Chemical, Petroleum and Environmental Engineering

The Effect of Nano Materials on Lost Circulation Control of Azkand Formation in Khabaz Oil Field

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ABSTRACT

Experimental tests were carried to control lost circulation in the Khabaz oil field using different types of LCMs including Nano-materials. A closed-loop circulation system was built to simulate the process of lost circulation into formations. Two dolomite plugs were used from different depths of the formation of Azkand in Khabaz oil field. The experimentations were carried out to study the effect of different types of LCMs, cross-linked copolymer (FLOSORB CE 300 S), SiO_2 NP, and Fe_2O_3 NP, on mud volume losses as a function of time.

The rheological measurements of the nanoparticles-reference mud system showed that both of the SiO_2 NP and Fe_2O_3 NP worked best at 1.2 g/l. and the closed-loop circulation system showed that SiO_2 NP cured losses by 48.3% and 37.5% for core plugs 5C1 and 1C2 respectively which is better than Fe_2O_3 NP loss curing which was 37.9% and 31.3% for core plugs 5C1 and 1C2 respectively.

On the other hand, FLOSORB CE 300 S- Fe_2O_3 NP-reference mud systems cured losses up to 69% with 0.25g/l FLOSORB CE 300 S and 79.3% with 0.5 g/l FLOSORB CE 300 S which is better than the FLOSORB CE 300 S- SiO_2 NP-reference mud systems results.

Keywords: Lost circulation, nanomaterials, drilling fluid

دراسة تأثير المواد النانوية للسيطرة على فقدان سائل الحفر في تكوين ازقند في حقل الخباز النفطي

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الخلاصة

في هذا البحث قد تم استخدام انواع مختلفة من مواد السيطرة على فقدان طين الحفر المحتوية على مواد نانوية لمعالجة مشكلة فقدان طين الحفر . وقد تم اجراء التجارب للسيطرة على مشكلة فقدان طين الحفر في حقل خباز النفطي. و قد تم انشاء منظومة

ضخ مغلقه لمحاكاة حالة فقدان طين الحفر . و تم اجراء التجارب على عينتين ماخوذتين من اعماق مختلفة لطبقة ازقند في حقل
خباز النفطي . و قد تم التوسع بالتجارب لدراسة تاثير الانواع المختلفة لمواد السيطرة على فقدان طين الحفر بخلط طين الحفر
مع الفلوسورب و اوكسيد الكبريت النانوي او اوكسيد الحديد النانوي و تم اخذ حجم الطين المفقود و فعاليته في الاداء مقارنة
بالزمن.
و قد تم قياس ريولوجية الجزيئات النانوية و بينت ان كلا اوكسيد الحديد النانوي و اوكسيد السيليكون النانوي كانوا فعالين عند
تركيز 1.2 غرام/لتر و قد بينت منظومة الضخ ان اوكسيد الحديد النانوي قد سجل خسارة بمقدار 48.3% و 37.5% لعينتي
ال 5C1 و ال 1C2 بالترتيب و الذي كانت نتائجه افضل من اوكسيد الحديد النانوي والذي كانت نسب فقدان الطين على نفس
العينتين 37.9% ل 5C1 و 31.3% لعينة 1C2
وعلى الصعيد الاخر فان خلط اوكسيد الحديد النانوي مع FLOSORB CE 300 S قد ادت لنتائج افضل حيث سجلت 69%
بخل 0.25 غرام /لتر من الفلوسورب ونسبة 79.3 % بخلط 0.5 غرام /لتر من FLOSORB CE 300 S مع اوكسيد
الحديد النانوي والتي كانت نتائج افضل بكثير من اوكسيد الكبريت مع الفلوسورب.
الكلمات الرئيسية: فقدان عمود طيين الحفر، اوكسيد الحديد النانوي، اوكسيد السلكون النانوي، FLOSORB CE 300 S.

1. INTRODUCTION

Lost circulation is the complete or incomplete loss of drilling fluids into the well (**Miranda, et al., 2017**). Unconsolidated or highly permeable formations, cavernous formation, natural fractured and induced fractures are the types of formation that caused lost circulation (**Howard & Scott, 1951**). Despite the fact that lost circulation has being known for decades, it still considered one of the core problems that cause non-productive time (NPT). Lost circulation can be classified according to the flow rate of lost fluid into seepage, partial, severe and total (**Daccord, et al., 2006**).

Normally lost circulation zones can be identified to some extent, based on how fast the drilling mud is being lost and how drilling is affected once the formation starts losing mud (**Datwani, 2012**). In general, there are four categories of formations that are in charge of lost circulation and they are: induced or natural formation fractures, cavernous or vugular formations, highly permeable formations, and unconsolidated formations (**Ali, et al., 1994**) and (**Datwani, 2012**).

Adding lost circulation materials to the mud system is a common practice when it comes to curing lost circulation in the thief zones. Lost circulation materials (LCMs) are used in general to reduce the loss rate, even though the process is not consistent because the materials are chosen by trial (**Razzaq & Kzar , 2016**). There is a wide range of materials that are used to cure lost circulation as single additives or as a blend of several materials. Nano-materials can be added to LCMs to cut fluid loss and make a thinner filter cake when compared to fluids having LCM (Borisov, et al., 2015). Where nanotechnology produces products that have many special properties, that can refine mud cake quality, maintain borehole stability, protect the reservoir, and meet the needs of drilling operations under complex geological environments (**Salam, et al., 2019**).

An important matter to take into consideration is the equipment and procedure used to evaluate the LCMs for severe lost circulation. A crucial step to investigate the feasibility of LCMs and the durability prior to field applications is done by laboratory evaluation (**Alsaba, et al., 2014**).

