NTNU – Trondheim
Norwegian University of
Science and Technology

Investigation of Nanoparticles for Enhanced Filtration Properties of Drilling Fluid

Frode Aarstad Riveland

Petroleum Geoscience and Engineering
Submission date: June 2013
Supervisor: Sigbjørn Sangesland, IPT

Norwegian University of Science and Technology
Department of Petroleum Engineering and Applied Geophysics

NTNU
Norges teknisk-naturvitenskapelige universitet
Studieprogram i Geofag og petroleumsteknologi
Study Programme in Earth Sciences and Petroleum Engineering

Fakultet for ingeniørvitenskap og teknologi
Faculty of Engineering and Technology

Institutt for petroleumsteknologi og anvendt geofysikk
Department of Petroleum Engineering and Applied Geophysics

HOVEDOPPGAVE/DIPLOMA THESIS/MASTER OF SCIENCE THESIS

Kandidatens navn/The candidate's name:	Frode Aarstad Riveland
Oppgavens tittel, norsk/Title of Thesis, Norwegian:	*Undersøkelse av filtrereringsegenskaper til borevæske ved anvendelse av nanopartikler*
Oppgavens tittel, engelsk/Title of Thesis, English	*Investigation of Nanoparticles for Enhanced Filtration Properties of Drilling Fluid*

Utfyllende tekst/Extended text:

Background:

In the past 10 years nanotechnology has contributed to new developments in the oil industry. The drilling fluid is already one of the most advanced fluids that exist, some of the drilling fluids tasks are to cool and lubricate the drilling bit, transport cuttings out of the well, prevent formation damage, keep the shale stable, be thermally stable, easy to pump, environment friendly and stabilize the pressure in the well. Within these ten years, nanotechnology has helped to improve the drilling fluid to overcome these tasks. The research is however in the starting phase and there is no commercial available option. Improved filter cake and rheological properties can lead to benefits like reduced formation damage and improved recovery, less friction and longer wells and more stable boreholes.

Task:

1) Perform laboratory tests with WBM and OBM using selected types of nanoparticles
2) Investigate further the most promising nanoparticles (nanofluid) using the MCR302 rheometer
3) Evaluate and compare the results and propose further work.

Supervisor	*Sigbjørn Sangesland*
Co-supervisor	*Pål Skalle, Jianying He*
Studieretning/Area of specialization:	*Petroleum Engineering, Drilling Technology*
Fagområde/Combination of subject:	*Drilling*
Tidsrom/Time interval:	*January 14 – June 10, 2013*

……………………………
Sigbjørn Sangesland

Acknowledgement

I would like to thank MI-SWACO, Grolman Nordic AS and Elkem for supplying material. I would also like to thank Jianying He and Pål Skalle who have been my co-supervisors for their support and advice. I appreciate the help and support I have received from Sigbjørn Sangesland who has been my supervisor and helped me with this report.

Sammendrag

De siste 20 åra har nanoteknologi bidratt til nyskaping innen medisin, elektronikk og bioteknologi. De siste 10 år har det vært fremgang også innen nanoteknologi i olje industrien. Borevæske er en av de mest avanserte væsker som finns. Oppgavene til borevæska er å smøre og kjøle borekrona, transportere borekaks ut av brønnen, hindre formasjons skade, holde formasjonen kjemisk stabil, være termisk stabil, lett å pumpe, miljøvennlig og stabilisere trykket fra formasjonen. Gjennom disse ti åra har det blitt gjort forskning for å forbedre borevæskas ytelse med nanoteknologi. Denne rapporten vil ha fokus på å forbedre filterkaka og dermed redusere filtrat tap til formasjonen og hvordan nanopartikler påvirker reologien i borevæska.

Hoved eksperimentet i denne rapporten har vært en standard 30minutters API lav temperatur lav trykk filter test for å se påvirkningen av nanopartikler for forbedret filterkake. Målet har vært å finne rett type nano partikler og rett konsentrasjon for best påvirkning på filterkaka. Det ble forsøkt forskjellige størrelser og typer nanopartikler, hovedegenskapene til de utprøvde partiklene var olje fuktende, vann fuktende, porøs og kompakt. Disse partiklene ble prøvd i oljebasert og vannbasert borevæske. Den høyeste reduksjonen i fluidtap for vannbasert borevæske var 22,5 %, denne typen nanopartikler gav også mindre heldige egenskaper til borevæska som større filterkake og høyere viskositet. Andre typer nanopartikler gav høyere fluidtap men samtidig lavere viskositet og høyere bristepunkt. Nanopartikler i oljebasert borevæske viste en reduksjon i fluidtap fra 0,8 til 0,5mL (62,5%), både referanseprøven og nanofluidet viste lite fluidtap og det er derfor satt spørsmålstegn til hvor store feilkildene har vært i dette forsøket. Dette nanofluidet gav samtidig ingen økning i filterkakens tjukkelse eller tetthet samtidig som det minsket viskositeten og gav et høyere bristepunkt.

Partiklene som muligens gav en reduksjon fra 0,8 til 0,5mL ble etterforsket videre i et mer presist reometer og det ble gjort tribologitester av nanofluidet. Det viste en høyere statisk friksjon og en ujevn dynamisk friksjon. Reometeret bekreftet også at nanofluidet hadde et høyere bristepunkt og en lavere viskositet ved høy skjær rate.

Filtrat testene med olje basert slam gav lave fluidtap som var en av utfordringene i denne rapporten. Det ble derfor foreslått videre arbeid med en annen test for å gi mindre feilkilder og gjør testene mer reel. Høy temperatur og høyt trykk filter test med keramisk filter er derfor foreslått. Forskjellige partikler med forskjellig overflate struktur burde bli testet, tidligere studier viser at jern baserte partikler har gitt gode resultater og anbefales derfor for videre testing. Andre studier viser til at partikler klumper seg og at overflate modifikasjon med silaner, alkoholer og organiske syrer er godt egnet til å bekjempe dette. Permeabilitets tester og retur permeabilitet bør også bli testet gjennom en kjerneprøve etter at fluidet har vist gode indikasjoner i HPHT eller LPLT tester. Jern og silica baserte nanopartikler har vist gode tribologiegenskaper i tidligere studier. En ball med plate test som den som ble gjort i denne studien kunne blitt studert nærmere med disse typer partikler.

Forsøkene viser at partikler som er blandbare gir generelt bedre borevæske. I denne studien ble det ikke funnet en spesiell type nano partikler eller konsentrasjon som forbedret borevæska på samtlige kriterier. Det ble foreslått at konsentrasjonen av nano partikler holder seg under 1 vekt prosent for praktiske og økonomiske årsaker.

Summary

In the past 20 years, the use of nanotechnology has contributed to new developments within medicine, electronics and biomaterials. In the past 10 years nanotechnology has also made progress in the energy industry. The drilling fluid is already one of the most advanced fluids that exist, some of the drilling fluids tasks are to cool and lubricate the drilling bit, transport cuttings out of the well, prevent formation damage, keep the formation chemical stable, be thermally stable, easy to pump, environment friendly and stabilize the pressure in the well. Within these ten years, research has been done on nanotechnology to improve the drilling fluid performance. This report will focus on improvement of the filter cake performance to reduce the fluid loss rate to the formation, and how the nanomaterial affects the drilling fluid rheology.

The main experiment to investigate the impact of nanoparticles to improve the filter cake was the API low temperature low pressure 30 min filter press test. The goal was to find the right type of nanoparticle and the optimum concentration. Different sizes and types of nanoparticles were tested. The main types were oil wet, water wet, porous and compact. These were tested in oil based and water based muds. For water based mud a reduction of 22,5 % was achieved but it also gave other complications like thicker mud cake and higher viscosity. Other nanoparticles gave a higher fluid loss rate but lower viscosity and higher yield point. The nanoparticles in the oil based mud showed a reduction in the fluid rate loss from 0,8 to 0,5 mL (62,5%). Both the base case and nanofluid showed little fluid loss and therefore the error sources might play a large role and the data are not trust worthy. This nanofluid showed no increase in filter cake thickness or in density, the fluid showed a higher yield point and a lower viscosity at high shear rate.

The particle that might have given the reduction from 0,8 to 0,5 was further investigated in a more precise rheometer and tribology test. It showed a higher static friction and an uneven dynamic friction. This new rheometer also confirmed that the nanofluid had a higher yield point and a lower viscosity at high shear rate.

The filtration tests with oil based mud gave low fluid loss rate which was one of the challenges in this report. It was therefore suggested for further work to do a different test to lower the sources of errors and make the test closer to real life. High pressure high temperature filter test with a ceramic filter was therefore suggested. Different particles should be tested and previously published studies shows good results with iron based particles and is suggested as one of the particle types that could be tested. Other papers also show a high degree of agglomeration among nanoparticles and that by surface modification with silanes, organic acids or alcohols could help. After a fluid has shown good indications with a LPLT or HPHT test flow through a core sample to test the permeability is suggested also to flow back to test the return permeability is advisable. Iron and silica based nanoparticles have shown good tribology properties in previous studies, ball on plates test as the one done in this study can also be investigated further with these kinds of nanoparticles.

This study shows that the particles that were miscible were favorable. It also concludes that there were not found a specific nanoparticle or concentration that significantly improved the drilling fluid. A suggestion was made to keep the concentration below 1 wt% for practical and economic reasons.

Table of Contents

List of Figures

List of Tables

1. Introduction

The demand for energy in the world is increasing hand in hand with the demand for oil. The easy oil is gone and the wells drilled now are increasingly more difficult. One of the most important factors for drilling advanced wells is the drilling fluid. Nanotechnology in drilling fluid may prove to be a new generation of drilling fluid. In drilling fluid the nanoparticles can reduce friction and wear and improve the rheology of the fluid. Nano technology could be used to enhance the mudcake, stabilize shale, protect against corrosion and enhance cement. The cost of lost drilling fluid can be significant, also loosing mud into the formation can decrease the production capability of the well due to increased skin factor. This study has tried to improve filtration properties of the drilling fluid with adding nanoparticles to it. The nanoparticles are supposed to go in between the larger particles and block the flow through them. The study has also looked at how the rheology profile changes with the addition of the particles.

1.1 Current solutions & Challenges

Currently only a handful papers have studied the effect that nanoparticles have on the filter cake. The common practice in the industry is to add macro materials or LCMs like bentonite, barite, calcium carbonate or graphite to build a decent filter cake. When allowed by the government, it is also possible to use an oil based mud which gives little fluid loss.

The fluid loss most often occurs in porous formation with pore throats between 0,1 µm-1mm where typically LCMs in the same size may play an important role. However in the pore size opening in shale are in the range of 10nm to 0,1µm where typical LCM may be too large and the nanoparticles can influence the fluid loss in a positive way with the right size, hydrodynamic properties and the area to volume ratio. [1]. The nanoparticles can also work together with the LCM in porous media, the smaller particles works together with the larger particles and block the fluid flow to enhance the seal. The challenge is to find the right particle which is inert to the surrounding environment and the right size range so the particles fit well together with both the particles in the fluid and the pore size of the formation. Also the cost is an issue, the concentration should therefore be as low as possible. The concentration should also be as low as possible due to the total solid concentration may affect the rate of penetration. Generally more solids in the fluid lower the rate of penetration.

1.2 Goals

Nanoparticles are defined as particles in the range of 1-100nm. A nanofluid is any kind of fluid, drilling fluid, drill-in fluid with suspended nanoparticles. The nanoparticles are smaller than the micro particles used in the mud today and have a higher area to volume ration which gives the surface properties more influence than the same particle with a larger size. The nanoparticles in drilling fluid may give better control of both the fluid loss to the formation and the initial spurt loss. This Master thesis aims to explore the drilling fluid with nanoparticles to gain experience of what the nanoparticles do to drilling fluids. The main focus will be to investigate what different nanoparticles do to the fluid loss properties of a drilling fluid. A reduction of the spurt loss and fluid loss can give less damage to the formation and total fluid loss. Thus the right combination of mud particles and nanoparticles can be economically beneficial. A secondary objective is to see how nanoparticles can affect the drilling fluid in other ways, see if there are any changes in rheological or tribological properties. Is there any unique signature for all nanoparticles or does each type of nanoparticle affects the drilling fluid in its own way. The paper will explore if nanoparticles can give rheological properties to the fluid to improve the energy loss when pumping, and in the meantime hold suspensions when the fluid is not circulating. Previous papers have shown that the nanoparticles can reduce wear and friction between steel and steel and paraffin as base fluid [18]. This study will look at tribology properties in a drilling fluid with good filtration properties on a ball and plates test.

1.3 Approach

There is not much work done on this subject, a couple of papers have independently investigated nanomaterial in drilling fluid for enhanced filtration properties two of the published papers are Maen Hussein [16] and Ammanullah M.D. [1]. In their studies they show that for certain kinds of fluids and nanoparticles there is a reduction of fluid loss. However, they fail to report what kind, what surface properties the nanoparticles have or their size. Due to the little information there is in this field, more laboratory experiments are therefore needed and this Master thesis will aim to do so.

2. Introduction to nanotechnology

Riveland 2012 [2] made an up to date report on studies on “Applications of Nanotechnology in Drilling and Completions” and wrote “Nano means billionth but comes from an ancient Greek word where it meant dwarf. Some describe nanotechnology to be engineering of functional systems at the molecular scale [3]. In most literature nanotechnology concerns systems in the range of 1-100nm. To put this in perspective the width of human hair is around 100 000nm, a blood cell is around 7 000nm, HIV-virus 100nm and a gold atom diameter 0.1nm [4]. This new science has in the last few decades made significant contributions to technologies in electronics, biotechnology, pharmaceutics and medicine. The nanotechnology is more than just the small size, according to Smalley and Yakobsonb (1998)[5] and Zhou et al. (2005) [6] the behavior of nanoparticles are significantly different from that of macro and micro sized particles even though they come from the same mother source.

Nanofluid is a fluid with at least one component in the size range of 1-100nm. Typical nanoparticles can be metals, metal oxides, carbides or fullerenes. These nanoparticles give desired properties to the fluid, like enhanced thermal conductivity, enhanced tribological effects, and enhance chemical or rheology improvements. The surface area of the nanoparticles may play a major role in these effects along with the surface chemistry. In Figure 1 it can be observed how much the surface area is increased over the same volume with different particle sizes and may indicate why the nanoparticles are so special.

