Sieve sizes of 300, 180, 125, 90, 75 microns and a no sieve pan were used. The highest percentage of weight retained was on the 150 micron sieve which indicates that maximum of the particles of the grass sample belong to the medium category of particle size classification. The frequency distribution curve (Fig. 5-11) of the grass sample shows that at and above 50% cumulative weight, the sample consists of fine particles with 6% of the sample retained on the pan (finest fraction). In order to determine the particle size of the finest fraction, the laser PSA is used with three attempts of measurements. Particle Size is read on the X-axis of Fig. 5-12 while the normal and frequency distributions are read on the Y-axis (right and left of the Y-axis respectively). The test revealed the average particle size of the finest fraction of grass at 50% net weight as 35 microns thus prompting to imply that this grass sample is also a suitable candidate to be used as an additive in the drilling fluid.

Table 5- 6: Sieve Analysis of Grass sample

Weight of Grass: 411.3 gms						
Sieve Size (μ)	Φ unit (dimensionless)	Sieve weight (gm)	Sieve + Date Seeds (gm)	Weight of Date Seeds Retained (gm)	Percent Weight Retained (%)	Total Percent Weight Retained (%)
300	1.74	371.6	426.1	54.5	13.25	13.25
180	2.47	356.2	528.4	172.2	41.87	55.18
125	3	346.8	433.5	86.7	21.08	76.20
90	3.47	338.7	370.7	32	7.78	83.98
75	3.74	339.7	379.3	39.6	9.63	93.60
No sieve	4.32	254.4	279.3	24.9	6.05	100

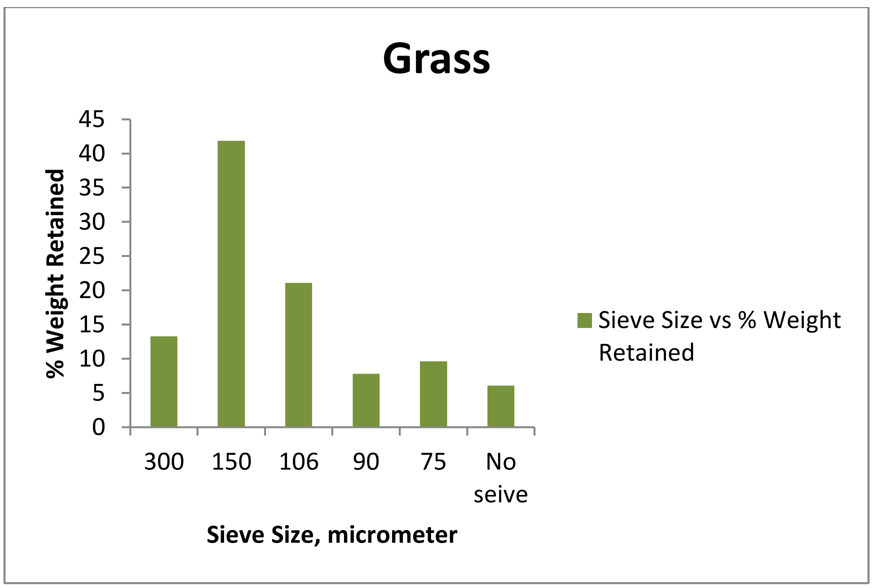

Figure 5- 10: Normal Distribution of Grass on various sieves

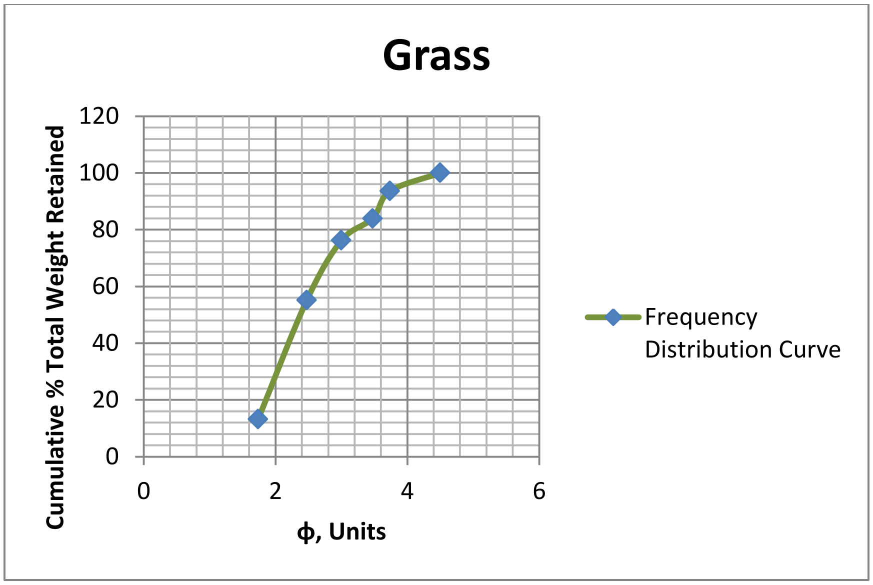

Figure 5- 11: Frequency Distribution Curve of Grass

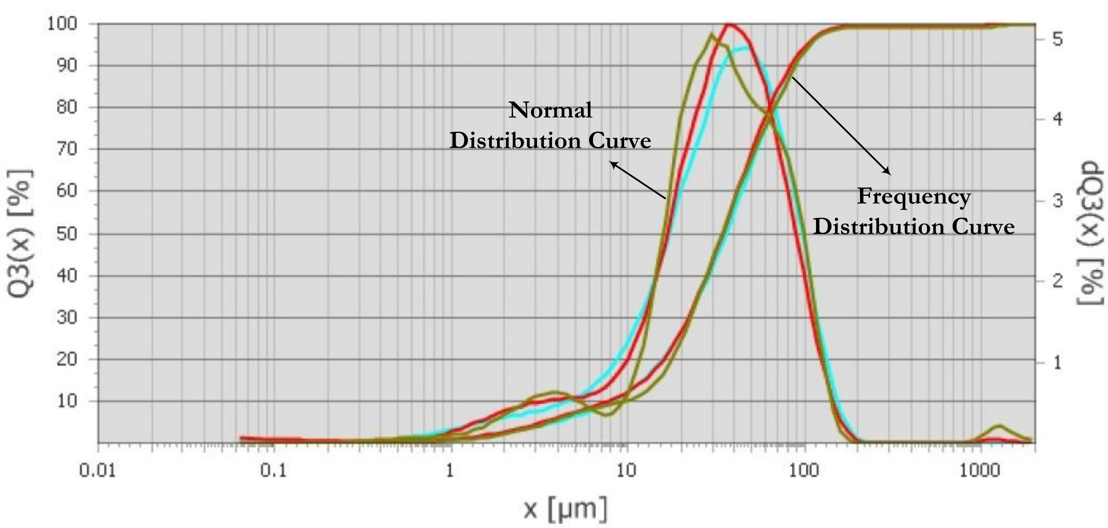

Figure 5- 12: Particle size distribution of Grass using a Particle Size Analyzer

Particle sizing is extremely important when considering the bridging abilities of materials. It is noted that particles smaller than the pore size of a geological formation bridge rock pores during mud circulation. This leads to the formation of a filter cake that prevents egress of fluids from the well during drilling. This is an important function of the mud as the cake protects the surrounding rock from damage while simultaneously preventing fluid loss and stabilizing the well. It is particularly vital when drilling through shales, which are highly prone to fluid invasion and difficult to drill without excessive fluid and associated pressure loss. Careful determination of the optimum particle size is essential as very small particulates may themselves penetrate the surrounding rock formation, blocking pores and cause an irreversible damage to the production zone.

5.5 Comparison of the three proposed additives with conventional mud additives:

5.5.1 Elemental Approach*:* XRF experiment conducted on the three specimens as mentioned earlier contained elements in their organic form. These elements include potassium, calcium, sulfur, silica, iron, chlorine, phosphorous, manganese, aluminium and titanium. These elements are used as compounds in the drilling fluid to perform various functions: Potassium is used in the drilling fluid as an Alkalinity Control agent (Potassium Chloride, KCl), Alkalinity and pH Control agent (Potassium Hydroxide, KOH), Weighing Agent (Potassium Formate, $CHKO_2$) etc. Calcium is also found in high percentage and is used as a Bridging and Weighing Agent as Calcium Carbonate ($CaCO_3$), as an inhibitor to control active shale and clay dispersion as Calcium Chloride ($CaCl_2$). Chlorine found in the sample could be used as a disinfectant to clean surface pipes as is used with source materials being Sodium Hypochlorite and Calcium Hypochlorite. It is also used as a polymer oxidizer for drilling, completion and work-over clean up in the form of Chlorine bleach. Silica found in the sample can be used to exhibit various functions. Silica is added to a drilling mud to change density, ionic strength, charge, etc. that are needed for critical drilling mud functions such as: drill-bit cooling, bit cleaning, effective cuttings removal to surface, downhole pressure control, and shale stabilization. Likewise, the use of silicate muds offers the advantages of prevention of bit-balling, differential sticking, and loss circulation in addition to the well-known use as a corrosion inhibitor. Phosphorous found in the sample could help reduce the pH of the mud as is done conventionally by phosphoric acid.

5.5.2 Particle Size Distribution Approach*:* The API specifies the range of particle size of barite for drilling mud applications. The fraction above 75 microns should be minimal and the percentage of material below 6 microns no higher than 30% by weight. Calcium carbonate, used as a bridging material and a weighting agent, often in preference to barite, is used in sizes ranging from less than 10 microns up to greater than 100 microns. Several researchers have used natural materials as additives, particularly as filtration control agents and lost circulation materials in the drilling fluid with varying particle sizes. Morris patented his work in the year 1962 with the use of peach seeds as a filtration control agent. He used a mixture containing particle sizes ranging from approximately 4 mesh size (4760 microns) to 200 mesh size (75 microns). This size grading of the peach seeds assured that all the particle sizes necessary for the efficient bridging of the porous subsurface formations would be present in the additive. The smaller seed particles would continually filter into porous formations until an effective mud sheath is formed by the larger seed particles. Lummus *et al.* (1971) used grounded nutshells and nut flour as fluid loss additives and patented their work. They came up with 20 mesh size (840 microns) to 100 mesh size (150 microns) nutshells and 100 mesh size nut flour to be the optimum particle size to avoid loss of fluid into the formation. Green (1984) came up with grounded and sized cocoa bean shells as lost circulation material in the drilling fluid. The lost circulation controller (cocoa bean shells) had a particle size distribution ranging from 2 mesh size (> 6730 microns) to 100 mesh size. Burts (1992) came up with a patent utilizing rice fraction as lost circulation material in the drilling fluid. He stated in his invention that the suitable particle size of rice fraction could be from 65 mesh (230 microns) to about 100 mesh but preferred it from about 65 mesh to 85 mesh (170 microns). Burts in the year 1994 came up with another patent and introduced corn cob outers as a lost circulation material and found out that a particle size of 85 mesh is suitable for his invention. Ghassemzadeh (2011) patented his work on fibers to be used as lost circulation materials in the drilling fluid. He used an optional average fine particle size of 5 to 15 microns, medium particles of an average size of about 20 to 150 microns and coarse particles having an average size of about 300 microns to 2500 microns. An observation of the results show that all the three samples contain particle sizes which comply well with those stated above. Thus, the author is of the opinion to study and develop a drilling fluid based on the three samples as the particles present in these samples can help clog the formation by means of a filter cake and prevent fluid loss as well as retard fluid invasion into and from the formation.

5.6 Analysis of Mud Rheology

5.6.1 Mud Formulations and measurement of mud properties

When developing a mud system in the laboratory, the units of measure most commonly used are grams for weight and cubic centimeters for volume. 1 barrel of mud in the field is equivalent to 350 ml in the laboratory. Thus adding 1 gram of material to 350 ml of fluid in the laboratory is equivalent to adding 1 lb_m of material to 1 bbl of mud in the field (Bourgoyne jr. *et al*, 1986).

A complete and comprehensive check is made on the formulated muds. The following tests were performed:

Density determination: The density of the mud was measured using a mud balance as shown in Figure 4-1. An 8.6 ppg mud was formulated for experimentation for all mixes.

Viscometer readings: Also performed on the mud are rheological experiments using a viscometer. Rotational speeds of 600, 300, 200, 100, 6 and 3 rpm were employed on the Fann Viscometer. Gel strengths were taken at 10 seconds and 10 minutes respectively while the plastic viscosity was determined from 600 and 300 rpm dial readings. Plastic viscosity (PV) relates to the portion of flow resistance caused by mechanical friction. For a good mud system, PV value should not be excessive. An excessive PV will result in an excessive equivalent circulation density (ECD). This ultimately results in an increased risk of loss circulation. Low PV will result in poor suspension of additive and weighing material in the mud. Yield Point (YP) and Gel Strengths (Gels) are also properties that should not be too high for a good mud system. If these properties are too high, the consequences will be the same as for high PV. If they are too low, the result would be poor cutting transport and an increased potential for barite setting or sag.

Low Temperature Filtration: Filtration tests are carried out using a low temperature filter press at a pressure of 100 psig. Filtrate loss is important because excessive filtrate loss can contribute to formation damage and differential pipe sticking.

Resistivity measurement: The resistivities of the formulated drilling muds are measured using a resistivity meter. The resistivity of water-based muds are measured and controlled whenever desired to permit better evaluation of formation characteristics from electrical logs. It is a known fact that composition of drilling fluids not only varies from well to well but may alter considerably during the drilling of a well. The suspended particles may consist of both resistant particles and conductive clays. The over-all effect of the suspended resistant solids is to increase the mud resistivity relative to the mud filtrate. The effect of the suspended clays may be conversely to decrease the mud resistivity. The combined effect of the two types of solids may be either to increase or decrease the resistivity of the mud, depending on the relative effective conductivities of the suspended clay and the filtrate.

pH Determination: pH is the relative acidity or alkalinity of a liquid. pH is an important parameter when drilling fluids are considered as it is the deciding factor for optimum control of mud systems, as is the detection and treatment of certain contaminants. A mud made with fresh water and bentonite has a pH of 8 to 9. Contamination will lead to either an increase or decrease in pH, which has to be corrected for proper functioning of a drilling fluid.

5.7 Preparation of Experimental Results

In developing the mud systems, a total of 54 (fifty four) formulations were tested, 36 (thirty six) of which were tested at ambient conditions and 18 (eighteen) of which were tested at high temperatures. For the formulations at ambient conditions, complete rheology check and filtration tests were performed. Densities, resistivities and pH were measured for all the mud formulations. Later, the best optimum concentration (based of filtration control) at different particle sizes of the three materials are tested at two high temperatures i.e., 160^{o}F and 200^{o}F. To understand and interpret the experimental results clearly, the results are divided into 2 stages. The first stage is to determine the amount or concentration of the proposed material (date seeds, grass and grass ash) needed for the formulation to be optimal. The second stage involves the testing of the muds for rheology at the optimum concentration at high temperatures.

5.8 Experimental Results of Date Seeds

These set of experiments are conducted on muds formulated with powdered date seeds. The particle sizes used here are 600 microns, 300 microns and 125 microns in order to incorporate coarse, medium and fine grains respectively in order to study the effect of different particle sizing on the properties of the formulated mud. The results in this section include rheology, filtration, pH and resistivity. The concentrations selected were as follows: 0.25 ppb, 0.5 ppb, 0.75 ppb and 1.0 ppb for 600 microns particle size; 0.25 ppb, 1.0 ppb, 1.5 ppb and 2.0 ppb for 300 microns and 125 microns particle size. It is suitable to mention that the basis of optimization is made exclusively on filtration characteristics i.e., the sample which leads to the least filtrate loss is considered as the most optimum concentration for that particle size.

5.8.1Results of Date Seeds at 600 microns particle size:

Table 5-7 shows the rheological profile of date seeds at 600 micron particle size. Mud system formulated with 1.0 ppb date seeds exhibit the highest PV whereas the highest YP is exhibited at 0.25 ppb and 0.5 ppb. All mud systems show good dial readings with values increasing progressively from 3 rpm dial speed to 600 rpm. Figure 5-13 shows the plot of Dial Readings, Viscosities, Yield Point and Gel Strengths versus Concentration.

Table 5- 7: Rheology of mud formulated with Date Seeds of 600 microns size

Speed, rpm	0 ppb	0.25 ppb	0.5 ppb	0.75 ppb	1.0 ppb
600	20	21	22	22.5	23
300	12	13	13.5	13.5	13.5
200	9	9.5	9.5	10	11
100	6	6.5	7	7.5	7.5
6	1.5	1.5	1.5	1.5	1.5
3	1	1.5	1.5	1.5	1.5

AV	10	10.5	11	11.25	11.5
PV	8	8	8.5	9	9.5
YP	4	5	5	4.5	4
Gel Strength, 10 sec	1	1	1	1	1
Gel Strength, 10 min	10	12	13	14	15

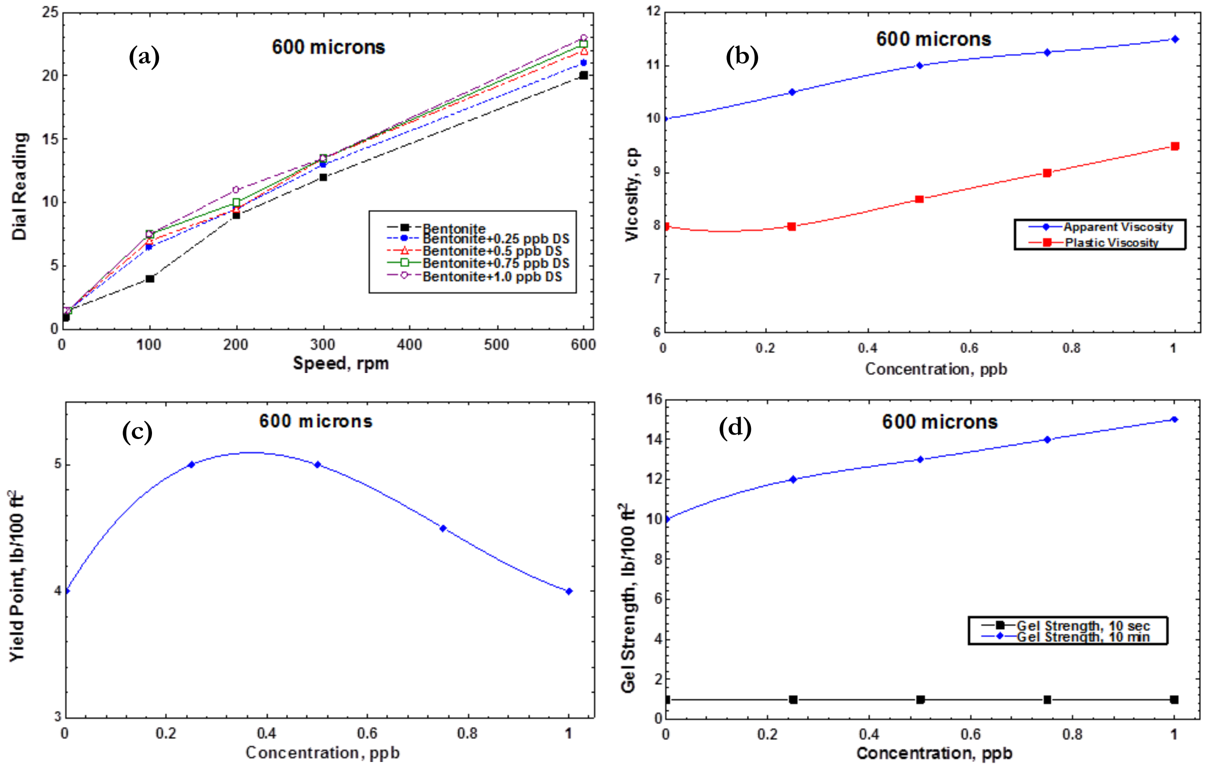

Figure 5- 13: Plot of rheological parameters of mud formulated with Date Seeds of 600 microns size

Figure 5-14 shows the filtration characteristics of the mud formulated using 600 micron size date seeds powder. It is seen in the plot that the filtration characteristics of the mud improve with the addition of date seeds obtaining an optimum value of 13 ml of filtrate loss (which is 13% reduction in water loss) at 0.75 ppb.