Lost circulation is a crucial problem in most of the Iraqi oil fields and the Khabaz oil field is one of these fields which has such a problem. Khabaz oil field is a small subsurface asymmetrical anticline with the northeast limb dipper than the southwest limb. Khabaz structure is positioned between Jambur and Bai Hassan oil fields. This oil field is located to the northwest of Kirkuk city northeast Iraq by approximately 23 km (**AlAtroshi, et al., 2018**).

In this research the effect of two nanomaterials on lost circulation was studied after constructing a closed-loop circulation system that was built to simulate the process of lost circulation into

formations and compare between them before and after combining with a new polymer. As well as, optimizing the lost circulation materials consisting with their effect on rheology properties.

2. EXPERIMENTAL WORK

2.1 Materials

The objective of the study is to compare the effectiveness of three different types of materials added to a reference mud to cure and treat lost circulation of Azkand formation. The ingredients of the reference mud system are bentonite (60 g), starch (5g), XC polymer (5 g), and soda ash (1g) (supplied by Iraqi Drilling Company). All the previously mentioned materials were mixed in one liter of freshwater which is similar to the composition of the mud used to drill the well that the plugs were extracted from. And the three additives are FLOSORB CE 300 S (a Cross-Linked Copolymer of Acrylamide and Potassium Acrylate that is supplied by SNF France), Fe_2O_3 with Nano-particle size range of (20 - 30) nm (supplied by Nanjing Nano Technology Co., Ltd.), and Nano Silica was used as LCM in Nano-particle size range of (3 - 30) nm (supplied by US Research Nanomaterials Inc.) and added to reference mud. The materials concentrations and functions are shown in **Table 1**.

The core samples were collected from Azkand formation C1 and C2 sub-formations, where two vuggy dolomite core plugs were obtained from well KZ-35 (Khabaz oil field), **Table 2.**

Table 1. Specifications of the Materials in Mud System.

Materials	Concentration (g/litre)	Primary Function	Solubility in Water
Bentonite	60	Viscosity and fluid- loss control	Insoluble
Soda Ash, Sodium Carbonate, Na_2CO_3	1	pH controller	Soluble
XC Polymer	5	Viscosity and fluid-loss control	Soluble
Starch	5	Fluid- loss control and viscosity	Insoluble
FLOSORB CE 300 S copolymer	0.25, 0.5, 2	LCM	Insoluble
Nano Silica (SiO_2)	0.4, 0.8, 1.2, 1.4	LCM	Insoluble

Table 2. Core plugs characteristics.

Core No	Formation	Depth (ft)	Length (cm)	Diameter (cm)	Permeability (md)	Appearance
1	Azkand C1	7241.3	4.4	3.77	83.69	
2	Azkand C2	7251.1	4.37	3.79	319.93	

2.2 Rheological and Filtration Properties Measurements

The rheological properties of the prepared drilling fluid, plastic viscosity (PV) in cP, yield point (YP) in $lb_f/100ft^2$, and gel strength in $lb_f/100ft^2$ were measured using OFITE model 900 viscometer. While average viscosity (AV) in cP was calculated using the following equation:

$$AV = \frac{\theta_{600}}{2}$$

Where, θ_{600} is a dial reading with viscometer operating at 600 rpm

The filter loss volume (at 7.5 and 30 min) and mud cake thickness in mm were measured using OFITE low-pressure filter press with deadweight hydraulic assembly.
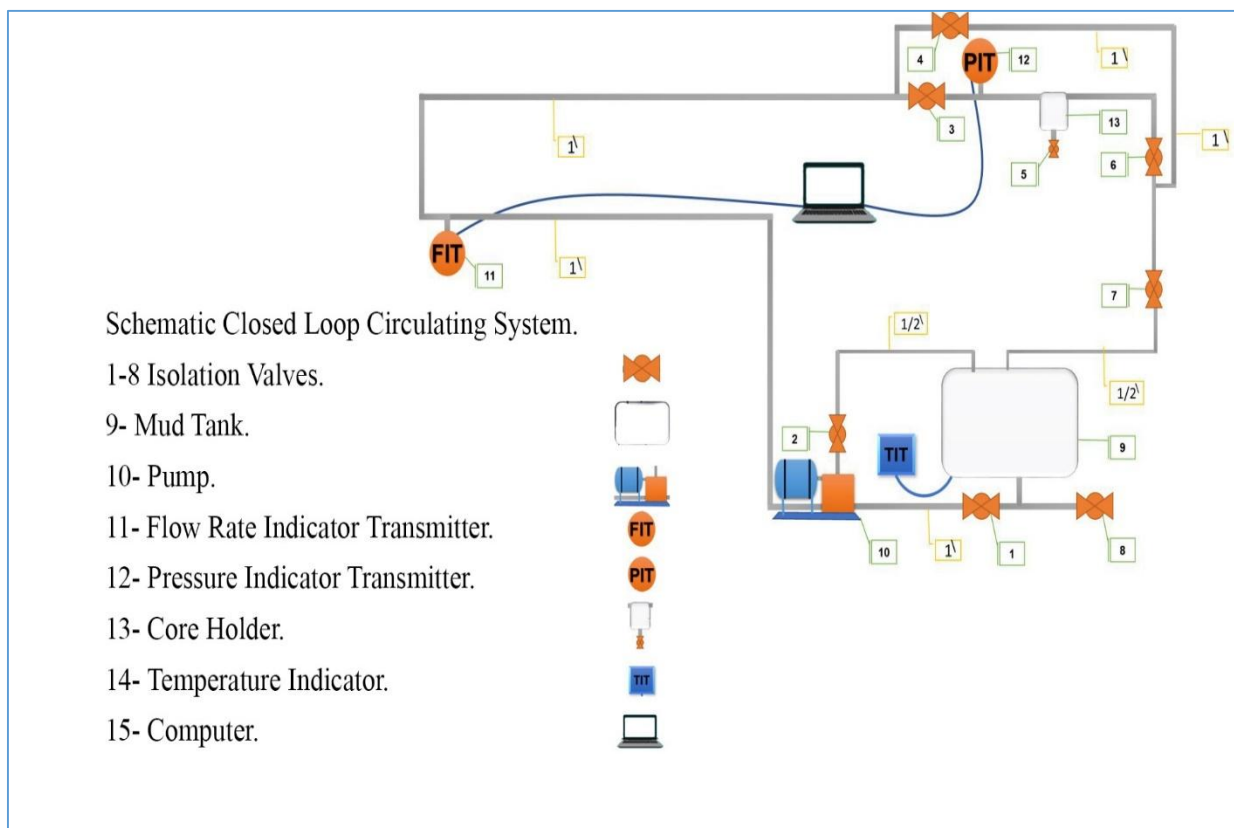
2.3 Experimental Apparatus

