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Improvement of drilling fluid capability by using natural loss circulation material (LCM)

تطوير قدرة سائل الحفر بإستخدام مادة طبيعية لمعالجة فقدان دورة سائل الحفر

Project submitted in partial fulfillment of requirement of the degree of B.sc In petroleum engineering

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مشروع تخرج مقدم إلى كلية هندسة وتكنولوجيا النفط-جامعة السودان للعلوم والتكنولوجيا

إنجاز جزئي لأحد متطلبات الحصول على درجة البكالريوس في الهندسة

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لللل إست



سورة الفتح الآيات (1-3)

Dedication

t is our genuine gratefulness and warmest that we dedicate this work to our families, relatives and friends.

We also dedicate this thesis to our friends who have supported us throughout the process. We also appreciate all they have done.

We dedicate this work and give special thank to our colleagues in the college all of them have been our best cheers.

To the spirit of Dr. Mohamed naeem

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Abstract

The problem of loss circulation has occurred during drilling operation through natural or induced fracture.

In Heglig area, Bamboo field exactly Amal formation has low cementing material in addition to high porosity (massive, well sorted, white sand stone), this lead it to fracture under any pressure above pore pressure. The type of loss at that area was partial loss about 400bbl/hr, this problem had solved by using CaCO3.

The objective of this project is to investigate the performance of three natural materials (mica, Azadirachta indica (neem) and balanites aegypytiaca (lalob)) as powder to solve the problem. Also to design local product with international standards that participate in oil industry, Minimize the quantity of barite used in mud design, prevent the occurrence of loss circulation events in the area of study and solve the LCM scarcity problem.

After resultant daily mud report from two different wells in order to determine the properties of drilling mud, the drilling mud prepared in the lab and simulated its properties and tested by API filtration. The three materials were grinded in powder form and screened to chemical one of Neem and Lalob. Then they tested by XRD to know the crystalline construction of Mica and chemical one of Neem and Lalob. After that they added to the mud and tested by API filtration, firstly each one alone to get minimum filtrate, secondly mixture of them to determine their percentages of any one in the last form of LCM and test the ability of them to use as LPM, all these tests without changing in the rheological properties of the drilling mud. After that poured the LCM in the loss circulation device to get the optimum weight that can be used to solve the problem. Finally the optimum weight has been reached and the objectives have done.

تجريد

مشكلة فقدان دورة سائل الحفر تحدث خلال عملية الحفر داخل الشقوق الطبيعية او المصطنعة.

في منطقة هجليج, حقل بامبو بالتحديد طبقة أمل لها مشكلة قلة المادة اللاحمة بالإضافة إلى المسامية الكبيرة (كتلية, ذو سحنة جيدة التصنيف, صخور رملية بيضاء) هذا يجعلها تتشقق مع تحت أي ضغط أعلى من ضغط الطبقة. نوع الفقدان الذي حدث هناك كان جزئيا حوالي 400 بر ميل/ساعة. وتم حل المشكلة باستخدام مادة .CaCO3.

يهدف هذا المشروع الى اختبار المواد الطبيعية الثلاثة (مايكا, نيم ولالوب) كبودرة لحل المشكلة. كذلك تصميم منتج محلي بمواصفات عالمية يستخدم في صناعة النفط, تقليل كمية البارايت, منع حدوث المشكلة و حل مشكلة ندرة مواد حل فقدان دورة سائل الحفر.

بعد استنتاج تقرير سائل الحفر اليومي لبئرين مختلفين لتحديد الخواص, تم تحضير سائل الحفر في المعمل وتمت مماثلة خواصه واختبر في جهاز. API filtration . المواد الثلاثة طحنت في شكل بودرة و غربلت لتحديد الحجم المناسب. ثم اجري اختبار CRD لمعرفة التركيب البلوري للمايكا والكيميائي للنيم واللالوب. بعد ذلك اضيفت إلى سائل الحفر وتم اختبار ها بواسطة جهاز nall filtration . في البداية كل مادة على حدة للحصول على اقل راشح, ثانيا: خليط من المواد لتحديد نسبها في المادة النهائية المادة النهائية المعمل وتم معائلة خواصه واللالوب. بعد ذلك اضيفت إلى سائل الحفر وتم راشح, ثانيا: خليط من المواد لتحديد نسبها في المادة النهائية DCM واختبار امكانية استخدامها در شمر بانيا: خليط من المواد لتحديد نسبها في المادة النهائية DCM واختبار المكانية استخدامها در شمر ثانيا: خليط من المواد لتحديد نسبها في المادة النهائية MCM واختبار المكانية استخدامها در شمر بانيا: خليط من المواد لتحديد نسبها في المادة النهائية MCM واختبار المكانية استخدامها در شمر ثانيا: خليط من المواد لتحديد نسبها في المادة النهائية MCM واختبار المكانية استخدامها در شمر بانيا: خليط من المواد لتحديد نسبها في المادة النهائية MCM واختبار المكانية استخدامها در شمر ثانيا: خليط من المواد لتحديد نسبها في المادة النهائية MCM واختبار المكانية استخدامها در الشح, ثانيا: خليط من المواد لتحديد نسبها في المادة النهائية المادة واختبار المائل الحفر بعد راشح, ثانيا: خليط من المواد لتحديد نسبها في المادة النهائية MCM واختبار المائل الحفر أر ألمانه وزن ونعيبر الخواص الريولوجية لسائل الحفر والك الخلا المشكلة. في النهاية تم الوصول لأنسب وزن وتحققت الأهداف.

Table of contents

Subjects	page
إستهلال	Ι
Dedication	II
Acknowledgement	III
Abstract	IV
تجريد	V
Table of contents	VI
Table of figures	Х
Table of tables	XI
Chapter 1	
1.1 General introduction.	1
1.2 Lost circulation materials (LCM)	3
1.2.1 Additives considerations	4
1.3 Problem statement	4
1.4 Project objective	4
Chapter 2	
2.1 the geology of heglig	6
2.2 The tectonic of the area.	7
2.3 Stratigraphy column description	9
2.4 Sedimentation.	10
2.4.1 Amal formation description (250-462)	11
2.5 Literature review	12
2.6 General description of Mica	13
2.6.1 Chemical properties	15
2.6.2 Physical properties	16
2.6.3 Quality classifications	17

2.6.4 RAHABA II Mica					
2.6.5 General conclusion and recommendation					
2.6.6 The quality and grades of Sheriek sheets Mica are quite good for					
marketing					
2.7 .Neem chemical and physical properties" azadirachta indica"	20				
2.7.1 Composition of neem seed oil and cake obtained after oil	21				
extraction					
2.7.1.1 Chemical properties of AISO and CPSO	21				
2.7.1.1.1 Fatty acid composition of AISO and CPSO	22				
2.7.1.1.2 (AIC and CPC)	23				
2.7.1.1.3 Proteins and amino acids of AIC and CPC	23				
2.7.1.1.4 Mineral fraction of AIC and CPC	24				
2. 8 Chemical and physical properties of Lalob" Balanites aegyptiaca"	25				
2.8.1 Physical properties	26				
2.8.2 Chemical contracture	26				
2.8.2.1 Polymers	26				
2.8.2.2 Acid value	26				
2.8.2.3 Saponification value	26				
2.8.2.4 Iodine value (Hanus method)	26				
2.8.2.5 fatty acids	27				
Chapter 3	<u> </u>				
3.1 Preparation of loss circulation materials	30				
3.1.1 MICA grinding	30				
3.1.2 Neem and Lalob grinding	32				
3.2 XRD test	33				
3.2.1 Description	34				
3.2.2 Procedure	34				
3.2.3 Objective of test	34				
	L				

3.3 Properties test	36
3.3.1 density measure	36
3.3.1.1 Description	37
3.3.1.2 Procedure:	37
3.3.2 hydrogen ion concentration PH	38
3.3.3 6 speed viscometers	38
3.3.3.1 Description	38
3.3.3.2 Procedure for apparent viscosity, plastic viscosity and yield	39
point determination	
3.3.4 API filtration	40
3.3.4.1 Description	40
3.3.4.2 Procedure	41
3.3.4.3 Reference mud	41
3.3.4.4 API filtration using Mica material	41
3.3.4.5 API filtration using Neem material	42
3.3.4.6 API filtration using Lalob material	42
3.3.4.7 API filtration using the mixture of materials	42
3.3.5 Lost circulation Materials Test Device (DL)	42
3.3.5.1 Purpose	42
3.3.5.2 Main technical parameters	42
3.3.5.3 Characters & Working Principles	43
Chapter 4	
4.1 The properties resultant	44
4.2 API filtration device to measure the mud filtrate	45
4.2.1 Firstly the powder of Mica	45
4.2.2 Secondly the powder of Neem seed	47
4.2.3 Thirdly the powder of Lalob seed	49
4.2.4 Fourthly the mixture of three materials	50

4.3 Lost Circulation Materials Test Device Experiments	52
4.3.1 Experiment 1	52
4.3.2 Experiment 2	53
4.3.3 Experiment No 3	53
4.3.4 Experiment No 4	54
4.3.5 Experiment No 5	55
4.3.6 Experiment No 6	56
4.4 Discussion	58
Chapter 5	
5.1 Conclusion	62
5.2 Recommendations	63
References	

Figure list

Figure	Description		
no.			
1-1	Circulation system	2	
2-1	Muglad basin	7	
2-2	Cross section map of bamboo field	8	
2-3	The stratigraphy column	11	
2-4	Mica sharps	15	
2-5	Neem seed	21	
2-6	Lalob seed	25	
2-7	The schematic clarify the chemical structure of lalob	28	
3-1	Mica powder	31	
3-2	RS200 device for grinding mica	31	
3-3	Seeds mill device	32	
3-4	Neem powder	33	
3-5	Lalob powder	33	
3-6	XRD device	34	
3-7	XRD for Mica powder	35	
3-8	XRD for Neem powder	35	
3-9	XRD for Lalob powder	36	
3-10	All materials that used	36	
3-11	Mud balance device	37	
3-12	PH paper	38	
3-13	6 speed viscometer device	39	
3-14	API filtration device	40	
3-15	loss circulation device	43	
4-1	The relation between time and filtrate using 2 gm from each	58	
	material		
4-2	The relation between time and filtrate using 4 gm from each	59	
	material		
4-3	The relation between time and filtrate using 5 gm from each	59	
	material		
4-4	The relation between time and filtrate using mixture of	60	
	materials		
4-5	The relation between time, volume lost and LCM weight at normal pressure	60	
4-6	The relation between time, volume lost and LCM weight at 100 psi	61	