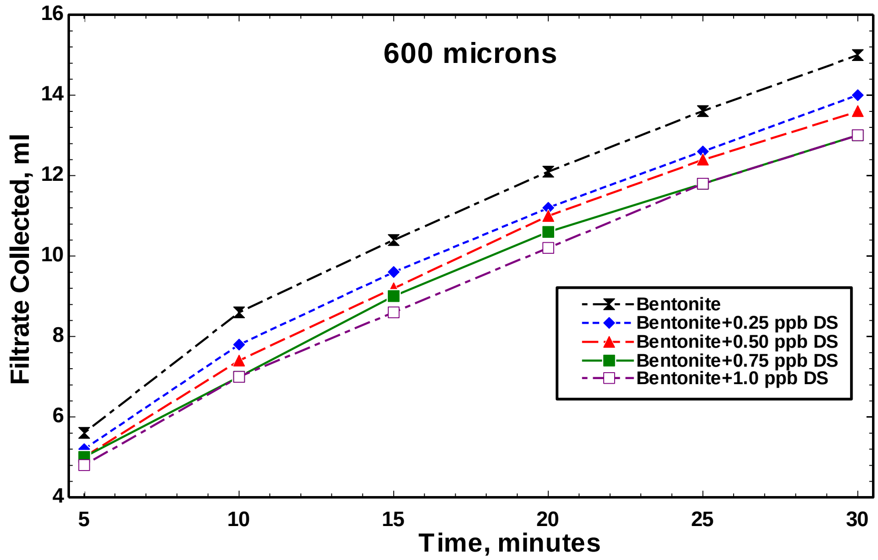

Figure 5- 14: Filtration Characteristics of the mud formulated with Date Seeds of 300 microns size

5.8.2 Results of Date Seeds at 300 microns particle size:

Table 5-8 shows the rheological profile of date seeds at 300 micron particle size. Mud system containing 0.25 ppb, 1.5 ppb and 2.0 ppb exhibit the highest PV whereas the YP is almost the same at all concentrations. All mud systems show good dial readings with values increasing progressively from 3 rpm dial speed to 600 rpm. Figure 5-15 shows the plot of Dial Readings, Viscosities, Yield Point and Gel Strengths versus Concentration.

Table 5- 8: Rheology of mud formulated with Date Seeds of 300 microns size

Speed, rpm	**0 ppb**	**0.25 ppb**	**1.0 ppb**	**1.5 ppb**	**2.0 ppb**
600	20	22	22	23	23
300	12	12.5	13	13.5	13.5
200	9	9.5	10	11	11.5
100	6	6.5	7	7.5	8
6	1.5	1.5	1.5	1.5	1.5
3	1	1.5	1.5	1.5	1.5
AV	10	11	11	11.5	11.5
PV	8	9.5	9	9.5	9.5
YP	4	3	4	4	4
Gel Strength, 10 sec	1	1	1	1	2
Gel Strength, 10 min	10	12	13	14	16

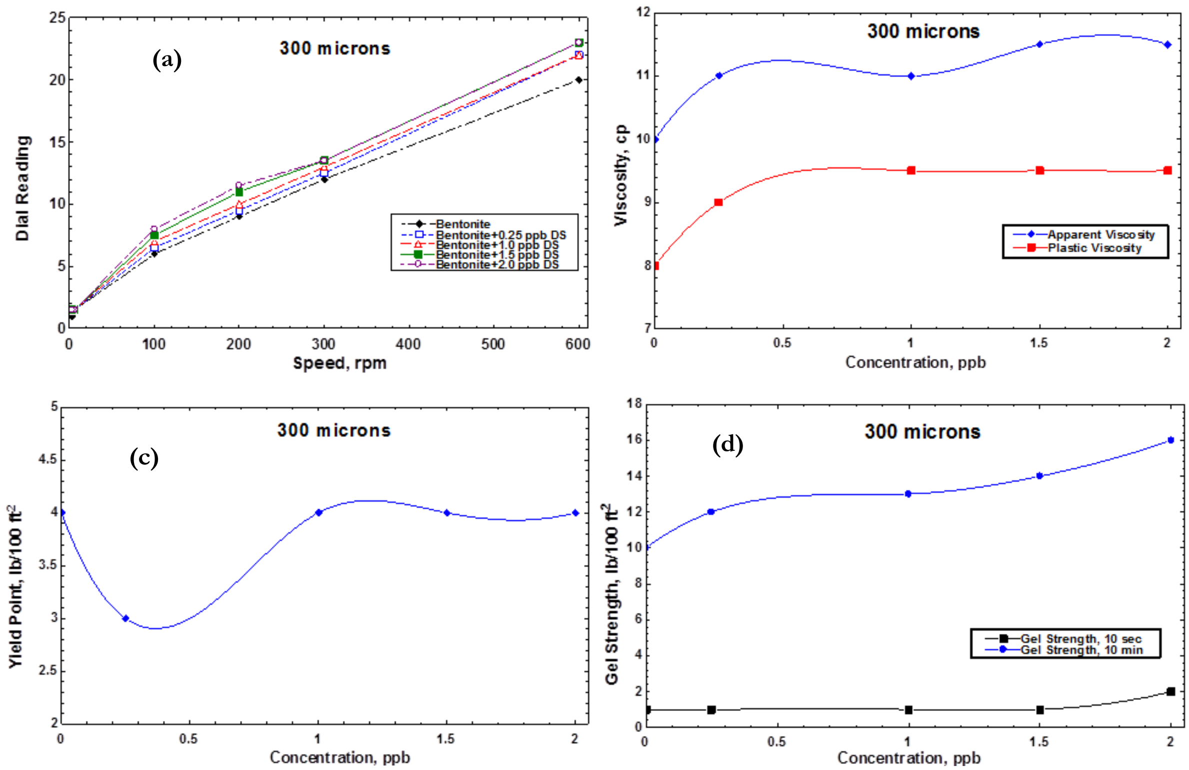

Figure 5- 15: Plot of rheological parameters of mud formulated with Date Seeds of 300 microns size

Figure 5-16 shows the filtration characteristics of the mud formulated using 300 micron size date seeds powder. It is seen in the plot that the filtration characteristics of the mud improve with the addition of date seeds obtaining value of 12 ml filtrate loss (which is 20% reduction in water loss) at an optimum concentration of 1.5 ppb.

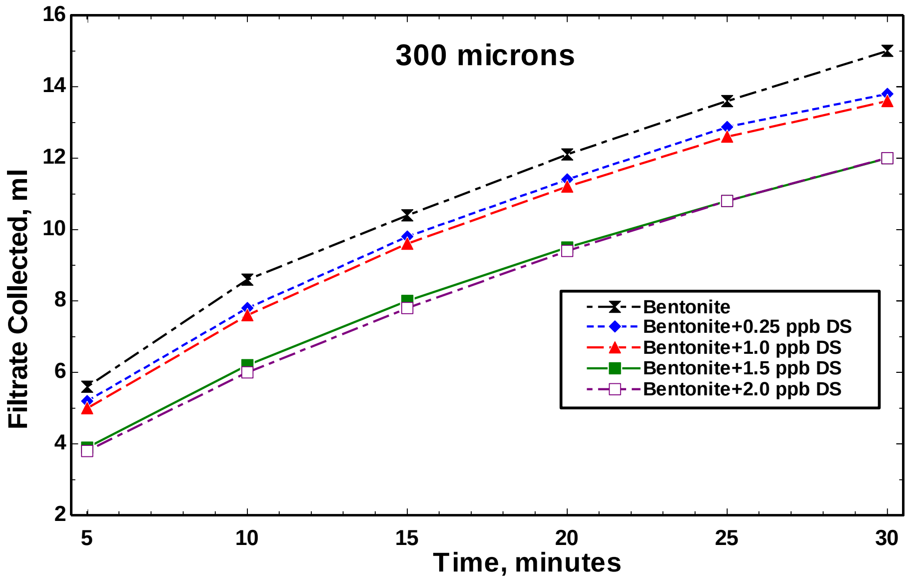

Figure 5- 16: Filtration Characteristics of the mud formulated with Date Seeds of 300 microns size

5.8.3 Results of Date Seeds at 125 microns particle size:

Table 5-9 shows the rheological profile of date seeds at 125 micron particle size. Mud system containing 2.0 ppb exhibit the highest PV whereas the YP is almost constant at all concentrations. All mud systems show good dial readings with values increasing progressively from 3 rpm dial speed to 600 rpm. Figure 5-17 shows the plot of Dial Readings, Viscosities, Yield Point and Gel Strengths versus Concentration.

Table 5- 9: Rheology of mud formulated with Date Seeds of 125 microns size

Speed, rpm	0 ppb	0.25 ppb	1.0 ppb	1.5 ppb	2.0 ppb
600	20	22	22	23	23.5
300	12	13	13	13.5	13.5
200	9	9.5	10	10.5	10.5
100	6	7	7	7	7
6	1.5	1.5	1.5	1.5	1.5
3	1	1.5	1.5	1.5	1.5
AV	10	11	11	11.5	11.75
PV	8	9	9	9.5	10
YP	4	4	4	4	3.5
Gel Strength, 10 sec	1	1	1	2	2
Gel Strength, 10 min	10	12	12	13	15

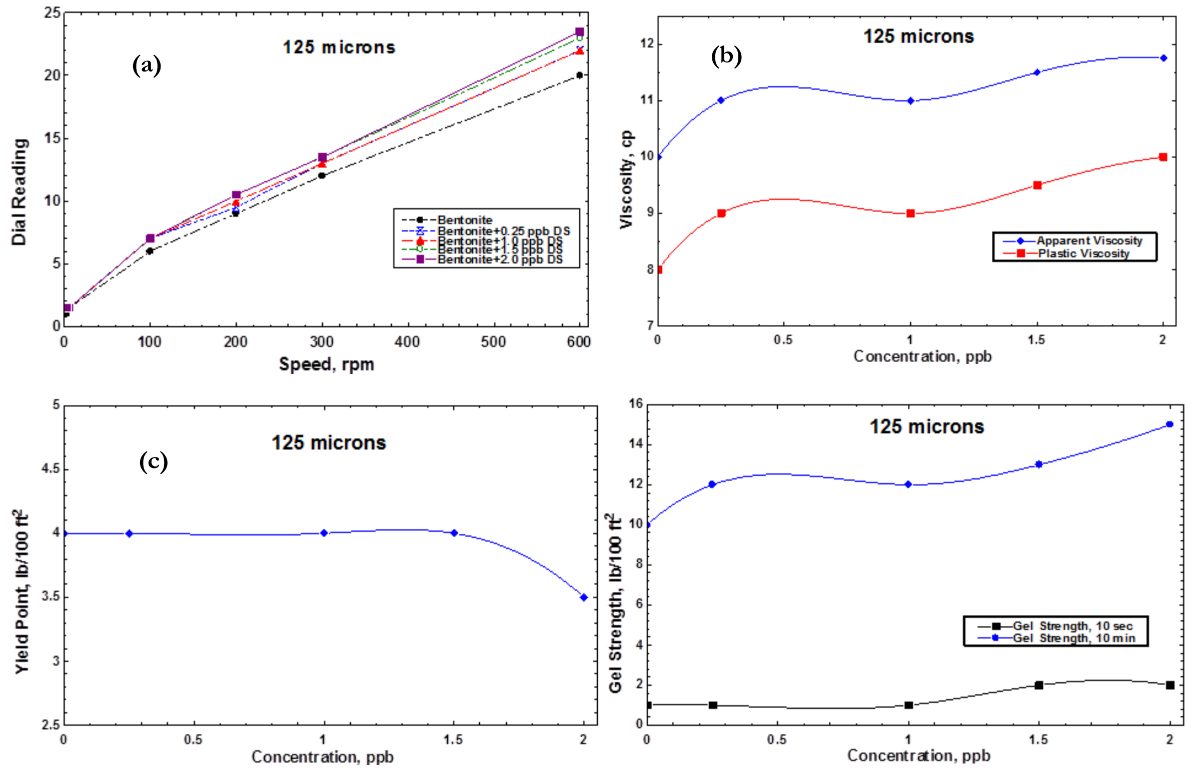

Figure 5- 17: Plot of rheological parameters for muds formulated with Date Seeds of 125 microns size

Figure 5-18 shows the filtration characteristics of the mud formulated using 125 micron size date seeds powder. It is seen in the plot that the filtration characteristics of the mud improve with the addition of date seeds obtaining value of 13.2 ml filtrate loss (which is 12% reduction in water loss) at an optimum concentration of 2.0 ppb.

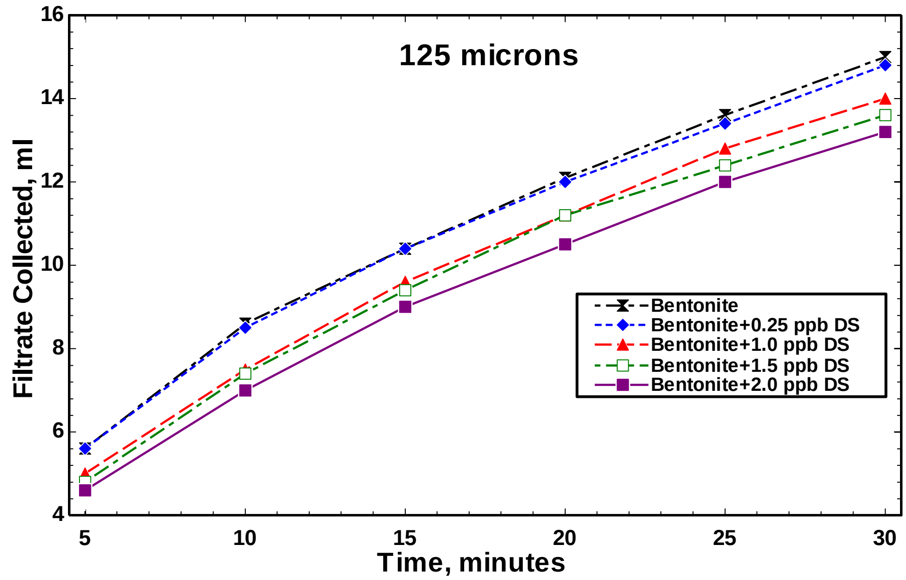

Figure 5- 18: Filtration Characteristics of the mud formulated with Date Seeds of 125 microns size

Effect of Date Seeds on the pH of the mud:

Figure 5-19 shows the effect of adding date seeds on the pH. The addition of date seeds lower the pH of the mud as well as the mud filtrate as evident from the below figure. This property of date seeds is good to treat contaminated muds whose pH has been raised above the unacceptable level.

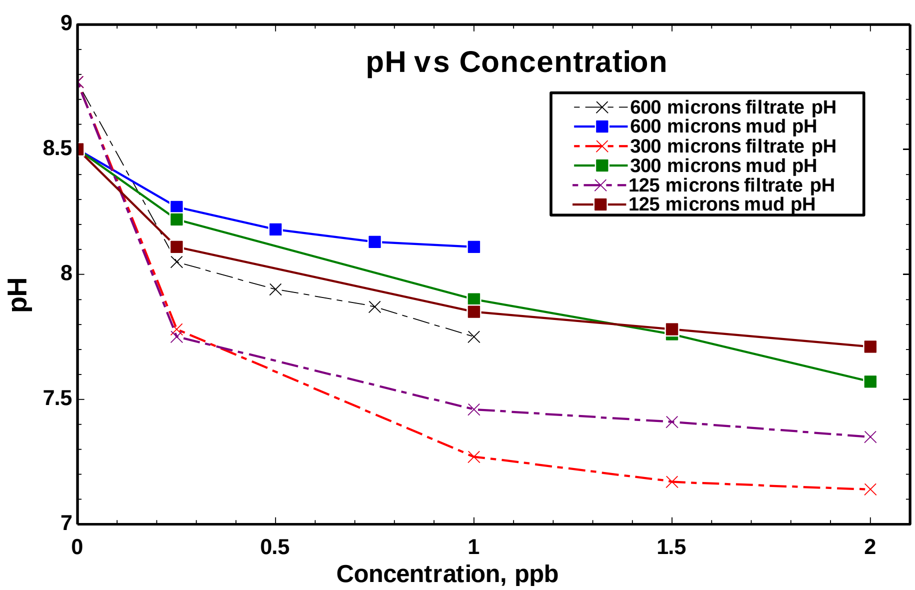

Figure 5- 19: Effect on pH with addition of Date Seeds

Effect of Date Seeds on the Resistivity of the mud:

Addition of date seeds to the mud lowers the resistivity of the mud cake as well as the mud filtrate as evident form the below plot (Fig. 5-20).

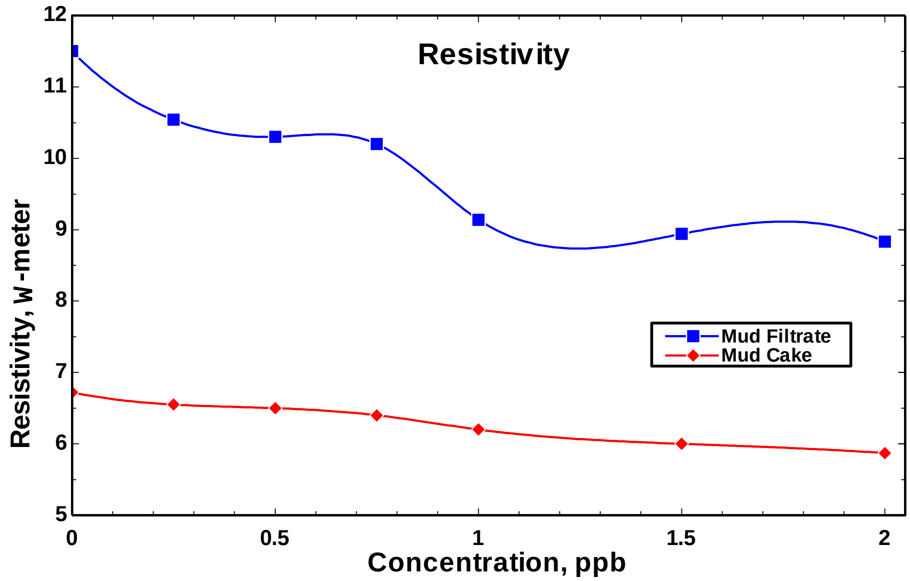

Figure 5- 20: Effect on Resistivities after the addition of Date Seeds

5.9 Experimental Results of Grass Ash

These set of experiments are conducted on muds formulated with grass ash. The particle sizes used here are 300 microns, 90 microns and 26 microns in order to encompass coarse, medium and fine grains respectively in order to study the effect of different particle sizing on the properties of the mud. The results in this section include rheology, filtration, pH and resistivity. The concentrations used here are 0.25 ppb, 0.5 ppb, 0.75 ppb and 1.0 ppb of grass ash at the three mentioned particle sizes. It is apt to mention that the basis of optimization made is solely on filtration characteristics i.e., the sample which leads to the least filtration loss is considered as the most optimum concentration for that particle size.

5.9.1 Results of Grass Ash at 300 microns particle size:

Table 5-10 shows the rheological profile of grass ash at 300 micron particle size. Mud system containing 1.0 ppb exhibit the highest PV and the YP gradually increases at all concentrations. All mud systems show good dial readings with values increasing progressively from 3 rpm dial speed to 600 rpm. Figure 5-21 shows the plot of Dial Readings, Viscosities, Yield Point and Gel Strengths versus Concentration.