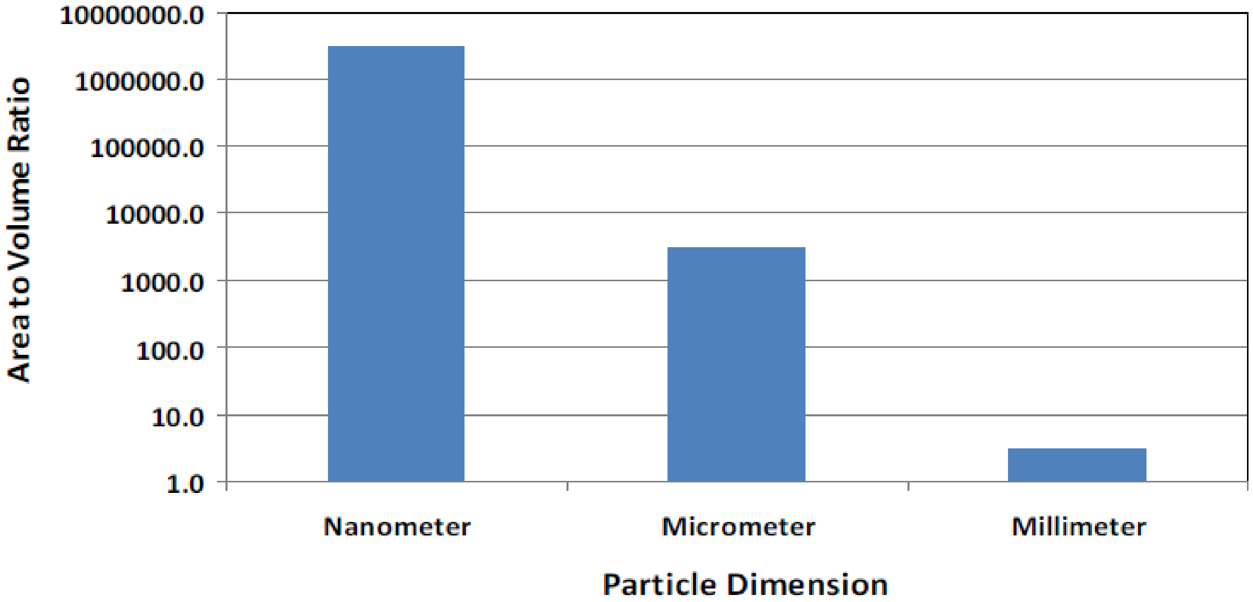

Figure 1 Area to volume ratio vs. spherical particles with diameter of 1nm, 1µm and 1mm [1]

2.1 Creating nanoparticles and fluid

Nanostructures can be made in a couple of ways. The original approach is the “bottom-up” approach. This approach fabricates the structure at atomic or molecular level with self-organizing chemical syntheses of each building block. This procedure can be done in either vapor, liquid or solid phase [7] and is them chemical method.

The second approach is the top-down, this is a physical method and depends on removal of bulk volume in order to create a nano structure. A common method in this approach is the lithographic, it uses either X- rays, ultraviolet light or ions to create the nano structure [7]. Other approaches are biomimetic and functional.

To prepare a nanofluid there are two common ways, a two-step method or the one-step method. The first step of the two-step method is to produce a fine nano powder, and the second step is to mix the nano powder with fluid. The two-step method is the most economical way to produce nanofluids, this is due to nanoparticles are already made on an industry scale. When the nanoparticles are mixed with the fluid it is important to disperse the particles properly. This is done by intensive magnetic force agitation, ultrasonic agitation, high-shear mixing, homogenizing or by ball milling [8]. Even though these methods are useful to disperse the nanoparticles in the fluid, the nanoparticles tend to aggregate with this method. The particles can aggregate both when they are in powder-form and when they have been added to the nanofluid [8]. This affects the stability of the nanofluids, but the stability can be improved by surfactants, surface modification and manipulation of zeta potential.

The one-step method was developed to improve the stability of the nanofluids by minimizing the agglomeration of the nanoparticles [9]. This is done by producing the nanoparticles directly into the nanofluid and thus avoiding the time-depending agglomeration that can occur while transporting, creating or stocking the nanoparticles. The downside of this method is the production cost, production amount, residual reactants in the nanofluid and the method is only compatible with low vapor pressure fluids [8].

2.2 Surface modification

The surface chemistry of a nanoparticle can change the properties of a nanofluid a lot due to the high surface area to volume ratio. By modifying the surface, the surface chemistry changes and thus the nanofluid properties can change [10]. Common surface modifying groups are organic acids, silanes or alcohols [11]. These surface modifying groups can improve the stability of the nanofluid by changing the zeta potential or by steric hindrance [10] (modify the surface molecular groups so other reactive groups on the particle cannot react [12]).

By use of surface modification the particles can be customized. For example the particles can be silanated with different functional groups in order to achieve desired properties. This can improve stability and performance in high brine and temperature to change the wettability of a reservoir [10]. The particles can also be surface modified to make them hydrophilic, hydrophobic or both.

Md. Amanullah et al. [1] wrote a paper in 2011 where they produced a mud with nanoparticles to deposit an ultra-thin, tight and well dispersed mudcake. They made it in a standard API test. Md. Amanullah et. al. [1] found that with the nanofluids that was used to make this mudcake gave no spurt loss. They also compared it to a bentonite mud. Bentonite is a clay mineral composed of three clay layers and is used for viscosity and filtration control [13]. In the same test the bentonite fluid showed a spurt loss of 2cc. One of the major factors to create formation damage in the production zone is the spurt loss which gives this nanofluid an interesting advantage.

The spurt loss in the formation causes damage when the particles in the spurt loss get stuck inside the pores of the rock. When it is impossible to get these particles out of the formation by cleaning or production, the damage is permanent. This will affect the return of investment on the well [1]. Another benefit of the thin external mudcake is the ease of cleaning. When there is no spurt loss, the cleaning fluid with the hydrodynamic force, is expected to effectively wash away the external mudcake. This is due to the fact that the cleaning fluid will have increased contact with the particles. With a cleaner wellbore the chances of getting a high quality cement job increases [1]. A poor cement job due to bad cleaning of the mud cake, can lead to lost reserves of oil, increased water production and worsen the productivity [14].

The mudcake they got was less than 1mm thick. A thinner mudcake can remove the differential sticking problem and thus decrease the nonproductive time and be good for economics. A thick, poorly packed mudcake can give a high torque and drag in a long horizontal well compared to this an ultrathin, well packed mudcake from a nanofluid has the possibility to reduce the torque and drag significantly [15].

The solid content in this fluid used by Amanullah 2011[1] was lower than the bentonite mud he compared it with. The solids content of the drilling fluid is a factor that may increase formation damage, reduce productivity index and decrease ROP(Rate of Penetration). With this in mind it is advisable to have as little solids as possible. From Figure 6 the solids content of the nanofluid is less than 1% (w/w), this shows that the nanofluid may play an important role to increase the ROP

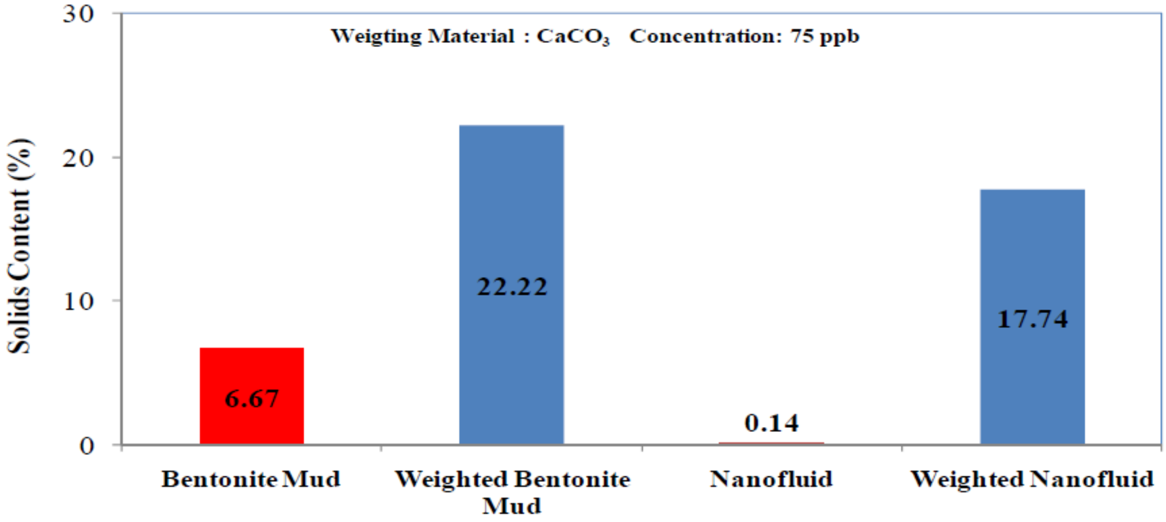

Figure 2 Solids Content of Nanofluid vs. Bentonite mud [1].

Maen Hussein [16] also did a study on the performance of drilling fluids using Nanoparticles. He used off-the-shelf available and in-house prepared particles like iron based, Calcium based and Barium based. They did experiments with both inverted drilling fluid, water based mud in a low pressure low temperature filter press. For the water based mud they reduced the amount of filtrate from 9% with iron based nanoparticles to 32% with calcium based particles. For the base case they used standard drilling fluid. In the LPLT filter press they also tested an invert emulsion with different nanoparticles. Here the reduction in fluid loss over 30 minutes was higher, from 55% with calcium based nanoparticles to 91,5% with iron based nanoparticles. In other words the invert drill fluid had a fluid loss of 11.0+- 0,3mL to drill fluid with iron based nanoparticles to 0.93+-0.1mL. A high pressure high temperature filter press was also used. The filter press had a temperature of 177°C (350°F) and a pressure of 500psi and the filtrate were collected in a 30 min time period, this apparatus is supposed to mimic the down hole conditions. The drilling fluid in this test was OBM with OWR 90/10, in these kinds of conditions Maen Hussein and his associates managed to reduce the filtrate loss with 86%. In the study they also tested the friction from the drilling fluid and got a reduction of 38 % with the calcium based particles and 59% reduction with the iron based particles. This friction reduction can result in extended wells with over 1000m as seen in Figure 3.

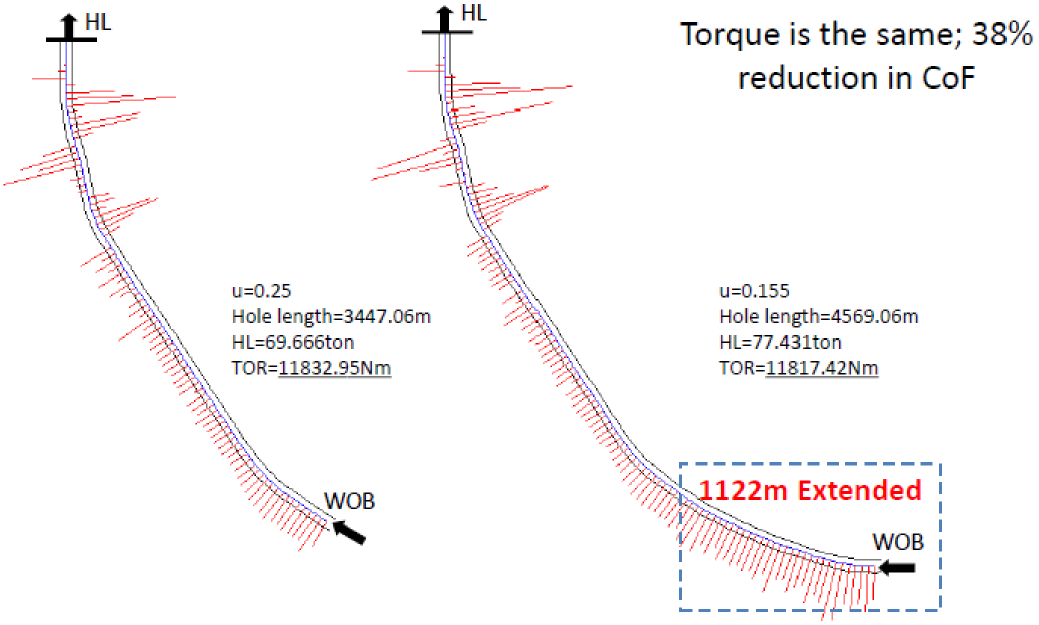

Figure 3 Simulation of 38 % reduction in friction [16]

Sushant Agarwal and his coworkers [17] did a study on Nanoparticle-Stabilized invert Drilling fluids for deep-hole drilling for oil and gas. They used Aerosil's nanoparticles R104 and R106 that respectively have mean size of 12nm and 7nm diameter as emulsifier to stabilize the emulsion in harsh conditions. The fluid contained nano clay as thickening additive that worked together with the nano silica for the stability properties. The test was heated and aged before testing the rheology. They use of 225°C aging cells for 96h before the rheology was tested. Judging by the time before the fluids separated the samples containing the nanoparticles was more stable than the samples without. The samples with nanoparticles could stay emulsified for a few months and the ones without any emulsifier were stable for a few hours. The emulsion showed more shear thinning properties with the added nano silica and kept its flow properties through the aging time.

A good way to reduce the torque and drag of the drill string is to reduce the friction between the drillstring and the casing or open hole sections. Peng 2010 did a study [18] with silica as oil additives in different sizes to find tribological properties of the fluid. They used a steel ball on ring tester to determine the optimal concentration and size of the silica particles. They made the particles by the sol-gel method and obtained particles ranging from 684nm to 58nm and the oil they used in this experiment was paraffin. They also changed the surface properties of the nano silica to better disperse in paraffin with Oleic acid.

Peng 2010 [18] explains that authors have suggested that in pure paraffin liquid the main wearing mechanisms are adhesion and contact fatigue in sliding friction. While in paraffin with nano silica particles the particles can easily penetrate into the rough surfaces between the two sides because of the nano size. The nanoparticles then forms a tribological film that can bear and separate the rubbing faces. This causes the spherical nano silica particles to alter the friction mechanism from sliding to a mix of sliding and rolling causing a drop in the friction coefficient. Peng [18] also explains that with experimental results it is this ball-bearing effect and polishing done by the nanoparticles that cause the reduction in both wear and friction.

Peng [18] found that in his test silica particles over 362nm was not able to go between the two rubbing sizes and therefore was not able to create the protective film causing an increase in friction and the particles started to create larger wear scars. For the particles under 362nm the film was created and both the wear scar diameter and friction coefficient goes down. The lower the diameter of the particles the lower the friction coefficient and wear scar diameter is. However Peng [18] did not use smaller particles than 58nm in diameter. Another thing to note in Figures 4 and Figure 5 is that as the particles size goes down the optimum wt% goes down to minimize the wear.

In Figure 6 it can also be observed that larger particles than 362nm creates larger wear scars than pure paraffin, and when the load exceeds 200N the pure paraffin increases significantly while the paraffin with particles only increases slightly. The figure shows that particle sizes under 362nm has better load carrying capacity than the pure paraffin, at 250 N the pure paraffin has a wear scar diameter of 1.6 mm and the paraffin with 58nm 0.2 wt % silica particles has 0.95mm wear scar diameter a reduction of 41%.