List of tables

Table	Description	Page			
no.		no.			
1-1	Types of loss	3			
2-1	Classify standard sizes of mica production operation	14			
2-2	Results of chemical analysis "Indian and Sudanese, Mica"				
2-3	Classification of rahaba II mica according to (A.S.T.M)	17			
2-4	Compared of the main characteristics of examined muscovite deposits with corresponding average data	19			
2-5	Chemical properties of AISO and CPSO	22			
2-6	Fatty acid composition of AISO and CPSO	22			
2-7	Proximate composition of AIC and CPC	22			
2-8	Amino acid composition of AIC and CPC	23			
2.0	Mineral fraction of AIC and CPC	23			
2^{-9}	Physical and chemical properties of laloh	24			
2.10	Fatty acid profile of lalob	20			
2-11 2-12	Percentage composition of fatty acids in Balanites kernel oil from	27			
$\Sigma^{-1}\Sigma$	different study districts	27			
2-13	The hydrocarbons in the lalob seed	28			
2-14	Composition of lalob seed	29			
2-15	Mineral composition of lalob pulp	29			
4-1	Mud reports screening	44			
4-2	Percentage of materials and filtrate for reference mud	45			
4-3	percentage of materials and filtrate for 2 gm of Mica	45			
4-4	percentage of materials and filtrate for 4 gm of Mica	46			
4-5	percentage of materials and filtrate for 5 gm of Mica	46			
4-6	percentage of materials and filtrate for 6 gm of Mica	46			
4-7	percentage of materials and filtrate for 2 gm of Neem	47			
4-8	percentage of materials and filtrate for 3 gm of Neem	47			
4-9	percentage of materials and filtrate for 4 gm of Neem	48			
4-10	percentage of materials and filtrate for 5 gm of Neem	48			
4-11	percentage of materials and filtrate for 2 gm of Lalob	49			
4-12	percentage of materials and filtrate for 3 gm of Lalob	49			
4-13	percentage of materials and filtrate for 4 gm of Lalob	49			
4-14	percentage of materials and filtrate for 5 gm of Lalob	50			
4-15	percentage of materials and filtrate of 2.2 gm mica, 4 gm neem, 8 gm lalob	50			
4-16	percentage of materials and filtrate of 2.2 gm mica, 5 gm neem and lalob	51			
4-17	percentage of materials and filtrate of 2.2 gm mica, 4 gm neem, 5 gm lalob	51			

4-18	Percentage of materials in final LCM	52
4-19	Percentage of materials in 20 gm of LCM	53
4-20	Percentage of materials	53
4-21	Percentage of materials in 50 gm of LCM	54
4-22	Quantity of lost mud and time of 50 gm LCM at normal pressure	54
4-23	Percentage of materials in 100 gm of LCM	54
4-24	The rheological properties of 100 gm LCM	55
4-25	Quantity of lost mud and time of 100 gm LCM at normal pressure	55
4-26	Quantity of lost mud and time of 100 gm LCM at pressure 100 psi	55
4-27	Percentage of materials in150 gm of LCM	55
4-28	The rheological properties of 150 gm LCM	56
4-29	Quantity of lost mud and time of 150 gm LCM at normal pressure	56
4-30	Quantity of lost mud and time of 150 gm LCM at normal pressure 100	56
	psi	
4-31	Percentage of materials in 200 gm of LCM	57
4-32	The rheological properties of 200 gm LCM	57
4-33	Quantity of lost mud and time of 200 gm LCM at normal pressure	57
4-34	Quantity of lost mud and time of 200 gm LCM at pressure 100psi	57

<u>Chapter 1</u>

Introduction

Chapter 1

Introduction

1.1 General introduction:

Drilling operation is one of the most important stages in petroleum industry. It consists of many parts like rotation system, hoist system and mud (drilling fluid) circulation system, figure (1-1).

Drilling fluids are used to provide some objectives such as:

- 1- Cooling and lubricating the equipments.
- 2- Clean the bottom hole and take cutting out of well.
- 3- Provide hydrostatic pressure to balance the formation pressure.
- 4- Transfer information from bottom to surface.
- 5- Suspend cutting when drilling stopped.



Figure (1-1): circulation system, (Adam T. Bourgoyne Jr. et.al, 1991)

Depending on the based fluid the API divided drilling fluid into three types: water based mud, oil based mud and gaseous based mud. Drilling fluid must be designed carefully to avoid most of well problems during drilling operation such as: pipe stuck, swelling, blow out and loss circulation.

Lost circulation costs the industry hundreds of millions of dollars each year in lost or delayed production and in spending to deal with drilling problems to repair faulty and replace wells irreparably damage by lost circulation (Jason T. Scorsone, et.al 2010).

Lost circulation is the reduced or loss of fluid flow up the formation- casing, or casing tubing annulus, when fluid is pumped down drill pipe or casing. Loss of fluid circulation is a familiar hazard when drilling and cementing in highly permeable reservoir , in depleted zones and in weak or natural fracture(due to poor cementing in study area), vuguler or cavernous formation. Stopping circulation losses before they

get out of control is crucial for safe and economically rewarding operations (Jason T. Scorsone, et.al 2010).

Although engineers define lost circulation in many ways, it may generally be classified as seepage when losses are less than10 bbl/hr. [1.5 m/hr] (Below) Partial lost returns involves losses greater than10 bbl/hr, but some fluid returns to surface. During total lost circulation no fluid comes out of the annulus. In this most severe case the borehole may not retain a fluid column even if the circulating pumps are turned off (Jason T. Scorsone, et.al 2010), table (1-1).

Type of loss	Severity of loss
Seepage	Less than 10 bbl/hr (1.5 m3/hr)
Partial lost return	Greater than 10 bbl/hr but some fluid return
Severe loss	Greater than 50 bbl/hr but few fluid return
Total lost circulation	No fluid comes out of the annulus

Table (1-1): types of loss (Jason T. Scorsone, et.al 2010).

The main reasons of loss circulation are either natural fractures in the formation or weak formation (low cementation material). The common treatment is using drilling fluid loss additives, (Jason T. Scorsone, et.al 2010).

1.2 Lost circulation materials (LCM)

The primary function of lost circulation additives is to block the zone of loss back in the formation away from the borehole face so that subsequent operation will not result in additional drilling fluid loss.

There are many difference types and particle sizes used as loss circulation materials (LCM). Products are both organic and synthetics man made materials. Examples of lost circulation materials are calcium carbonate, crushed mica, cellulosic plant, particles, graphite and dolomites. Plant materials that have been used are kenaf, walnut hulls, peanut hulls, coconut coir and number of others. Polymers are also sometimes used to increase the viscosity through these are most costly they are more compatible with several types of fluids systems. There are a great variety of additives which physically plug or seal the losses including sawdust, flaked cellophane and crushed or ground gypsum. Other common and cheaper additives are shredded newspaper and cotton seed hulls. Cotton seed hulls are less preferred as they may cause wear to pump swabs and springs. Both of these are generally only used when either fresh or brine water is being used for drilling fluid,(Plank J.P, et.al 1991).

1.2.1 Additives considerations:

Several factors are considered in what additives are uses,(Plank J.P, et.al 1991).

- The well bore size that is currently being drilled.
- The type of drilling fluid in use as the additives must be compatible.
- The depth of the well in regards to geological stability.
- The depth of the well in regards to production zones, plugging production zone is not desired out come.
- Drill bit nozzles sizes. If the additives will not go through the drill bit they cannot be used.
- Other drill string mechanical equipment such as a mud motor, agitator or mud tools.
- Economic constraints.

1.3 Problem statement:

Weak formations consider dangerous state that it will fracture under pressure above pore pressure. Calcium carbonate was used to solve the problem. In this project we study this problem in (bambo field Amal formation) and use mixture of LCM by using particle materials of Mica Azadirachta indica and balanites aegypytiaca as powder to solve the problem.

1.4 Project objectives:-

The main objective of project is to evaluate the effect of mixture of LCM by using particle materials of Mica, Azadirachta indica and balanites aegypytiaca as powder to solve the problem.

Other sub objectives:-

1- Design local product with international standards that participate in oil industry.

2- Minimize the quantity of barite used in mud design.

- 3- Prevent the occurrence of loss circulation events in the area of study.
- 4- Solve the LCM scarcity problem.

Chapter 1 is an introduction about drilling fluid, problem statements, and objectives of research, Chapter 2 literature review, Chapter 3 methodology of the project, Chapter 4 presence result and discussion and Chapters 5 conclude with the conclusions and recommendations

Chapter 2

Literature review

Chapter 2

Literature review

2.1 The geology of Heglig:-

The Muglad basin is the largest known rift basin in the Sudan interior, running northwest–southeast and covering about 120,000 km². The basin is around 800 km long and 200 km wide, (GNPOC planning section, 2015), figure (2-1).

The area is divided into four producing fields namely Bamboo main, Bamboo West, Bamboo South and bamboo east. The first discovery well was drilled in 1982 by Chevron. Bamboo main is an anticline, having structural closure and the other fields (West, east & South) are situated on the up thrown blocks of NW- SE fault providing lateral seal for the hydrocarbon accumulation. Greater Bamboo (Bamboo main, West, South and East) are major fields in block 2A with 51 producer wells. These fields are currently producing a total of 48,951 blpd liquid, which constitute 6.2% of GNPOC oil production. The field is experiencing a rising trend on water cut. Rapid increasing in water cut is effecting the oil production and also becoming a cause of concern for water disposal. These fields are multi blocks, multi layered sandstone reservoirs of late cretaceous age with aerial extent. The depth varies from 1000 to 1700 m. Average porosity and permeability is good. Fields have strong aquifer support in Bentiu as the major reservoir. The viscosity of the fields is relatively high and varies from 120-1100 cp. The area is a part of the Late Jurassic to Early Cretaceous Sudanese Muglad Rift Complex. A thick sequence of Mesozoic to Tertiary sediments has been penetrated in Bamboo area. 51/52 wells were drilled in the 3D area and 2D areas of Bamboo main field of block 2A Muglad basin GNPOC concession (GNPOC planning section, 2015).