A closed-loop circulation system was built to simulate the process of lost circulation into formations **Figure 1** illustrates the closed-loop circulation system and its schematic is shown in **Figure 2.**
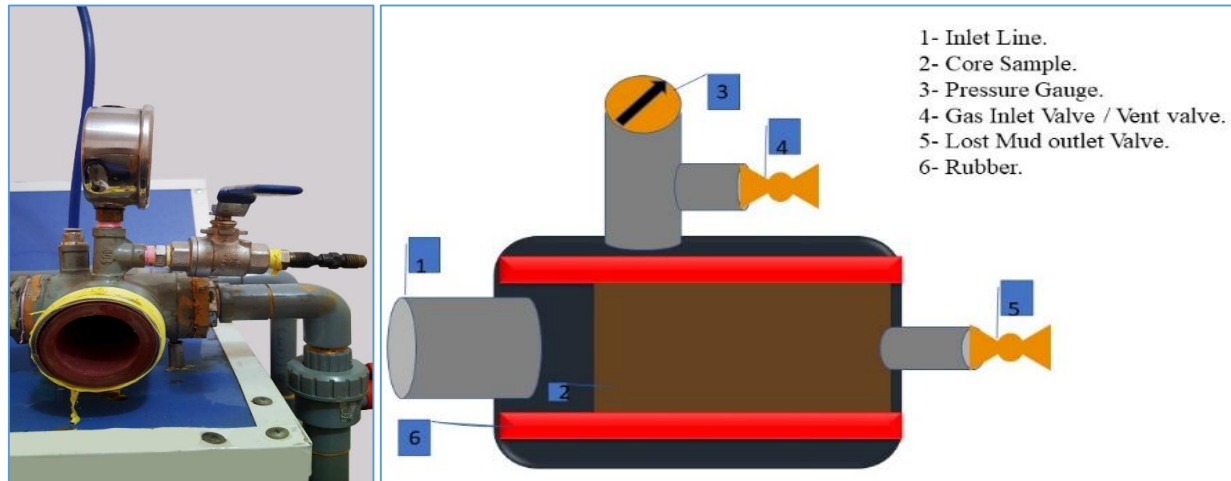
A stainless-steel core holder was also built as a dynamic mud loss cell similar to standard Hassler type core holder that can hold a single 4.4 cm in diameter core plug. The core plug sited inside a rubber sleeve and then sealed together by air pressure applied by an air compressor to avoid the bypassing of the injected mud to the spaces between the core sample and the rubber sleeve as shown in **Figure 3**.
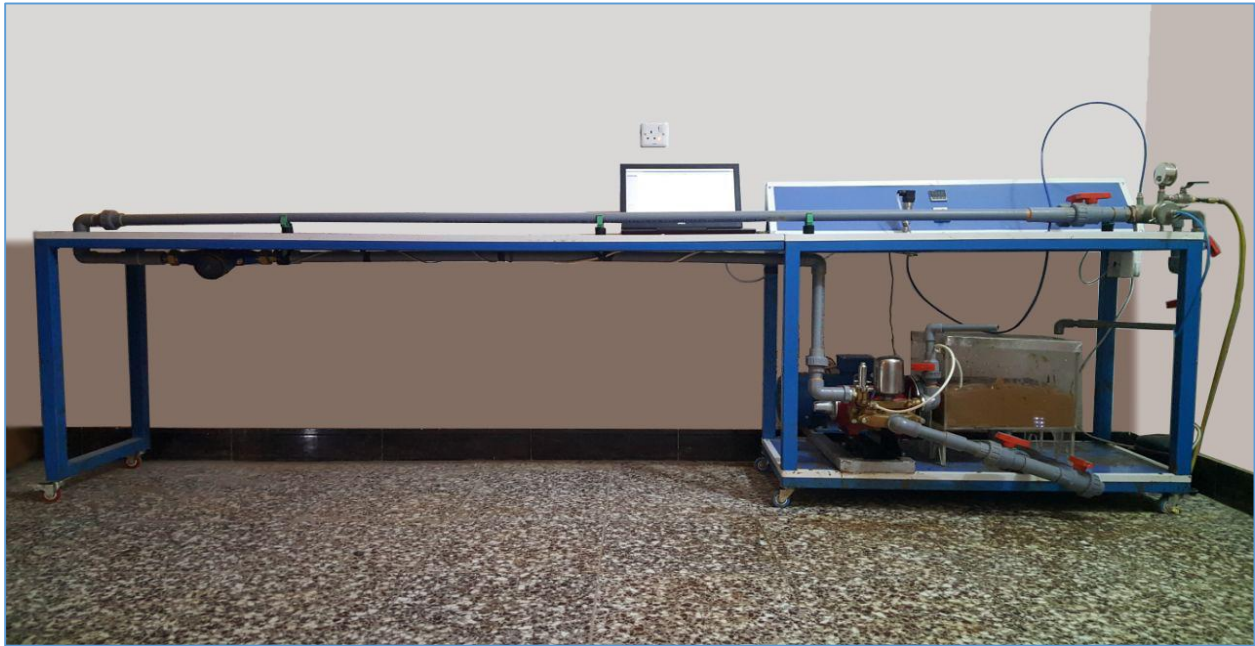
Figure 1. Main components of the closed-loop circulation system.