Table 5- 10: Rheology of mud formulated with Grass Ash of 300 microns size

Speed, rpm	**0 ppb**	**0.25 ppb**	**0.5 ppb**	**0.75 ppb**	**1.0 ppb**
600	20	21	22	24	25.5
300	12	13.5	14.5	16	17
200	9	10.5	11.5	12.5	15
100	6	7.5	8.5	10	11.5
6	1.5	2.5	3.5	5.5	6.5
3	1	2	3	5	6
AV	10	10.5	11	12	12.75
PV	8	7.5	7.5	8	8.5
YP	4	6	7	8	8.5
Gel Strength, 10 sec	1	4	6	8	9
Gel Strength, 10 min	10	18	19	24	28

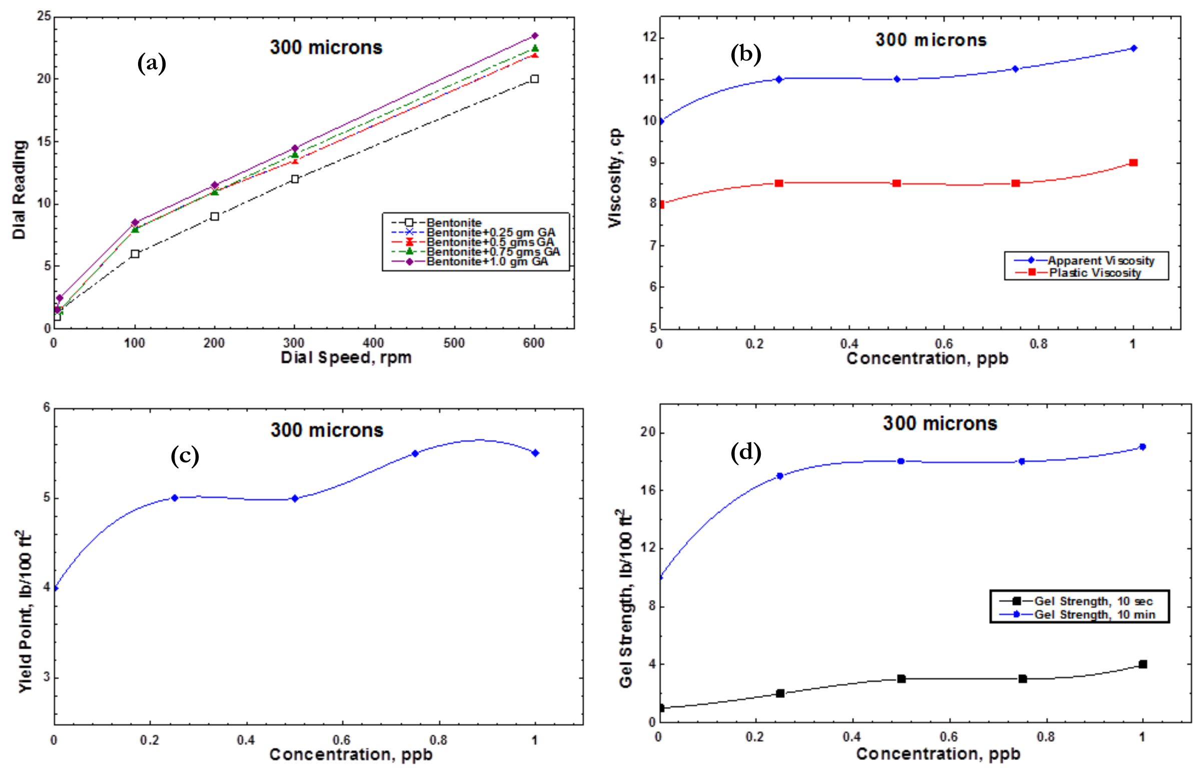

Figure 5- 21: Plot of rheological parameters for muds formulated with Grass Ash of 300 microns size

Figure 5-22 shows the filtration characteristics of the mud formulated using 300 micron size grass ash powder. It is seen in the plot that the filtration characteristics of the mud improve with the addition of grass ash obtaining value of 11.9 ml filtrate loss (which is 20.67% reduction in water loss) at an optimum concentration of 1.0 ppb.

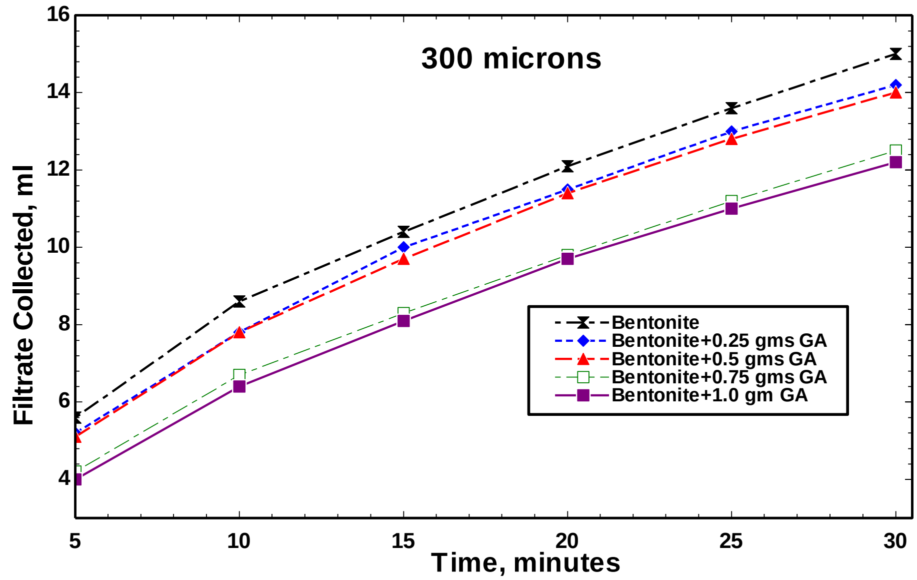

Figure 5- 22: Filtration Characteristics of mud formulated with Grass Ash of 300 microns size

5.9.2 Results of Grass Ash at 90 microns particle size:

Table 5-11 shows the rheological profile of grass ash at 90 micron particle size. Mud system containing 1.0 ppb exhibit the highest PV and the YP gradually increases at all concentrations. All mud systems show good dial readings with values increasing progressively from 3 rpm dial speed to 600 rpm. Figure 5-23 shows the plot of Dial Readings, Viscosities, Yield Point and Gel Strengths versus Concentration.

Table 5- 11: Rheology of mud formulated with Grass Ash of 90 microns size

Speed, rpm	0 ppb	0.25 ppb	0.5 ppb	0.75 ppb	1.0 ppb
600	20	21	22	23	25
300	12	12.5	14.5	15	16.5
200	9	10.5	12.5	13	14
100	6	7.5	9	9.5	11
6	1.5	1.5	3	5	5.5
3	1	1.5	2.5	4.5	4.5
AV	10	10.5	11	11.5	12.5
PV	8	8.5	7.5	8	8.5
YP	4	4	7	7	8
Gel Strength, 10 sec	1	3	6	8	9
Gel Strength, 10 min	10	17	20	21	24

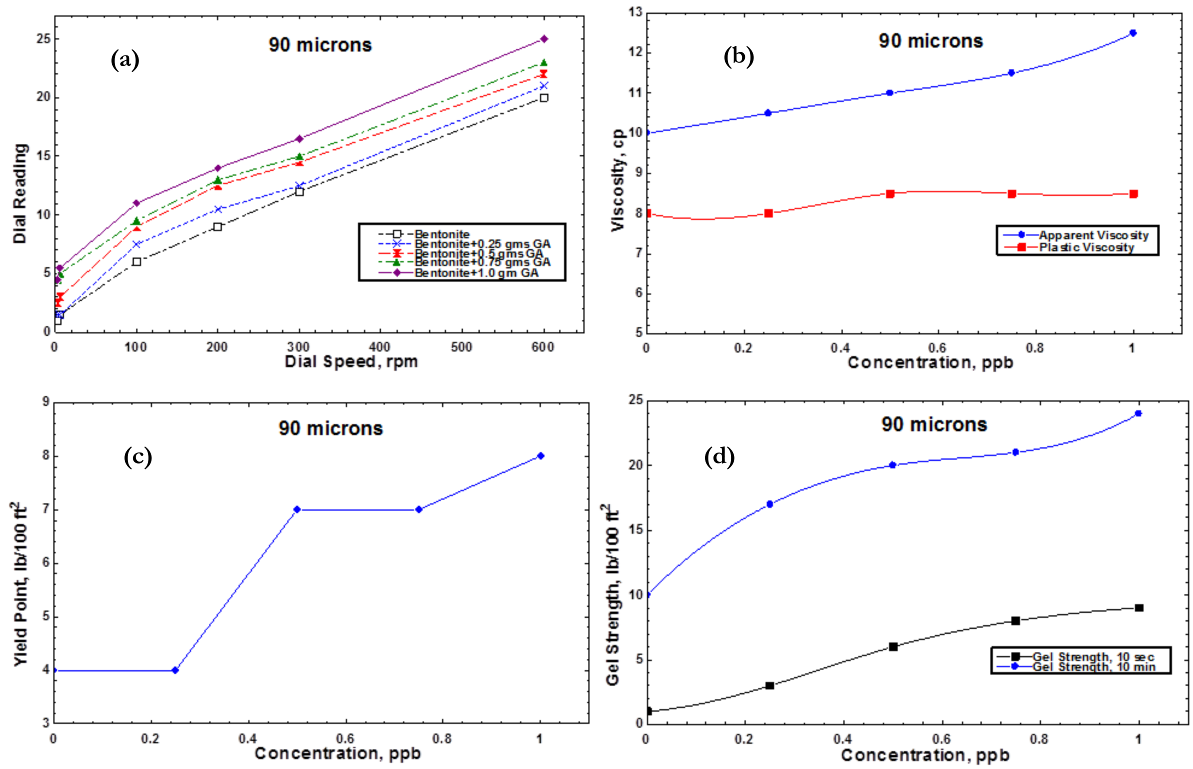

Figure 5- 23: Plot of rheological parameters for muds formulated with Grass Ash of 90 microns size

Figure 5-24 shows the filtration characteristics of the mud formulated using 90 micron size grass ash powder. It is seen in the plot that the filtration characteristics of the mud improve with the addition of grass ash obtaining value of 11.8 ml filtrate loss (which is 21.33% reduction in water loss) at an optimum concentration of 1.0 ppb

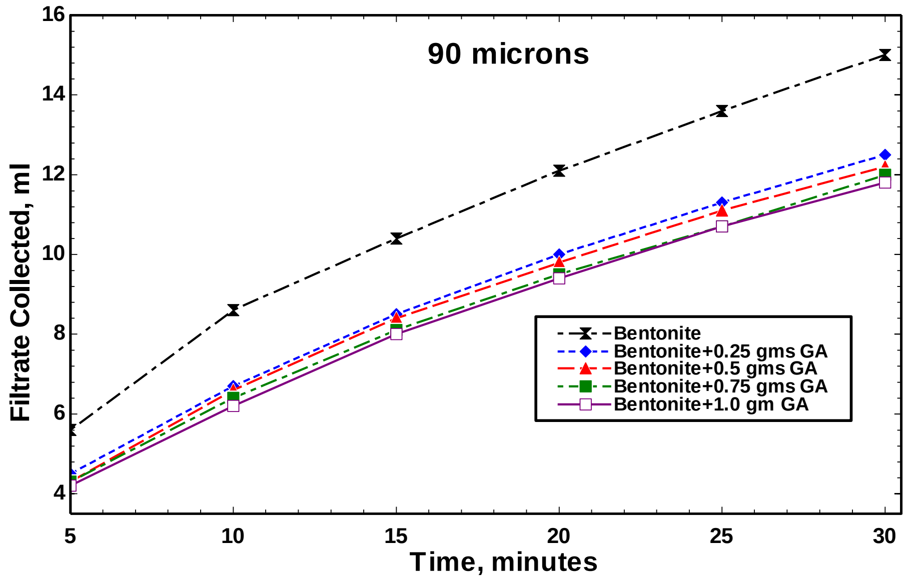

Figure 5- 24: Filtration Characteristics of mud formulated with Grass Ash of 90 microns size

<u>5.9.3 Results of Grass Ash at 26 microns particle size:</u>

Table 5-12 shows the rheological profile of grass ash at 26 micron particle size. Mud system containing 1.0 ppb exhibit the highest PV and the YP progressively increases at all concentrations. All mud systems show good dial readings with values increasing gradually from 3 rpm dial speed to 600 rpm. Figure 5-25 shows the plot of Viscosities, Yield Point and Gel Strengths versus Concentration.

Table 5- 12: Rheology of mud formulated with Grass Ash of 26 microns size

Speed, rpm	**0 ppb**	**0.25 ppb**	**0.5 ppb**	**0.75 ppb**	**1.0 ppb**
600	20	21	22	24	25.5
300	12	13.5	14.5	16	17
200	9	10.5	11.5	12.5	15
100	6	7.5	8.5	10	11.5
6	1.5	2.5	3.5	5.5	6.5
3	1	2	3	5	6
AV	10	10.5	11	12	12.75
PV	8	7.5	7.5	8	8.5
YP	4	6	7	8	8.5
Gel Strength, 10 sec	1	4	6	8	9
Gel Strength, 10 min	10	18	19	24	28

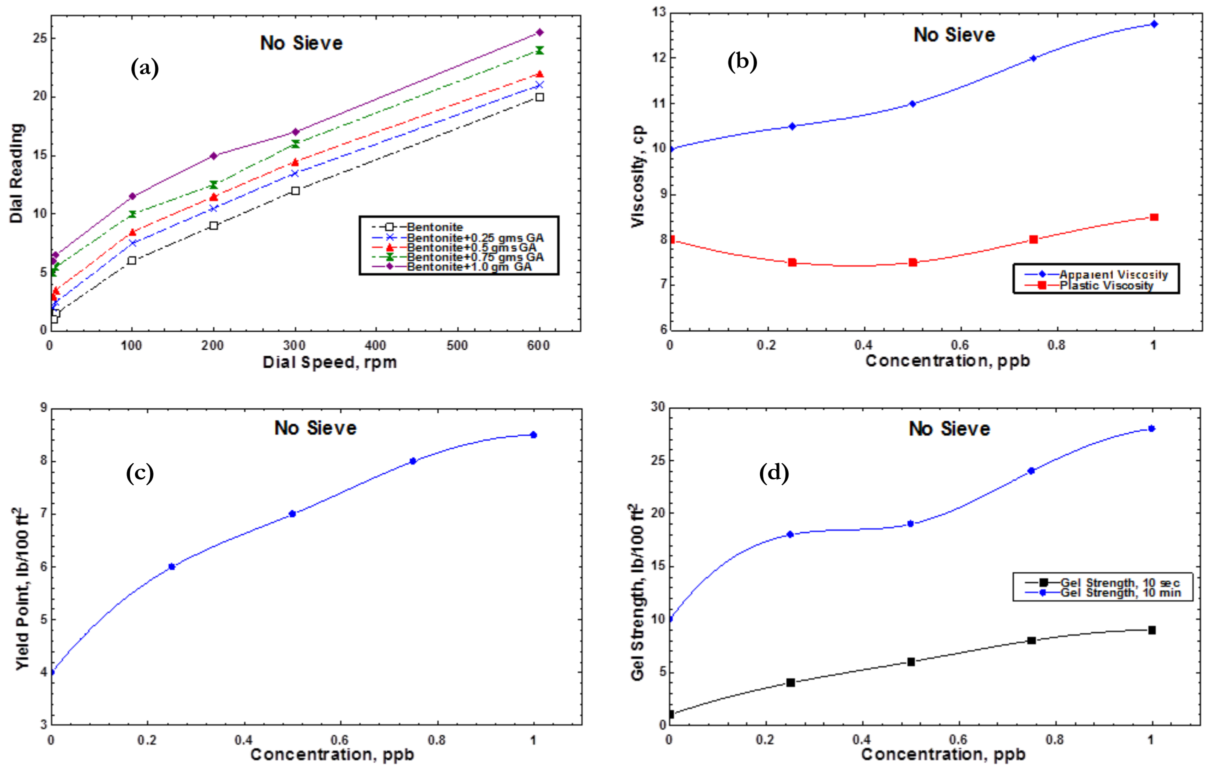

Figure 5- 25: Plot of rheological parameters for muds formulated with Grass Ash of 26 microns size

Figure 5-26 shows the filtration characteristics of the mud formulated using 90 micron size grass ash powder. It is seen in the plot that the filtration characteristics of the mud improve with the addition of grass ash obtaining value of 11.9 ml filtrate loss (which is 20.67% reduction in water loss) at an optimum concentration of 1.0 ppb

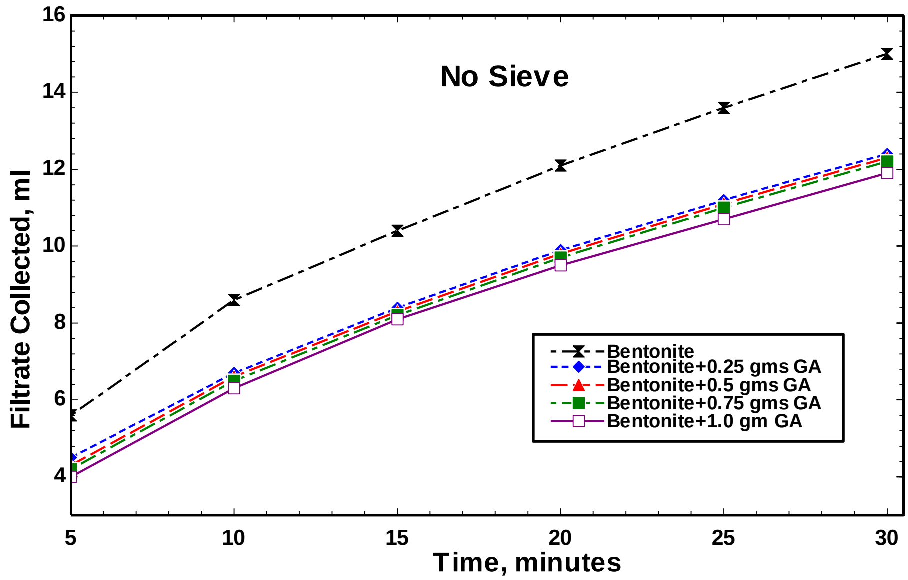

Figure 5- 26: Filtration Characteristics of mud formulated with Grass Ash of 26 microns size

Effect of Grass Ash on the pH of the mud:

Figure 5-27 shows the effect of adding grass ash on the pH. The addition of grass ash increases the pH of the mud as well as the mud filtrate as evident from the below figure. This property of grass ash is good to treat contaminated muds whose pH has lowered below the optimum level.

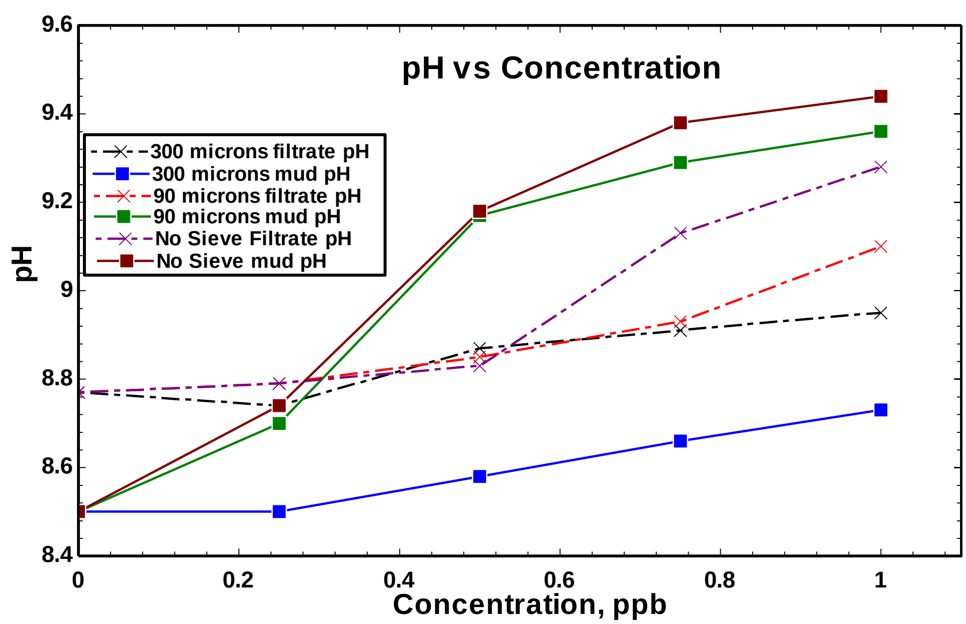

Figure 5- 27: Effect on pH with addition of Grass Ash

Effect of Grass Ash on the Resistivity of the mud:

Addition of grass ash to the mud lowers the resistivity of the mud cake as well as the mud filtrate as evident form the plot (Fig. 5-28) below.