The primary hydrocarbon reservoir is Bentiu while Aradeiba is the secondary reservoir. The field is located in the Greater Bamboo 3D (GNPOC planning section, 2015).

Seismic area of Block 2A of GNPOC concession in Muglad basin. Structurally, Bamboo main is an anticline, having structural closure and the other fields (West, east & south) is situated on the up thrown blocks of NW- SE fault providing lateral seal for the hydrocarbon accumulation. It is a fault bounding structure. The structure of Bamboo is located at the tilted up thrown side of the normal fault block.



A - A' Cross Section

Figure (2-1) Muglad basin (GNPOC, 2015)

2.2 The tectonic of the area:-

In 1988 schull divided the structure of the Sudan into 5 phases depending on the geophysical data, well information and regional geology. The 5 phases: prephases, three rifting phases and sag phases. The three rifting happened during the early Cretaceous, late Cretaceous and early middle tertiary. (McHargue et al., 1992). These episodes of rifting resulted in the deposition of a sedimentary section up to 13 km thick in the deep troughs comprising that three depositional cycles the basin stratigraphy : commonly the rift sediments consider as fine grained and the sag phase sediments are coarser grained the strategraphic frame work of the muglad basin shows unconformities with a slight angular discordance that terminate the cycles, which probably reflect the uplift due to fault block rotation; the extent of the erosion, however, is uncertain. The positioning of the unconformities within the unfossiliferous sections is contentious, and proposed that both the Bentiu and Amal formations straddle unconformities. (McHargue et al., 1992) figure (2-2).



Figure (2-2): cross section map of bamboo field,(Sudapet CO, 2015).

2.3 Stratigraphy column description:-

In general, the synrift sediments are fine-grained and the more recent thermal subsidence phase (the sag phase) sediments are coarser-grained. The general stratigraphic framework for the interior Muglad basin shows unconformities with a slight angular discordance that terminates the cycles, which probably reflect the uplift due to fault block rotation; the extent of the erosion, however, is uncertain. The positioning of the unconformities within the unfossiliferous sections is contentious, also proposed that both the Bentiu and Amal formations straddle unconformities. The first depositional cycle (Early Cretaceous) consists mainly of suboxic organic-rich shales comprising the main lacustrine source beds of the Sharaf and Abu Gabra formations, which are overlain in the sag phase by the medium- to coarse-grained sandstones of the Bentiu Formation. The second depositional cycle (Late Cretaceous-Paleocene) is the Darfur Group, comprising fluvial and deltaic clay stones at the bottom (Aradeiba Formation) and thin sandstone beds (Zarga and Ghazal formations), thickening toward the top of the section (Baraka Formation) and overlain by the coarser Amal Formation. The thin intercalating sandstones in the Darfur Group are the main reservoirs in the Unity field. The Kordofan Group (Oligocene-late Eocene), which forms the third depositional cycle, consists of the largely shaly Nayil and Tendi formations and culminates in the coarse sandstones of the Adok Formation. The Miocene-Holocene Zeraf Formation unconformable overlies the Adok and probably represents fluvial reworking of these earlier deposits, (McHargue et al., 1992).

2.4 Sedimentation:- Amal formation predominantly massive white coarse grain quartz sand interbeded clayers. It consider as the sag of the second cycle .It was sedimented at the Tertiary period (Paleocene). The sandstone is buff, occasionally tan, poorly consolidated, fine to medium grained, subangular, to subrounded and moderately sorted.





2.4.1 Amal formation description (250-462ft):-

From (250-285ft): the sand stone is transparent to translucent, unconsolidated, fine grain, trace medium grain, sub angular to sub rounded, well sorted, quartz, trace mica, rare carbonaceous matter, fair porosity, no shows, with minor interbeded, **clay stone**: light brownish grey, minor dark grey, soft, occasional firm, sub blocky, earthy, trace micro micaceous, rare chlorite (GNPOC, final well report, bamboo west-7).

From (285-307ft): clay stone interbeded with sand stone (GNPOC, final well report, bamboo west-7).

From (307-356ft): sandstone is transparent to translucent, trace orange, unconsolidated, fine to coarse, grained, common coarse grained, sub rounded to rounded, poorly sorted, quartz, trace mica, trace pyrite, rare carbonaceous matter, fair to good porosity, no shows with minor interbeded, **clay stone**: light brownish grey, minor dark grey, soft, sub blocky, occasional massive, earthy, minor sticky, trace micro micaceous,(GNPOC, final well report, bamboo west-7).

From (356-390ft): sandstone: clear, translucent, trace orange, unconsolidated, predominant coarse grain, occasional medium grained, sub granular to sub rounded, well sorted, quartz, trace pyrite, rare carbonaceous matter, good porosity, no shows, (GNPOC, final well report, bamboo west-7).

From (390-420ft): sandstone: clear, translucent, trace orange, unconsolidated, abundant medium grained, minor fine grained, trace coarse grained, sub granular to sub rounded, moderate sorted, quartz, trace pyrite, trace iron staining, fair porosity, no shows, (GNPOC, final well report, bamboo west-7).

From (420-462ft): sandstone: clear, translucent, trace orange, unconsolidated, predominant medium grain, occasional coarse grained, sub granular to sub rounded, well sorted, quartz, trace pyrite, rare iron staining, good porosity, no shows, (GNPOC, final well report, bamboo west-7).

2.5 Literature review:

The further that indicate the lost circulation in Amal resultant to that reduces formation pore pressure, then when it drilled by minimum hydrostatic pressure the mud fracture the formation and lost circulation of mud.

Most granular material proposed as loss circulation material in many wells. The reason of using granular material was its wide range of applicability and the unique feature which this type of processing produced, (Gockel C.E Brinemann, 1987).

Sulfonate polymer used as fluid loss additive in water based drilling fluid. The main advantage of polymer was temperature stability up to 400° F, salt tolerance up to saturation, hardness tolerance up to 75000 ppm calcium ions and complete neutral effect on viscosity, (Plank J.P, et al, 1988).

Paper presented about maximizing drilling time by minimizing circulation losses. That paper reviewed the newly developed high fluid –loss, high-strength (HFHS) lost circulation solution and compared it to other technologies that are currently offered in the field for similar applications, and found that it exhibits superior resistance to shear over a wide density range, and also when mixed in different base fluid, (Jason T.Scorsone, et al, 2010).

Review of lost circulation materials and treatments with an update classification presented. The paper depends on both physical and chemical properties and the application of materials to classify it into: granular, flaky, fibrous, mixture of LCMs, High Fluid Loss LCMs squeeze, Swellable/Hydratable LCMs Combinations, nanoparticles, (Murtada Alsaba and Runar Nygaard, 2014).

2.6 General description of Mica:-

Mica is a group name for sheet-like silicate minerals of which muscovite biotite and phologopite are the most important minerals, chemically mica is a complex silicate of sodium potassium and aluminum, however the chemical composition is not of important of any industry except of some uses of ground mica where the content of iron oxide present as an impurity should be very low. Muscovite occurs in a range of colors from the so called "ruby" to light and dark green with numerous other intermediate shades ruby color is usually preferred for some industries. Muscovite mica is not affected by heat till temperature of 1050 F, where it is starts' loosing it is water of crystallization ti is completely loose water at (1500-1800) F. Mica is widely distributed through the world, but commercially valuable book and block mica concentrated are scarce. The most important mica deposits in sudan occurred in the rubatab area, nile province. They extend along the western bank of the nile between berber and Abu Hamed, but the most interesting mica outcrops concentrate in the region between zuma and zuaira villages and up to jebel Rahaba and Jebel Razam to the west of the Nile. The area is composed of metasediments of the basement complex intruded by different types of igneous rocks. It is believed that the mica bearing pegmatitic is associated with the young granitic intrusions in the area. An experimental project for mica production was started in march, 1974. The project started with two leased areas Rahaba I and Rahab II, (Geological& Mineral Resources Department. "Mica Deposits in the sudan").

Rahaba II mine opened as underground .The calculated ore reserves down to depth of thirty five meters are 90,000 tons of mica books. The development of the mine now reached 22 meters in depth and gave very good results ,since march 1978 Rahaba II is considered as commercial body the other mine Rahaba I is under development and the

result till now are very encouragement its expected that in six month time Rahaba I will be another commercial unit. The topography of the rubatab area is characterized by amonotony of low relief. Several features namely gebel Bafd1 in the extreme north, G. Razzam, G. umm Sheriba, G. Absol, G. Qarn Dam, El Tor, G.Danab EL-kelb, G.Nakharu and G. Umm Arafibia in the extreme south; G.Barga to the south – east of the shereik, rise through this low- flying undulating monotonous country. These comprise Elkoro, Abusalam, Abusaiyal, Abusol ,Elrahaba (EL I th-nein) ELHad, Dam EL Tor, Umm Sarih and a few Khors. The process that the company used for cutting mica was, after began of mica project it was trained the local labors on knife cut, they did this to avoid air inclusion and they were trained and can be considered skills labors. Bates Company was prepared mica after mining, it was transported to the cutting centers, they used knife cut to avoid air inclusion. Then they putted the standard bellow to define the criteria that they used to determine it is size, area and minimum diameters of one side, (Geological& Mineral Resources Department. "Mica Deposits in the sudan"), table (2-1)

Table	(2-1):	Classify	standard	sizes	of	mica	production	operation,	(M.L.
KABE	SH, 19	60)							

SIZING(diameter)	AREA(sq in)	Minimum diameters of one side (in)
1	24	3
2	15	2
3	10	2
4	6	1.5
5	3	1
5.5	2.55	7/8
6	1	3/4



Figure of (2-4): mica sharps

2.6.1 Chemical properties:

In the chemical composition muscovite is a silicate of aluminum and potassium, but usually small amount of magnesium and iron are present. Also for most industrial purposes the physical properties of mica are more important than chemical composition for some uses the latter is an important concentration, (M.L. KABESH, 1960)

Table: (2-2) results of chemical analysis "Indian and Sudanese, Mica", (M.L. KABESH, 1960)

COMPONENT	SUDANESE Rubatab muscovite %	INDIAN %
AL2O3	35.1	36.72
F2O3	2.3	0.95
TIO2	0.4	-
CAO,BAO	-	0.21
SIO2	45	45.57
K2O	9.8	8.81
NA2O	1	0.62
Mature H2O	4.6	5.05

Muscovite is chemically stable and is not easily affected by oil, water or acid and can be heated to dual redness with little ill effect, (M.L. KABESH, 1960).