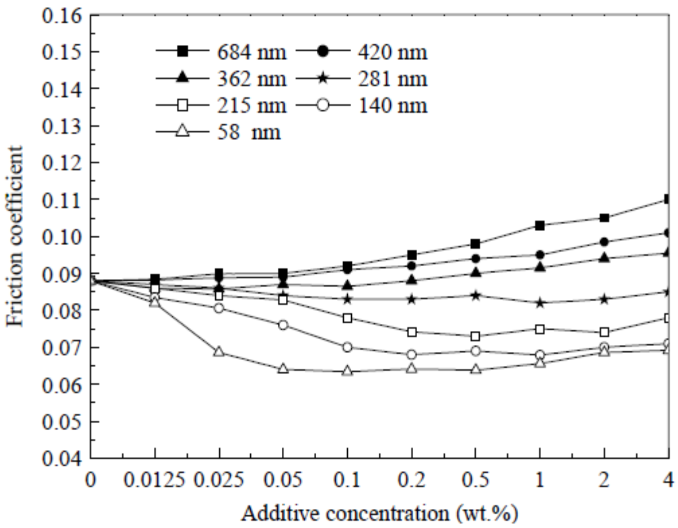

Figure 5 Friction coefficient vs additive concentration [18]

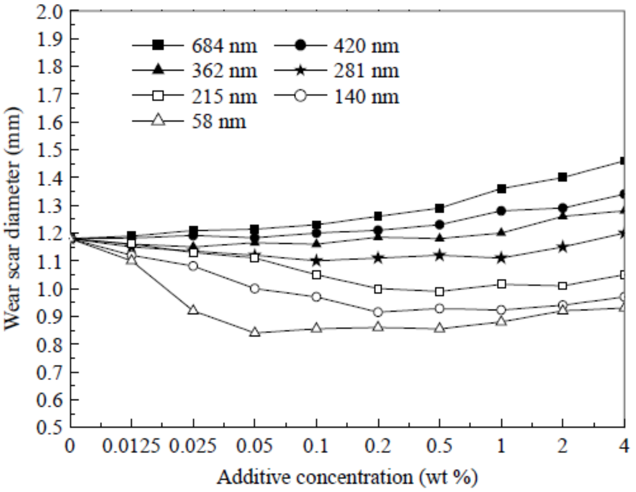

Figure 4 Wear scar diameter vs additive concentration [18]

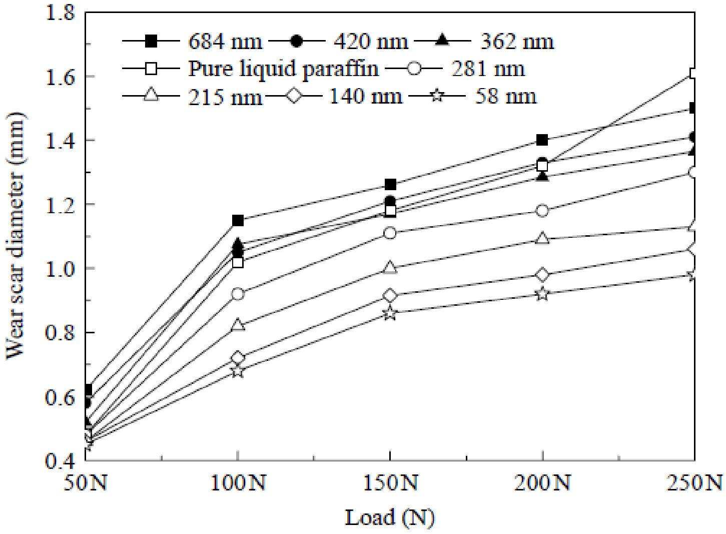

Figure 6Weaer scar diameter vs load (N) [18]

YU He-long et. al. [19] did a study on copper nano-particles with oil as the base fluid in a high temperature environment. The nanoparticles were surface modified with an organic layer to protect the particles from oxidation and improve their dispersability. They found that the copper particles would melt because of the high temperature and pressure between the two surfaces. This gave a copper film and a significant reduction in both wear and friction coefficient. The below figures shows how friction is reduced with nanoparticles in different temperatures"

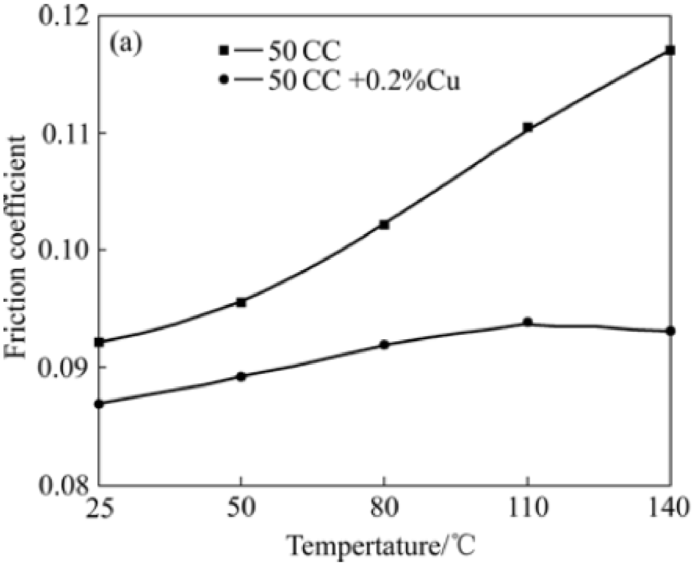

Figure 7 Friction coefficient vs. Temperature [19]

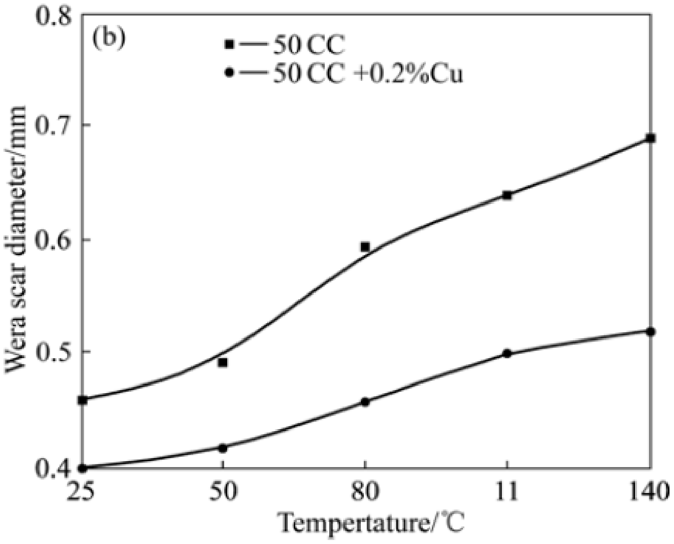

Figure 8 Wear scar diameter vs temperature [19]

3. Experiments and theory behind

There are different kinds of drilling fluids, the simplest of them are a mix of water and clay. The wells being drill now calls for more advanced drilling fluid because of depth, pressure, temperature and geology.

The main tasks of the drilling fluids are always the same. One of the main tasks for the drilling fluid is to act as the first pressure barrier against the pressurized fluids in the reservoir. In deep wells water alone has far too low density to make sure that the well is drilled overbalanced. For deep wells drilling fluid may be required to have twice the density of water. Barite is a typical additive to control the density of the drilling fluid.

Another important task of the drilling fluid is to bring back cuttings from the formation to the top of the well. In regular water the viscosity is too low to transport cuttings back to the top, or to make sure the cuttings don't fall deeper into the well in case the fluid circulation stops. Common viscosifyers are clays typical bentonite or polymers. The mud is also important to remove cuttings from under the drill bit and that's where most of the pump pressure is used.

Other tasks are stabilizing the wellbore, cool and lubricate the drill bit and string, bring back information to the surface and avoid losing mud into the formation. The fluid can react with certain types of formation, or the pressure can cause the rock to crack, leading to massive loss of fluid into the formation. Avoid losing mud to the formation is extra important when drilling in the reservoir. If a large amount of drilling fluid goes into the reservoir, the mud can contaminate the area around the well so that the well is nonproductive and has to be abandoned. Even if a small amount of fluid goes into the formation it can plug the formation and decrease the permeability. Worse permeability can reduce the income of the well. Good filtration properties for the fluid are thus important.

It's not only water that can be used in drilling fluid. There are several different mud systems. The easiest is a non-dispersed mud, which can be natural muds or lightly treated muds. These muds are mostly used for top hole or shallow drilling.

A dispersed mud system is used at deeper sections of the well. This can have two or three phases, water and solids or water, oil and solids. These types of mud are good filtrate reducers.

Calcium or magnesium treated muds may inhibit the swelling of clays and shale. Also to inhibit hole enlargement and to prevent formation damage [20].

Polymer can also be used to increase the viscosity of the fluid, reduce the filtrate loss and stabilization of the formation. There are many types of polymer, they can be cross linked or bio polymers. Generally for polymers the concentration needed to obtain a good effect is very low [20].

3.1 Filtration theory

Fluid loss is defined as loss of a mud filtrate into a permeable formation that is being drilled. The loss is due to a positive differential pressure between the fluid column in the wellbore and the formation fluid pressure. When there is a positive differential pressure between the two fluids fluid flows into the formation while most of the solids deposit on the wellbore wall, the solids build what is commonly called mud cake or filter cake. As more and more solids builds up, less and less fluid goes into the formation and the filter cake forms a barrier between the two pressurized fluids. The initial fluid that flows into the formation before any solids have built any filter cake is called spurt loss. After this initial loss the fluid loss is called continuous fluid loss [21].

Some of the common experiments to conduct fluid loss tests are API static filtration test (most common is the low pressure low temperature, LPLT, second is with high temperature high pressure, HPHT) and dynamic filtration test. The dynamic filtration tests are only done in laboratory conditions. The dynamic test circulates mud flow while the solids are deposited and the fluid is filtrated. The static tests are done while the fluid is at rest. The low temperature and low pressure can be done in room temperature with a 7 bar differential pressure. The HPHT is done 34,5 bar and 150 degrees Celsius [21].

3.1.1 Factors affecting fluid loss

When a filter cake has been made the fluid flows through a porous media, therefore a good starting point is the Darcy equation:

$$\frac{dQ_f}{dt} = \frac{kA\Delta P}{\mu h}$$ EQ 1.1

Where

- Q_f is the amount of filtrate volume
- k is the filter cake permeability
- A is the filtration area
- ΔP is the differential pressure
- t is the time of filtration
- μ is filtrate viscosity
- h is the thickness of the filtrate cake

If the assumption is made that all mud volume Q_m is filtrated. A volume and material balance can be written [21]

$Q_m = Q_f + h*A$ EQ 1.2

$Q_s = f_{vc}*h*A$ EQ 1.3

Where

- Q_m is the total volume of mud filtrated.
- Q_s is the volume of solids in the deposited mud cake
- f_{vc} is the volume fraction of solids in the filter cake.

The volume fraction of the solids in the mud can be written:

$$f_{vm} = \frac{Q_s}{Q_m} \quad \text{EQ 1.4}$$

Inputting equation EQ 1.2 and EQ 1.3 in to EQ 1.4 gives us EQ 1.5

$$f_{vm} = f_{vc} + \frac{h*A}{Q_f+h*A} \quad \text{EQ 1.5}$$

Solving this for the mud cake thickness h, gives:

$$h = \frac{Q_f}{A[\left(\frac{f_{vc}}{f_{vm}}\right)-1]} \quad \text{EQ 1.6}$$

Inputting equation EQ 1.6 to darcy equation 1.1 and assuming that Q is the only time dependent parameter and integrating gives [21]:

$$Q_f = A\sqrt{\frac{2k\left[\left(\frac{f_{vc}}{f_{vm}}\right)-1\right]*\Delta P*t}{\mu}} + Q_s \quad \text{EQ 1.7}$$

Where Q_s is a constant of the integration, equal to spurt loss at t=0.

From this derivation it can be seen that filtrate volume is dependent on the following parameters:

- Time
- Differential pressure
- Solids (amount, type, size and size distribution)
- Permeability of mud cake
- Filtrate viscosity

As seen from equation 1.7 the time is proportional to the square of the filtrate volume if the spurt loss is 0. To start the restriction of flow and a mud cake build up there has to be a spurt loss. Therefore a common mistake in testing is to test only the first 7,5 minutes and multiply it by 2 to get the total 30 minutes of filtrate volume. This practice assumes zero spurt loss which is never true, but the reasoning behind it is that the square root of the ratio between 30 and 7,5 is 2. In some cases the spurt loss can be close to zero and in some cases it can be significant and thus the error becomes significant as well [21].

The pressure differential in the equation is not only determined by the how much the pressure difference increases but also the compressibility of the mud cake. With a highly compressible filter cake, the permeability of the filter cake will be reduced and thus give less increase in the fluid loss than if it were a non-compressible filter cake giving no reduction in the permeability [21].

The solids are a significant part of what dictate the filtrate loss. The size distribution, amount and the properties of the solids are a part of what gives the permeability. The solids give the factor$\left[\frac{f_{vc}}{f_{vm}} - 1\right]$ as well, an increase in solids will decrease the fluid loss and in the meantime decrease the rate of penetration when drilling [21].

It is a given that the filter cake permeability is a part of what contribute to the fluid loss. With higher permeability the higher filtrate volume will be created. As mentioned the solids are important when packing the filter cake to a tight and low permeability filter cake. Shape, size and deformability of the solids must be taken into account when designing a fluid with good filter properties [21].

In the equation 1.7 it can be observed that the filtrate volume loss is inverse proportional to the filtrate viscosity. A higher viscosity in the filtrate can therefore reduce the filtrate loss. This can be obtained by increasing the viscosity of the mud, but this is not always practical because it can cause other drilling related problems [21].

There are many different additives to the mud to reduce the filter loss. Some of the more common ones are bentonite which increases viscosity, reduces the filter loss and adds more solids to the mud. Bentonite gives a good base filter cake. Other additives that reduce the filtration and increase the viscosity are the sodium poly acrylate, CMC (carboxymethylcellulose) and other polymers. CMC reduces the filtration rate by minimizing flocculation and by coating the solids. There are even viscosity thinners that reduce the filtration rate. Lignosulfonate is such an additive and reduces the filtration by deflocculating the mud, but increases the viscosity of the filtrate. Liginite is another thinner that also deflocculates the mud and plugs the void spaces in the filter cake [21].

The temperature also has an effect on the filter loss properties. Higher temperature gives the mud lower viscosity and from equation 1.7 we can see that it gives higher fluid loss. The temperature effect can somewhat be reduced by the compressibility of the mud cake especially if there are a high differential pressure. An incompressible mudcake such as in bentonite muds the filtration rates are more or less the same at the same differential pressures. On the other hand with other additives like polymer the filter rates are pressure related [21].

The packing of the material in the mud cake determines how well the cake is compacted. In the mud there can be coarse particles like native clays, shale, barite and silt. If there are such particles the filter cake will be less compacted and give a higher filter loss than if all the particles are of finer particles like a mud with good quality bentonite, PAC (polyanionic cellulose), and barite. When drilling there will always be native solids in the mud, but with good mud control it is possible to hinder that one size dominates the particle size distribution (PSD). A well balanced size distribution will give the best possible mud with regards to the filter cake properties. When the filter cake starts to form, depending on the PSD small channels will appear. The smaller channels the higher pressure is needed to press the fluid through. Thus small particles and a wide spread in the PSD result in a densely packed filter cake with reduced filtration [21].

3.1.2 Zeta potential

Zeta potential is the measured point between two particles in solution and the strength of the electric charge between them. All particles in solution have an electric charge either positive or negative charge with a hydrodynamic diameter. Positive electric charge corresponds to a positive zeta potential and vice versa. Equal electric charges repel each other and opposite attract each other, so a large zeta potential (positive or negative) will make a solution of particles more stable because the particles will repel each other. On the other hand in brines or ionic solutions a large zeta potential will destabilize the solution due to the strong attraction between the particles and the ions [19]. Paul McElfresh, Marodi Wood and Daniel Ector 2012 [10] wrote that the manipulation of zeta potential and charge density is critical to stabilize the particles in high brine and temperature. The zeta potential can be manipulated by changing pH or by surface modification of the particle. Nano silica particles tend to be negatively charged and lowering the pH value of the fluid will cause the hydronium ions to shield the negatively charged nano silica particles and thus neutralize the zeta potential [19].