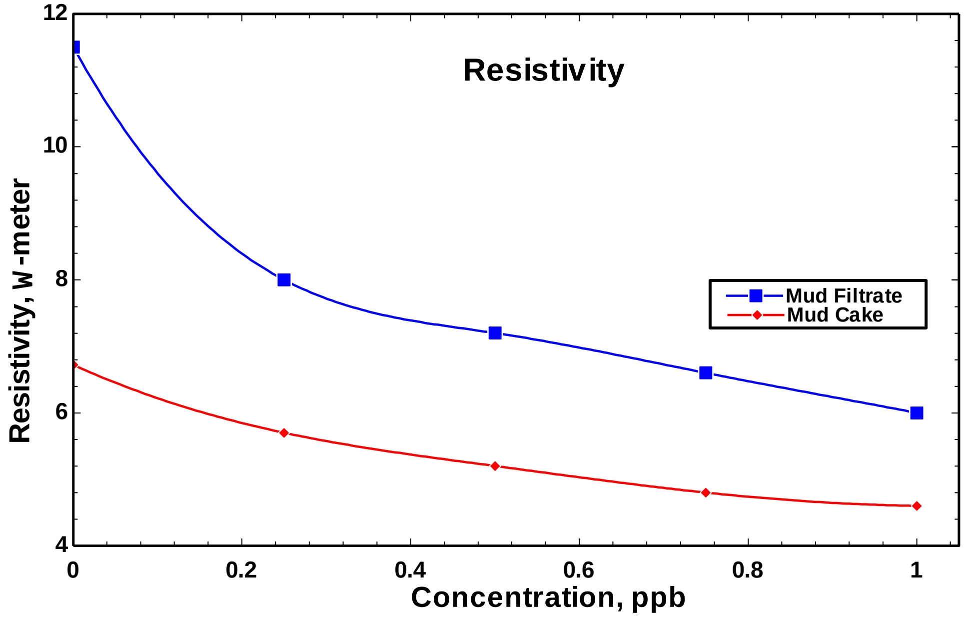

Figure 5- 28: Effect on Resistivities after the addition of Grass Ash

5.10 Experimental Results of Grass

These set of experiments are conducted on muds formulated with powdered grass. The particle sizes used here are 300 microns, 90 microns and 35 microns in order to contain coarse, medium and fine grains respectively in order to study the effect of different particle sizing on the properties of the mud. The results in this section include rheology, filtration, pH and resistivity. The concentrations used here are 0.25 ppb, 0.5 ppb, 0.75 ppb and 1.0 ppb of powdered grass at the three mentioned particle sizes. It is apt to mention that the basis of optimization made is specially on filtration characteristics i.e., the sample which leads to the least filtration loss is considered as the most optimum concentration for that particle size.

5.10.1 Results of Grass at 300 microns particle size:

Table 5-13 shows the rheological profile of grass ash at 300 micron particle size. The PV is almost constant at all concentrations whereas the YP gradually increases at higher concentrations. All mud systems show good dial readings with values increasing progressively from 3 rpm dial speed to 600 rpm. Figure 5-29 shows the plot of Viscosities, Yield Point and Gel Strengths versus Concentration.

Table 5- 13: Rheology of mud formulated with Grass of 300 microns size

Speed, rpm	0 ppb	0.25 ppb	0.5 ppb	0.75 ppb	1.0 ppb
600	20	21	21	21.5	22
300	12	12.5	12.5	13	13.5
200	9	9.5	10	10.5	11
100	6	6.5	7	7	7
6	1.5	1.5	2	2.5	2.5
3	1	1.5	1.5	2	2
AV	10	10.5	10.5	10.75	11
PV	8	8.5	8.5	8.5	8.5
YP	4	4	4	4.5	5
Gel Strength, 10 sec	1	1.5	2	2.5	3
Gel Strength, 10 min	10	15	15	15	16

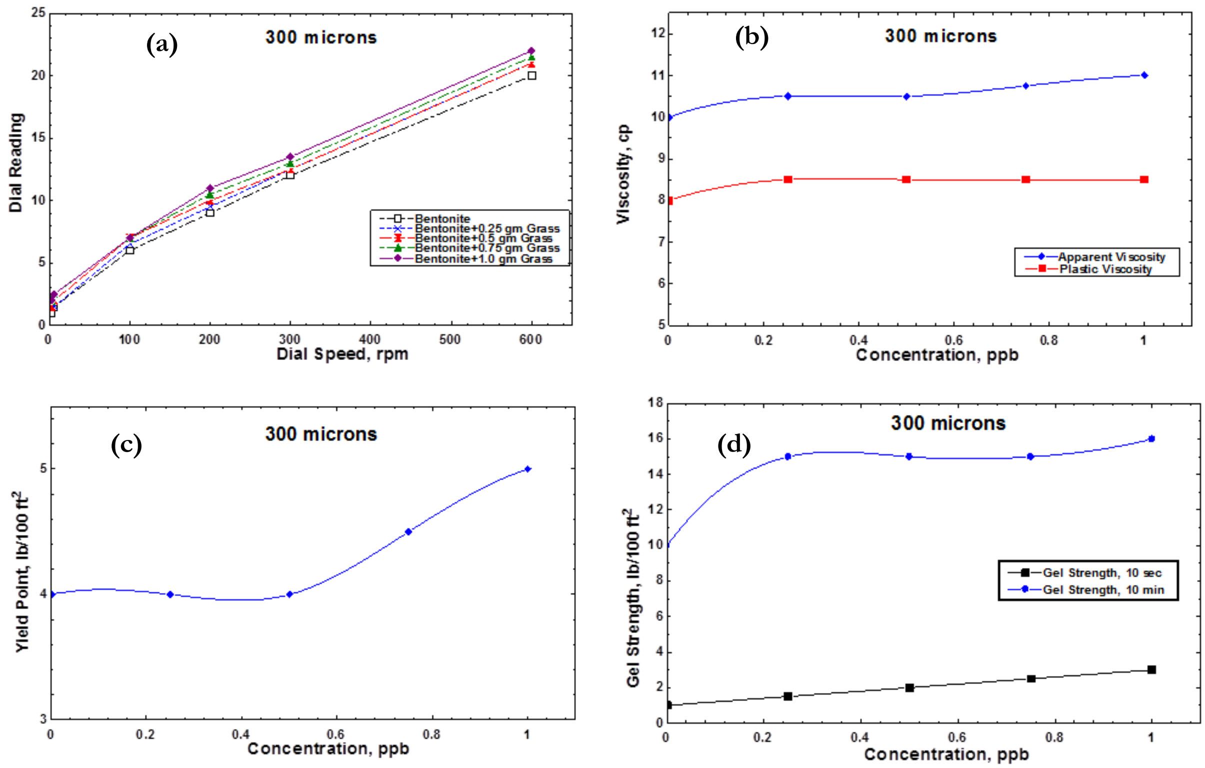

Figure 5- 29: Plot of rheological parameters for muds formulated with Grass of 300 microns size

Figure 5-30 shows the filtration characteristics of the mud formulated using 300 micron size grass ash powder. It is seen in the plot that the filtration characteristics of the mud improve with the addition of grass obtaining value of 11.3 ml filtrate loss (which is almost 25% reduction in water loss) at an optimum concentration of 1.0 ppb

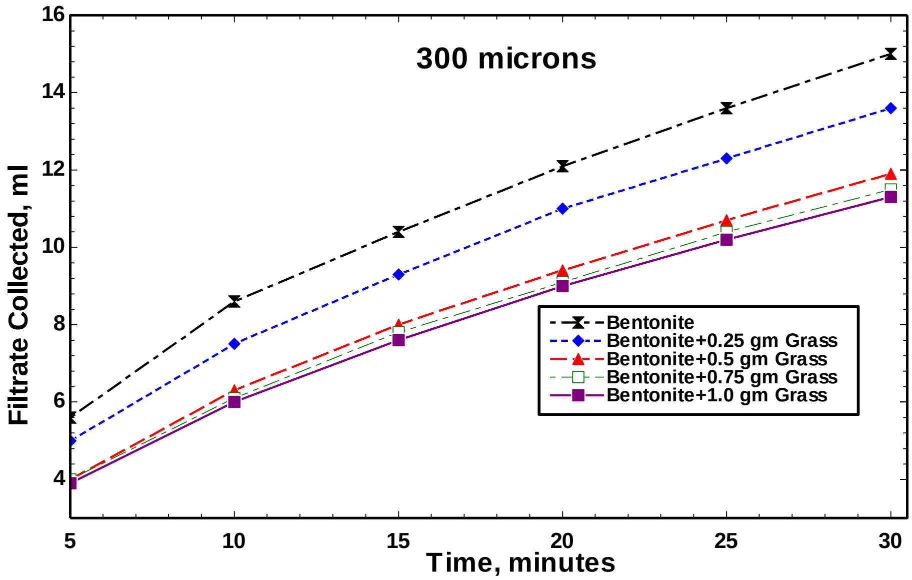

Figure 5- 30: Filtration Characteristics of mud formulated with Grass of 300 microns size

5.10.2 Results of Grass at 90 microns particle size:

Table 5-14 shows the rheological profile of grass at 90 micron particle size. The PV is almost constant at lower concentrations and increases at high concentrations of 0.75 ppb and 1.0 ppb whereas the YP gradually increases at higher concentrations. The gel strengths at both 10 seconds and 10 minutes also increase gradually. All mud systems show good dial readings with values increasing progressively from 3 rpm dial speed to 600 rpm. Figure 5-31 shows the plot of Viscosities, Yield Point and Gel Strengths versus Concentration.

Table 5- 14: Rheology of mud formulated with Grass of 90 microns size

Speed, rpm	0 ppb	0.25 ppb	0.5 ppb	0.75 ppb	1.0 ppb
600	20	20.5	20.5	21	21.5
300	12	12.5	12.5	12.5	13
200	9	10	10	10	10
100	6	6.5	6.5	7	7.5
6	1.5	1.5	1.5	2	2
3	1	1.5	1.5	1.5	1.5
AV	10	10.25	10.25	10.5	10.75
PV	8	8	8	8.5	8.5
YP	4	4.5	4.5	4	4.5
Gel Strength, 10 sec	1	1.5	2	3	3
Gel Strength, 10 min	10	14	14	15	16

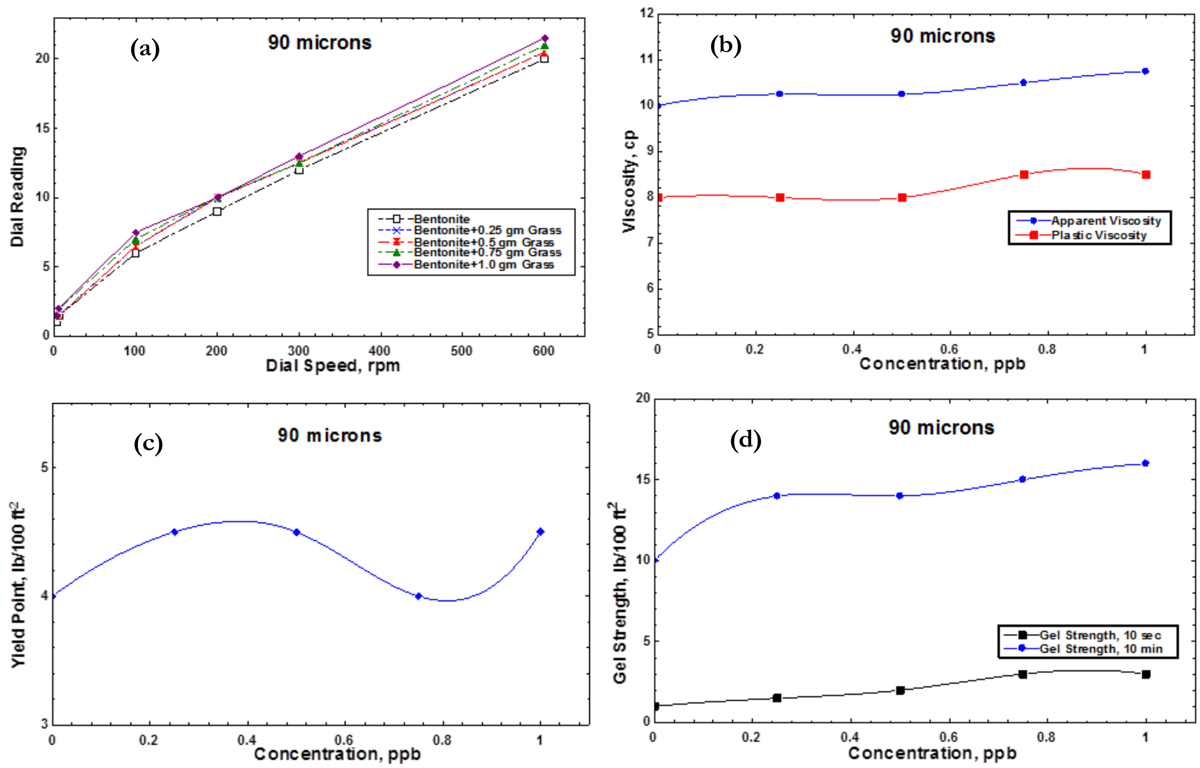

Figure 5- 31: Plot of rheological parameters for muds formulated with Grass of 90 microns size

Figure 5-32 shows the filtration characteristics of the mud formulated using 90 micron size grass powder. It is seen in the plot that the filtration characteristics of the mud improve with the addition of grass obtaining value of 11.5 ml filtrate loss (which is almost 23% reduction in water loss) at an optimum concentration of 1.0 ppb.

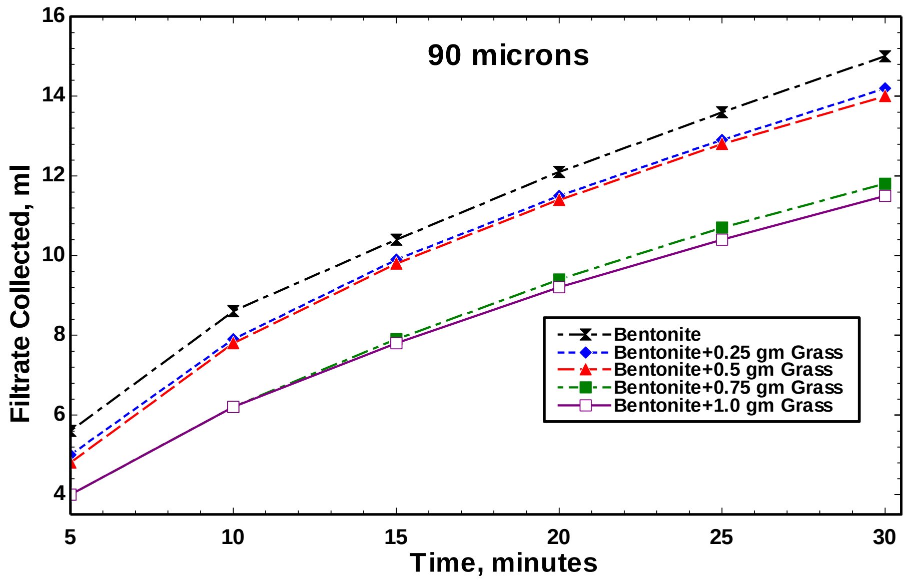

Figure 5- 32: Filtration Characteristics of mud formulated with Grass of 90 microns size

5.10.3 Results of Grass at 35 microns particle size:

Table 5-15 shows the rheological profile of grass at 35 micron particle size. The PV increases at higher concentrations whereas the YP gradually increases as the concentration is increased. The gel strengths at both 10 seconds and 10 minutes also increase steadily. All mud systems show good dial readings with values increasing progressively from 3 rpm dial speed to 600 rpm. Figure 5-33 shows the plot of Viscosities, Yield Point and Gel Strengths versus Concentration.

Table 5- 15: Rheology of mud formulated with Grass of 35 microns size

Speed, rpm	**0 ppb**	**0.25 ppb**	**0.5 ppb**	**0.75 ppb**	**1.0 ppb**
600	20	20.5	21	21.5	22.5
300	12	12	12.5	13	13.5
200	9	9	9.5	9.5	10
100	6	6.5	7	7.5	7.5
6	1.5	1.5	1.5	2	2
3	1	1.5	1.5	1.5	1.5
AV	10	10.25	10.5	10.75	11.25
PV	8	8.5	8.5	8.5	9
YP	4	3.5	4	4.5	4.5
Gel Strength, 10 sec	1	1	1	2	2.5
Gel Strength, 10 min	10	14	15	16	17

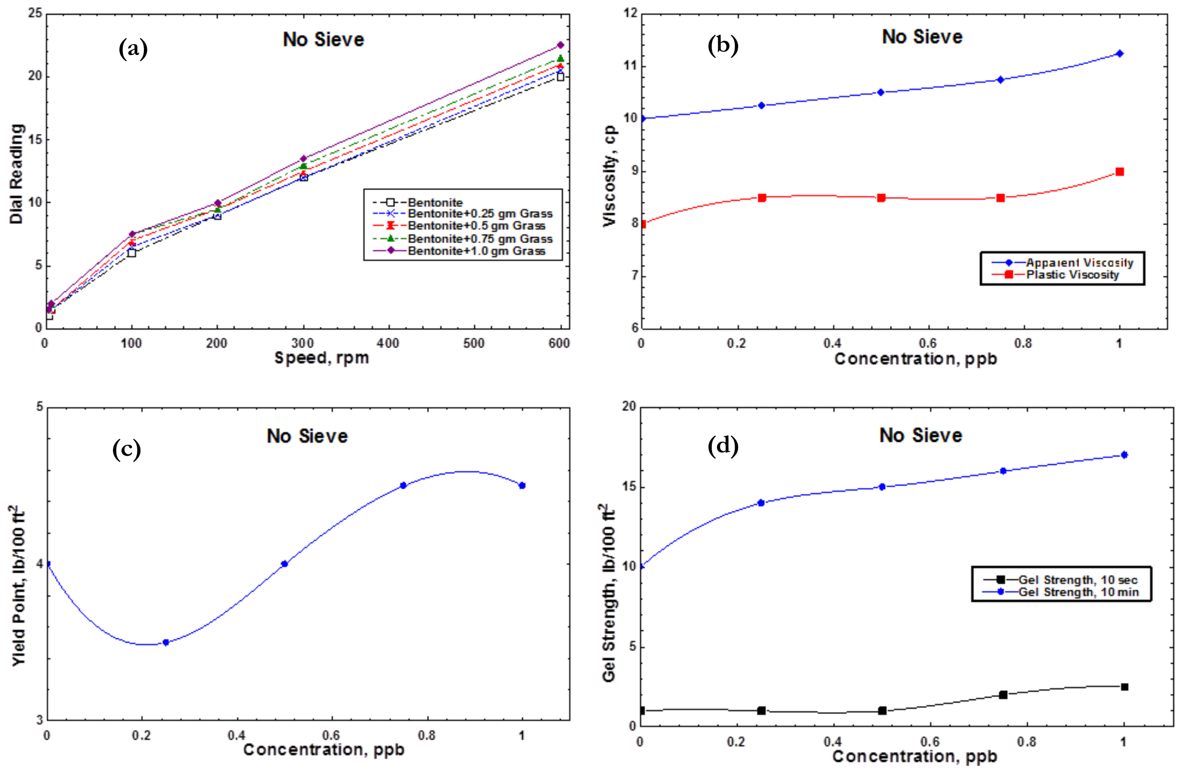

Figure 5- 33: Plot of rheological parameters for muds formulated with Grass of 35 microns size

Figure 5-34 shows the filtration characteristics of the mud formulated using 35 micron size grass powder. It is seen in the plot that the filtration characteristics of the mud improve with the addition of grass obtaining value of 12.1 ml filtrate loss (which is almost 19% reduction in water loss) at an optimum concentration of 1.0 ppb.