2.6.2 Physical properties:-

The physical properties of muscovite however are those which determine it is value cleavage flexibility with which it can be cut, color, staining, size, resistant to heat and electrical current. It is the last factor that the muscovite is superior to most other mica.

The de-electrical properties, unfortunately, the de electrical strength, the de electrical constant, the power factor and the volume resistant of the rahaba II muscovite have not been studied in detail.

The flexibility of the Rahaba mica should be described as moderate to good and the thermal properties are good .Some pinholes, commercially known as "pin pits", were recognized in a few samples. The Rahaba muscovite is hard and it is possess good splitting properties. To describe mica diaphaneity the company was take the samples from depths 2-3 meters were with very mineral stains, vegetable stains or cross graining, Some silver stains were present due to air bubbles and patches gave them a silvery appearance by reflected lights.

They divided the diaphaneity to two classes according to defect due impurities and structural defect due to impurities, these are further divided in to two sub-classes stains and spots, the most common impurities films of clay staining takes place in the zone of surface weathering by the penetration of muddy water between the laminae. The solution penetrate large areas of crystals and work between many of the laminae is generally obtained at some distance from the surface and is abcent where mining reaches hard and un altered pegmatite body.

Inclusion plates of of brittle silica are sometimes found lying between the lamination of mica and forcing them apart this inclusion of silica as muchas one cm thick.

Thickness examination of mica was checked by standard thickness seven mills and more (mill=1/1000 in). Bellow 7 mills rejected to be opened to consider films and book form splitting, (M.L. KABESH, 1960).

2.6.3 Quality classifications:-

Although the standardization of the quality of mica is controversial subject attempt have been made to define and describe latterly the various commercially quality of mica as a previously stated, the rahaba II mica should be classified as ruby mica.

According to the standard specification of the American society for the testing of material (A.S.T.M), the rahaba II mica falls largely under the "good stained "and "stained" and sometimes heavily stained, table (2-3).

Table (2-3): Classification of rahaba II mica according to (A.S.T.M) (M.L.KABESH, 1960).

1	Clear or slightly stained	CL/SS
2	Fair	F.S
3	Good stained market	G.S.M
4	Good stained first	G.S.T
5	Stained first	ST-I
6	Stained A	ST-A
7	Stained B	ST-B
8	Heavy stained	H-S
9	Densely stained	D.S
10	Walsher mica	

SIZE or (grade) mica was graded by bates company (1971) according to be commercially valuable, mica book must of such size as be permits trimming to at least (1.5*2) square in chess for sheet mica. All mica that will not split or rift to this minimum dimension is called scrap mica. Mica in the Rahaba II usually graded to ten sizes which mainly conform to worldwide practice, (M.L. KABESH, 1960).

2.6.4 RAHABA II MICA:

As seen in hand specimen is muscovite. They classified it as ruby mica although some samples are considered between ruby and non ruby mica (but rahaba II almost consisted of ruby mica more than non ruby, this is the reason why it selected and preferred rahaba II).

Historically evident from the previous minerals that all types and grades of muscovite mica are available in the area and from the previous study that's also containing scrap.

Trenching stripping and open cast method as done by the company showed that muscovite books are distributed with different pattern in the different parts of the same pegmatite (that was distributed in rahaba II).These was Controlled by chemical and physical properties.

The reason that the Bates company was select rahaba II mica was due to it is concentration of pockets of book mica attaining larger size and feldspar rich, the same rule was applied (i.e. mica disseminated in ground mass of k feldspar either fresh or altered also disseminated in quartz mica, (Geological& Mineral Resources Department. "Mica Deposits in the sudan").

The recovery of Rahaba II mica was estimated at 2000 ton ,then Bite company produced about 700 ton till 1971 and the production continued in difference ranges, (ar.m.wikipedia.org/wiki, ministry of mineral publication, seen at 9:48 am 11.march.2015, last altered 2015).

2.6.5 General conclusion and recommendation:

This study was done by Bates Company in the shereik area especially in rahaba II, table (2-4).

 Table (2-4): Compared of the main characteristics of examined muscovite

 deposits with corresponding average data, (M.L. KABESH, 1960).

|--|

Rahaba11	17.8% 290	0.4	42.5	
Rahabal	16% 280	0.2	50	
Abaza1	5.5% 91	0.1	48	
Abaza11	3.6 % 59	>0.1	38	
Kanisa	1.8% 29	>0.1	40	
Brazil	5-20UP TO 40			1960
India				1960
USA	2-6UP TO30-40			1960

2.6.6 The quality and grades of Sheriek sheets Mica are quite good for marketing:

It is supposed that in populated areas with adequate transportation facilities, muscovite deposits with reserves of 100-200 tons of mica may be regarded as worthy of exploitation (Treabovaniya, 1967).

Taking into consideration that during detailed prospecting and exploration of rahaba1 and rahaba11,mica deposit having more than 20 tons of muscovite were extracted, it is reasonable to assume that the above mentioned reserve of mica exist here. To be marketable, mica has to meet the world standard and be produced at a primary cost lower than the market price .this mean that to profitable, a mica enter price must be well organized, equipped and staffed with skilled laborers. The later is especially important because initial treatment of the mica and its preparation for marketing are almost exclusively manual work.

In Sudan ,the processing of mica is not atraditional occupation as it is ,for example, in India there for ,any plans to exploit mica deposits in the shereik area must include the training of staff in the treatment of mica.this is only economically viable on a long term employment basis, (that is woeking mica deposits during aprotracted period of time). The next step in the exploration of the most promising mica deposits-rahaba11, rahaba1, abaza1, perhaps elkoro-is tracking in depth the pegmatite bodies, drilling the mica bearing zone must be following by sinking pits or small shafts. the localities rahaba and daghma deserve further examination .it is recommended that they be mapped(scale1:500)and that bulk sample of mica –bearing rocks be taken to determine the quantity ,quality and grades of the muscovite there. It's advisable to prospect in rahaba and after wards, the elkoro pegmatite fields (scale 1; 500), using geophysical methods of investigation (magneto metric and radiometric surveys).

New mica bearing pegmatite bodies ,including some which are unexposed, probably will be discovered during the course of this work feldspar will be a natural byproduct of exploitation of mica deposits and its utilization will make the mining more profitable .therefore , it's necessary to test the technological properties of shereiks feldspar and to estimate it reserve. shereik region in the broad sense, that is beyond the area investigated by the project .is worthy of the geologist attention the presence of old gold working on marry island, north of shereik and jebel abu nahl granitic ring complex in the upper reaches of khor rahaba indicate that some other economics metals and minerals, in addition to those described in this report ,can be found in the area. Geological mapping and prospecting(scal1:500) including magneto metric and radiometric surveys and mineralogical examination of heavy fraction of materials filling the valleys of dry water corses are recommended for further investigation of this part of the northern province of the Sudan.

For the time being, muscovite is the main is the main economic mineral of the shereik area but, a number of other economic rock and minerals were recorded, and still other will probably be discovered in the further, (Geological& Mineral Resources Department. "Mica Deposits in the sudan").

2.7 Neem chemical and physical properties" azadirachta indica":

In African savanna there are several types of forest tree species used for food, wood or Traditional medicinal purpose also there is a large variety of oil-bearing plants used for Food, lighting and for industrial purposes.Meliaceae families are well known in Africa because it has many suitable uses and cover Size range from forest trees to small shrubs.Theneemtree (Azadirachta indica) is native to tropical South East Asia and belongs to Meliaceae family. It is fast growing ,can survive drought , poor soil and very hot temperature up to 44°C and as low as 4°C. It is a tall tree,up to 30
meters high with leafy spreading branches. The seed of neem (figure 2-5)is about 1.5 cm long.Neem (azadirachtaindica) is an ever green tree and used for medicinal purpose and pest control.The neem seeds contain about (30-50)% of oil and other many active components,Neem fatty acids (FAs) comrise oleic,stearic,palmitic and linoleic acids, mainly used for pharmaceutical industries. The fatty acid composition of the oil may vary from tree to tree because of genetic make-up (Faye, 2010; singh et al., 1999).Neem cake has been used as animal feed for a long time we

report here on the chemical composition of A. indica oils (AISO and CPSO) and cakes (AIC and CPC): FA composition, acidity, iodine values, amino acids, and carbohydrates, (Mulholland et al, 2000).



Figure (2-5): Neem seeds before grinding

2.7.1 Composition of neem seed oil and cake obtained after oil extraction:

Fresh A.indica was collected and the seeds were separated manually, cleaned and dried. The dried seeds were ground in a mill and screened through a mesh Tindo Se'bastien Djenontin, et al, 2012).

2.7.1.1 Chemical properties of AISO and CPSO:

After extracting the oil from powdered whole seeds by using extractor, the chemical properties of AISO and CPSO were determined, (Tindo Se'bastien Djenontin, et al, 2012), table (2-5).

Table (2-5): Chemical properties of AISO and CPSO, (Tindo Se'bastien Djenontin, et al,2012).

	Property	AISO,this work	AISO ^b	CPSO,this work	CPSO ^C
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Oil content(wt%dry almond)	44 ±1.3	30-51.6	61.5±3.1	48-52
Oleic acidity (wt%)	6.8 ±0.9	3.9-5.6	6.4±0.5	14.0
Saponification value(mgKOH)	167 ±1.4	195-202	188±3.2	195-201
Peroxide value(meq O2kg-1)	5.3 ±0.5	6.0-7.2	15.5±0.5	-
Iodine value	75 ±1	69-76	69±1	37-70
Unsaponifiable(wt%)	1 ±0.1	0.5-2	1.2±0.1	0.8-3
Total phospholipids(wt%)	0.3 ±0.1	-	0.04±0.01	-

2.7.1.1.1 Fatty acid composition of AISO and CPSO:

Fatty acid were obtained through a two steps method with sodium methoxyde and HCl as catalysts, and then analyzed by capillary column gas chromatography (GC) (Hewlett Packard HP 6890) equipped with a Flame-ionization detector (FID), Tindo Se'bastien Djenontin, et al, 2012) table (2-6).