3.2 Rheology

Viscosity is the fluids internal resistance to its forced flow, or in other words how thick the fluid is. As have been shown previously the viscosity has an influence on the fluid loss properties. The viscosity however influences different tasks in the circulating system. For the fluid to have good cleaning properties the viscosity of the drilling fluid should be as low as possible. When the drilling cuttings have been removed from under the bit the viscosity should be high to transport the cuttings all the way to the top of the well. Especially in highly deviated wells the viscosity needs to be high due to the smaller path the particles can fall. It is also preferable that the drilling fluid is low for the surface pumps. The pumps require less energy when the viscosity is low. Too high viscosity can also lead to severe drilling problems when running the drill string up or down (surge and swab problems). The drilling fluid must be designed with these problems in mind [21].

Rheology is the study of the deformation of fluids, the core elements are viscosity, friction pressure loss and the fluids velocity profile. A fluid does not necessary have one determined viscosity, it can vary depending on the shear rate. Only Newtonian fluids have a determined viscosity, the most typical Newtonian fluid is water. However drilling fluids are not always Newtonian, most often they are non-Newtonian. There are different kinds of non-Newtonian fluids, to determine what kinds it is a rheology profile must be made. This is made by measuring the shear stress versus shear rate. Shear stress τ is defined as an applied force, F, acting along a unit surface area, A. γ is the shear rate. It is defined as the velocity gradient or in other words, the change in velocity of a fluid moving in the x-axis with respect to another layer a unit distance away along a perpendicular axis, typically the y-axis or the r-axis in a polar coordinate system [21]. Mathematically they can be described:

Shear rate:

$$\gamma = \frac{dv}{dy} = \frac{dv}{dr} \qquad \text{EQ 1.8}$$

Shear stress:

$\tau = \frac{F}{A}$ EQ 1.9

Newtonian fluids are fluids that can be fully described by the equation

$\tau = \mu * \gamma$ EQ1.10

which gives a straight line from origin with shear rate on the x-axis and shear stress on the y-axis in a Cartesian plot. Non-Newtonian fluids are all the fluids that do not behave according to equation 1.10. The easiest non-Newtonian fluids to describe are the ones that can be described by the Bingham model which is a straight line but does not go through the origin on a Cartesian plot with shear rate and shear stress on the axis. The equation is similar to the one describing Newtonian fluids but with a yield stress. The yield stress determines how much stress the fluid can take before it starts to move or gives a shear rate [21]. The Bingham model describes the fluid with the equation:

$\tau = \tau_y + \mu_p * \gamma$ EQ1.11

Where the μ_p is the plastic viscosity and τ_y is the yield stress.

Another common equation that describes non-Newtonian fluids is the Power law. These fluids are known as pseudoplastic or shear thinning fluids. If the shear stress of these fluids is plotted on a log-log paper they would draw a straight line. The equation for that describes the shear stress for these fluids is:

$\tau = K * \gamma^m$ EQ1.12

Where the K is the consistency index and the m is the exponent or the power law index. The viscosity from this relationship is defined as:

$\mu_a = K * \gamma^{m-1}$ EQ1.13

The μ_a is the apparent viscosity and it decreases as the shear rate gets higher, this is why the fluid is sometimes called shear thinning fluid. If the fluid has both shear thinning properties and a yield stress the shear stress could be described by the yield power law which is almost the same as the power law, the difference is the addition of the yield stress term.

Viscoelastic fluids can be compared with both solids and regular fluids. When applying constant strain to a solid the stress will be equal as long as you continue to apply the strain as seen in Figure 9 called elastic. With regular fluids however the stress will only follow when the strain is applied. For the viscoelastic fluids the response in stress will be a mix of the two, the stress will increase when the strain is applied and then go back to zero stress after a relaxation time depending on the fluid.

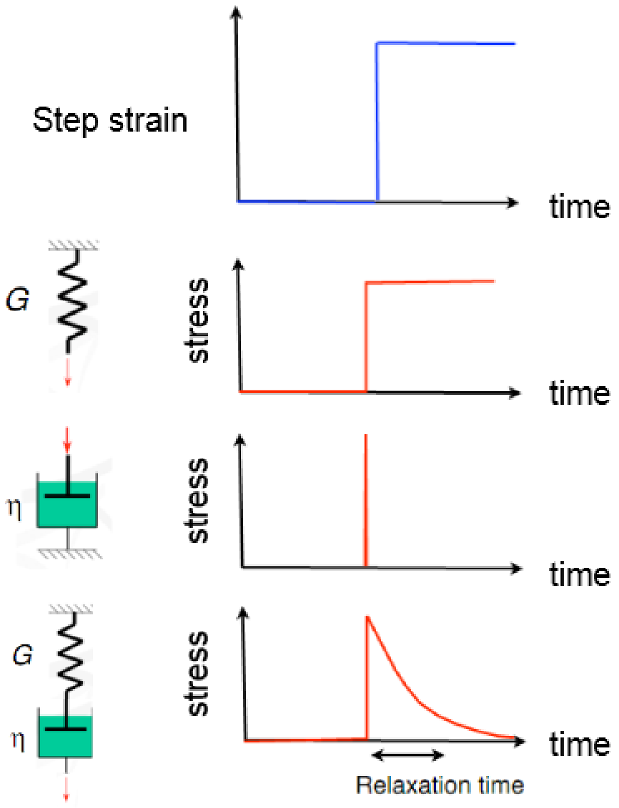

Figure 9 Explanation of viscoelastic fluids [22]

A common way to explain the relationship of viscoelastic fluids is from changing the strain that gives the shear modulus, loss modulus and storage modulus [23].
$G^* = G' + iG''$ EQ1.14

Where the G^* is the complex shear modulus, G' is the storage modulus and G'' is the loss modulus. Another way to describe the relationship is

$$\tan(\partial) = \frac{G''}{G'} \quad \text{EQ1.15}$$

The $\tan(\partial)$ describes the balance between the loss and storage modulus, where a value higher than 1 is a more liquid like structure of the fluid and a value below 1 describes the fluid more solid like. When the loss modulus is higher than the storage modulus the fluid can flow and where it is the same is called the flow point [22].

3.3 Description of experiments

3.3.1 Making of the fluid

In these tests two types of fluids were made, the first one is an oil based fluid which was ordered and prepared by MI-SWACO a Schlumberger company. The fluid they made is based on the companies Versatec system with micronized barite. The chemical breakdown of the fluid is:

- Base oil (external phase)
- $CaCl_2$ Brine (internal phase)
- emulsifier
- Viscosifyers- Organophilic clay
- Lime
- Fluid loss additive and particles –$CaCO_3$
- Weight material Micronized Barite

The fluid has an oil water ratio of 80:20, the density was chosen to be 1.20 SG. The micron sized barite was chosen over the standard barite because they were thought to better work with the nanoparticles due to a more consistent spread in the particle size distribution. The average diameter of a micronized barite is 1µm. To prepare the fluid, about 350mL from the already prepared oil based mud was weighed. From this, the correct amount of nanoparticles could be measured and was based on 0.1, 0.5 and 1% of the fluid weight. The nanoparticles were measured in a weight with an error of 0.001 grams. After the nanoparticles were measured in a petri dish, it was poured in a Hamilton beach mixer and mixed for 2 minutes. The fluids that contained nanoparticles were also mixed with a hand hold supersonic mixer for 2 additional minutes. The fluid was now ready for tests.

The second fluid was water based mud and was made from scratch in the lab. This started with weighing the water up to the specified amount. The first additive to the mud was 5wt% bentonite, this was mixed until the fluid was observed to be smooth but if this was the only additive it was mixed for 5minutes. Then barite and the polymer were added. The pH was tested by pH paper and if needed a base solution consisting of 0.1M of NaOH was added to increase the pH value. The nanoparticles were always the last additive to the mud. The weight of amount of nanoparticles was based on calculation of the total weight of the mud. The final preparations of the mud was mixing in a Silverson mixer for 5 minutes followed by a new measuring of pH value and mixing by a hand held supersonic mixer for another 5 minutes.

3.3.2 The nano additives

The nanoparticles can mainly be divided into two different categories water wet and oil wet particles. The nanoparticles in these experiments have also been divided into size and microstructure (if they are porous or compact). There have been two suppliers of the nanoparticles, the main contributor have been Aerosil. The other company who has supplied materials is Elkem Silicon Company. Below is a table of all the nanoparticles and some of their properties.

Table 1 The nano additives and their properties

Name	Mean size	Type	Manufacturer
300	7nm	Water wet, porous	Aerosil
130	16nm	Water wet, porous	Aerosil
999	100nm	Water wet porous	Aerosil
R812S	7nm	Oil wet, porous	Aerosil
R972	16nm	Oil wet, porous	Aerosil
R972V	16nm	Oil wet, compact	Aerosil
Kettlitz Si-69 treated silane	120nm	Oil wet, compact	Elkem
3(trimethoxysilyl)propyl methacrylate	120 nm	Oil wet, compact	Elkem
PMMA	15µm	PMMA	Microbeads

3.3.3 Density test

The density was tested after the fluid was mixed, a weight with the sensitivity of 0,001 grams was used together with a glass pycnometer of 10 mL. The pycnometer was cleaned on the outside when fluid was inside and inside after the test was over.

3.3.4 FANN 35 test

The second test of the fluid was a viscosity test. The FANN model 35 viscometer is widely known as the standard of the industry for drilling fluid viscosity measurements. This is a test which they do to drilling fluids on rigs around the world. A common way to measure the rheology profile of a fluid in field is to use the FANN model 35 viscometer. The test sample is placed in a cell with a rotor and a stator. By rotating the rotor or outer cylinder a drag is created, the fluid transfers the rotational force to the stator or the bob. The bob is connected to a spring and the torque on the bob which is the shear stress is measured.

First thing that was done was to make sure that all the parts were clean especially the rotor and the stator due to the small gap between them. Then the container was filled up with the test fluid to the amount indicated by a small line in the container which is 350mL. After that the container was mounted into the viscometer and raised to where the rotor lines and the fluid matched each other. The test was then started from 3rpm going to 100, 300, 6, 200, 600 rpm which converts to 5, 10, 170, 341, 511 and 1022 /s shear rate. When a shear rate was selected and the rotor was spinning the shear stress was visually observed when it stabilized. The viscosity measured by the FANN model 35 viscometer measures the shear stress caused by a given shear rate. The shear rate is calculated from the rpm and the geometry of the test equipment, it is therefore important to be precise when filling the container with test fluid. The viscometer has changeable rotors and bobs, in these experiments the standard B1 and R1 bob and rotor was used. With these parts the conversion from rpm to shear rate is a factor of 1.7023.

Figure 10 FANN 35 viscometer

3.3.4 Filtration test

The easiest way to determine the filtration properties of a fluid is to use a filter press. In the experiments done in this report a standard API filter press with compressed air was used. The pressure source delivered air with a pressure of 7 bars. All the parts of the filter press can be seen in figure 11. The experiments were always started with thorough cleaning and drying of the base cap, rubber gasket, screen and the filter cell. The cell was then sealed to the base cap and filled with mud with approximately 6mm from the top. After that the cell was carefully placed into the frame and the regulator from the pressure source was gradually opened (within 3 seconds). This was the most critical step as the cell sometimes was leaking. If that were the case the cell was disassembled and the experiment was started over again. If the cell was not leaking the timer was started and the filtrate volume was measured after 1, 2, 3, 5, 7.5, 10, 15, 20, 30 minutes. The drilling fluid was discarded and the height of the mud cake was measured to the closest 0.5 mm.

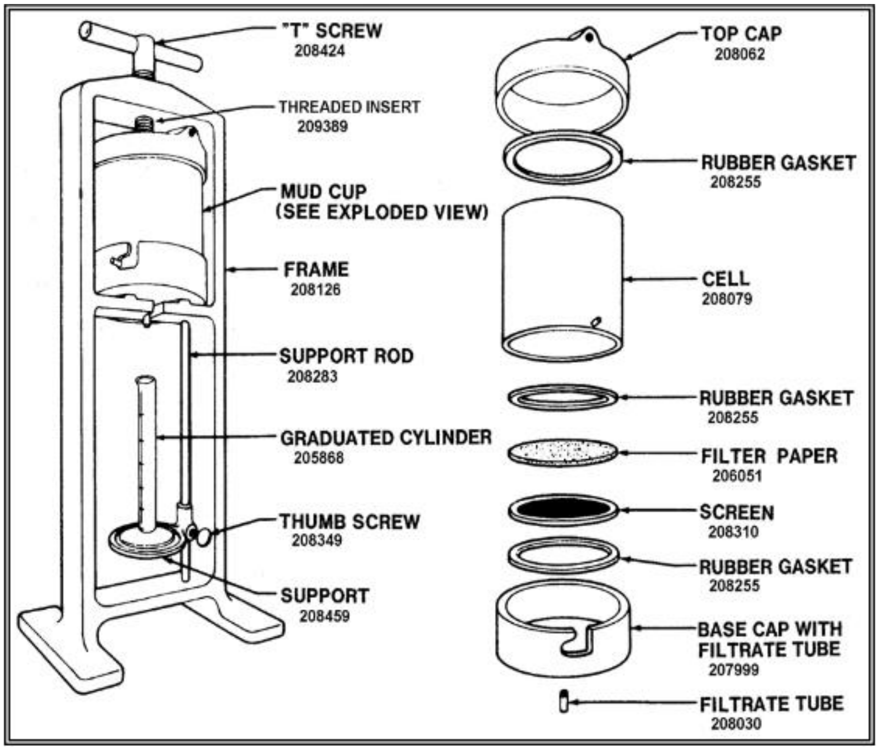

Figure 11 LPLT Filter press

3.3.5 PaarPhysica MCR 302 Rheometer tests

The MCR 302 rheometer is an advanced new rheometer on the market. It can provide torque down to 10nNm for rotation and 2nNm for oscillation with a torque resolution of 0,1nNm. The temperature and pressure can vary between -150°C to 1000°C and to 1000 bar with the right accessories, it is needless to say that this rheometer is far more advanced than the FANN 35. Without any accessories the rheometer can vary the temperature between -20 and 200 °C. This apparatus was used to do a more into depth analysis of fluids where the FANN 35 viscometer failed to do so. This rheometer has also been used to do tribology analysis.

Figure 12 MCR 302 rheometer with static plate cell

To do a full rheological analysis not more than 2mL per test was needed, but to optimize mixing of the nanoparticles into the fluid a larger batch was made. The measuring system consists of a static flat plate at the bottom where the fluid rests that can vary temperature rapidly. On top of the flat plate goes a tool, the tool that was used in these experiments was a parallel plate. The fluid was first placed in the middle of the flat plate, the parallel plate was then lowered to measuring position which was 1mm and the excess fluid removed. The tests were started and measured flow point, storage modulus, loss modulus and shear stress. The test ran from 0,01 to 100 % strain in an oscillating modus. A thixotropic test was also done to find how long the fluid had to rest before it gained back its properties, it was ten minutes and the graph is shown in the appendix.

After waiting 10 minutes for the fluid to rest the same procedure was followed to measure the shear rate vs. shear stress from 0,1 /s to 600 /s shear rate. The rheometer software automatically calculated the viscosity at the given shear rates.