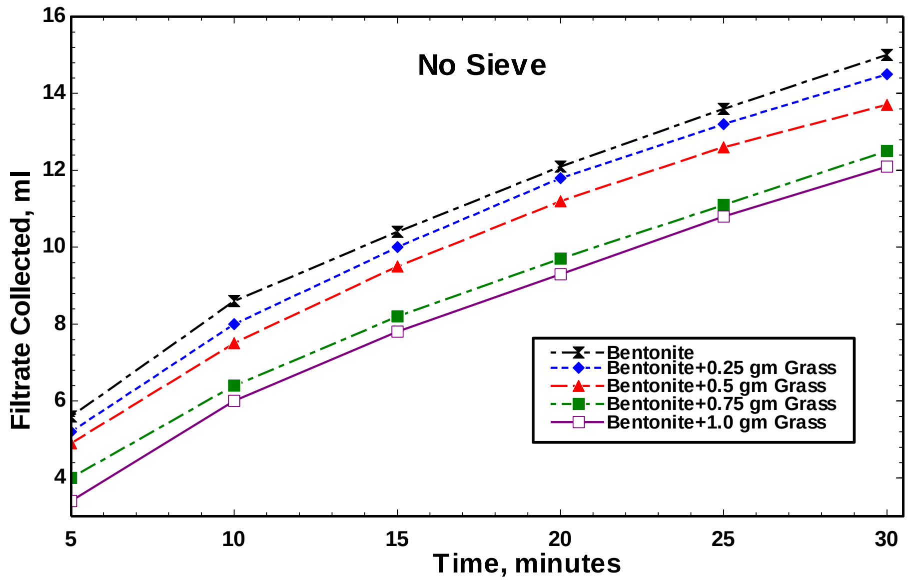

Figure 5- 34: Filtration Characteristics of mud formulated with Grass of 35 microns size

Effect of Grass on the pH of the mud:

Figure 5-35 shows the effect of adding grass on the pH. The addition of grass decreases the pH of the mud as well as the mud filtrate as evident from the below figure. This property of grass is good to treat contaminated muds whose pH has been raised above the optimum level.

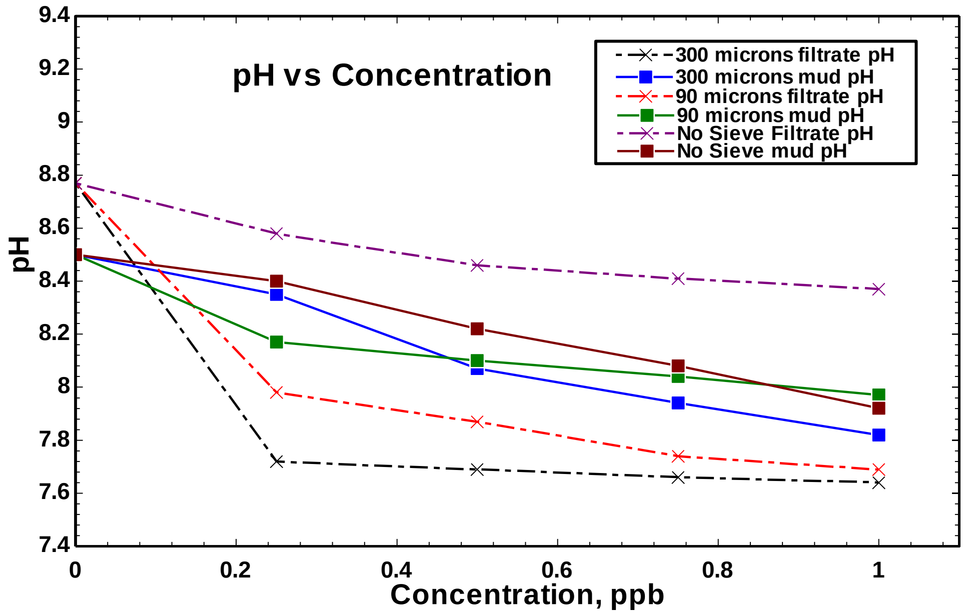

Figure 5- 35: Effect on pH with addition of Grass

Effect of Grass on the Resistivity of the mud:

Addition of grass to the mud lowers the resistivity of the mud cake as well as the mud filtrate as seen from the plot (Fig. 5-36) below.

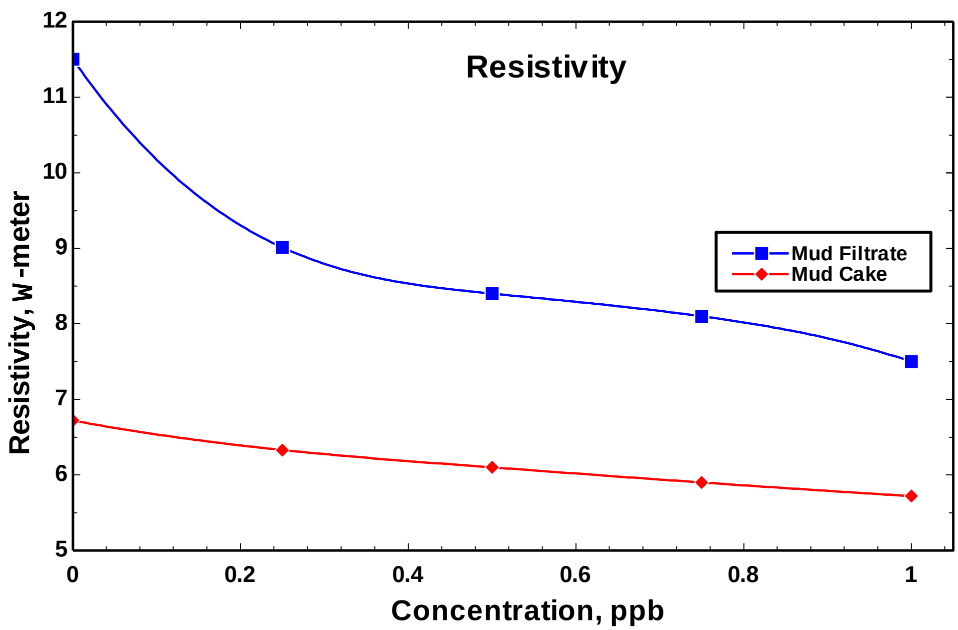

Figure 5- 36: Effect on Resistivities after the addition of Grass

5.11 Comparison of the three proposed additives muds with Modified Starch mud

To validate the results of rheology and filtration, a comparison is made between the muds prepared using powdered date seeds, grass ash and grass with a mud prepared using a commercial additive viz. in our case modified starch. Table 5-16 shows the rheological parameters of the muds prepared using modified starch. The same can be seen in Figure 5-37 which illustrates the consistency plot of modified starch muds.

Table 5- 16: Rheology of muds formulated using Modified Starch

Speed, rpm	0 ppb	0.25 ppb	0.5 ppb	0.75 ppb	1.0 ppb
600	20	23	24	24	26
300	13	15	16	16	17
200	11	12.5	12.5	12.5	13
100	7	8	8	8	9
6	2	2	2	2	2.5
3	1.5	1.5	1.5	1.5	2
AV	10	11.5	12	12	13
PV	7	8	8	8	9
YP	6	7	8	8	8
Gel Strength, 10 sec	2	3	3	3	4
Gel Strength, 10 min	8	9	11	11	12

It can be seen from Figure 5-37 that modified starch muds bear close resemblance to muds formulated with powdered date seeds, grass ash and grass based on the rheology with the fact that at all concentrations, the muds obey Bingham Plastic model. However, the modified starch muds exhibit better readings when compared to their counterparts.

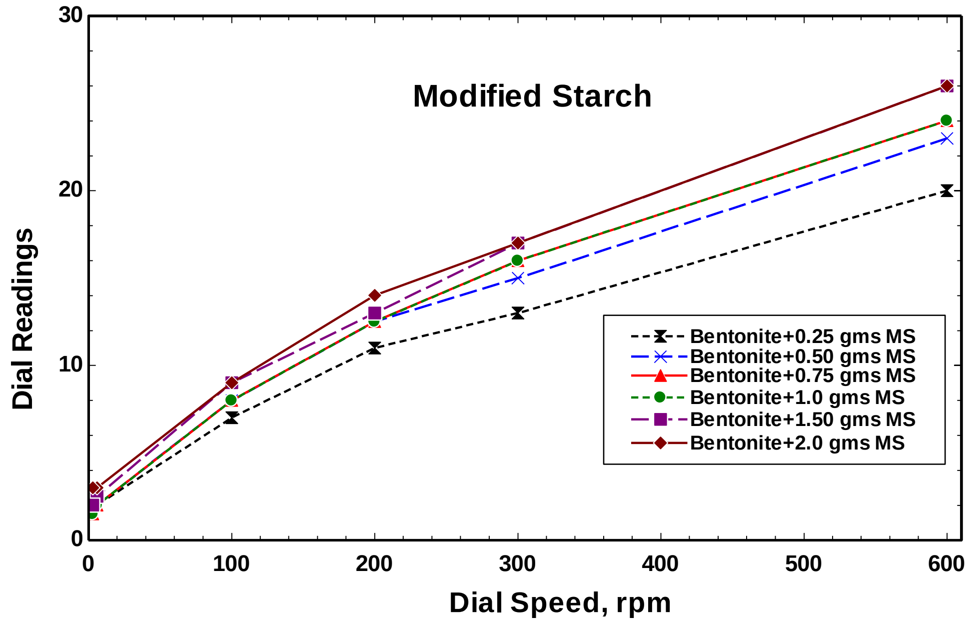

Figure 5- 37: Consistency curves of Modified Starch muds at various concentrations

It is thus prompted that the rheological properties of the proposed materials be compared with that of modified starch. The following sections depict this comparison.

5.11.1 Comparison of Date Seeds muds with Modified Starch muds:

An evaluation is carried out on the viscosities, yield point and filtration characteristics of date seeds muds and modified starch muds. Figure 5-38 shows the comparison of apparent viscosities, Figure 5-39 illustrates the comparison of plastic viscosities, Figure 5-40 demonstrates the comparison of yield point and Figure 5-41 shows the comparison of the filtration characteristics. From all figures 5-39 and 5-41, it can be explicitly derived that date seeds mud performed better than the modified starch muds when plastic viscosity and filtration are concerned. These two parameters are of utmost concern to a drilling fluids engineer as they play an integral part during drilling operations. Also, from Figure 5-40, it is seen that the yield point of modified starch mud is almost two folds than that of date seeds mud at higher concentrations make it superior to the latter one on the yield point basis.

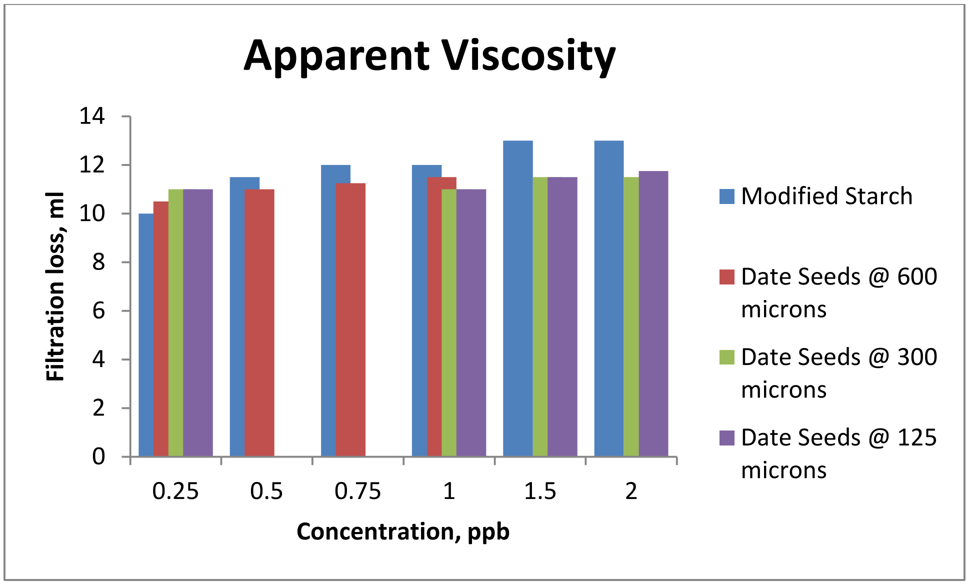

Figure 5- 38: Comparison of Apparent Viscosities of Date Seeds muds and Modified Starch muds

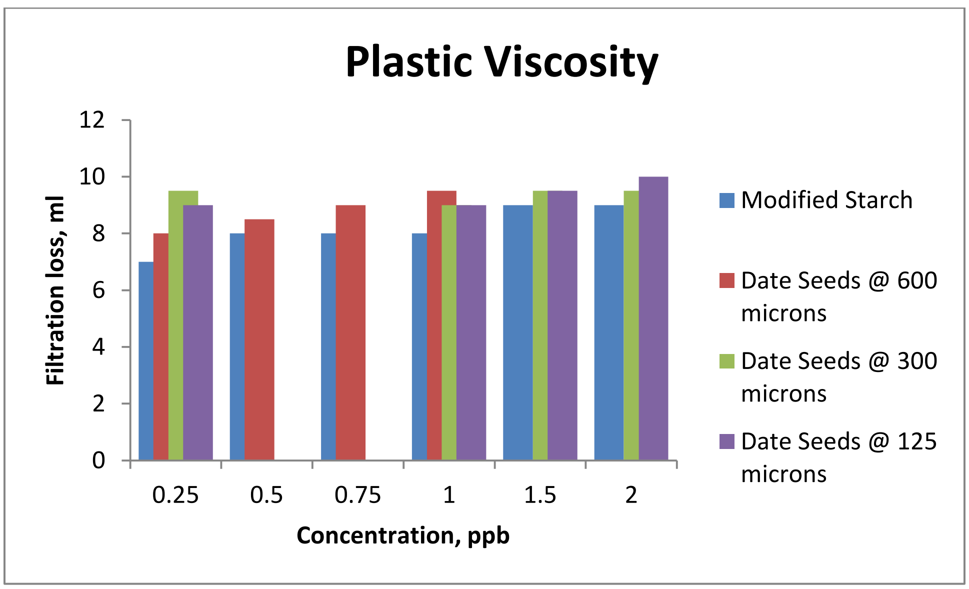

Figure 5- 39: Comparison of Plastic Viscosities of Date Seeds muds and Modified Starch muds

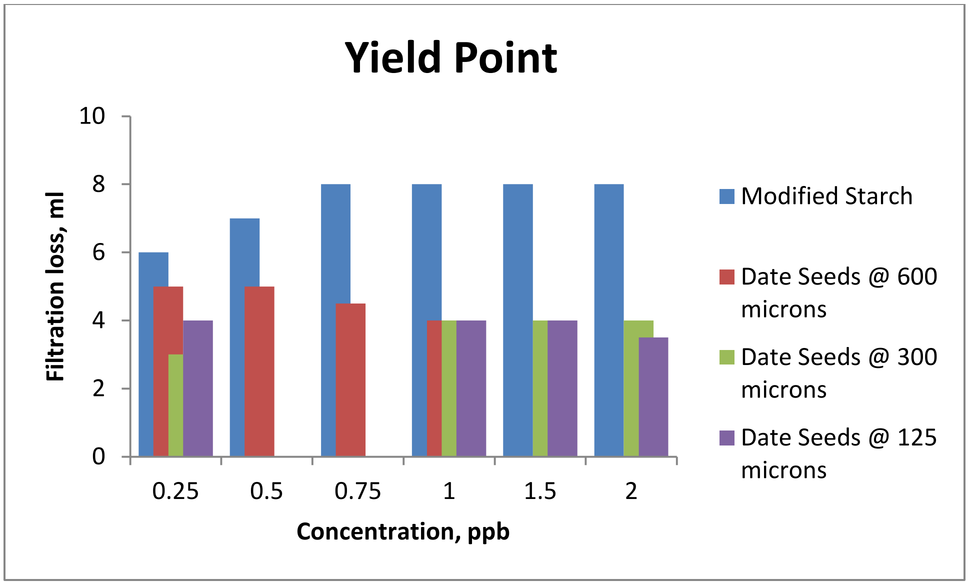

Figure 5- 40: Comparison of Yield Point of Date Seeds muds and Modified Starch muds

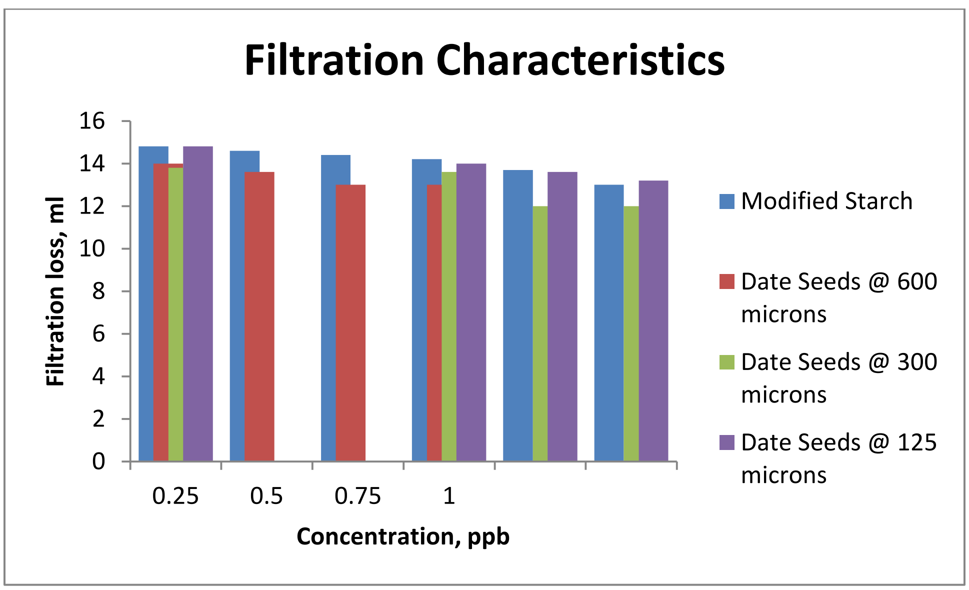

Figure 5- 41: Comparison of the Filtration Characteristics of Date Seeds muds and Modified Starch muds

5.11.2 Comparison of Grass Ash muds with Modified Starch muds:

An assessment is carried out on the viscosities, yield point and filtration characteristics of grass ash muds and modified starch muds. Figure 5-42 shows the comparison of apparent viscosities, Figure 5-43 illustrates the comparison of plastic viscosities, Figure 5-44 demonstrates the comparison of yield point and Figure 5-45 shows the comparison of the filtration characteristics. It can be observed from all the figures that grass ash muds performed much better than the modified starch muds in all aspects of viscosities, yield point and filtration.