Table (2-6): Fatty acid composition of AISO and CPSO, Tindo Se'bastien Djenontin, et al, 2012).

Fatty acid (wt%)	AISO,this work	AISO ^b	CPSO,this work	CPSO ^c
Palmitic acid C16:0	17.8±0.1	17.3-34.3	21.1±0.4	22.6-26.4
Palmitoleic acid	0.2±0.0	0.12-0.13	0.4±0.0	0.5-2.4
C16:1				
Stearic acid C18:0	17.4±0.5	6.6-24.0	8.5±0.2	6.2-11.9
Oleic acid C18:1	43.5±0.4	25.4-57.9	59.1±1.0	37.1-48.9
Linoleic acid C18:2	18.7±0.2	6.2-24.3	8.1±0.3	14.4-25.4
Linoleic acid C18:3	0.6±0.0	0.37-0.51	0.1±0.0	0.5-0.8
Arachidic acid C20:0	1.3±0.1	1.24-1.3	1.2±0.1	1.5-2.9
Gadoleic acid C20:1	0.1±0.0	-	0.2±0.0	-
Behenic acid C22:0	0.3±0.1	0.23-1.73	nd	-
Unknowns (%)	0.01	-	1.3	-

Nd : not detected

b Source: Kaushik and Vir (2000) and Faye (2010).

c Source: Vieux et al. (1970) and Miralles (1983).

2.7.1.1.2 (AIC and CPC):

After extracting the oil, the powdered seeds were analyzed in order to know some properties like: Moisture, protein, lipid, ash, total carbohydrate, sugar, starch and crude fibre contents. The minerals in defatted cakes were analyzed from solution obtained by first dry – ashing the seed flour at 550 \circ C, (Tindo Se'bastien Djenontin, et al, 2012), Table (3-7).

Table (2-7): Proximate composition of AIC and CPC, (Tindo Se'bastien Djenontin, et al,2012).

Constituent (wt%)	AIC,this work	AIC ^{b,c}	CPC,this work	CPC ^d
Moisture	4.6±0.3	9.03-10.24 ^b	7.6±0.3	12.5-12.7
Ash	5.3±0.2	4.45-18 ^b	8.3±0.5	4.8-7.2
Oil	6.4±0.1	6.4 ^b	19.2±0.1	4.5-9.9
Proteins	31.5±0.5	12.25-50 ^b	20.2±0.5	16.8-26.3
Starch	1.10.4	-	2.9±0.4	-
Total sugars	9.6±0.4	-	12.4±0.1	-
Crude fibre	4.1±0.1	3.31-8 ^b	12.1±0.1	26.1-35.1
Lignins	1.9±0.1	2.15 - 2.18 ^c	7.7±0.2	-
Total	81	26-94 ^b	95	64.7-91.2

b Source: Rao (1987), Saxena et al. (2010)and Gowda and Sastry (2000).

c Source: Faye (2010).

d source: Vieux et al. (1970) and Miralles (1983).

2.7.1.1.3 Proteins and amino acids of AIC and CPC:

The proportion of proteins in AIC (31wt%) is comparable to that of other samples of neem (Saxena et al., 2010 and Faye, 2010) we found that it is lower than the protein content of soybean cake (Lusas and Hernandez, 1997), but it is not far from the seeds of balanites aegyptiaca (36.8wt%) (Cook et al., 2000).

Table (2-8): Amino acid composition of AIC and CPC, (Tindo Se'bastien Djenontin, et al,2012).

Amino acid(g100g)	AIC,this work	AIC ^c	CPC,this work
Arginine	2.7±0.1	0.57-0.73	2.0±0.1
Histidine	0.5±0.1	0.16-0.21	0.5±0.1
Isoleucine	1.2±0.1	0.33-0.6	0.7±0.2

Leucine	2.4±0.2	0.71-0.95	1.6±0.1
Lysine	1.2±0.1	0.28	0.9±0.2
Methionine	0.4±0.1	0.14-0.7	0.2±0.0
Phenylalanine	1.4±0.2	0.62-0.8	1.0±0.2
Threonine	1.0±0.2	0.3-0.5	0.8±0.1
Valine	1.8±0.2	0.48-0.76	0.9±0.1
Alanine	1.6±0.1	0.46	1.1±0.1
Aspartic acid	2.7±0.3	1.17-1.31	2.0±0.2
Glutamic acid	8.6±0.3	2.40-2.42	4.7±0.2
Glycine	1.4±0.1	0.39-1.08	1.1±0.1
Serine	1.4±0.1	0.46-0.58	1.1±0.1
Tyrosine	0.7±0.1	0.26	0.6±0.0
Cystine	0.1±0.0	0.34-1.73	0.2±0.0
Proline	1.6±0.1	0.84	1.9±0.2
Unknowns	0.8±0.2	-	nd
Total proteins	31.5±0.5	12.25-19.40	20.2±0.5

nd: not detected

bDry weight of defatted cake.

c Source: Gowda and Sastry (2000).

The amino acids composition of AIC is close to that of soy bean according to Lusas and Hernandez (1997). It shows the possible use as animal feed (Saxena et al., Faye, 2010), and consider as cheaper protein source.

2.7.1.1.4 Mineral fraction of AIC and CPC:

AIC contain, 5.3 wt% of ash as shown in Table below, also rich in nitrogenated and the presence of N, P, and K especially for AISO would allow it is use as fertilizer, (Tindo Se'bastien Djenontin, et al, 2012), table (2-9).

Mineral	AIC,this work	AIC ^C	CPC, this work	CPC ^d
N (wt%)	8.1±0.1	4.7	40±.1	-
P (wt%)	0.8±0.0	0.30-0.5	0.7±0.0	-
Ca (wt%)	0.3±0.0	0.3-0.5	0.2±0.1	-
Mg (wt%)	0.5±0.0	0.3-0.96	0.5±0.0	-

 Table (2-9): Mineral fraction of AIC and CPC

K (wt%)	2.6±0.0	0.3-0.44	3.6±0.1	-
Na (ppm)	202.0±2.0	400	1574±15	-
Fe(ppm)	107±1.0	75-2705	97±2.0	-
Mn(ppm)	31±2.0	25-70	16±2.0	-
Cu(ppm)	25±0.5	19-33	32.0±2.0	-
Zn (ppm)	56±2.0	19-56	41.0±0.5	-
Ash (wt%)	5.3±0.3	5.5-13.9	8.3±0.5	4.8-7.2

a Values are expressed as mean of three samples each analyzed in triplicate.

b Dry weight of defatted cake.

c Source: Schmutterer (1995), Faye (2010), Rao (1987), Saxena et al. (2010), Gowda and Sastry (2000).

d Source: Vieux et al. (1970) and Miralles (1983).

The present study provides the composition of two selected oil seeds of two meliaceae, A. indica and C.procera, it also determine components amounting and chemical properties of the seeds of neem, (Tindo Se'bastien Djenontin, et al, 2012).

2.8 Chemical and physical properties of Lalob" Balanites aegyptiaca":-

Lalob which known as balanites aegyptiaca is one of the desert tree and has many uses as edible seed (figure 2-6), wood of fuel, charcoal, timber and fodder. It has high medicine value (Sheded; Pulford and Hamed; 2006).the outer part of the seed used to cure skin diseases, hypoglycemic agent, root bark as anti malaria and promising for HIV (Alashaal et al,2010; Cook et al,1998).



Figure (2-6): Lalob seed

2.8.1 Physical properties:-

Color: was described by using the color nomenclature namely, red, orange (mixture of red and orange), yellow, green (mixture of green and yellow) in the addition of bright or dull, (C.A.OKIA et al, 2013)

Refractive index: was defined by using Bellingham Stanly refract meter, (model number A86006), (C.A.OKIA et al, 2013)

Viscosity: was defined by using viscometer standardized using viscosity standard fluid from Brookfield, (C.A.OKIA et al, 2013)

2.8.2 Chemical contracture:-

2.8.2.1 Polymers:-

- **Cellulose**: It is cracked in the presence of high acidic media, and then it is swelled when it is absorbed water, has no effect on sandstone and does not ferment during time.
- Lignin: Is a complex polymer of aromatics alcohol. It is an organic compound commonly found in the cell wall of plant cell making them hard and protective. It is affected by basicity of the media.
- **Hemi cellulose**: Has a random amorphous structure with little strength. It is easily hydrolyzed by dilute acid or base as well as myriad enzymes.

2.8.2.2 Acid value: this test was done by to find the acid value of lalob seed, (C.A.OKIA et al, 2013), table (2-10).

2.8.2.3 Saponification value: the saponification value measure the number of milligrams of KOH required to saponificate 1 gram of fat under certain condition. This test was done to find the saponification value of lalob seed. (C.A.OKIA et al, 2013), table (2-10).

2.8.2.4 Iodine value (Hanus method): the iodine number measure the quantity of lode in 1 gram of subtends. This test was done to find the iodine value of lalob seed by Hanus meyhod, (C.A.OKIA et al, 2013), table (2-10).

|--|

Property	Katakwi	Adjumani	Moroto district	Mean
	district	district		

Physical						
Color	Yel-Gr+9.9Y	Yel-Gr+9.3Y	Yel-Gr+13.3Y	Yel-		
				Gr+10.8Y		
Refractive index	1.461	1.46	1.46	1.46		
Viscosity	22.60	18.94	23.04	21.53		
Chemical						
Saponification value	192.80	185.55	180.50	186.28		
(mgKOH g-1)						
Acid value(mgKOH g-1)	1.41	1.33	1.95	1.56		
Iodine value(Ig 100g-1)	98.02	100.04	103.03	100.52		

2.8.2.5 Fatty acids: The fatty acids are carboxylic acid have straight chain, may be find in the form of saturated or unsaturated acid, to find the fatty acid value of lalob seed, by (C.A.OKIA et al, 2013), table (2-11) (2-12).