The tribology tests are done after the ball on plate principle. Steel plates were chosen and are supposed to represent casing, the moving ball that pushes against the three plates represent the moving drillstring. In the rheometer the static plate used for rheology measurement have been replaced to a tribology cell

that can mount the steel plates and the fluid as seen in Figure 13. The mounted cell with the three steel plates is filled half up with fluid. In the figure the tool which holds the ball can also be seen. It is the friction between the rotating or static steel ball and the three plates that is measured.
To measure the static friction the tool with the ball pushes down on the plates and the fluid with a force of 10Nm for 1 min and then applies torque until the tool moves with a minimum speed of 0,001mm/s. When the tool had this rotational speed the test was stopped and the static friction was calculated.

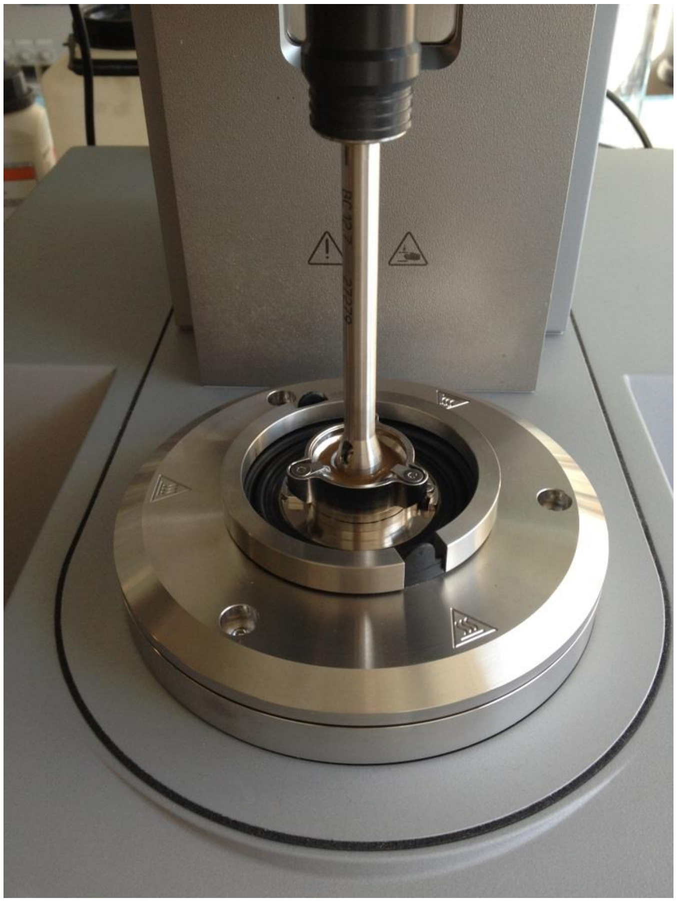

Figure 13 MCR 302 rheometer with tribological cell

To get the Stribeck curve or the sliding friction factor versus sliding speed the tool was lowered to the steel plates again with a normal force of 10Nm and started to slide with a speed of 0,05mm/s to 280mm/s which represents 0,1 to 600 rpm. The sliding friction factor is calculated for each rpm and plotted versus the speed.

4. Results & Discussion

4.1 Water based mud

The below table (Table 2) shows key numbers from the experiments done with the water based mud that contained 5wt% Bentonite, 5wt% barite, 0,02wt % HEC and had a pH value of 10. The nano additives can be divided into oil wet and water wet particles. It can be seen that the water wet particles give a higher fluid loss than the base case with no nanoparticles. The worst case was 1wt % 7nm, it gave an increase of almost 50% to 29,5 mL and an increase in the filter cake thickness. On the other hand it gave an increase in the yield point with a small decrease in the viscosity. A decrease in the viscosity and an increased yield point is something that can be beneficial for drilling fluid because it can increase how large solids the fluid can carry before settling starts, a decrease in the viscosity can reduce the energy needed to pump the fluid and also clean the well better. Another good thing that can be observed for the water wet particles or miscible nanoparticles are that the change in density is low.

The oil wet particles are not miscible with the water based mud. It is possible mix the fluid with the particles with high energy mixing in the Silverson mixer but then air is mixed in to it. That created a lighter mud where the nanoparticles could be mixed with the mud inside air bubbles. This can be seen on the density column, the fluid expanded and the density dropped. The oil wet particles showed better fluid loss properties. The fluid that showed the best fluid loss properties is the fluid with 5wt% 16nm oil wet particles. A decrease of 22,5 % from the base fluid. The filter cake for this fluid shows a doubling in thickness, the filter cake is shown in Figure 14. The 5wt% additive changed the rheology of the fluid as well, both the viscosity and the yield point increased significantly.

Figure 14 Filter cake of 5 wt% oil wet particle in WBM

The fluid with the best results with the water based mud is the one with 2wt % oil wet 16nm particle. That fluid shows a decrease in amount fluid loss of 7,5 % and a decrease in the filter cake thickness to 1,8mm. There are also an increase in the viscosity and the yield point, but not as much as the increase for the 5wt % fluid. As in all the oil wet particles in water based mud there are decrease in density, this fluid shows a decrease of 15,9%. Another thing to notice is that for the immiscible particles, it does not appear that the size matters much due to the similar results between the 16nm and 7nm. This can be due to the way the particles are mixed into the mud, the air bubbles are larger than the particles, so the small particle size does not affect the properties of the fluid.

Table 2 Water based mud with different nanoparticles

5% Bentonite, 5% barite, pH10, 0.02 wt%HEC						
Particle type	Fluid loss 7,5min [mL]	Fluid loss 30min[mL]	Filter cake thickness [mm]	Viscosity [cP, mPas]	Yield point [Pa]	Density [SG]
no nanoparticles	10,5	20	2,3	14	6,6	1,07
1 wt % 100 nm nano	12,5	24,5	2,1	15	5,6	1,08
1 wt % 7nm nano	15	29,5	3,4	13	9,7	1,06
1 wt % 16nm oil wet	11	21	2,3	17	10,2	0,98
2 wt % 16nm oil wet	9,5	18,5	1,8	22	11,7	0,9
5 wt % 16nm oil wet	8	15,5	4,6	57,9	32,7	0,67
1 wt % 7nm oil wet	10	19,5	2	18	12,8	0,97
1 wt % PMMA	11	21	2,2	12,5	6,1	1,07
2 wt % PMMA	10,5	20	2,2	16,5	10	1,07

The last material that was tried was the PMMA, they showed no significant change in any of the parameters given in the table. The parameter that changed the most was the viscosity which fell 4 mPas for the 1 % concentration increase in PMMA . The fluid loss, filter cake thickness and the density remained almost the same as the base case.

All the data points for the filtrate test of the base case are shown in the graph below. As described previously the volume loss at 30 min should be close to the double of the volume loss at 7,5, here it is 20 and 10,5 respectfully. This and the fact that it follows the square root of time indicated by the half slope in Figure 16 shows that the fluid loss follow equation and we have Darcy flow through the system.

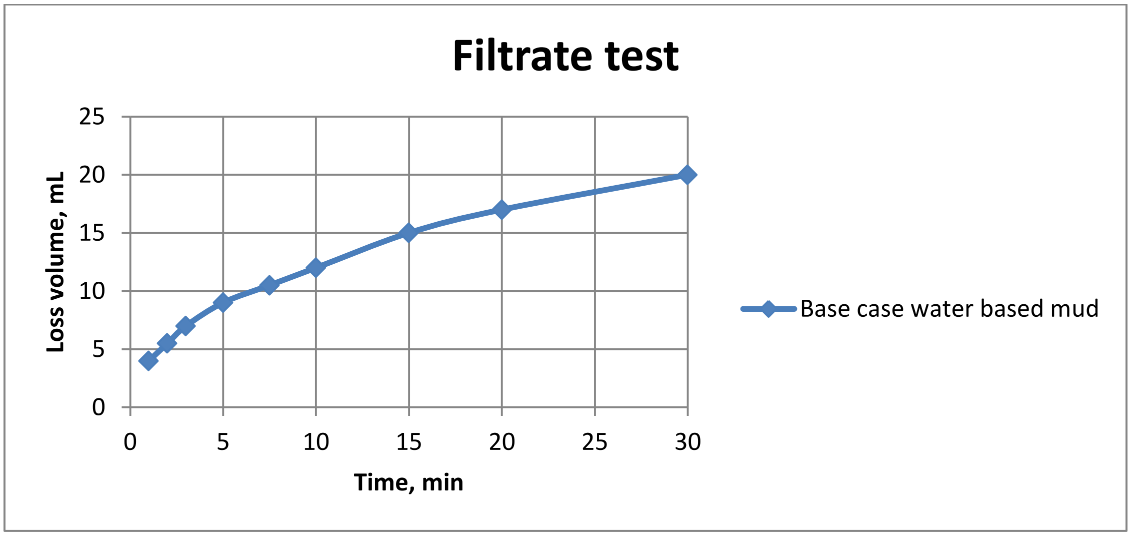

Figure 15 Filter test for base case water based mud, cartesian plot

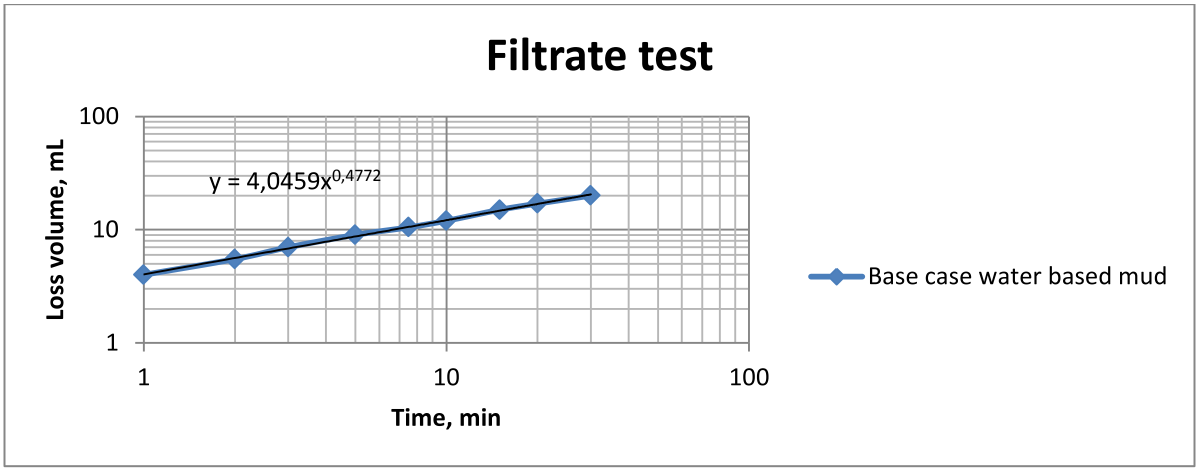

Figure 16 Filter test for base case water based mud, half slope on log- log plot

The rheology profile of the base case of the water based mud is shown in Figure 17, it shows a shear thinning fluid and a Bingham model describing it. The plastic viscosity in the Bingham model is the slope of the line or the tangent in the any given point. A common point to measure the viscosity is at 300rpm or at 511 /s shear rate. If we compare this with the base case of the oil based mud we can see that the shear stress is smaller at the same shear rates, the viscosity is also less.

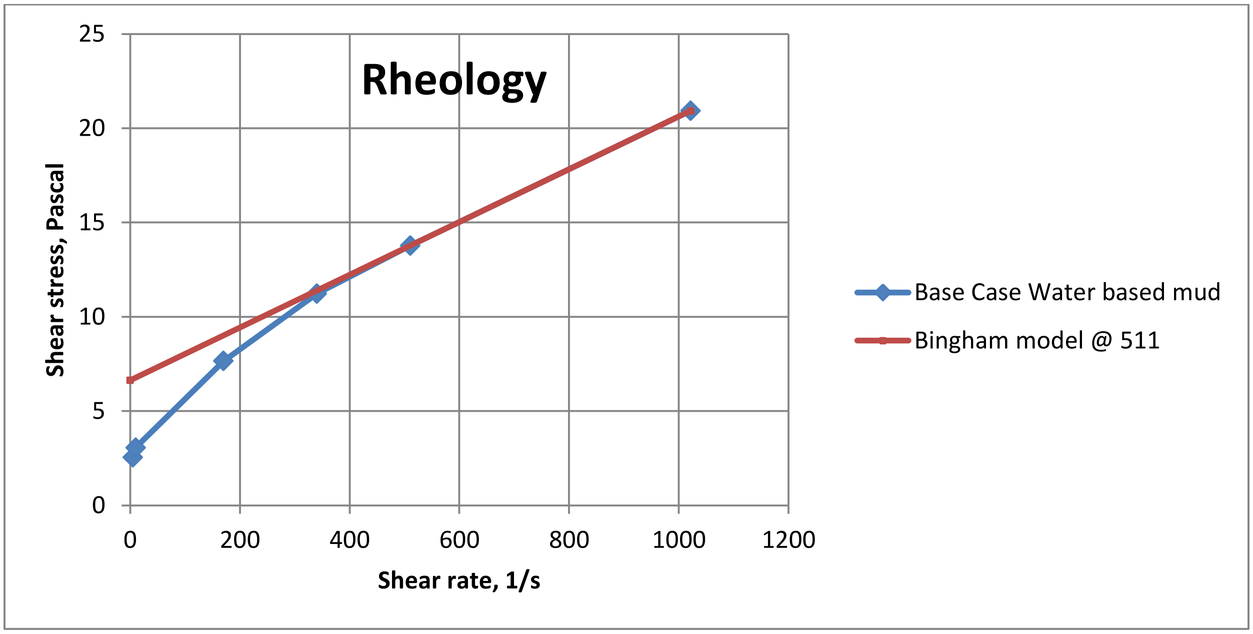

Figure 17 Rheology plot for base case WBM

The best filtrate test with water based mud and nanoparticles are the one with 2% 16nm oil wet porous particles. The graph is shown in Figure 18. This graph like the previous with no nanoparticles shows Darcy flow through porous media due to the volume loss is proportional related with the square root of time. The 7,5 min volume is 9,5 and the final volume is 18,5 which also indicate this.