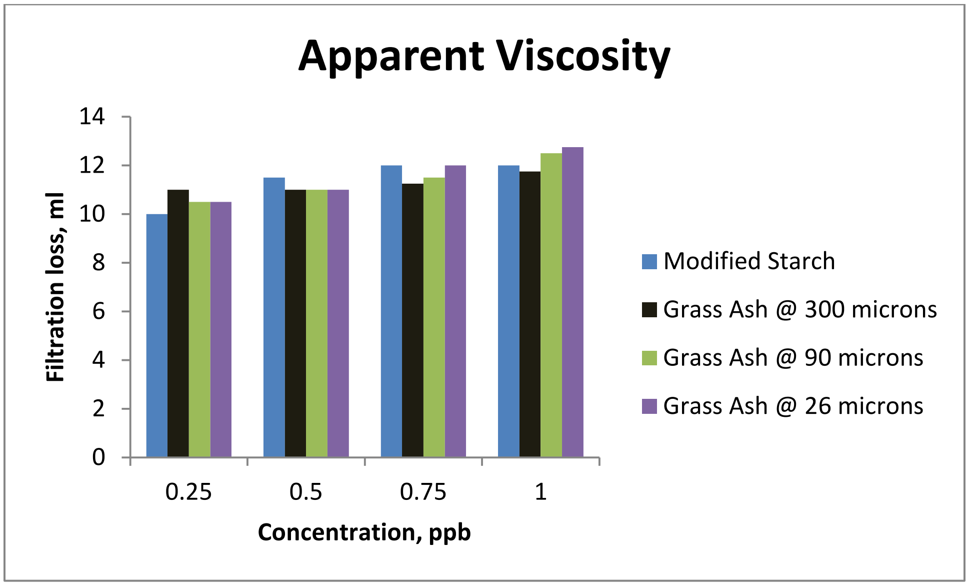

Figure 5- 42: Comparison of Apparent Viscosities of Grass Ash muds and Modified Starch muds

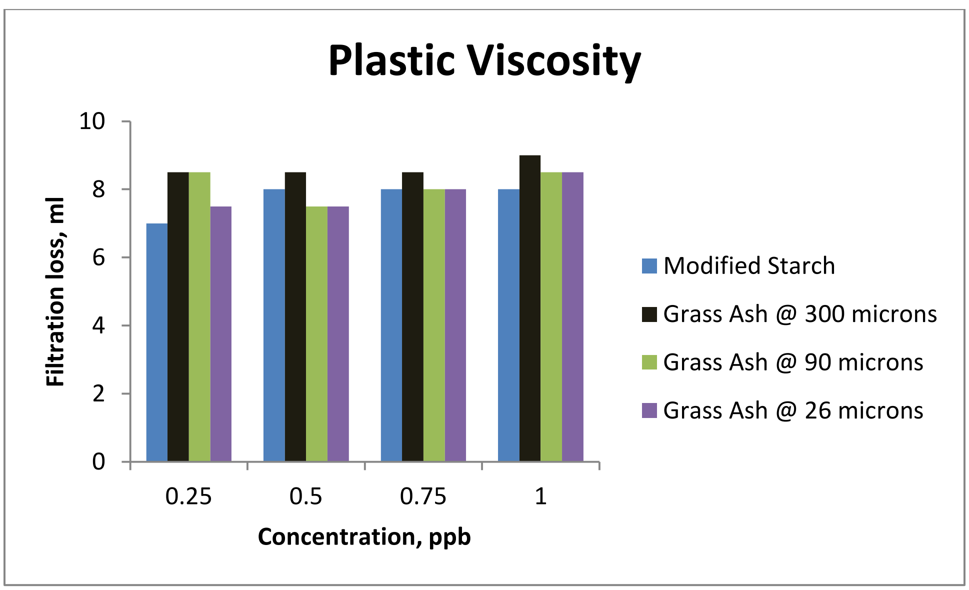

Figure 5- 43: Comparison of Plastic Viscosities of Grass Ash muds and Modified Starch muds

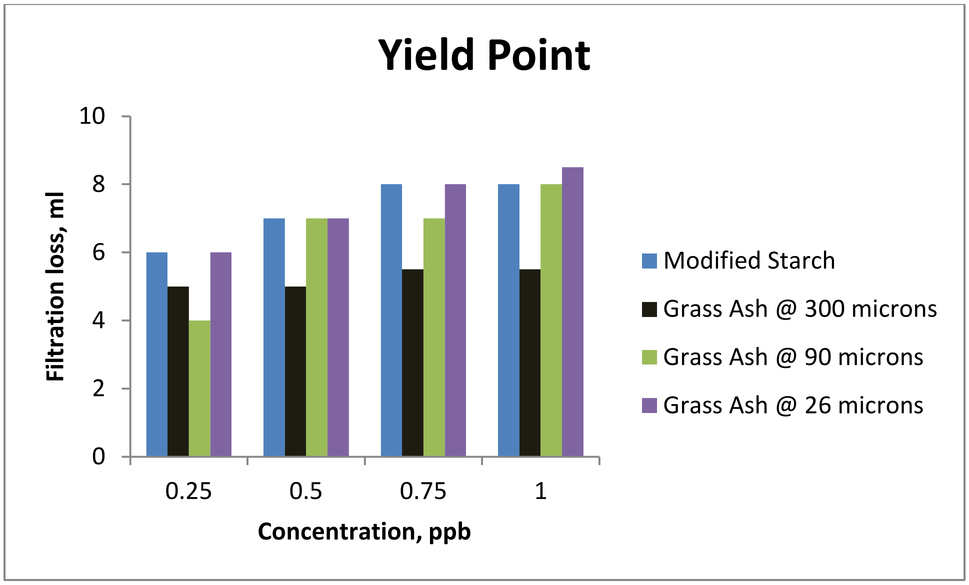

Figure 5- 44: Comparison of Yield Point of Grass Ash muds and Modified Starch muds

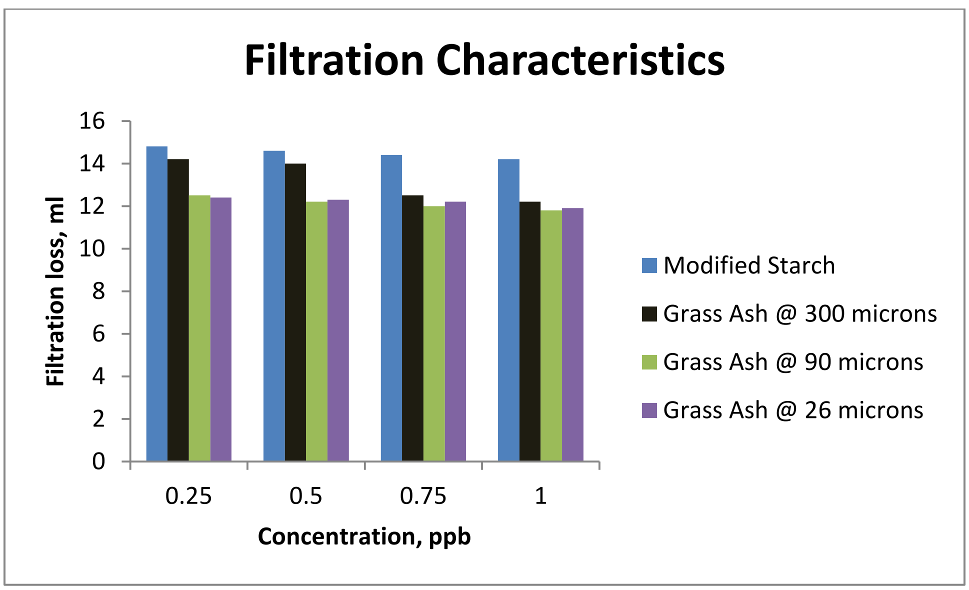

Figure 5- 45: Comparison of the Filtration Characteristics of Grass Ash muds and Modified Starch muds

5.11.3 Comparison of Grass muds with Modified Starch muds:

A comparison is made between the viscosities, yield point and filtration characteristics of grass ash muds and modified starch muds. Figure 5-46 shows the comparison of apparent viscosities, Figure 5-47 illustrates the comparison of plastic viscosities, Figure 5-48 demonstrates the comparison of yield point and Figure 5-49 shows the comparison of the filtration characteristics. It is seen from Figures 5-47 and 5-49, the plastic viscosities and filtration characteristics of the grass muds are superior to the modified starch muds. However, the yield point of modified starch muds are found to be nearly twice than the grass muds at higher concentrations.

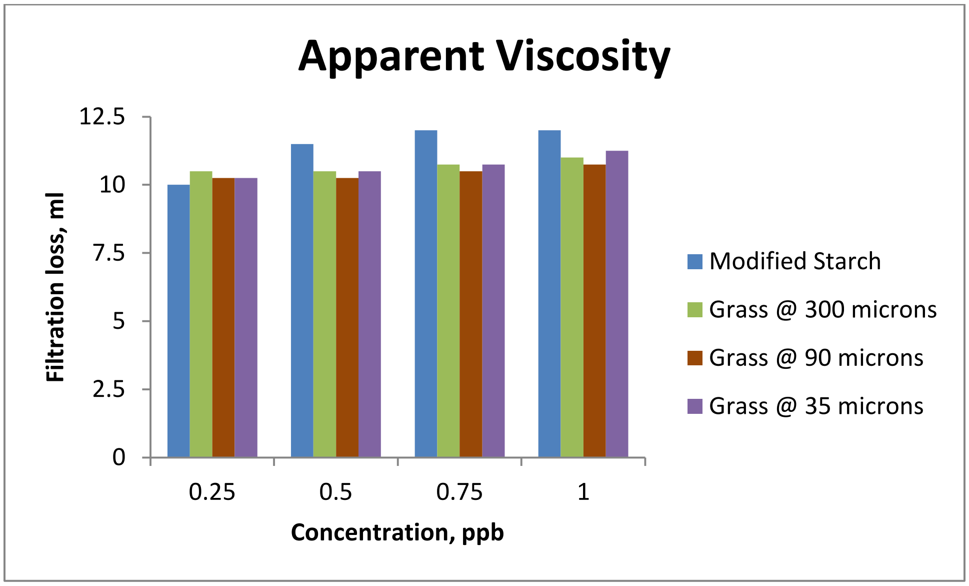

Figure 5- 46: Comparison of Apparent Viscosities of Grass muds and Modified Starch muds

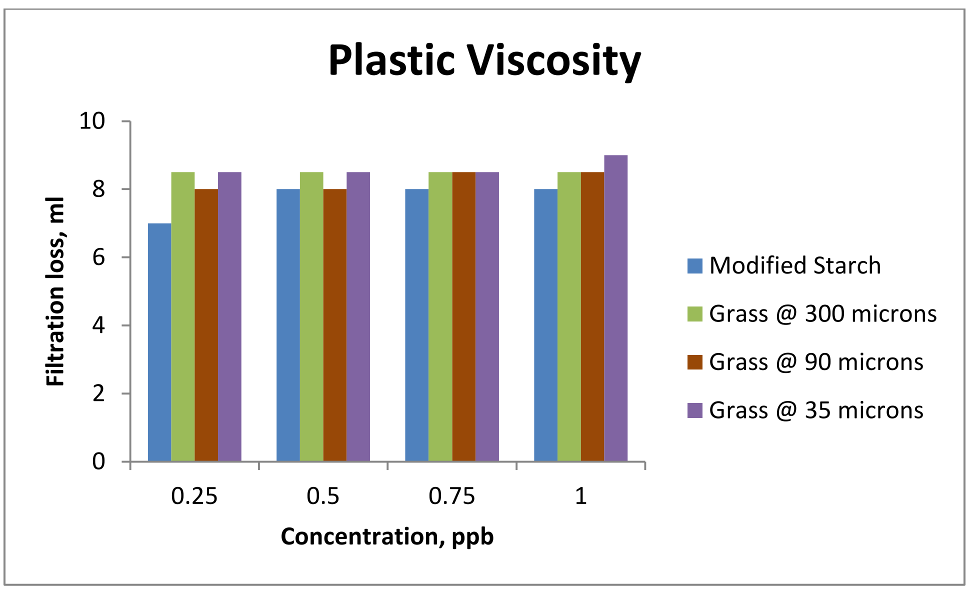

Figure 5- 47: Comparison of Plastic Viscosities of Grass muds and Modified Starch muds

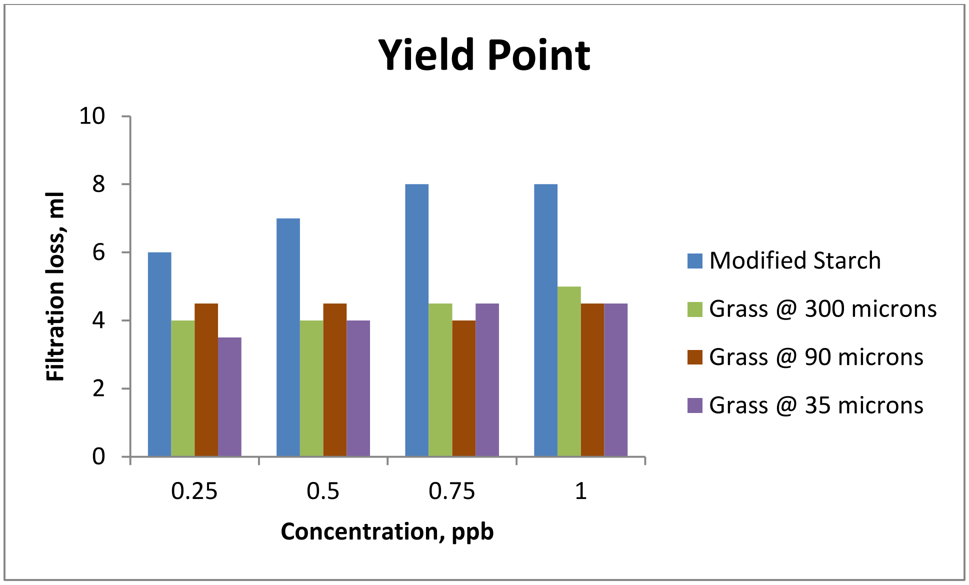

Figure 5- 48: Comparison of Yield Point of Grass muds and Modified Starch muds

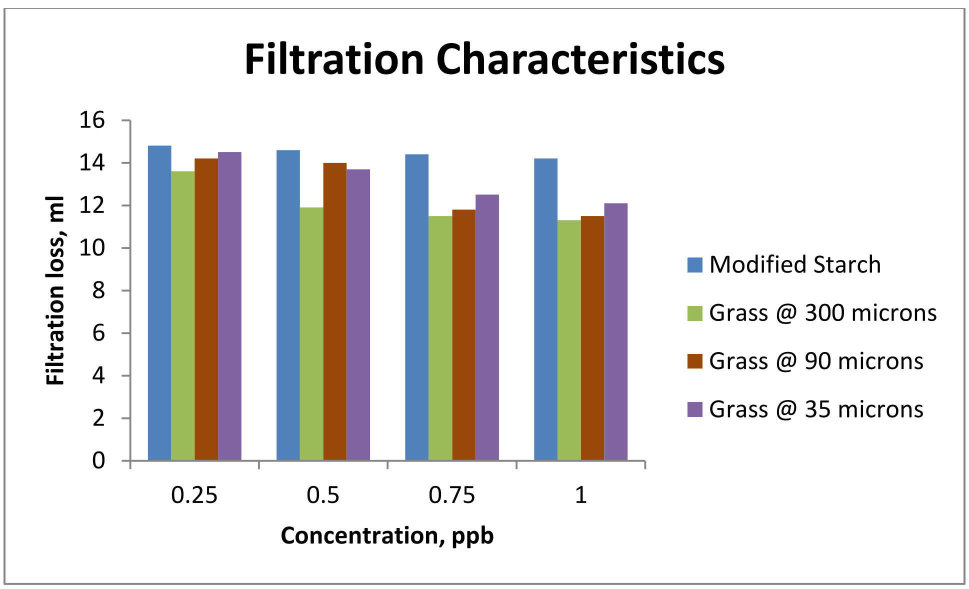

Figure 5- 49: Comparison of the Filtration Characteristics of Grass muds and Modified Starch muds

5.12 Experimental Results of Date Seeds, Grass Ash and Grass at high temperatures

The behavior of drilling fluids at high temperature, particularly water based drilling fluids, is unpredictable, and, indeed not yet fully understood by researchers. Even quite small differences in composition can make considerable differences in behavior, so that it is necessary to test each mud individually in order to obtain reliable data. Annis (1967) studied the rheology of water based muds at high temperature. Hiller (1963) studied the effect of high pressure as well, but found that it was minor. An increase in temperature decreases the viscosity of the liquid phase; an increase in pressure increases the density of the liquid phase and therefore increases the viscosity.

5.12.1 Effect of high temperature on Date Seeds:

From the previous tests on date seeds conducted at ambient conditions, it is deduced that for the three particle sizes used i.e. 600 microns, 300 microns and 125 microns, the optimum concentrations were 0.75 ppb, 1.5 ppb and 2.0 ppb respectively. A further step is taken towards conducting experiments on rheology for the three samples formulated, using the above mentioned optimal concentrations at high temperatures of 160^{o}F and 200^{o}F. Note that higher temperatures above 200^{o}F are avoided because no high temperature additives were used in the formulation.

Table 5- 17: High temperature rheology of mud formulated with Date Seeds of 600 microns size

Speed, rpm	**Room Temperature**	**160^{o}F**	**200^{o}F**
600	22.5	17	19
300	13.5	11	12.5
200	10	9	10
100	7.5	6.5	7.5
6	1.5	2.5	3
3	1.5	2	2.5
AV	11.25	8.5	9.5
PV	9	6	6.5
YP	4.5	5	6
YP/PV	0.5	0.83	0.92
Gel Strength, 10 sec	1	3	4
Gel Strength, 10 min	14	12	14

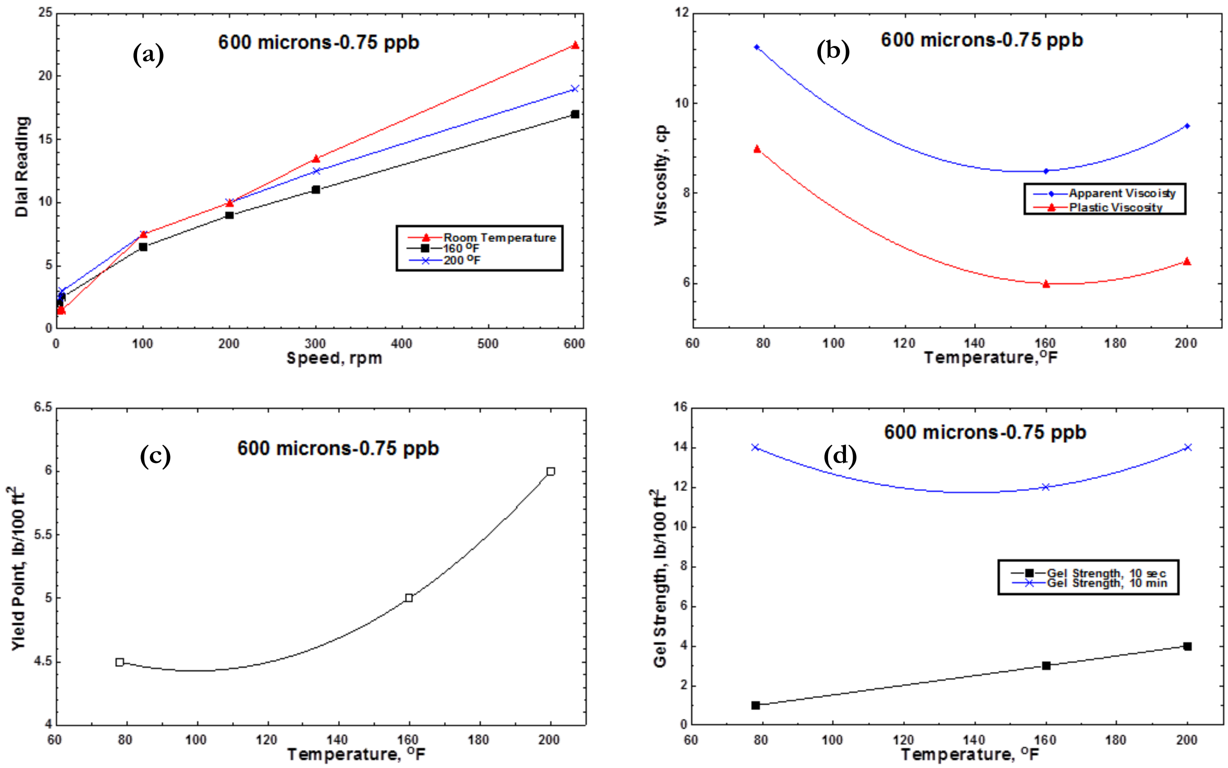

Figure 5- 50: Effect of high temperature on rheological parameters for muds formulated with Date Seeds of 600 microns size

The above figure (Fig. 5-50) shows the plot of viscosities, yield point and gel strength versus temperature at 600 microns particle size. It is seen that the viscosities decrease as the temperature is increased up to 200^{o}F. However, it is observed that the yield point increases as the temperature increases which is the characteristic of a good mud. The gel strengths are also noted to be increasing. From Table 5-17, it is seen that the yield point to plastic viscosity ratio increases gradually at high temperatures which is usually good for hole stability and the mud gel easily breaks when circulation is restarted.