Schematic Closed Loop Circulating System.

1-8 Isolation Valves.

9- Mud Tank.

10- Pump.

11- Flow Rate Indicator Transmitter.

12- Pressure Indicator Transmitter.

13- Core Holder.

14- Temperature Indicator.

15- Computer.

Figure 2. Schematic Closed Loop Circulating System.

Figure 3. The core holder. Where 1- inlet line, 2- core sample, 3- pressure gauge, 4- gas inlet valve, vent valve, 5- lost mud outlet valve, 6- rubber.

2.4 Test Procedure

The full test procedure is explained in (Shibeeb, et al., 2019) and can be summarized by preparing 8 – 10 liters of drilling mud and pumping it in the closed-loop system and into the core plug that is fixed inside the core holder for 120 min and measure the volume of mud that flowed through the plug. This procedure then is repeated for all the mud systems and for both of the core plugs.

3. RESULTS AND DISCUSSION
3.1 Rheological and filtration properties.

The rheological and filtration properties of each type of mud were measured and illustrated in **Table 3** to **Table 6**. First, the reference mud properties were measured and gave relatively high rheological properties.

Adding 0.4, 0.8, 1.2, and 1.6 g/l SiO_2 NP concentration to the reference drilling mud caused a significant increase in rheological properties as shown in **Table 3**. The results indicated that 1.2 g/l of SiO_2 NP exhibited the highest rheological properties (plastic viscosity and gel strength) and showed a reduction filter cake thickness (increased its firmness) in comparison with other concentrations. That was the reason for proceeding with this concentration for the next step of losses curing. Trying with the same Fe_2O_3 NP concentrations, the results showed that 1.2 g/l of Fe_2O_3 NP gave the peak rheological properties when added to the reference mud as shown in **Table 5**.

The rheological and filtration properties of 1.2 g/l SiO_2 NP with 0.25 and 0.5 g/l FLOSORB CE 300 S mixture are illustrated in **Table 5**. FLOSORB CE 300 S addition caused an increase in rheological properties even at low concentrations, but with low effect on filtration properties.

FLOSORB CE 300 S with two concentrations 0.25 and 0.5 g/l were used to prepare a mixture of LCMs; their rheological and filtration properties are shown in **Table 6**. High rheological properties were obtained even with low FLOSORB CE 300 S concentrations while it has a low effect on filtration properties.

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Combined nanomaterials with FLOSORB CE 300 S cause a significant effect on rheological properties which helped in controlling lost circulation. High rheological properties of this mixture of two different LCMs could make it difficult to pump it as drilling fluid so it is recommended to use it as pills which can often cure lost circulation.

Table3. Rheological and filtration properties of reference mud with different concentration of SiO2 NP.

Properties	Reference Mud	Reference Mud+0.4 g/l SiO$_2$ NP	Reference Mud+0.8 g/l SiO$_2$ NP	Reference Mud+1.2 g/l SiO$_2$ NP	Reference Mud+1.6 g/l SiO$_2$ NP
Density, SG	1.02	1.02	1.01	1.005	1.01
10 sec gel, lb$_f$/100 ft^2	78	85	92	109	106
10 min gel, lb$_f$/100 ft^2	117	121	126	145	137
Filter Volume, ml	7.4	7.2	6.6	6.8	6
Mud Cake Thickness, mm	1.1	1.05	1	0. 87	1.1
pH	10.66	10.63	10.6	10.56	10.73
YP, lb$_f$/100 ft^2	116	122	127.9	146	129
PV, cP	13	14	14	19	13.2

Table 4. Rheological and filtration properties of treated mud with 1.2 g/l SiO2 NP and different concentrations of the cross-linked copolymer.

Properties	Reference Mud	Reference Mud+1.2 g/l SiO$_2$ NP +0.25g/l FLOSORB CE 300S	Reference Mud+1.2 g/l SiO$_2$ NP +0.5g/l FLOSORB CE 300S
Density, SG	1.02	1.02	1.01
10 sec gel, lb$_f$/100 ft^2	78	92	98

10 min gel, lb$_f$ /100 ft^2	117	148	153
Filter Volume, ml	7.4	7.4	7
Mud Cake Thickness, mm	1.1	1.5	1.5
pH	10.66	10.43	10.12
YP, lb$_f$/100 ft^2	116	157	161
PV, cP	13	11	14

Table 5. Rheological and filtration properties of reference mud with different concentrations of Fe2O3 NP.