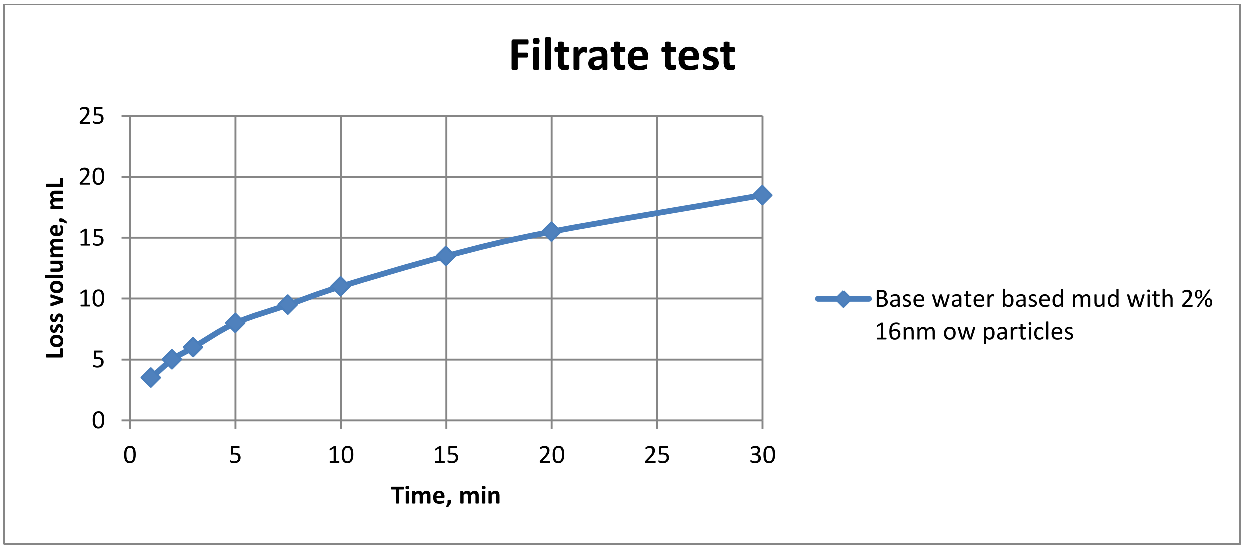

Figure 18 Filtrate test WBM with 2% 16nm ow particles

The rheology profile of the nanofluid is similar to the base case. The difference between them is at low shear stress and the higher viscosity. Due to the high degree of shear thinning effect of this fluid there is a difference in the Bingham model and the data from this experiment. The difference is 11,8 Pa for the Bingham model at 5 /s shear rate and 2,8 Pa for the real data. A more precise rheometer is needed to find the real yield points of the fluid, the shear rate of 5 /s is the closest point to yield point. Comparing this point with the real data of the fluid without nanoparticles we can see that they actually are very similar even though the Bingham model for the two fluids indicates that the fluid with these nanoparticles have yield points that differ from each other 6,6 and 11,7 Pa. The graphs for the fluid loss and the rheology for all the fluids in the table are given in appendix A.

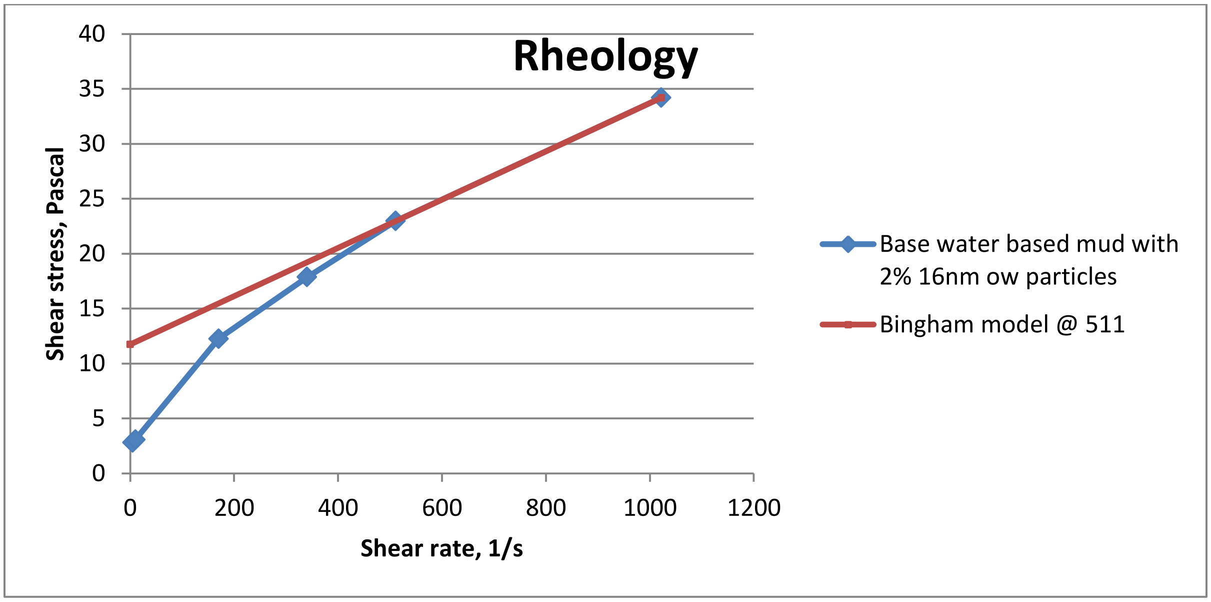

Figure 19 Rheology profile for WBM with 2% 16nm ow particles

4.1.2 Other water based muds

For the oil wet particles to be miscible to the water based oil, methanol was tried. First the oil wet particles were mixed into the methanol, than the blend was mixed into the water based mud like the other fluids. The result is given in table 2. The table shows that the fluid loss is greater than the base case, the fluid also shows a higher viscosity, a lower density and filter cake thickness and similar yield point. In the filter cake there was observed small lumps showed in Figure 20. Nothing in this fluid composition gave good results and was therefore not investigated further.

Figure 20 Filter cake of OMB with 5% bentonite, 9% methanol and 1% 16nm ow particles

Table 3 Other water based muds with different particles types with their properties

Fluid	Particle type	Fluid loss 7,5min [mL]	Fluid loss 30min[mL]	Filter cake [mm]	Viscosity [cP, mPas]	Yield point [Pa]	Density [SG]
Water, Bentonite 5 wt %	No nanoparticle	11,5	21	2	7	2	1,03
Water, bentonite 5 wt %, methanol 9 wt %	1 wt % oil wet 16nm Particle	12	24	1,8	9	2	0,96
Water, Bentonite 5 wt %	5 wt % water wet 100nm	16	30,5	3	*	*	1,02
Water, bentonite 5 wt%, Barite 5 %	No nanoparticle	10,5	20,5	2	8	1,3	1,06
Water, bentonite 5 wt%, Barite 5 %	1 % water wet 100nm	12	23,5	2,2	9,5	2	1,06

For the 5 wt % 100nm particles, there was a significant increase of fluid loss to 30,5mL, the filter cake also increased in thickness. It was not possible to measure the viscosity or yield point of this fluid in the FANN viscometer due to what is thought to be highly thixotropic properties. When the fluid was sheared at 600rpm or a 1022 shear rate the Dial reading on the FANN viscometer varied from 18 to 25 several times in 5 minutes giving inconclusive results.

Looking at what the barite particles does to the fluids, it can be seen that the particles changes the density of the fluid. Other properties remain unchanged. The base fluid described in Table 2 is with the polymer HEC, in Table 3 is a fluid without the polymer. It can be seen that it does not influence the fluid loss property of the fluid and only affects the rheology. The fluid with the nanoparticles with 100nm fumed particles shows no improvement with fluid loss.

4.2 Oil based mud

Compared to the water based mud we can see that the oil based mud gives significantly less filter loss. At the first glance on the table below, we can see that there are not a lot of changes compared to each other and it is therefore difficult to evaluate the improvement of nanoparticles. A more detailed discussion of the evaluation of the OBM and the error range in this experiment is found under chapter 4.5 and 4.6. The density of the different fluids changes with maximum 0.03 and with one less significant digit, it wouldn't change at all. Another thing to notice is the change in the rheology, the fluids with nanoparticles show a decrease in both the viscosity and the Yield point with some exceptions.

The filter loss properties for the oil based mud and the compacted 16nm particles are not much different from the base case. The worst case is with 1 wt % nano additives , but there might as well be no difference due to the inaccuracy of the experiment. There is also a large difference between the fluid with 2 wt % nano additive and the rest with regards to the yield point and the viscosity. We also see that with less of the nano additive the viscosity is going down.

Some of the fluids with particles from Elkem showed a reduction in fluid loss. The fluid with 1% Sil had the lowest fluid loss of all the fluids tried in these experiments. It had only a loss of 0,2mL after 7,5 minutes as well as the viscosity shows a decrease. This was therefore the one fluid that was chosen for further experiments in a more precise rheometer and tribology tests.

When looking at the fluids below, it is possible that the measurements are not precise enough. That the values ranging in the area of 0,5-1,2 depends on errors in the measurement system or a human error. Looking at the fluids with 3- (trimethoxysilyl) propyl methacrylate we can see that the fluid with 1% has the lowest loss volume, going down to half the amount of nanoparticles the fluid loss volume goes up to 1 mL which is more that the base case. Going further down to 0,1% the loss volume goes down again to 0,6 mL.

Table 4 OBM with differnt particle types

Particle Type	Yield point [Pa]	Viscosity [cP, mPas]	Filter loss 7,5 min [mL]	Filter loss 30min [mL]	Density [SG]
no nano	11,2	34,5	0,7	0,8	1,21
2 wt % 16nm compact	12,8	35	0,5	1	1,20
1 wt % 16nm compact	6,6	28	0,7	1,2	1,20
0,5 wt % 16nm compact	5,1	25	0,5	1	1,21
0,1 wt % 16nm compact	4,6	25	0,6	0,8	1,18
1% T120 3li	5,1	24	0,6	1	1,20
0,5% T120 3li	5,1	23	0,2	0,6	1,20
0,1% T120 3li	4,1	24	0,3	0,6	1,19
1% sil	4,6	24	0,2	0,5	1,20
0,5% sil	4,6	23	0,6	1	1,20
0,1% sil	4,1	24	0,2	0,6	1,20
1% R812S 16nm	11,2	30	0,4	1	1,19
0,5% R812S 16nm	7,1	26	0,5	1	1,19
0,1% R812S 16nm	5,1	24	0,2	0,6	1,19
1% R972 7nm	7,7	30	0,3	0,8	1,20

The same porous oil wet particles from the water based mud was also tried for the oil mud. In the oil mud these particles are miscible and therefore do not create air bubbles in the mud. For the 1% and the 0,5% of R812S there is an increase of 0,2 mL and a reduction for the 0,1 %. This indicates as well that the oil based mud does not let trough enough fluid to get good results with this experiment. Looking at the viscosity for the porous particles we can see that they differ from the rest, especially for the R812S which has the same yield point and a lower viscosity compared to the base case with no nanoparticles. For the porous nanoparticles it can be observed that with less and less amount of nanoparticles both the yield point and the viscosity go down.

A closer look at the base case with oil based mud and no nanomaterial in Figure 22 which displays the filter loss of the fluid it can be seen that the graph does not act according to flow through porous media and equation 1.7. This further suggests that the apparatus or measuring procedures are at fault for the inconsistent results with the oil based mud. On the other hand it could be that the rate is measured inaccurate and too few times to see if flow follows the equation. It could also be that the results are accurate, if so the graph can be divided into 3 stages. The first stage where the fluid increases to 0,7mL and then we have a plateau followed by a small increase the last 10mins. The start could indicate that there is Darcy flow and then followed by a good seal and no flow. The last stage could indicate small leakage in the seal.

The rheology graph of the base case for the oil based mud shows that the fluid follows Yield Power law nicely. In the graph the Bingham model is chosen because it is common in the industry, fits most muds well and most important that it is easy to compare the viscosity. The graph shows a difference in the first data point and the model of 11,4 against 1,8 Pa or 9,6 Pa.

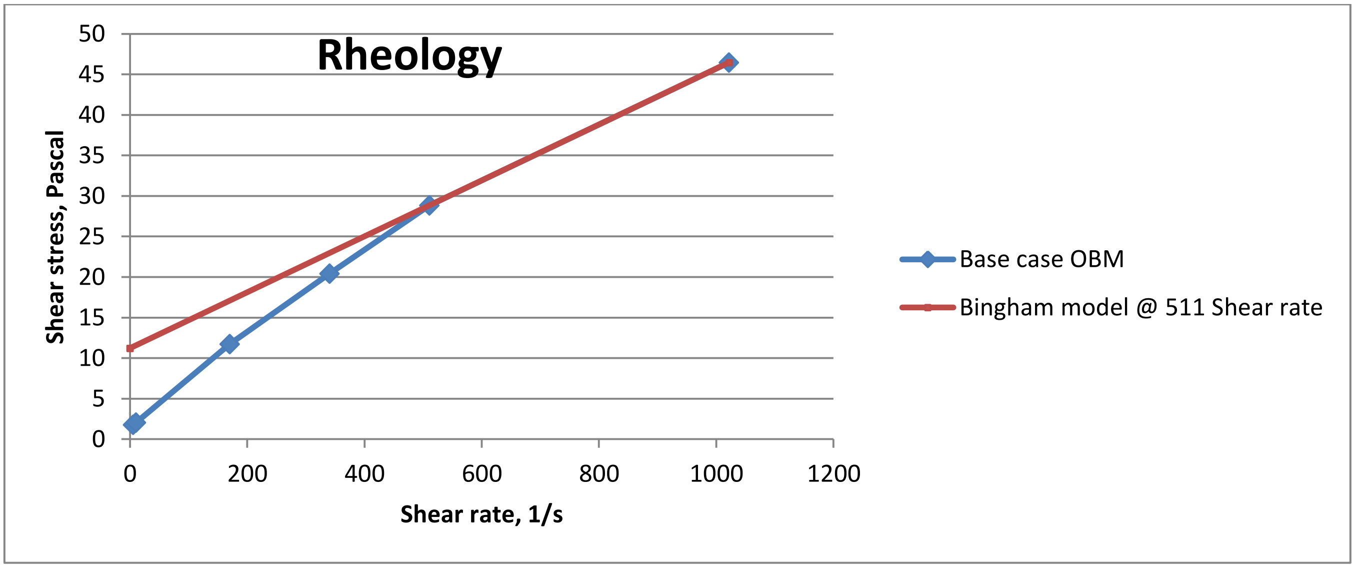

Figure 21 Rheology profile of OBM base case

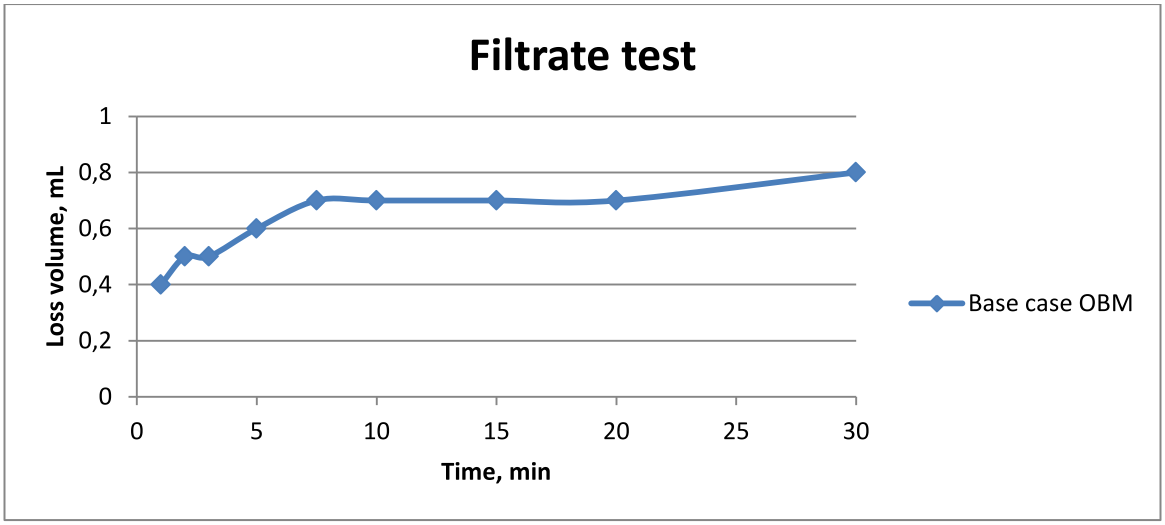

Figure 22 Filtrate test of OBM base case

Looking closer at a fluid with nanoparticles, where the fluid loss is less we have Figure 23. This graph shows a l spurt loss of 0,1 after 1 min. It slowly increases to 0,2 mL in within 10 minutes. After the 10 minutes it loses 0,1 mL every 5 minutes for the last 20 minutes. If the measured volumes where more precise it could be that we could see the rate 0,1mL/5min from the start of the test. Indicating that there is a small leakage where the fluid does not flow through a porous volume and therefore creates a steady flow of 0,1 mL/5min. In the case that these are the true values it could point to the effect of the nanoparticles, which in this case is an earlier seal or plateau at a lower value. The last stage in this graph could be that the seal has broken and the flow is continues and not through the porous media. More discussion of the error sources and suggestion for improvement is found under chapter 4.5 and 4.6.