Table 5- 18: High temperature rheology of mud formulated with Date Seeds of 300 microns size

Speed, rpm	Room Temperature	160^{o}F	200^{o}F
600	23	18.5	20
300	13.5	11.5	13
200	11	9	10.5
100	7.5	7	7.5
6	1.5	1.5	2.5

3	1.5	1.5	2
AV	11.5	9.25	10
PV	9.5	7	7
YP	4	4.5	6
YP/PV	0.42	0.64	0.86
Gel Strength, 10 sec	1	2	11
Gel Strength, 10 min	14	9	20

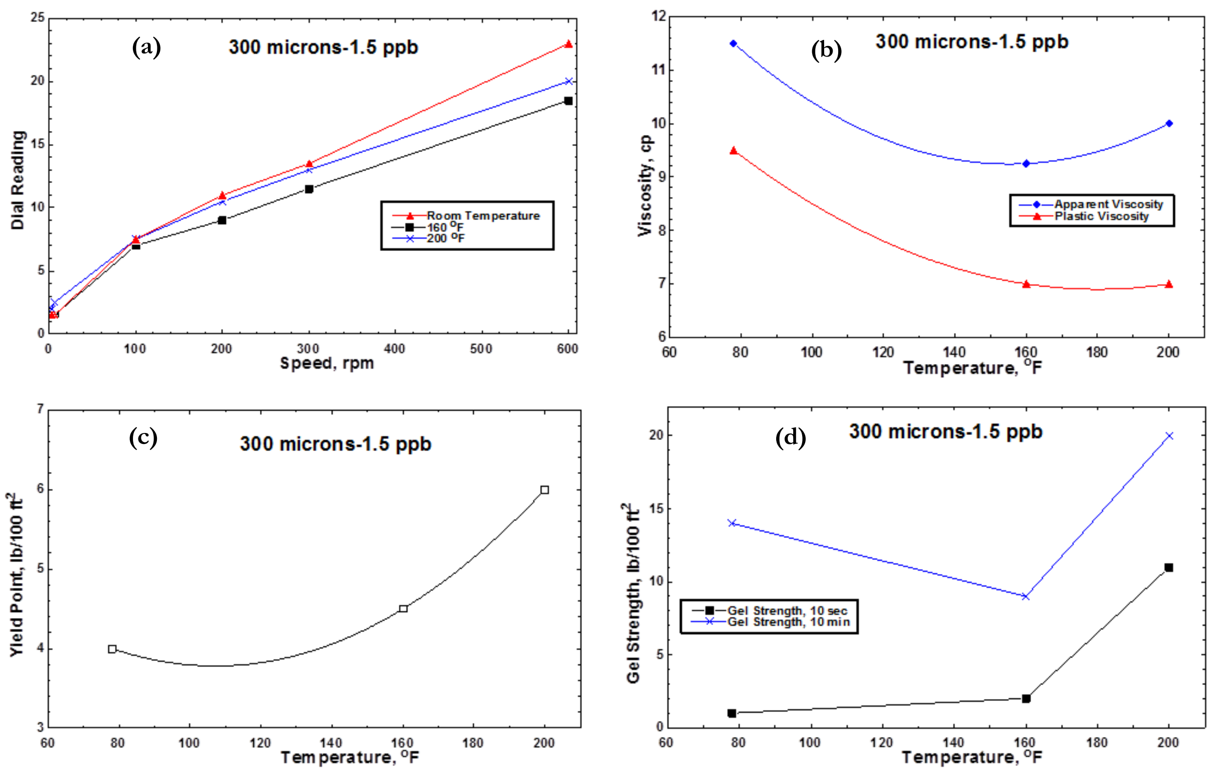

Figure 5- 51: Effect of high temperature on rheological parameters for muds formulated with Date Seeds of 300 microns size

The figure (Fig. 5-51) above shows the plot of viscosities, yield point and gel strength versus temperature at 300 microns particle size. It is observed that the viscosities decrease as the temperature is increased up to 200°F. However, it is seen that the yield point increases as the temperature increases which is the characteristic of a good mud. The gel strengths are also noted to be increasing. From Table 5-18, it is seen that the yield point to plastic viscosity ratio increases gradually at high temperatures which is usually good for hole stability and ensures that the mud gel easily breaks when circulation is restarted.

Table 5- 19: High temperature rheology of mud formulated with Date Seeds of 125 microns size

Speed, rpm	Room Temperature	160°F	200°F
600	23.5	14.5	15.5
300	13.5	9	9.5
200	10.5	7	7.5
100	7	5	6
6	1.5	1.5	1.5
3	1.5	1	1.5
AV	11.75	7.25	7.75
PV	10	5.5	6
YP	3.5	3.5	3.5
YP/PV	0.35	0.64	0.58
Gel Strength, 10 sec	2	1	5
Gel Strength, 10 min	15	7	14

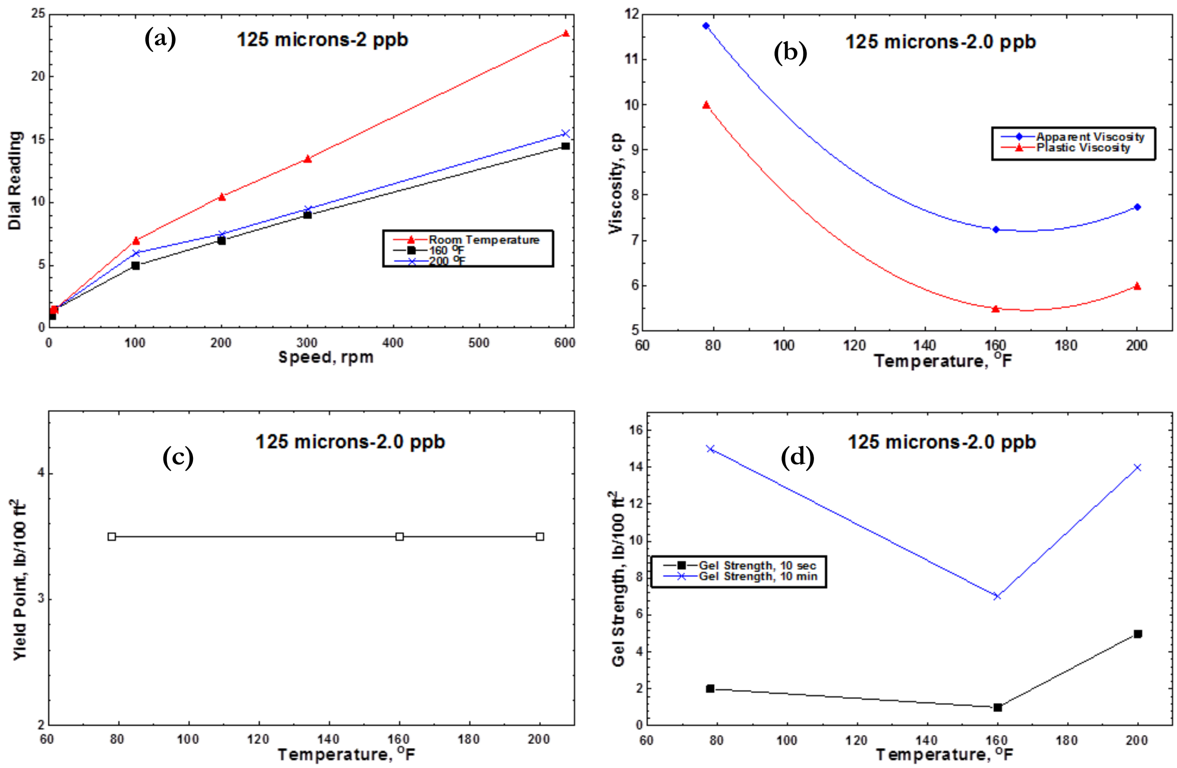

Figure 5- 52: Effect of high temperature on rheological parameters for muds formulated with Date Seeds of 125 microns size

Figure 5-52 above shows the plot of viscosities, yield point and gel strength versus temperature at 125 microns particle size. It is observed that the viscosities decrease as the temperature is increased up to 200^{o}F whereas the yield point remains constant. The gel strengths are also noted to be increasing.

5.12.2 Effect of high temperature on Grass Ash:

From the experiments conducted on grass ash at ambient conditions, it is inferred that for the three particle sizes used i.e. 300 microns, 90 microns and 26 microns, the optimum concentrations were found to be 1.0 ppb. Tests on rheology are conducted at temperatures 160^{o}F and 200^{o}F for the three samples at their optimal concentrations. Note that higher temperatures above 200^{o}F are avoided because no high temperature additives were used in the formulation.

Table 5- 20: High temperature rheology of mud formulated with Grass Ash of 300 microns size

Speed, rpm	**Room Temperature**	**160^{o}F**	**200^{o}F**
600	23.5	20	22.5
300	14.5	13.5	16
200	11.5	11	13
100	8.5	8.5	10
6	2.5	4	5.5
3	1.5	3.5	4.5
AV	11.75	10	11.25
PV	9	6.5	6.5
YP	5.5	7	9.5
YP/PV	0.61	1.08	1.46
Gel Strength, 10 sec	4	6	7
Gel Strength, 10 min	19	23	26

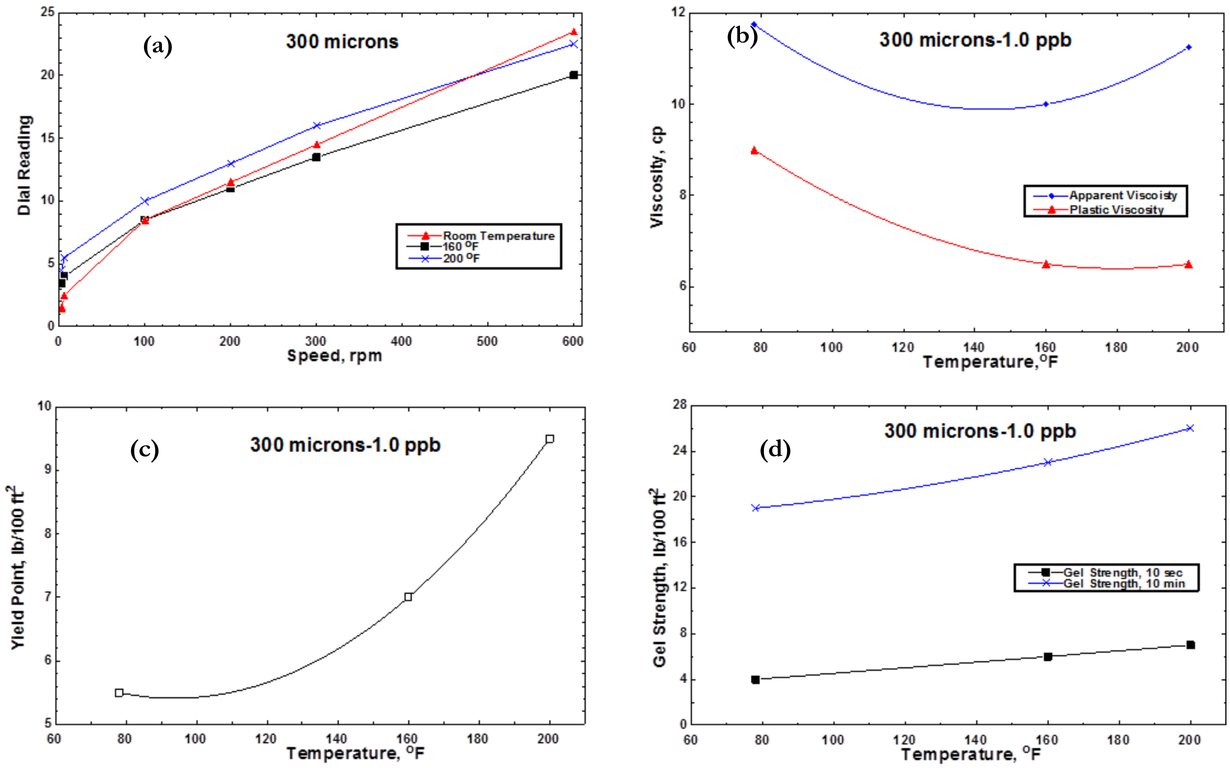

Figure 5- 53: Effect of high temperature on rheological parameters for muds formulated with Grass Ash of 300 microns size

The figure above (Fig. 5-53) shows the plot of viscosities, yield point and gel strength versus temperature at 300 microns particle size. It is observed that the viscosities decrease as the temperature is increased up to 200°F. However, it is seen that the yield point increases as the temperature increases which is the characteristic of a good mud. The gel strengths are also noted to be increasing. From Table 5-20, it is seen that the yield point to plastic viscosity ratio increases gradually (and is above 1) at high temperatures which is usually good for hole stability and ensures that the mud gel easily breaks when circulation is restarted.

Table 5- 21: High temperature rheology of mud formulated with Grass Ash of 90 microns size

Speed, rpm	Room Temperature	160°F	200°F
600	25	24	27
300	16.5	17	18.5
200	14	14	14.5
100	11	11.5	11
6	5.5	6.5	4.5
3	4.5	6	4.5
AV	12.5	12	13.5
PV	8.5	7	8.5
YP	8	10	10
YP/PV	0.94	1.43	1.18
Gel Strength, 10 sec	9	8	9
Gel Strength, 10 min	24	28	30

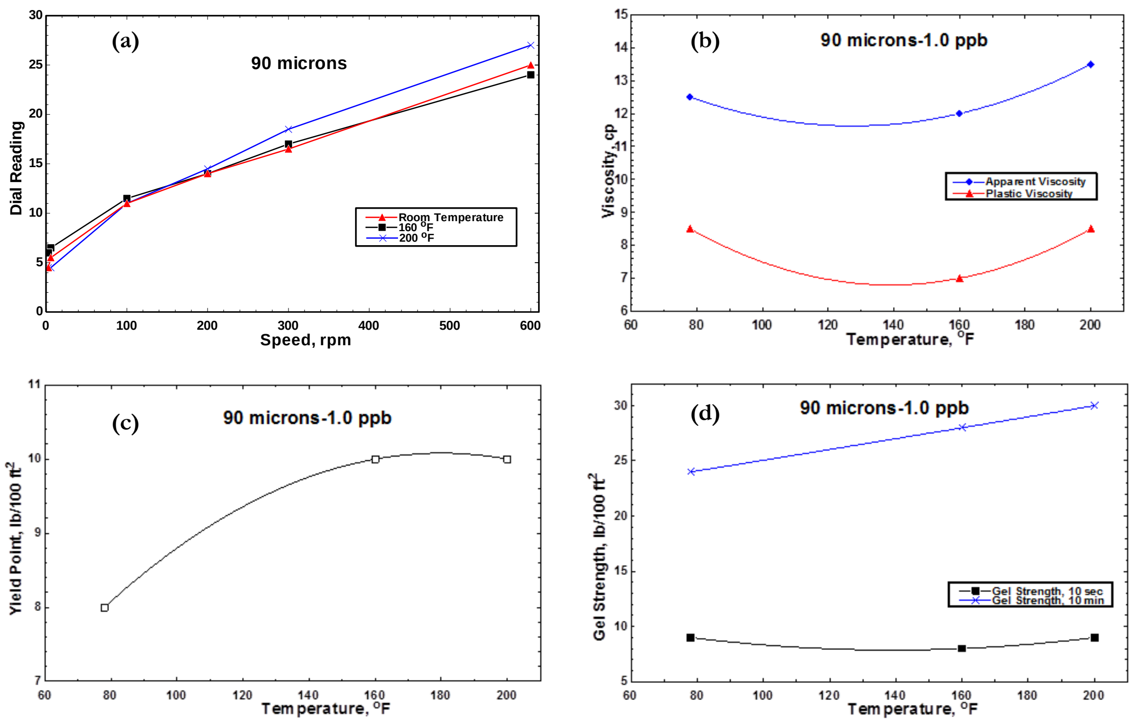

Figure 5- 54: Effect of high temperature on rheological parameters for muds formulated with Grass Ash of 90 microns size

Figure 5-54 shows the plot of viscosities, yield point and gel strength versus temperature at 90 microns particle size. It is observed that the plastic viscosity value increases after a dip as the temperature is increased up to 200°F. The apparent viscosity value at 200°F is greater than the value at room temperature. This may be attributed to the loss of water viscosity at high temperature making the mud denser. However, it is seen that the yield point increases as the temperature increases which is the characteristic of a good mud. The 10 minute gel strength is also noted to be increasing. From Table 5-21, it is seen that the yield point to plastic viscosity ratio increases gradually and is above 1, which is desirable where borehole stability is concerned.

Table 5- 22: High temperature rheology of mud formulated with Grass Ash of 26 microns size

Speed, rpm	**Room Temperature**	**160°F**	**200°F**
600	25.5	27	31
300	17	18.5	21.5
200	15	15	16.5
100	11.5	12	12
6	6.5	5	6
3	6	4.5	5.5
AV	12.75	13.5	15.5
PV	8.5	8.5	9.5
YP	8.5	10	12
YP/PV	1	1.18	1.26
Gel Strength, 10 sec	9	7	9
Gel Strength, 10 min	28	29	33

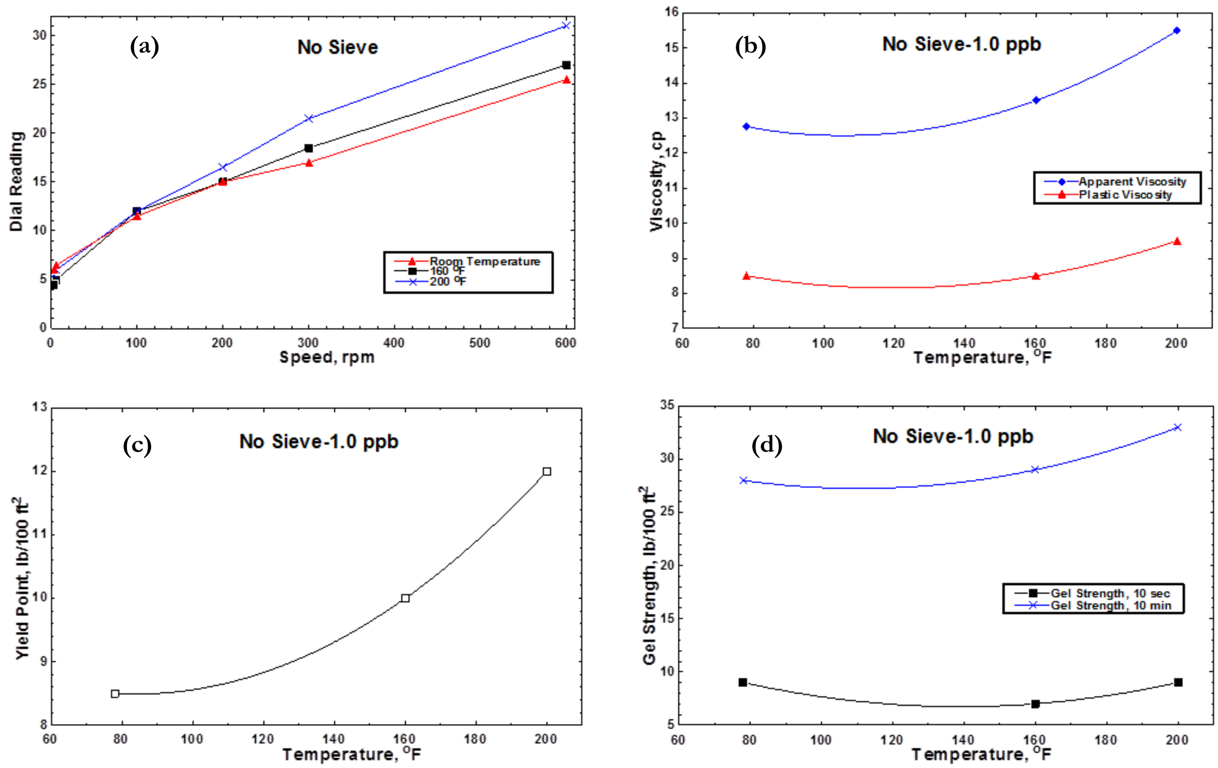

Figure 5- 55: Effect of high temperature on rheological parameters for muds formulated with Grass Ash of 26 microns size

The figure (Fig. 5-55) above shows the plot of viscosities, yield point and gel strength versus temperature at 26 microns particle size. It is observed that viscosities increase as the temperature is increased up to 200^{o}F. This may be attributed to the loss of water viscosity at high temperature making the mud denser. However, it is seen that the yield point increases as the temperature increases which is the characteristic of a good mud. The 10 minute gel strength is also noted to be increasing. From Table 5-22, it is seen that the yield point to plastic viscosity ratio increases gradually and is above 1, which is desirable where borehole stability is concerned.