Properties	Reference Mud	Reference Mud+0.4 g/l Fe2O3 NP	Reference Mud+0.8 g/l Fe$_2$O$_3$ NP	Reference Mud+1.2 g/l Fe$_2$O$_3$ NP	Reference Mud+1.6 g/l Fe$_2$O$_3$ NP
Density, SG	1.02	1	0.99	0.98	0.99
10 sec gel, lb$_f$ /100 ft^2	78	78	118	102	92
10 min gel, lb$_f$ /100 ft^2	117	109	139	132	118
Filter Volume, ml	7.4	7.8	7.3	7.1	8.2
Mud Cake Thickness, mm	1.1	1.5	1.1	1	1.5
pH	10.66	10.63	10.66	10.68	10.66
YP, lb$_f$/100 ft^2	116	121	179	178	148
PV, cP	13	12.4	12.1	11.8	12.8

Table6. Rheological and filtration properties of treated mud with 1.2 g/l Fe2O3 NP and different concentrations of cross-linked copolymer

Properties	Reference Mud	Reference Mud+1.2 g/l Fe$_2$O$_3$ NP +0.25g/l FLOSORB CE 300S	Reference Mud+1.2 g/l Fe$_2$O$_3$ NP +0.5g/l FLOSORB CE 300S
Density, SG	1.02	0.98	0.98
10 sec gel, lb$_f$ /100 ft^2	78	83	94
10 min gel, lb$_f$ /100 ft^2	117	135	147
Filter Volume, ml	7.4	7.7	7.9
Mud Cake Thickness, mm	1.1	1.2	1.5
pH	10.66	10.64	10.42
YP, lb$_f$/100 ft^2	116	127	142
PV, cP	13	10.9	9.2

3.2 Lost Circulation System Results

Adding LCMs cause a decrease in the mud loss volume with time to some level as presented in the following sections. The reduction in the lost volume depends mainly on the type and concentration of LCMs. Also, the lost volume with time varies from one core sample to another, where the rate of losses tends to increase for the core plugs with higher permeability.

Adding 1.2 g/l SiO$_2$ NP reduced losses by 48.3 % and 37.5% for core plugs 1 and 2 respectively, while 1.2 g/l Fe$_2$O$_3$ NP addition to the reference mud caused a reduction in the lost volume of 37.9 % and 31.3 % for core plugs 1 and 2 respectively as shown in **Figure 4** and **Figure 5**. 1.2 g/l SiO$_2$ NP cured losses better than Fe$_2$O$_3$ NP when added to the same reference mud system. This is resulted from having higher rheological properties and a better mud cake characteristic for the SiO$_2$ NP-reference mud system.

When introducing FLOSORB CE 300 S to the NP-reference mud systems the results were different, where FLOSORB CE 300 S- Fe$_2$O$_3$ NP- reference mud systems cured the losses better than the FLOSORB CE 300 S- SiO$_2$ NP- reference mud systems. Using a 0.25 g/l FLOSORB CE 300 S in the FLOSORB CE 300 S- Fe$_2$O$_3$ NP- reference mud systems reduced losses by 69 % and 46.9 % for core plugs 1 and 2 respectively when compared combining the same

concentration of FLOSORB CE 300 S with FLOSORB CE 300 S- SiO_2 NP- reference mud systems that reduced losses by 58.6 % and 43.8 % for core plugs 1 and 2 respectively as shown in **Figure 6** and **Figure 7**.

While using a 0.5 g/l FLOSORB CE 300 S in the FLOSORB CE 300 S- Fe_2O_3 NP- reference mud systems gave similar results trend by reduced losses by 79.3 % and 56.3 % for core plugs 1 and 2 respectively when compared combining the same concentration of FLOSORB CE 300 S with FLOSORB CE 300 S- SiO_2 NP- reference mud systems that reduced losses by 65.5 % and 50 % for core plugs 1 and 2 respectively as shown in **Figure *8*** and

Figure 9.

FLOSORB CE 300 S as a lost circulation material was tested and gave better results than one of the traditional LCMs (cotton seeds) (Shibeeb, et al., 2019), but when it was combined with nanomaterials it gave superior results overusing one LCM on its own whether it was FLOSORB CE 300 S, SiO_2 NP or Fe_2O_3 NP.