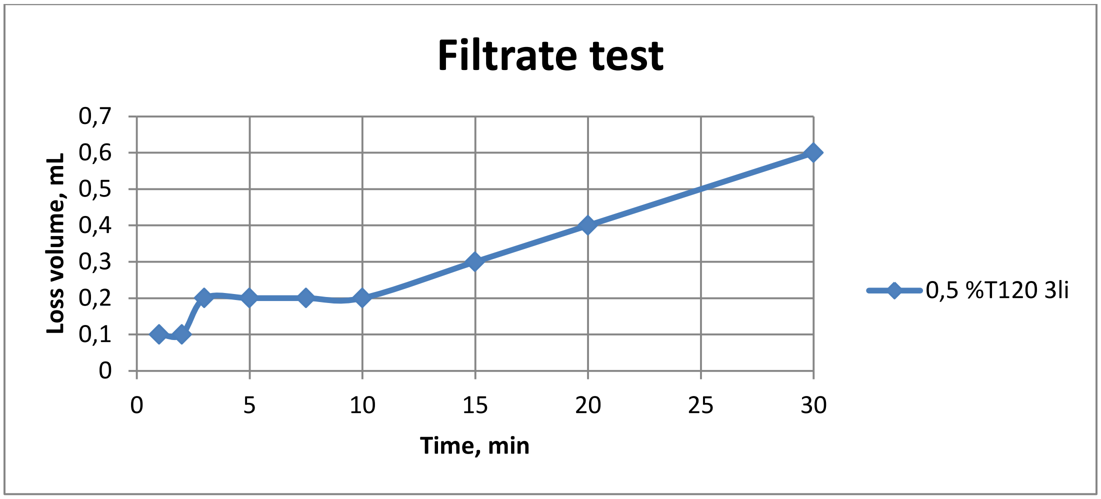

Figure 23 Filtrate test OBM with 0,5% T120 particles

The graph of the rheology of the 0,5 % T120 3li is shown below(Figure 24). The graph shows that the real data are more consistent with the Bingham model than the model was for the fluid without the nanoparticles. This is due to that the new fluid is less shear thinning than the fluid without any nanoparticles. The difference in shear stress for the 3rpm or 5 /s shear rate is 5,2 Pa for the model and 1,5 Pa for the real data or 3,7 Pa. Again this indicates that the fluid with this concentration of nanoparticles is less shear thinning as the difference in shear stress is less. It is also notable to see that the model as well as the data indicates that the real yield point is less than the fluid without any nanoparticles given in Table 4.

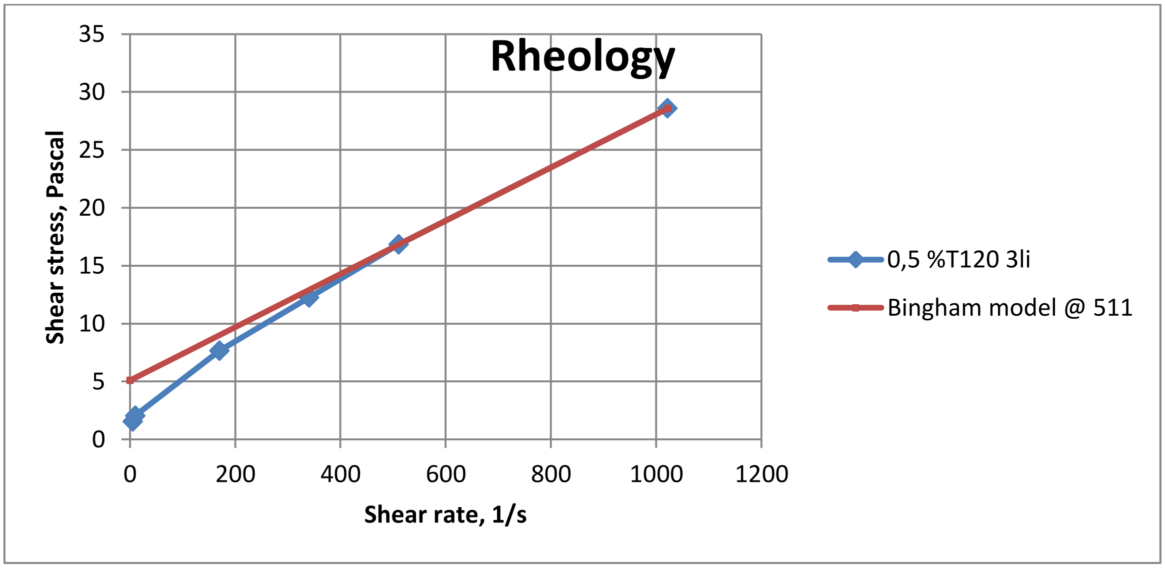

Figure 24 Rheology profile 0,5 % T120 3li

4.3 Improved Rheology tests

The overall better fluid was chosen to be further tested with the more advanced rheology apparatus. The previous experiments showed that the oil based mud with 1% Si-69 silane which is shorted sil in the figures it had good filtration properties as well as better rheological properties, this fluid was therefore chosen to be further investigated.

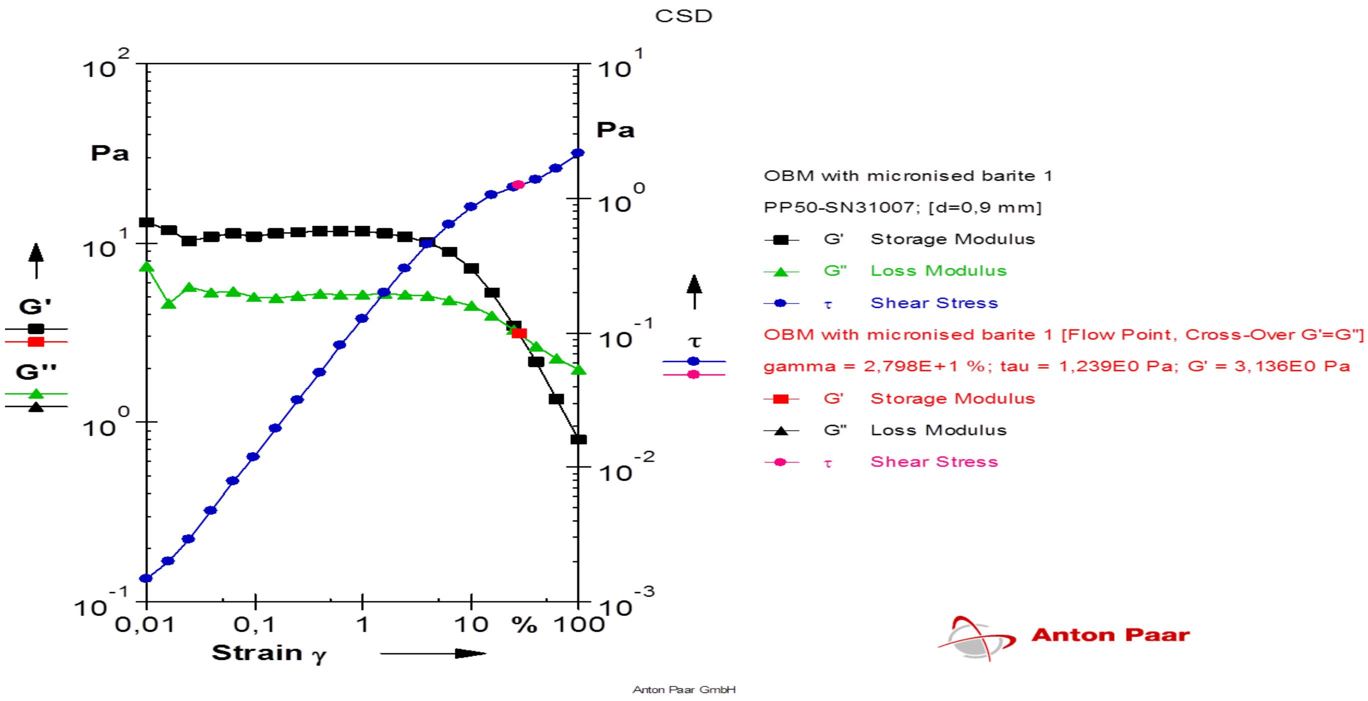

Figure 25 Amplitude sweep for OBM base case

The above figure shows the relationship between the storage modulus and the loss modulus, it also shows the shear stress with the axes to the right. The fluid is the base case for the oil based mud. The flow point of the oil based mud is in red. The flow point is where the storage and the loss modulus is equal it has a value close to 3 Pa. At this point the shear stress is 1,2 Pa indicated by the red square.

The plot below (Figure 26) shows the same graph for the new fluid with nanoparticles. It shows that the nanofluid has a higher value of both modules when they cross than the base case and that the shear stress at the flow point is higher. This indicates that the new drilling fluid can take higher shear before settling starts. In the flow point the storage and loss modulus is 3,68 Pa against the 3 Pa for the base case, while the shear stress at the flow point is 1,3 against 1,2 Pa. The shear stress at the flow point is the true yield point and not the calculated yield point found previously in Table 4.

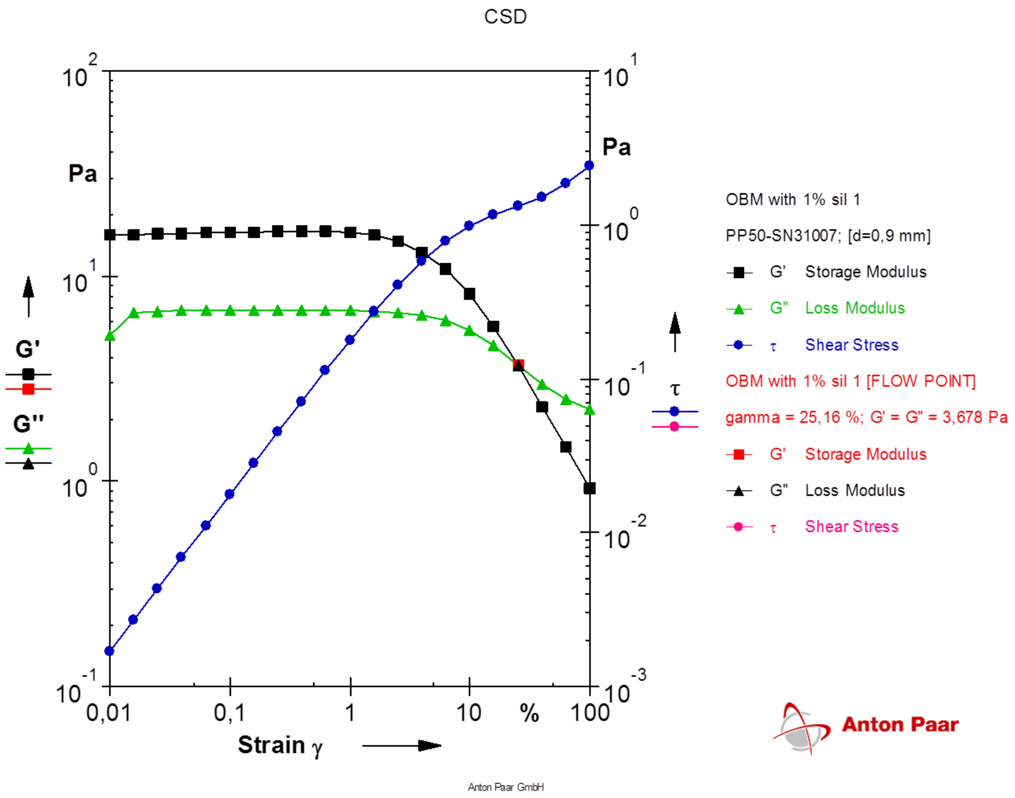

Figure 26 Amplitude sweep OBM with 1 % sil

The next figure shows the shear stress over the shear rate. The starting point in this experiment was 0,1 /s shear rate which also indicates that the yield point for the nanofluid is higher than the base case. It can also be seen that the new nanofluid has a slightly lower slope than the base case, which means that the viscosity is slightly lower.

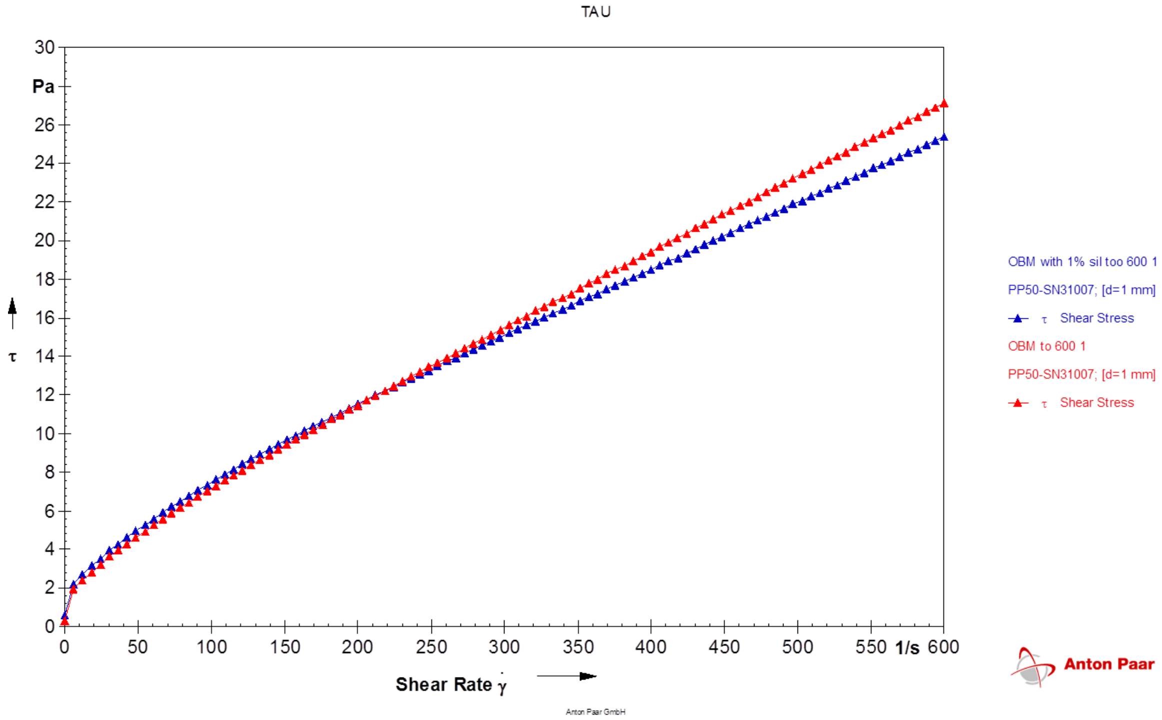

Figure 27 Shear stres vs. Shear rate for base case OBM and OBM with 1% sil

In Figure 28 and Figure 29 the viscosity of both fluids are plotted against the shear rate. On the first figure the first two points are interesting because it is related to the power the rig pump has to have in order to get the mud circulation started. These points are important to be low because it is often the limiting factor if the pump can use the fluid. The next figure is the same graph only zoomed in on the rest of the data. That graph shows that the new fluid actually has a higher viscosity before 200 /s. After 250 /s shear rate the viscosity is lower than the base case. With the FANN viscometer the viscosity was only calculated for the 511 /s where the new fluid shows 5 mPas lower than the base case. The previous difference were calculated to 10 mPas lower, the new measurement is more trustworthy due to the more precise and automatic apparatus.