Weight ±SE Fatty acid Palmitic 15.40 ± 0.26 Stearic 19.01±0.29 Oleic 25.74±0.35 Linoleic $39.85{\pm}0.48$ 34.41 ± 1.80 Saturated Unsaturated 65.59 ± 6.92 Unsaturated/saturated acid ratio 1.91

Table (2-11): Fatty acid profile of lalob, (C.A.OKIA et al, 2013)

Table (2-12): Percentage composition	of fatty acids	in Balanites	kernel o	il from
different study districts, (C.A.OKIA et	t al, 2013).			

Chemical constituent	Acetone	Methnolic
	extract	extract
Saponins	+ve	+ve
Tannins	+ve	+ve
Flavonids	-ve	-ve
Alkaloids	-ve	-ve
Glycosides	-ve	-ve
Volatile oil	+ve	+ve
Terpens	+ve	+ve

The schematic clarify the chemical structure of lalob, (C.A.OKIA et al, 2013), figure (2-7).



Figure (2-7): The schematic clarify the chemical structure of lalob, (C.A.OKIA et al, 2013)

Other content that lalob consist of them such as hydrocarbons, ash and minerals, tables (2-13) to (2-15).

carbon number	Unripe	Partially ripe	Ripe
n16	Т	Т	Т
n17	Т	Т	Т
n18	Т	Т	Т
n19	1.8	2.6	4.1
n20	1.8	2.3	2.4
n21	1.8	3.2	4.5
n22	1.8	1.9	2.4
n23	10.5	18.8	21.3
n24	5.4	4.5	4.1
n25	28.2	23.6	21.3
n26	4.7	3.9	5.3
i27	Т	Т	Т
n27	24.7	20.7	16.5
n28	3.6	4.9	4.5
n29	13.8	9.7	8.3
n30	1.8	2.3	2.8
n31	Т	Т	2.4
n32	Т	1.7	Т
N33	-	Т	Т

Table (2-13): the hydrocarbons in the lalob seed, (C.A.OKIA et al, 2013).

Parameter		Content(mean±SEM)
	Total sugar	42.57±0.35
Organic element(g 100 g-1 DM)	Proteins	9.57±0.01
	Fat	0.41±0.01
Ash(mg 100 g-1 DM)	-	9.06±0.26
Humidity (%)	-	16.31±1.07
Titratable acidity(meq 100 g-1)	-	17.5±0.1
	Ascorbic acid	6.87±0.1
Vitamins(mg 100 g-1 DM)	Carotene	-
	Tocopherol	-

Table (2-14): composition of lalob seed, (C.A.OKIA et al, 2013).

 Table (2-15): Mineral composition of lalob pulp, (Moustapha Bassimbé Sagnal, et al., 2014)

Mineral	Content ± SEM(mg 100 g-1	Mineral	Content ± SEM(mg 100 g-1
	dm)		dm)
K	2220 ± 0.06	Fe	4.94 ± 0.01
Ca	141 ± 0.01	Zn	0.65 ± 0.01
Mg	73 ± 0.03	Cu	0.39 ± 0.01
Na	48 ± 0.02	Mn	0.33 ± 0.01
Р	48 ± 0.02	Se	0.05 ± 0.01

<u>Chapter 3</u> <u>Methodology</u>

Chapter 3

Methodology

Mud circulation system is considered as necessary unit in the drilling operation, and drilling fluid that used in normal drilling to formation when there is no problemoccurs during drilling operation. When prospecting problem or suddenly happened, that compels us to modify the mud properties by adding additives to exceed it. Generally problem that counterpoised us in **Amal** formation represent in (loss circulation). After resultant to daily mud report from two different wells inorder to determine the optimum properties for the drilling fluid.

Depending on the properties of surface section of bamboo area in (heglig field, muglad basin)the data correlated that's resultant from the reports and obtained. After placement the data, the major material quantified and weighted to prepare the drilling mud represented in (barite, bentonite, pac lv, water).

3.1Preparation of loss circulation materials:

Neem and Lalob seed are collected from national forests seeds center and Mica is obtained from ministry of minerals. After that these materials are formed in powder form by grinding those using different devices.

3.1.1 MICA grinding:-

The grinding process of mica is done in stages after cutting it into sharps ,and it will grinded in uniform shapes in small sharps as possible and then insert it in cyclic metal called "hole" then entered it in grinding machine called **Vibration mill device** (**RS200**).

After grinding then it produced a powder from more parts of mica sharps ,then more of sharps still need grinding to be powder we put it again in the hole for the second time to transform it completely to more soft powder as minimum as possible depending on its sizes and surface area, figure (3-1).



Figure (3-1): mica powder (ministry of mineral lab)



Figure (3-2): vibration mill device for grinding mica (ministry of minerals)

3.1.2 Neem and Lalob grinding:-

Neem and lalob seeds are being collected from National Seed Center. The grinding mechanism of these materials began after cleaning them carefully and removes the upper cover and other undesirable substances from them.

In grinding process used conventional classic mill (figure (3-3)) that used to grind varies substances in common markets. Seeds are placed in the orifice of the mill and the process of grinding spent about a quarter hour (15 min) for each material alone.

The next step is to sizing the powder particle of those materials using sieve shakers. Neem and lalob powder are putted in 355micron mesh screen and shaked carefully in order to collect the suitable size of powder which pass through the mesh screen. Also Mica was screened with that sieve but, 375micron mesh screen; figure (3-4) (3-5).

Then prepared the drilling fluid by mixture device to mix the certain quantity of materials (bentonite, barite, pac-lv, mica neem and lalob) with water after weighting using sensitive balance, and then the properties are detected as the details



Figure (3-3): seeds mill device(Omdurman market)



Figure (3-4): Neem powder



Figure (3-5): Lalob seed and powder

3.2XRD test:

X-ray diffraction (XRD) is one of the most important characterizations tools used in solid state chemistry and materials science. X-rays enabled scientists toidentify crystalline structure at the atomic level.X-ray diffraction has been in use in two main areas, for theFingerprint characterization of crystalline materials and the determination of their structure. Also can used to un crystallized material such as (neem and lalob) seeds, figure (3-7) (3-8) (3-9).

3.2.1 Description:-

The device (figure 3-6) consist two basics part:-

1-a monochromatic beam

2-filter

There is a source emitted electron then electron are accelerated by a high voltage x-ray produced when a high speed electron hit the metal target.



Figure (3-6): XRD device

3.2.2 Procedure:-

The material is powdered in pure powder, then the grained size talked about 1/2 g of sampleThepowder packed tightly in the sample holder.

X-rays are produced by bombarding a metal target (Cu, Mo usually) with a beam of electrons emitted from a hot filament (often tungsten). The incident beam ionized electrons of the target atom and X-rays are emitted through the powder as the resultant vacancies are filled by electrons dropping then reflected

3.2.3 Objective of test:-

identification the material (neem ,mica ,lalaob),X-ray crystallography determined the material structural, Lattice, parameter, Phaseidentity,Phase ,purity ,Crystallinity Crystal structure and Percent phase composition. Then the structure refinement result illustrated in figure & table.



Figure (3-7): XRD for Mica powder (CPL)



Figure (3-8): XRD for Neem powder (CPL)



Figure (3-9): XRD for Lalob powder (CPL)

3.3 Properties tests:-

The materials were prepared in powder form that to be satisfying the laboratory tests. Then they weighted to add to mud, figure (3-10).



Figure (3-10): all materials that used (drilling fluid lab, CPET)

3.3.1 Density measure:-

Measure density of drill fluid (mud weight)

The density (commonly referred to mud weight) is measured with a mud balance of sufficient accuracy to measure within 0.1 lb/gal(0.5 lb/ft³ or 5 psi/1000 ft of depth.). For practical purpose density means weight per unit volume of mud may beexpressed in hydrostatic pressure gradient in psi per 1000 of vertical depth.

3.3.1.1 Description:

The mud balance principally of a base on which rests a graduated arm with cup, lid, knife edge, level vial, rider and counter weight. The constant volume cup is a fixed to one end of the graduated arm, which has a counterweight at the other end. The cup and arm oscillation a plane perpendicular to the horizontal knife edge then balanced by moving the rider along the arm, figure (3-11).



Figure (3-11): mud balance device

3.3.1.2 Procedure:

The lids are removed from the cup, and completely fill the cup with the mud for test. also the lid is replaced and rotated until firmly seated, then mud is expelled through the hole in the lid and washed from the outside of the cup, the balance arm is placed on the base, with the knife edge resting on the fulcrum., the rider moved until the graduated arm level, as indicated by vial on the beam., the density or weight of the mud is read At the edge of the rider closest to the cup the result is reported in lb/gal.

3.3.2 Hydrogen ion concentration PH

Measured the value of acidity and alkalinity in the drilling fluid mud by used sunflower (ph) paper its standard paper as green color, submersed in the mud and compared the paper color to standard color contributed to standard number Then matches and read directly.



Figure (3-12): PH paper

3.3.3 6 speed viscometers

Measured (viscosity, plastic viscosity, and yield point) by used:-

3.3.3.1 Description:

Direct-indicating viscometers are rotational types of instruments powered by an electric motor or a hand crank. Drilling fluid is contained in the annular space between two concentriccylinders; figure (3-13).

The outer cylinder or rotor sleeve is driven at a constant RPM (rotational velocity). The rotation of the rotor sleeve in the fluid produces a torque on the bob or inner cylinder. A torsion spring restrains the movement of thebob, and a dial attached to the bob indicates displacement of the bob. Instrument constants have been adjusted so that plastic viscosity and yield point are obtained by using readings from rotor sleeve speed of 600 and 300 rpm.





3.3.3.2 Procedure for apparent viscosity, plastic viscosity and yield pointdetermination:

The mud is placed in a thermo cup and surface of mud is adjusted to scribed line on the rotor sleeve. .the motor is started by placing the switch in the high-speed position with the gear shift all the way down. Thenwaiting for a steady indicator dial value, and record the 600 rpm, the switch is changed to the 300-RPM speed, then waitingfor a steady value and record 300-RPM reading. Plastic viscosity in centipoises = 600 reading minus 300 reading. Yield Point in lb/100 ft2 = 300 reading minus plastic viscosity in centipoises. Apparent viscosity in centipoises = 600 reading divided by 2.