Due to SiO_2 NP- reference mud systems having better mud cake characteristics and higher rheological properties it gave better results than the Fe_2O_3 NP- reference mud systems. On the other hand, FLOSORB CE 300 S- Fe_2O_3 NP- reference mud system gave firmer and thinner mud cake that helps the FLOSORB CE 300 S to seal the fractures and the interconnected channels than the FLOSORB CE 300 S- SiO_2 NP- reference mud system.
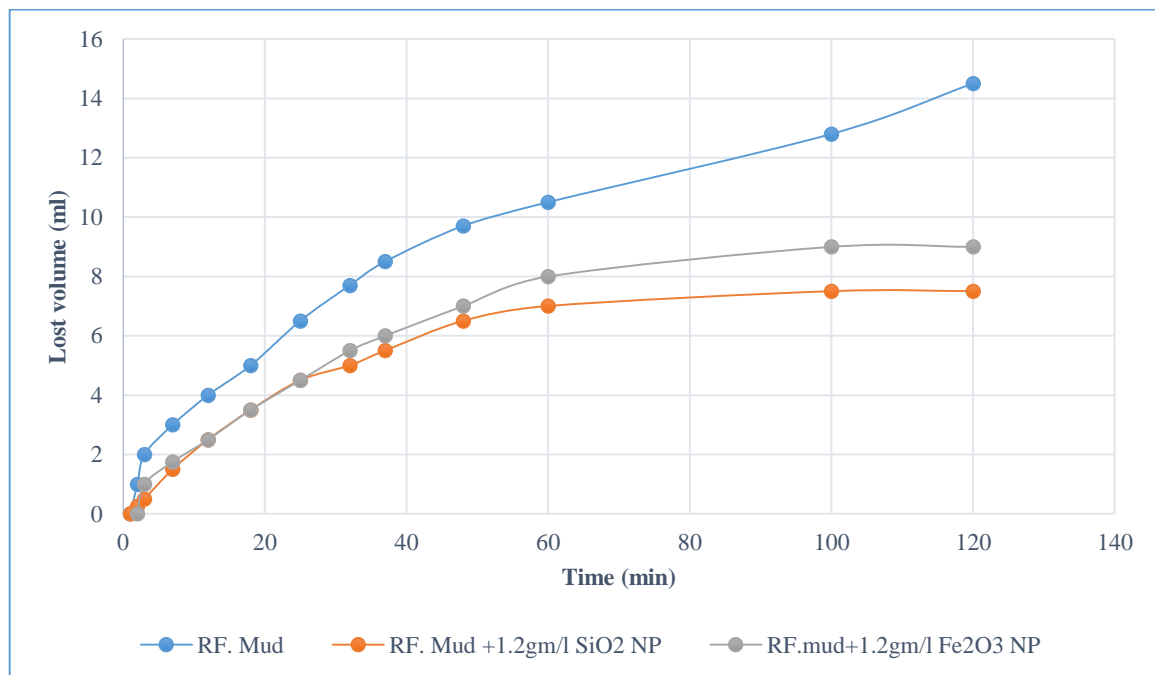