5.12.3 Effect of high temperature on Grass:

From the experiments conducted on grass at ambient conditions, it is concluded that for the three particle sizes used i.e. 300 microns, 90 microns and 26 microns, the optimum concentrations were found to be 1.0 ppb. Tests on rheology are conducted at temperatures of 160^{o}F and 200^{o}F for the three samples at their optimal concentrations. It is again to be noted that higher temperatures above 200^{o}F are avoided as no high temperature additives were used in the formulation.

Table 5- 23: High temperature rheology of mud formulated with Grass of 300 microns size

Speed, rpm	**Room Temperature**	**160^{o}F**	**200^{o}F**
600	22	18	20
300	13.5	12.5	14.5
200	11	10	11
100	7	7	8
6	2.5	3.5	4.5
3	2	3	4
AV	11	9	10
PV	8.5	5.5	5.5
YP	5	7	9
YP/PV	0.59	1.27	1.64
Gel Strength, 10 sec	3	5	7
Gel Strength, 10 min	16	18	21

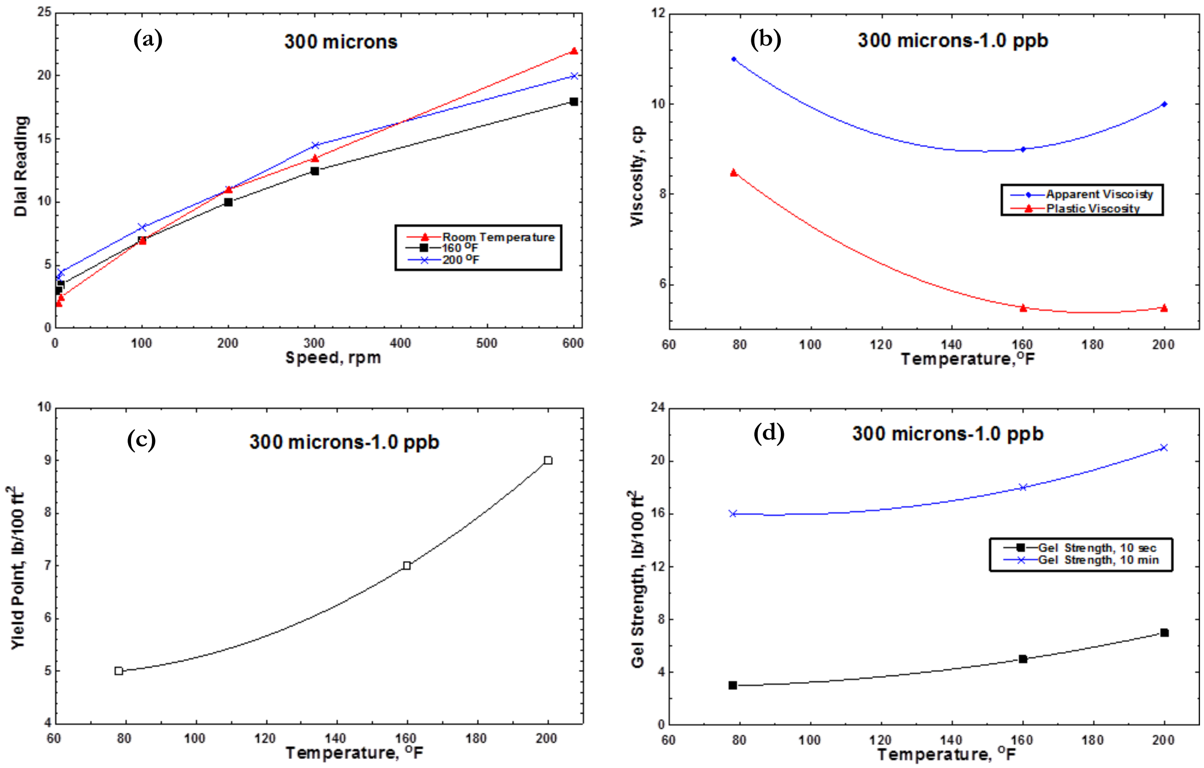

Figure 5- 56: Effect of high temperature on rheological parameters for muds formulated with Grass of 300 microns size

Figure 5-56 shows the plot of viscosities, yield point and gel strength versus temperature at 300 microns particle size. It is observed that the viscosities decrease as the temperature is increased up to 200^{o}F. However, it is seen that the yield point increases as the temperature increases which is the characteristic of a good mud. The gel strengths are also noted to be increasing. From Table 5-23, it is seen that the yield point to plastic viscosity ratio increases gradually (and is above 1) at high temperatures which is usually good for hole stability and ensures that the mud gel easily breaks when circulation is restarted.

Table 5- 24: High temperature rheology of mud formulated with Grass of 90 microns size

Speed, rpm	Room Temperature	160°F	200°F
600	21.5	19	20.5
300	13	12.5	14
200	10	10	11
100	7.5	7	8
6	2	3	3.5
3	1.5	2.5	3
AV	10.75	9.5	10.25
PV	8.5	6.5	6.5
YP	4.5	6	7.5
YP/PV	0.53	0.92	1.15
Gel strength, 10 sec	3	4	6
Gel Strength, 10 min	16	18	19

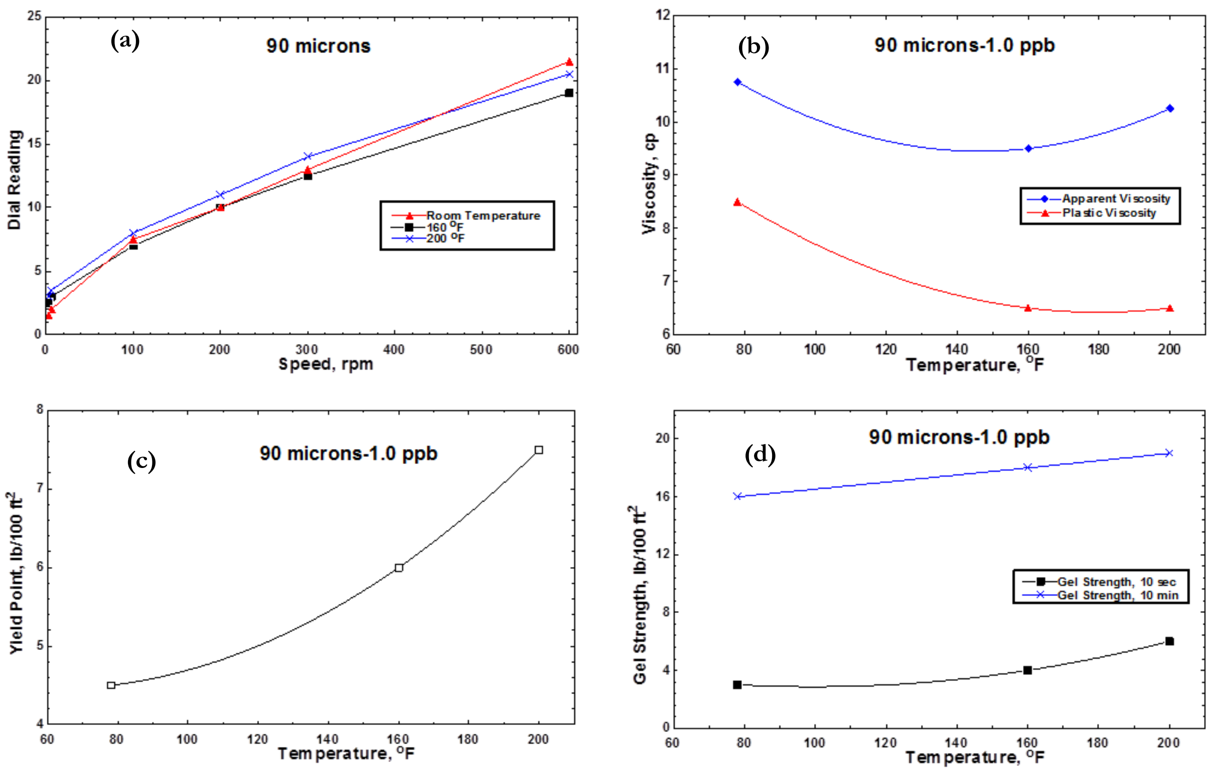

Figure 5- 57: Effect of high temperature on rheological parameters for muds formulated with Grass of 90 microns size

The figure (Fig. 5-57) shows the plot of viscosities, yield point and gel strength versus temperature at 90 microns particle size. It is observed that the viscosities decrease as the temperature is increased up to 200^{o}F. However, it is seen that the yield point increases as the temperature increases which is the characteristic of a good mud. The gel strengths are also noted to be increasing. From Table 5-24, it is seen that the yield point to plastic viscosity ratio increases gradually (and is above 1) at high temperatures which is usually good for hole stability and ensures that the mud gel easily breaks when circulation is restarted.

Table 5- 25: High temperature rheology of mud formulated with Grass of 36 microns size

Speed, rpm	**Room Temperature**	**160^{o}F**	**200^{o}F**
600	22.5	19.5	20
300	13.5	12.5	13.5
200	10	10	11
100	7.5	7.5	8
6	2	3.5	3.5
3	1.5	3	3
AV	11.25	9.75	10
PV	9	7	6.5
YP	4.5	5.5	7
YP/PV	0.5	0.79	1.08
Gel Strength, 10 sec	2.5	5	6
Gel Strength, 10 min	17	18	20

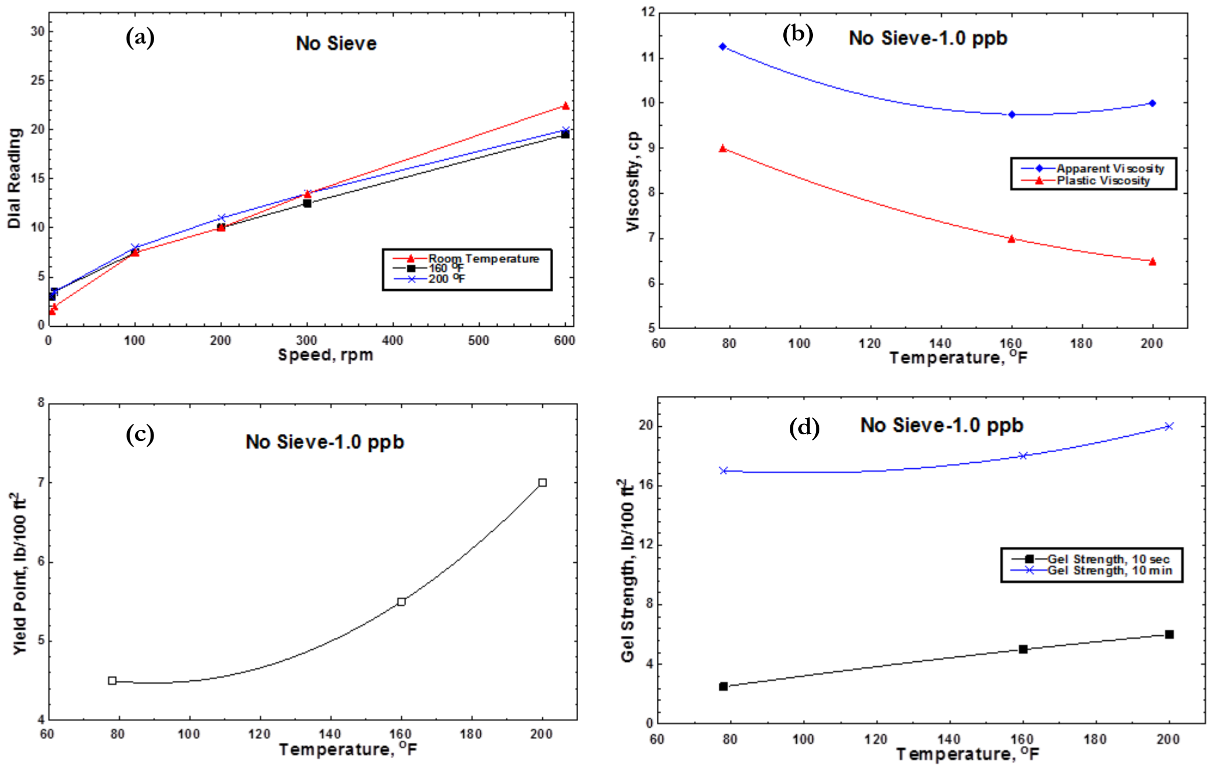

Figure 5- 58: Effect of high temperature on rheological parameters for muds formulated with Grass of 35 microns size

Figure 5-58 shows the plot of viscosities, yield point and gel strength versus temperature at 35 microns particle size. It is observed that the viscosities decrease as the temperature is increased up to 200°F. However, it is seen that the yield point increases as the temperature increases which is the characteristic of a good mud. The gel strengths are also noted to be increasing. From Table 5-25, it is seen that the yield point to plastic viscosity ratio increases gradually (and is above 1) at high temperatures which is usually good for hole stability and ensures that the mud gel easily breaks when circulation is restarted.

5.13 Research Highlights

This section presents the findings of the conducted research. It is to be noted that the sole focus of this work is to find the application of date seeds, grass and grass ash as environmentally-friendly substitutes to the existing toxic chemicals used by the oil industry. In tables 5-26 and 5-27, the commonly used pH and filtration control additives are listed with the amount of damage they cause to the human body. Date seeds, grass and grass ash can be used to substitute any of these toxic additives from the drilling fluid.

Table 5- 26: Commonly used pH control additives versus the proposed pH control additives

Commonly used Additives	Function	Remarks on conventional pH controller	Remarks on proposed pH controller
Sodium Hydroxide (Caustic Soda), NaOH	Increases pH	1. Corrosive, toxic and a major potential hazard upon contact to skin and eyes. 2. Ingestion can cause severe burning and pain in lips, mouth, tongue, throat and stomach. Death can result from ingestion. 3. Causes burns and scarring. Can cause serious damage to all body tissues contacted	1. Date Seeds and grass performed to lower the pH of the mud whereas grass ash tended to increase the pH. 2. No damage is caused to the skin, eyes or other parts of the body while in contact with the proposed natural additives. 3. Date Seeds and Grass can be used to lower the pH (alkalinity controllers) whereas grass ash can be used to control the acidity.
Potassium Hydroxide, KOH	Increases pH	1. Very hazardous in case of skin contact (corrosive, irritant), of eye contact (irritant, corrosive), of ingestion, of inhalation. 2. The amount of tissue damage depends on length of contact. Eye contact can result in corneal damage or blindness. 3. Skin contact can produce inflammation and blistering. Inhalation of dust will produce irritation to gastro-intestinal or respiratory tract, characterized by	

		burning, sneezing and coughing. 4. Severe over-exposure can produce lung damage, choking, unconsciousness or death. 5. Inflammation of the eye is characterized by redness, watering, and itching. Skin inflammation is characterized by itching, scaling, reddening, or, occasionally, blistering.	
Calcium Hydroxide, $Ca(OH)_2$	Increases pH, flocculates bentonite dispersions	1. Very hazardous in case of eye contact (irritant). Hazardous in case of skin contact (irritant), of eye contact (corrosive), of ingestion, of inhalation. Corrosive to eyes and skin. The amount of tissue damage depends on length of contact. 2. Eye contact can result in corneal damage or blindness. Inflammation of the eye is characterized by redness, watering, and itching. 3. Skin contact can produce inflammation and blistering. 4. Inhalation of dust will produce irritation to gastro-intestinal or respiratory tract, characterized by burning, sneezing and coughing. 5. Severe overexposure can produce lung damage, choking,	

		unconsciousness or death.	
Sodium Bicarbonate, $NaHCO_3$	Precipitates calcium and reduces pH	Slightly hazardous in case of skin contact (irritant), of eye contact (irritant), of ingestion, of inhalation.	
Citric acid	pH Reducer, precipitates calcium when treating cement contamination	1. Causes severe eye irritation and possible injury. 2. Causes skin irritation. May cause skin sensitization, an allergic reaction, which becomes evident upon re-exposure to this material. 3. May cause gastrointestinal irritation with nausea, vomiting and diarrhea. Excessive intake of citric acid may cause erosion of the teeth. 4. Causes respiratory tract irritation. 5. Repeated exposure may cause sensitization dermatitis.	
Sodium Carbonate (Soda Ash), Na_2CO_3	Increases pH	1. Severely irritating to eyes. Avoid contact with eyes. 2. Repeated exposure may cause skin dryness or cracking. Wash thoroughly after handling. 3. Inhalation of dust in high concentration may cause irritation of respiratory system. 4. Although low in toxicity, ingestion may cause nausea, vomiting, and diarrhea.	

Table 5- 27: Commonly used Filtration Control Additives versus the proposed Filtration Control Additives

Commonly used Additives	Function	Remarks on Conventional Filtration Control additives	Remarks on proposed Filtration Control additives
Sodium Carboxymethyl cellulose	Viscosifier and Filtration Control	Hazardous in case of skin contact (irritant), of eye contact (irritant), of ingestion, of inhalation.	1. Date seeds, grass and grass ash are not hazardous in case of skin or eye contact, ingestion or inhalation. 2. No irritation is caused when contacted with the proposed additives. 3. Date seeds, Grass and Grass ash performed good to lower the amount of filter loss, thus mimicking the action of filtration control agents.
Xanthan Gum	Viscosifier and Filtration Control	Hazardous in case of skin contact (irritant), of eye contact (irritant), of ingestion, of inhalation.	
Polyanionic Cellulose	Filtration Control	Dust causes mild eye irritation. It may cause respiratory irritation if inhaled.	
Starch	Filtration Control	1. May cause eye irritation. 2. May cause skin irritation. Low hazard for usual industrial handling. 3. May cause irritation of the digestive tract. Low hazard for usual industrial handling. 4. May cause respiratory tract irritation. Low hazard for usual industrial handling.	
Hydroxymethyl Cellulose	Filtration Control	1. Dust may cause irritation of respiratory tract, experienced as nasal discomfort and discharge. 2. Dust may cause discomfort in the eye and slight excess redness of the conjunctiva.	

CHAPTER 6

CONCLUSIONS AND RECOMMENDATIONS

6.1 Conclusions

Based on the experimental findings through this research work, the following conclusions are drawn:

1) Date Seeds, Grass ash and grass can be used as environmentally-friendly additives for water based muds.
2) The developed mud systems have zero level of toxicity.
3) The muds formulated with the said additives at different particle sizing exhibited improvement in filtration control.
4) Date Seeds and Grass lowered the pH of the mud whereas Grass ash tends to increase the pH. This attribute of the natural additives can be used to replace the current toxic chemicals used for alkalinity and acidity control.
5) All the three materials lowered the resistivity of the mud filtrate which would find application during electrical logging.
6) The muds formulated with date seeds, grass ash and grass exhibited thermal stability at high temperatures of 160^{o}F and 200^{o}F.

6.2 Recommendations

The following recommendations are made for future research toward the development of a more competitive and comprehensive sustainable mud system:

1) Tests can be carried out using these additives with a salt water-based system.
2) Efforts should be directed towards the formulation of an oil-based mud system with date seeds or grass or grass ash as natural additives.
3) Studies can be undertaken for filter cake characterization in detail.
4) Cost analysis should be done to establish the economic viability of the mud system formulated with the proposed natural additives.
5) A low cost commercial mud system can be formulated with the addition of other additives.

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