Herewith obtained the optimum mud which has used in bamboo, exactly for amal formation. To prepossess the loss in amal through drilling operation, here addingadditive represent in (mica, neem,lalob)in order to solve the problem of drilling fluid loss circulation.

In the step where we used to add loss circulation material (lcm) as additive to encounter the poor cementing fracture ,the ratio of barite is minimized to exceed the effect of dense fluid and replaced apart of it by (neem seeds, lalob seeds and mica) as powder gradually . Then reached to the optimum drilling mud properties and filtration that they reached to it in the area of study

3.3.4 API filtration:

3.3.4.1 Description:

The filtration or wall-building property of a mud is determined by means of a filter press. The test consists of determining the rate at which fluid is forced through the filter paper. The test is run under specified conditions of time, temperature and pressure. The thickness of the solid filter cake deposited is measured after the test. The filter press being used should meet specifications as designated in the API Recommended Practice and conducted in the manner suggested. The API fluid loss is conducted at surface temperature at 100 psi pressure, and is recorded as the number of milliliters lost in 30 min.



Figure (3-14): API filtration device

3.3.4.2 Procedure:

The pressure was prepared by compressor (100 psi), then the lid was removed from the bottom of the clean and dry cell, and the O-ring was placed in an undamaged groove and then was inverted to fill. The cell was filled with reference mud, then filter paper was placed on top of O-ring, after that the lid was placed on the filter paper with the flange of the lid between the flanges of the cell and turned until hand tight. The cell was turned over and the male cell coupling was inserted into the female filter press coupling and was turned either direction to engage. Then suitable graduated cylinder was placed under the filter opening to receive the filtrate. After that the pressure was applied to the cell by opening inlet valve. The filtrate was observed every certain period (1, 5, 3, 7.5, 10, 30 min) and the volume recorded in tables.

All these steps repeated to the three materials every one alone to reach the optimum quantity of it to achieve minimum filtrate, after that mixture of them used and the results recorded in the below tables.

3.3.4.3 Reference mud:

The purpose of filtration test is to determine the percentage of each material (neem,lalaob,mica) individually in the (lcm) the mechanism occurred through these test focusing on increasing in the percentage of each material (neem,lalaob,mica) and decreasing barite percentage until obtaining the optimum result with balancing between the rheological properties of mud and reference mud rheological properties ,after that an initial (lcm) is formed by mixing the optimum percentage of these material .the experiment is repeated until obtained the optimum result .

3.3.4.4API filtration using Mica material:-

The percentage of the barite reduced and grinded mica sharps in powder form then obtained drilling fluid properties approximately near the optimum and compared it with the reference mud used in bamboo area.

3.3.4.5API filtration usingNeem material:-

The percentage of the barite and ad grained neem seeds in powder form and obtained drilling fluid properties consider optimum then compared with the reference mud used in bambo area.

3.3.4.6API filtration usingLalob material:-

Thepercentage of the barite reduced and grindedlalob seeds in powder form and obtained drilling fluid properties approximately near to the optimum then compared it with the reference mud used in bamboo area.

3.3.4.7 API filtration usingmixture of materials:-

The test was repeated many times with changing the percentages of the materials until reach the best result which recorded.

3.3.5 Lost circulation Materials Test Device (DL):

The calculation of the lost circulation material is based on it is characters. For this testing with special pressure provide the lost circulation material (mud), test the effect of jam-up o every mud to select the correctly lost circulation material.

3.3.5.1 Purpose:

This device can be used to simulate lost value of lost circulation material under the different stratum, rift and pressure, and measure the value and depth of osmosis of the lost circulation material. It will be used to test the effect off jam-up of every material.

3.3.5.2 Main technical parameters:

Feed cylinder capacity: 4000ml

Working pressure:	0-7MPa
Depth:	0-80mm
Weight:	56Kg

Overall size: 4000*520*1060

3.3.5.3 Characters & Working Principles:

Feed the testing material into cylinder of the device, and then test the effect of jam-up of every material with the different pressure. This device has the characters of small size, simple structure, and easy operation. It can be used to test all kinds of lost values with different situation.



Chapter 4

Results and discussion

Chapter 4

Result and discussion

According to the data from geology of study area and literature review some results (from laboratory experiments that mentioned in previous chapter) concluded in this chapter, after that these results discussed.

4.1 The properties resultant:

As mention in chapter 3 after comparison of two well's data to get the optimum tabulated field values, table (4-1).

Categories	Well 1	Well 2	Well compare
Contractor company	Gc	Gc	Gc
DF type	GEL	Gel	Gel
Losses type	Partial	Partial	Partial
Density	8.8	9	8.8
Viscosity	48	38	50
Ph	9	9	9
PV	18	16	17
Gel strength	3/4	4/5	4/5
API filtrate	5.6	5	5.3
API cake	0.5	0.5	0.5
Sand content	3	3	3
600/300	60/42	56/41	56/41
ҮР	24	25	25

Table (4-1): mud reports screening (Great wall Co. MDR)

4.2 API filtration device to measure the mud filtrate

The way to obtain the optimum weight of each material is trying up to acceptable result, before using the mud with materials the reference mud must be tested, table (4-2).

Table (4-2). percentage of materials and mitrate for reference muu (la	Table	(4-2):	percentage	of materials	s and filtrate	for reference	mud	(lab
--	-------	--------	------------	--------------	----------------	---------------	-----	------

Component	Weight	Time (min)	Volume (ml)
Bentonite (gm.)	76.14	1	1.8
Barite (gm.)	15.2	3	2.8
Pac-lv (gm.)	9	5	3.6
Water (ml)	1100	7.5	4.5
		10	5.2
		30	9.1

After that test each material alone to reach the optimum concentrations percentage in the LCM, and the mixture of them, tables (4-11)-(4-17)

4.2.1 Firstly the powder of Mica:

Table (4-3): percentage of materials and filtrate for 2 gm of Mica

Component	Weight	Time (min)
Bentonite(gm.)	76.14	1
Barite(gm)	10.2	3
	0	5
Pac-lv(gm.)	9	3
Mica (gm.)	2	7.5
Water (ml)	1100	10
		30

Time (min)	Volume (ml)
1	2
3	3.2
5	4.1
7.5	5.07
10	6.27
30	10.14

Table (4-4): percentage of materials and filtrate for 4 gm of Mica

Component	Weight	Tin	ne (min)	Volume (ml)
Bentonite(gm.)	76.14		1	1.8
Barite(gm.)	10.2		3	3.4
Pac-lv(gm.)	9		5	4.3
Mica (gm.)	4		7.5	5.1
Water (ml)	1100		10	6.3
	I		30	10

Table (4-5): percentage of materials and filtrate for 5 gm of Mica

Component	Weight	
Bentonite(gm.)	76.14	
Barite(gm.)	10.2	
Pac-lv(gm.)	9	
Mica (gm.)	5	
Water (ml)	1100	

Time (min)	Volume (ml)
1	1.8
3	3
5	4
7.5	4.4
10	5.6
30	9.8

Table (4-6): percentage of materials and filtrate for 6 gm of Mica

Component	Weight	Time (min)	Volume (ml)
Bentonite(gm.)	76.14	1	2.4
Barite(gm.)	10.2	3	3.5
Pac-lv(gm.)	9	5	4.52
Mica (gm.)	6	7.5	4.93
Water (ml)	1100	10	6.13
		30	9.9

Depending on the method which mentioned in chapter 3 this results gained from experiments

4.2.2 Secondly the powder of Neem seed:

Table (4-7): percentage of materials and filtrate for 2 gm of Neem

Component	Weight	
Water (ml)	1100	
Bentonite(gm.)	76.14	
Barite (gm.)	10.2	
Pac-lv(gm.)	9	
Neem (gm.)	2	

	Time (min)	Volume (ml)
	1	1.8
	3	3.6
	5	3.8
	7.5	4.9
	10	5.8
	30	9.8

Table (4-8): percentage of materials and filtrate for 3 gm of Neem

Component	Weight	Time (min)	Volume (ml)
Water (ml)	1100	_	
Bentonite(gm.)	76.14	1	2
Barite (gm.)	10.2	3	3.4
Pac-lv(gm.)	9	5	3.8
Neem (gm.)	3	7.5	4.8
		10	5.6
		30	9.6

Table (4-9): percentage of materials and filtrate for 4 gm of Neem

Component	Weight	Time (min)	Volume (ml)

Water (ml)	1100		
Bentonite(gm.)	76.14	1	1.9
Barite (gm.)	10.2	3	2.6
Pac-lv(gm.)	9	5	3
Neem (gm.)	4	7.5	3.4
		10	5.2
		30	6.8

Table (4-10): percentage of materials and filtrate for 5 gm Neem

Component	Weight	Time (min)	Volume (ml)
Water (ml)	1100		
Bentonite(gm.)	76.14	1	1.7
Barite (gm.)	10.2	3	2.4
Pac-lv(gm.)	9	5	3
Neem (gm.)	5	7.5	4.4
	I	10	3.5
		30	6.5

4.2.3 Thirdly the powder of Lalob seed:

Table (4-11): percentage of materials and filtrate for 2 gm of Lalob

Component	Weight	Time (min)	Volume (ml)
Bentonite(gm.)	76.14	1	1.7
Barite(gm.)	10.2	3	2.4
Pac-lv(gm.)	9	5	3.1

Lalob (gm.)	2	7.5	3.4
Water (ml)	1100	10	4.4
		30	7.2

Table (4-12): percentage of materials and filtrate for 3 gm of Lalob

Component	Weight	
Bentonite(gm.)	76.14	
Barite(gm.)	10.2	
Pac-lv(gm.)	9	
Lalob (gm.)	3	
Water (ml)	1100	

Time (min)	Volume (ml)
1	1.68
3	2.1
5	3
7.5	3.6
10	4.2
30	7.6

Table (4-13): percentage of materials and filtrate for 4 gm of Lalob

Component	Weight	Tim	e (min)	Volume (ml)
Bentonite(gm.)	76.14		1	1.2
Barite(gm.)	10.2		3	1.9
Pac-lv(gm.)	9		5	2.1
Lalob (gm.)	4		7.5	2.65
Water (ml)	1100		10	2.95
			30	5.3