Figure 4. A comparison between the effect of adding 1.2 g/l of SiO2 NP and 1.2 g/l of Fe2O3 NP to the reference mud for core plug 1.
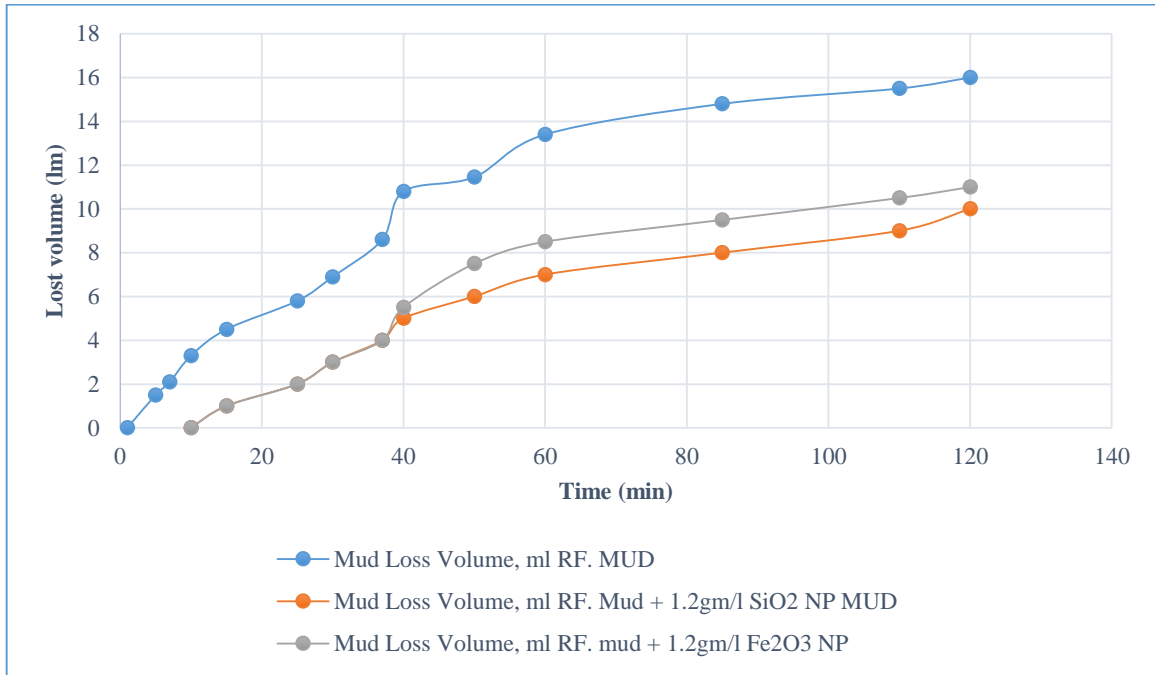
Figure 5. A comparison between the effect of adding 1.2 g/l of SiO2 NP and 1.2 g/l of Fe2O3 NP to the reference mud for core plug 2.
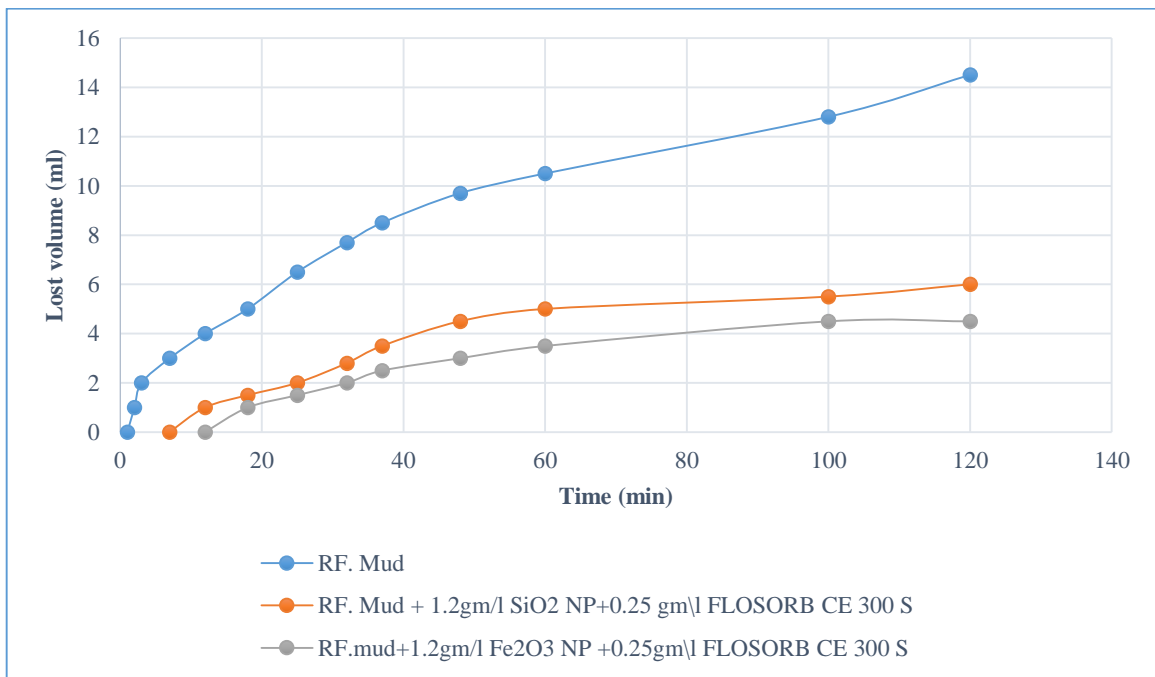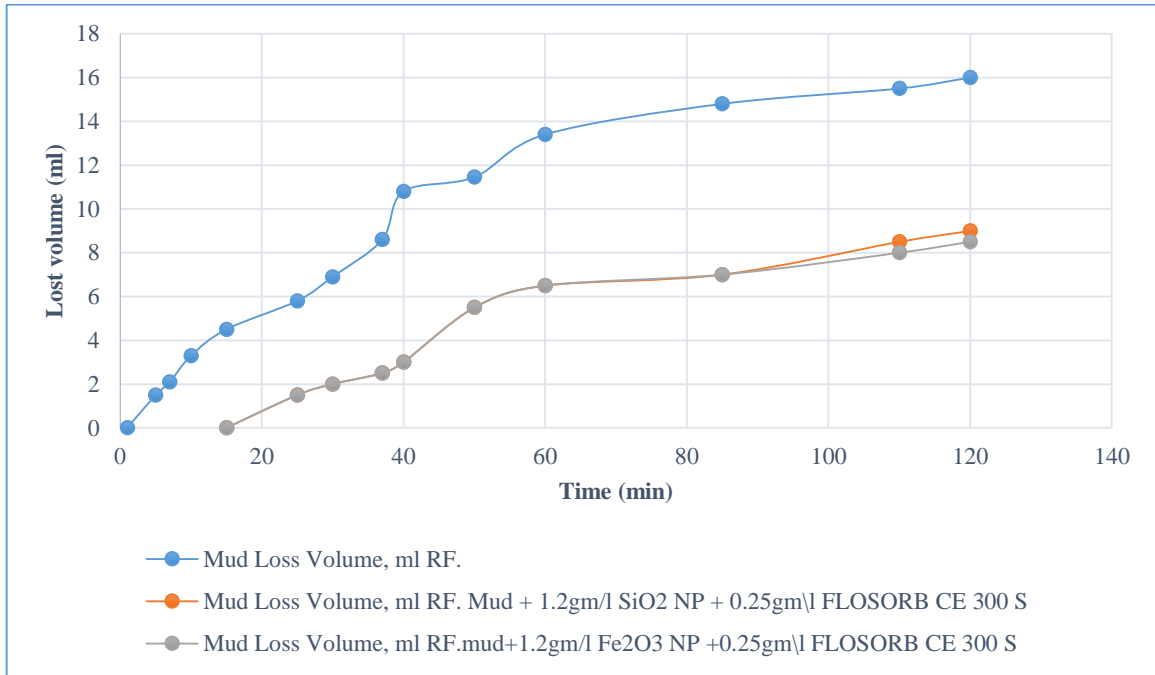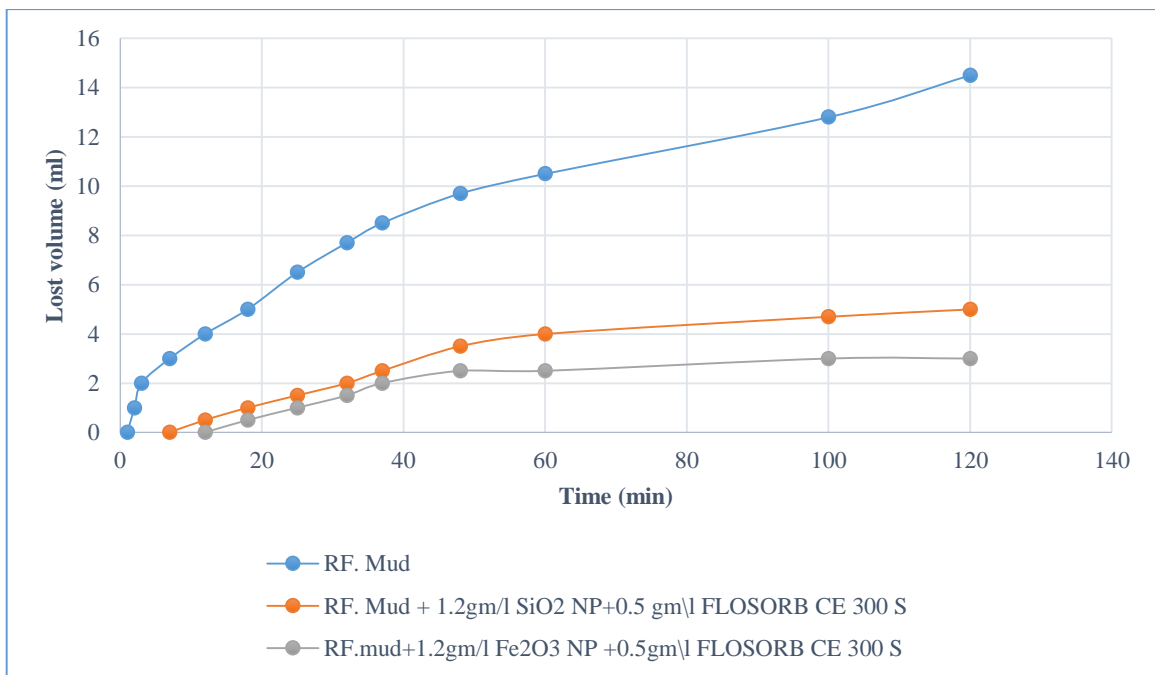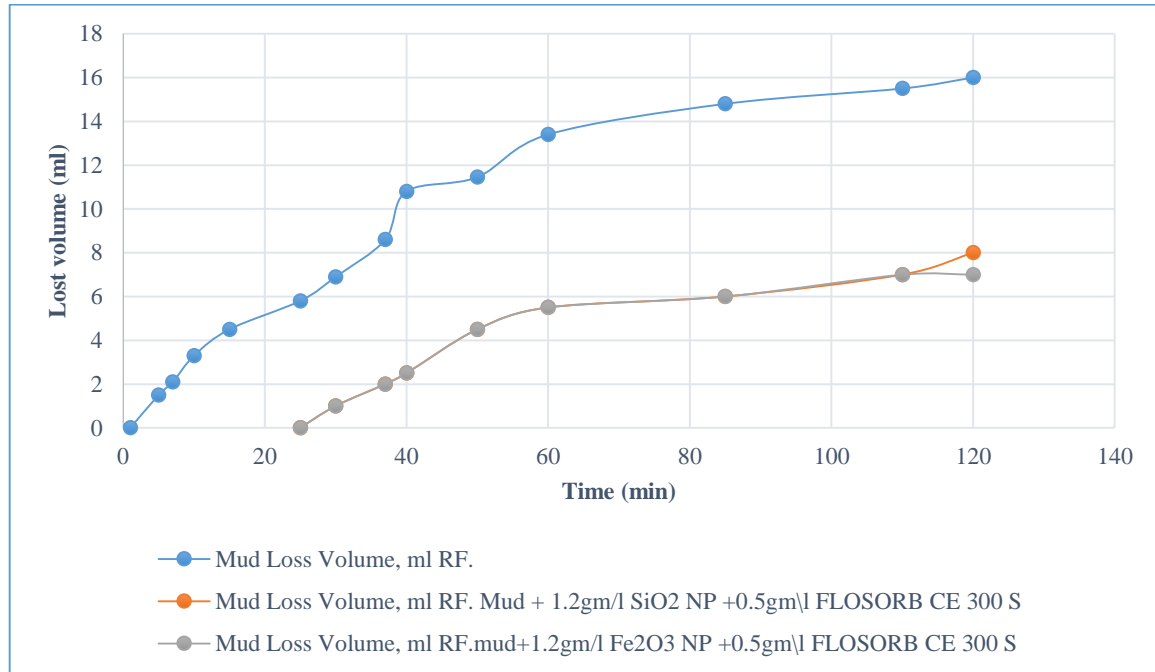

Figure 6. A comparison between the effect of adding RF. mud+ 1.2 g/l SiO2 NP + 0.25 g/l FLOSORB CE 300 and RF. mud+ 1.2 g/l Fe2O3 NP + 0.25 g/l FLOSORB CE 300 to the reference mud for core plug 1.

Figure 7.A comparison between the effect of adding RF. mud+ 1.2 g/l SiO2 NP + 0.25 g/l FLOSORB CE 300 and RF. mud+ 1.2 g/l Fe2O3 NP + 0.25 g/l FLOSORB CE 300 to the reference mud for core plug 2.

Figure 8. A comparison between the effect of adding RF. mud+ 1.2 g/l SiO2 NP + 0.5 g/l FLOSORB CE 300 and RF. mud+ 1.2 g/l Fe2O3 NP + 0.5 g/l FLOSORB CE 300 to the reference mud 1.

Figure 9.A comparison between the effect of adding RF. mud+ 1.2 g/l SiO2 NP + 0.5 g/l FLOSORB CE 300 and RF. mud+ 1.2 g/l Fe2O3 NP + 0.5 g/l FLOSORB CE 300 to the reference mud 2.

4. CONCLUSIONS

1. The performance of SiO_2 NP as LCM added to the reference mud is better than Fe_2O_3 NP the reduction in fluid loss is up to of 37.9 % for Fe_2O_3 NP the and 48.3 % SiO_2 NP. Where the reduction in mud volume losses with SiO_2 NP is greater than Fe_2O_3 NP.

2. Combined two types of LCMs are well capable of controlling lost circulation. Where an enhancement was observed with SiO_2 NP - FLOSORB CE 300 S mixture and Fe_2O_3 NP- FLOSORB CE 300 S mixture.

3. A mixture of Fe_2O_3 NP- FLOSORB CE 300 S is more efficient than SiO_2 NP - FLOSORB CE 300 S even with a low concentration of FLOSORB CE 300 S in the mixture.

4. Using multiple LCMs cures losses better than using single LCM.

5. ABBREVIATION

LCM: Lost circulation materials.
NP: Nano-particles.
PV: Plastic viscosity.
RF mud: Reference mud.
SG: Specific gravity.
YP: Yield point.

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