Table (4-14): percentage of materials and filtrate for 5 gm of Lalob

Component	Weight	Time (min)	Volume (ml)
Bentonite(gm.)	76.14	1	1
Barite(gm.)	10.2	3	1.8
Pac-lv(gm.)	9	5	2
Lalob (gm.)	5	7.5	2.6
Water (ml)	1100	10	2.9

|--|

4.2.4 Fourthly the mixture of three materials:

Using different percentage of three materials and try to reach the optimum one without change in the rheological properties, tables (4-15) to (4-17)

Table (4-15): percentage of materials and filtrate of 2.2mica, 4 gm neem, 8 gm gm lalob 1

Component	Weight	Time (min)	Volume (ml)
Water (ml)	1100		
Bentonite(gm.)	76.14	1	2
Barite (gm.)	10.2	3	3.1
Pac-lv(gm.)	9	5	4
Neem (gm.)	4	7.5	4.8
Lalob (gm.)	8	10	5.6
Mica (gm.)	2.2	30	9.6

Table (4-16): percentage of materials and filtrate of 2.2mica, 5 gm neem and lalob

Component	Weight	Time (min)	Volume (ml)
Water (ml)	1100		
Bentonite(gm.)	76.14	1	1.5
Barite (gm.)	10.2	3	2.9
Pac-lv(gm.)	9	5	3.9
Neem (gm.)	5	7.5	4.8

Lalob (gm.)	5	10	5.4
Mica (gm.)	2.2	30	9.6

Table (4-17): percentage of materials and filtrate of 2.2mica, 4 gm neem, 5 gm lalob

Component	Weight	Time (min)	Volume (ml)
Water (ml)	1100		
Bentonite(gm.)	76.14	1	1.6
Barite (gm.)	10.2	3	2.8
Pac-lv(gm.)	9	5	3.6
Neem (gm.)	4	7.5	4.4
Lalob (gm.)	5	10	5.1
Mica (gm.)	2.2	30	8.8

4.3 Lost Circulation Materials Test Device Experiments:

According to the properties of the formation and the mud the loss circulation device was used to evaluate the performance of the LCM. All these experiments were done in the drilling fluids lab of CPET, SUST and the results illustrated in tables (4-18) (4-34).

4.3.1 Experiment 1:

Proceeding drilling fluid lab to access minimum loss of drilling fluid or close the portion throughout additives

Prepared drilling fluid used same mixtures of raw materials before, then specifying the ratios of these materials configuring it and it found as detailed below:-

Table (4-18): percentage of materials in final LCM

Materials	Ratios
Balanities aegyptica	44.64%
Azadiratcha indica	35.71%
Mica	19.64%

2. After preparing the mud using the same as the past additives, **LOST CIRCULATION DEVICE** prepared then cleaned accurately before using it.

3. Poured the drilling mud (size: 4000ml) to the loss circulation device, the poses of the materials was powder added.

Result:

The result was **total loss** at normal pressure and when exposure to 100 psi (6.8 bar), then we cleaned the device for the next attempt.

4.3.2 Experiment 2:

Drilling mud was prepared same as the one before, then to encounter the problem before (total loss), we weighted lost circulation materials to get other form of results.

Minimize the volume of water in the drilling mud and appreciate suitable ratios of the additives according to the table above, it was approximately adding **20** gm of loss circulation materials. The ratios of these materials are clarifying in the table below:-

Table (4-19): percentage of materials in 20 gm of LCM

Materials	Weight(gm)
Balanites aegyptiaca	9
Azadirachta inidca	6.6
Mica	4.4

4.3.3 Experiment No 3:

Drilling mud prepared according to the description in the table below:-

Table (4-20): percentage of materials

Materials	Weight(gm)
Bentonite	278.6
Pac-lv	32.8
Azadiratcha indica	14.6
Balanities aegyptica	14.6
Barite	18.2
Mica	8

Water volume = 4000 ml*

Weighting **50 gm** of loss circulation materials and minimizing the volume of water.

Table (4-21): percentage of materials in 50 gm of LCM

Materials	Weight(gm)
Azadiratcha indica	18
Balanities aegyptica	22
Mica	9.9

Water volume = 3950 ml*

The results:

In normal pressure the loss device closed at time and volume as described below:

Table (4-22): quantity of lost mud and time of 50 gm LCM at normal pressure

Time(min)	Volume(ml)
1.5	1400

4.3.4 Experiment No 4:

Then after that added **50 gm** of loss circulation materials (LCM) accumulatively to the previous drilling mud as illustrated in the table below:

Table (4-23): percentage of materials in 100 gm of LCM

Materials	Weight(gm)
Azadiratcha indica	36
Balanities aegyptica	44
Mica	19.8

Water volume = 3900 ml*

Table (4-24): the rheological properties of 100 gm LCM

Mud properties	
Density	8.92Ib/gal
PV	25
YP	23
PH	8
In normal pressure the **LCM** materials closed the stratum fracture at the time and volume described, table (4-25)

Table (4-25): quantity of lost mud and time of 100 gm LCM at normal pressure

Time(min)	Volume(ml)
45sec	900

Table (4-26): quantity of lost mud and time of 100 gm LCM at pressure100 psi

Time(min)	Volume(ml)
1	1600

4.3.5 Experiment No 5:

Adding other **50gm** of loss circulation materials to the past drilling mud and the form of the additives accumulatively detailed in the table (4-27).

Table (4-27): percentage of materials in150 gm of LCM

Materials	Weight(gm)
Azadiratcha indica	54
Balanities aegyptica	66
Mica	29.7

Water volume = 3850 ml*

Table (4-28): the rheological properties of 150 gm LCM

Mud prosperities		
Density	8.94Ib/gal	
PV	29	
YP	29	
РН	8	

After calibrating the mud properties it was poured to the feed cylinder of the Lost Circulation Test Device, then waiting **30 minutes** till the mud settles in the device to simulate the same process happens in the wells.

Table (4-29): quantity of lost mud and time of 150 gm LCM at normal pressure

Time (min)	Volume(ml)
21 sec	820

Table (4-30): quantity of lost mud and time of 150 gm LCM at pressure100 psi

Time (min)	Volume(ml)
33 sec	1300

4.3.6 Experiment No 6:

To reach to the results that can close the portion totally or minimize the fluid losses as minimum as possible, we weighted other 50gm of loss circulation materials and it was accumulate calculated with the past adding tables results.

Table (4-31): percentage of materials in 200 gm of LCM

Materials	Weight(gm)
Azadiratcha indica	72
Balanities aegyptica	88
Mica	39.6

Water volume =3800 ml*

Table (4-32): the rheological properties of 200 gm LCM

Mud properties	
Density	8.95Ib/gal
PV	33
YP	35
PH	8

Table (4-33): quantity of lost mud and time of 200 gm LCM at normal pressure

Time (min)	Volume(ml)
18 sec	750

Table (4-34): quantity of lost mud and time of 200 gm LCM at pressure100psi

Time(min)	Volume(ml)
30sec	1150

4.4 Discussion:

From plotting of filtration volume verses time for each specific weight of (mica ,neem ,lalob ,mixture) .it was observed that the volume of filtration decrease while weight increase, then the optimum volume of filter loss obtained from 5gm of mica was 9.8 ml ,5gm of neem was 6.8ml and 5gm lalob was 5ml (figures (4-1)-(4-3)). From mixture plotting, perfect percentage of LCM obtained 2.2gm mica,4gm neem and 5gm lalob the volume of filtrate is 8.8 ml. representation in % (mica19.64% ,neem35.71%, lalaob44.64%)figure(4-4).



Figure (4-1): The relation between time and filtrate using 2 gm from each material



Figure (4-2): The relation between time and filtrate using 4 gm from each material



Figure (4-3): The relation between time and filtrate using 5 gm from each material



Figure (4-4): The relation between time and filtrate using mixture of materials

At the condition of normal pressure the optimum result of (100, 150, 200) gm obtained 750 ml.



Figure (4-5): The relation between time, volume lost and LCM weight at normal pressure

When plotted the volume of mud lost verses time for specific mixture weight 100 gm ,150 gm ,200 gm ,significantly the volume decrease inversely with weight, at condition of exposure 100 psi to the device the optimum result obtained 1150 ml



Figure (4-5): The relation between time, volume lost and LCM weight at 100 psi

<u>Chapter 5</u>

Conclusion and recommendations

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Conclusion and recommendations

5.1 Conclusion:

In this research a mixture of neem powder, lalob powder and mica used as LCM material, by easy scan XRD test found that the crystallography structure of mica as powder and the chemical structure for organic materials(neem, lalob) as powder, this test found that the mixture powder (neem, lalob and mica) applicable to use as LCM.

Screening two well data then simulate them in laboratory to set the rheological properties. Then the mud prepared in the lab and the rheological properties achieved. After that this mud tested by API filtration test to measure the filtrate. After that the three materials added to the mud to test by API filtration device (to determine the percentages of each material (mica, neem and lalob), and to test the ability of them to work as LPM) and loss circulation device.

After the API filtration tests for each material found that the optimum weight of Mica was 5 gm. And the optimum one of Neem was 5 gm, and 5gm of Lalob. For Mixture the quantities of materials together found that the optimum weights were 2.2gm (19.64%) Mica, 4gm (35.71%) Neem, 5gm (44.65%) Lalob. all these quantities without change in rheological properties.

The next step was using the loss circulation device with different weight too get the optimum one, it found that the optimum weight of LCM (Mixture of three materials) was 150gm, without change in rheological properties.

5.2 Recommendations:

- More studies using X-ray diffraction (XRD) test to determine the change in chemical composition.
- To gain more applicable results that require to use Particle plugging apparatus (PPA) and HPHT fluid loss apparatus in conjunction with slotted/tapered discs to evaluate LCM performance for corrective treatments.
- For final result we recommend to apply pilot test on this materials.
- ✤ Use different sizes of materials together.
- ♦ Grind the materials in nano size and use it.
- Study how the usage of these materials can reduce the non productive time (NPT).
- Calculate the cost of using these materials.

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