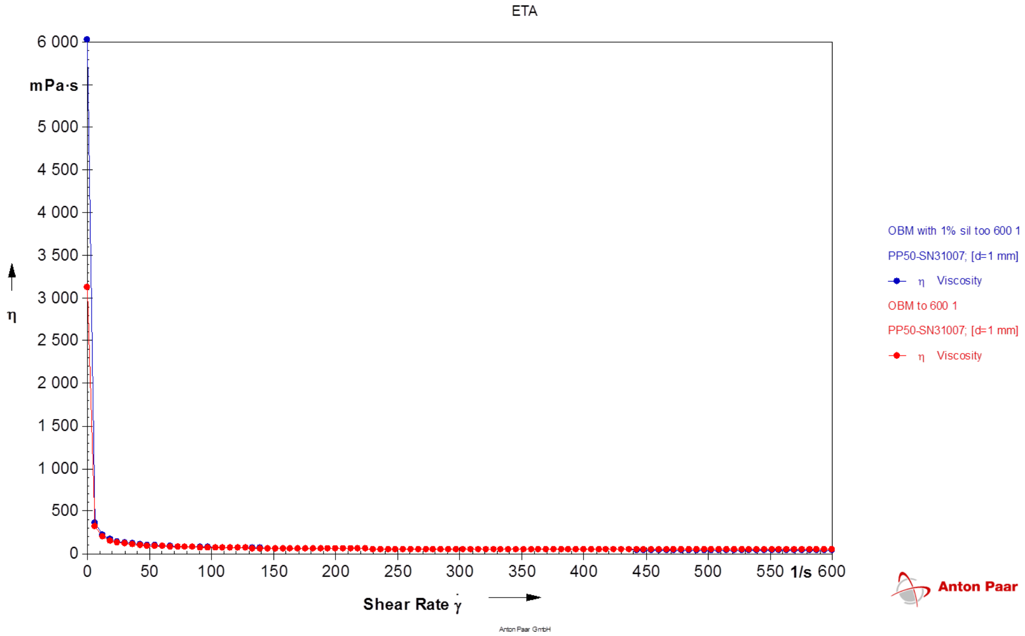

Figure 28 Viscosity vs. Shear rate for OBM base case and with 1% sil

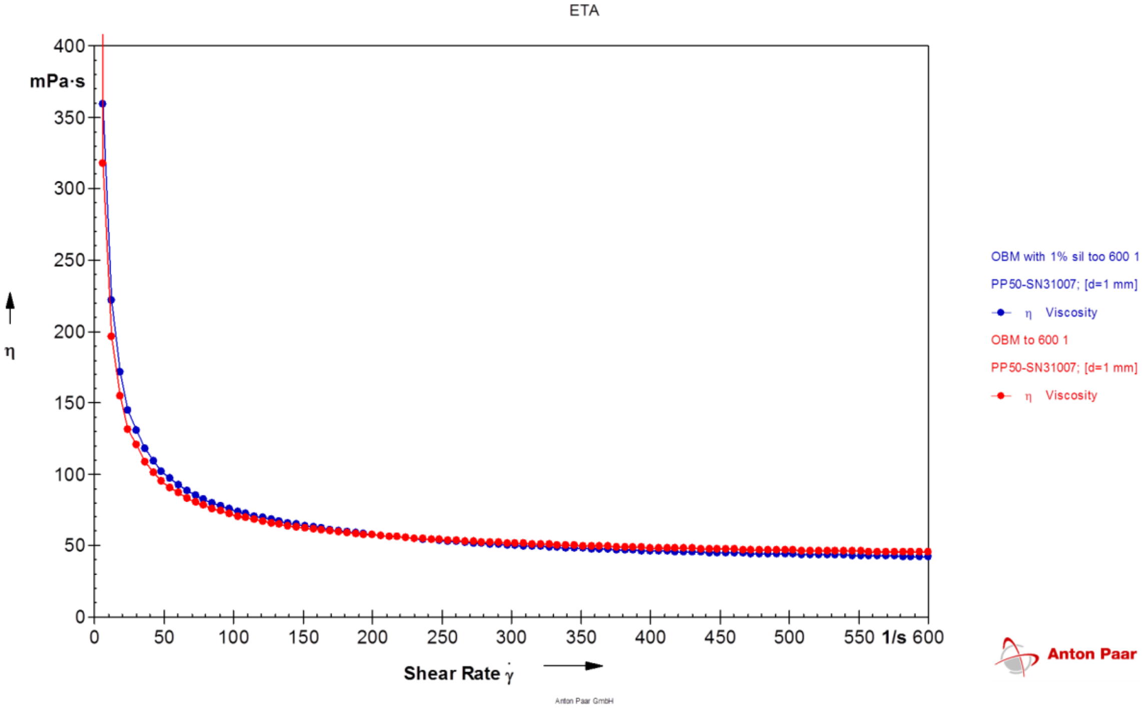

Figure 29 Viscosity vs. Shear rate for OBM base case and with 1% sil, zoomed

4.4 Tribology tests

The below figure (Figure 30) shows the two fluids again, the graph may be easiest to read with increasing y axis. The test gives more and more torque until the tool starts to move with a speed of 0,001mm/s. The first speeds versus the friction factors is noise when the two parameters starts to make a slope then we have a static friction factor. It can be observed that the base case has a lower static friction factor than the new nanofluid. The base case has a static friction factor of 0,19 and the nanofluid 0,22. This indicates that it is easier to start to move the drillstring with the base case fluid. The static friction factor is the highest friction factor and therefore may be the limiting property for how long a well can be.

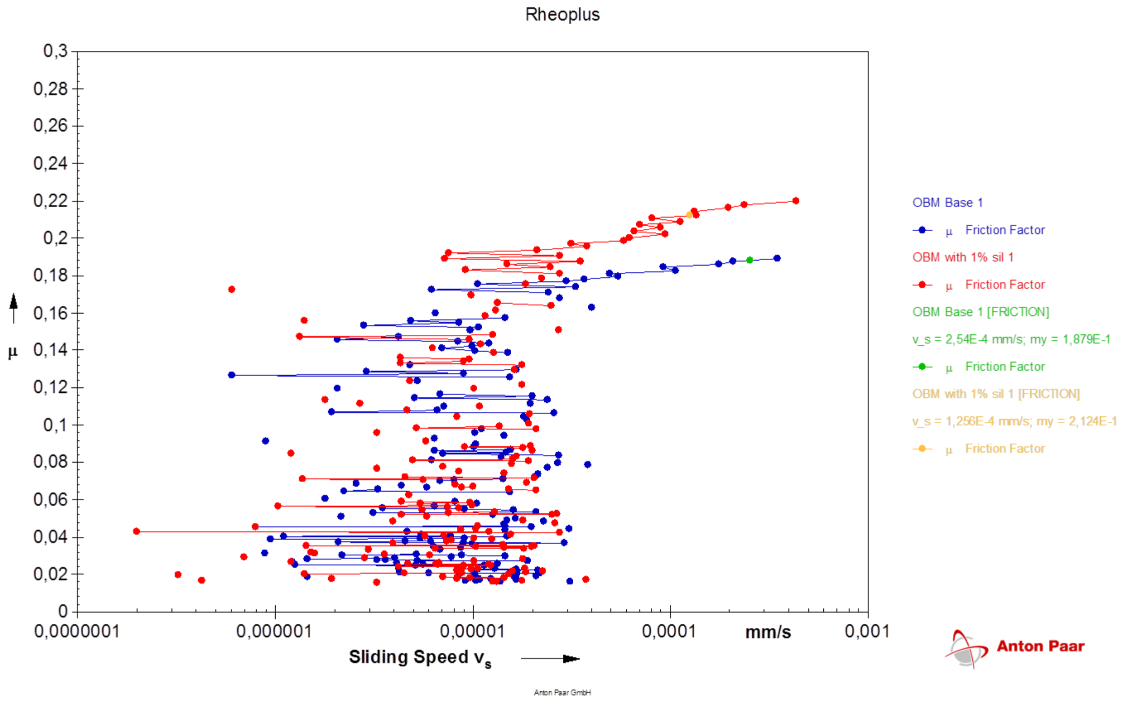

Figure 30 Static friction for OBM base case and with 1% sil

The next figure shows the rest of the friction factor, from slow rotation to 300rpm. The base case for oil based mud shows a stable friction factor from 0,18 to 0,12 with the high rotation. The new nanofluid shows varying friction factor, at first there is a higher factor than was measured as the static friction factor. The lowest friction factor is also at low rotation speed, at 0,5 rpm the friction factor is down to 0,09 which is lower than the base case at any speed. The friction factor goes up again to around 0,2 and then stabilizes along with the base case. The nanoparticles show that friction factor behaves more dynamically. The temperature is room temperature and is the same for all the tribology experiments.

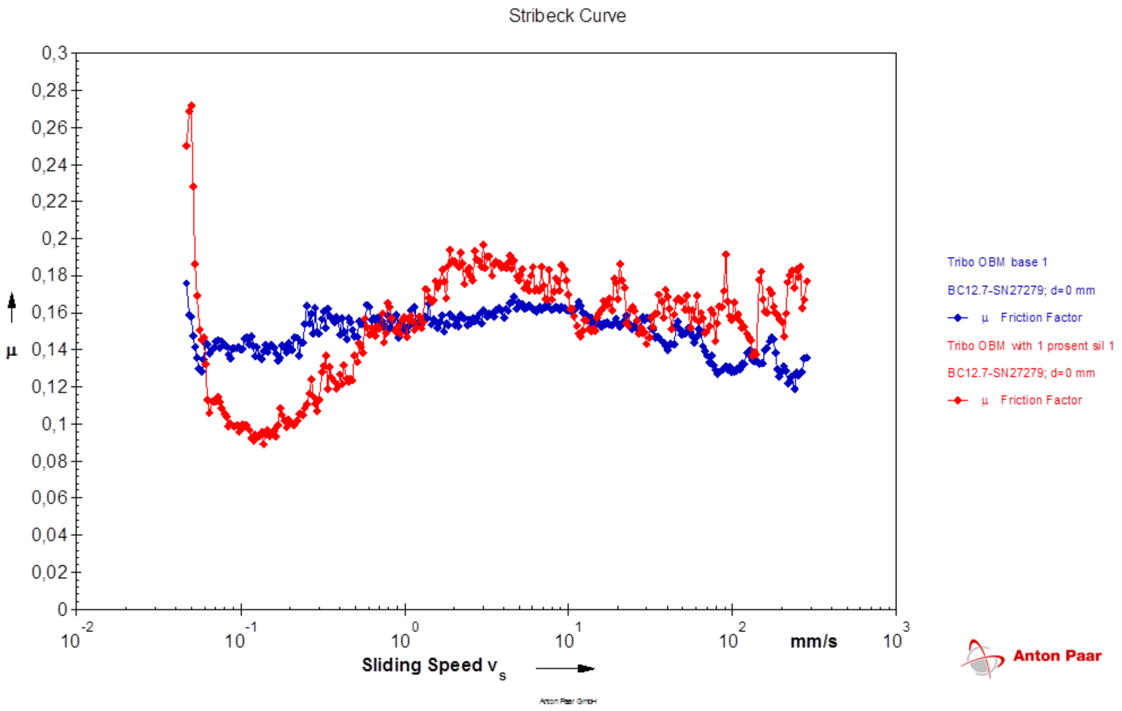

Figure 31 Stribeck curve, friction factor vs. Sliding speed for OBM base case and fluid with 1% sil

4.5 Error sources and its significance

There may be many sources of error in these experiments, in this chapter the main sources will be presented test by test. One of the main faults that which worsen the sensitivity of all the experiments is that the tests per fluid were only done once. This however does not ruin the goal of finding indications of which and in what cases nanomaterial can improve a drilling fluid, it allows for more tests to be done. More tests of the fluid that shows improvements are therefore strongly suggested.

4.5.1 Mixing the fluids

The first place where there might occur differences between the tests, is the making of the fluid. The oil based mud was made 4 days before it was used for the first time. The rest of the tests were finished after one week from the first test was finished. Only visual observations were made to assure that there was no sag or change in the fluid. There is a chance that both the sun and heat changed the fluid over time like water separating from oil. The heavier particles in the fluid might also settle during this time affecting the tests that ran over the week. When pouring the fluid from the large container to the mixing cup only the fluid on top of the container might have been poured out. The larger particles may then have accumulated on the bottom. If this was a significant source of error there may have been evidence in the density tests, there is no indication of this in the results.

Mixing of nanoparticles in the lab was only done for the water based mud. A timer was used when mixing so there is expected only small errors in this part. The weight that was used to measure the different additives had a sensitivity of 0,001 grams. One thing that might cause an error is the waiting time between the fluids was finished mixing and to the FANN viscosity test was executed. There was not used a timer from the fluid was finished mixing and to when the viscosity test was executed in the low stress rate. This could possibly cause a problem due to unknown thixotropic properties of the fluids. A thixotropic test should perhaps been done on the different fluids to estimate the wait time before adding more strain to the fluids as is done in the viscosity test. The difference between the FANN viscometer and the MCR 302 shows that this might be a significant error.

4.5.2 Filtration test

Looking at the results from the water based mud it doesn't look like there have been a significant source of error. It is difficult to estimate the sensitivity on the filtrate loss test other than the measuring container for the water based fluid had a sensitivity of 2 mL. The sensitivity could be worse due to the way the experiment was set up. When looking at the results for the oil based mud it is easier to see that there may be something wrong with the sensitivity due to the spread in the results that do not follow the amount of added nanoparticles. Some of the results show that with an increase in the nanoparticles the fluid loss goes up and vice versa for different particles. There is no constancy in the amount of increased nanoparticles and the change of the filtrate loss. With the low filtrate loss it is therefore possible that small error sources can dictate the outcome of that test. A variable small amount of water could have found its way into the test cell if bad drying occurred or when wetting the filtrate paper. If this was true it could explain the inconstancy in the results. If this was water in the base cap of the cell it does not explain that in some cases there were large spurt loss and in other cases the fluid did not show before the end and still end up with almost the same result.

Another error source could be how the paper was placed in the test cell. If there was a small fold on the paper when the rubber gasket was placed in the test cell this could have given a small leakage. Damage to the paper could also cause this. An opening with a higher permeability than through the paper could make the promising nanoparticles look like they gave too high filtrate loss. An easy way to avoid this would be to do the same test with the same concentration multiple times. In the oil based mud results there were little fluid loss and therefore it is difficult to say if this was a cause to higher fluid loss in this case. For the water based mud the losses was up to 50% greater than the base case, but in those cases the fluid loss concurred with the increase of nanoparticles. It is therefore safer to say that the high loss amount was due to the nanoparticles, but the error could still influence the result and give higher fluid loss.

These error sources and the results for the oil based mud with low filtrate loss raises the question if the results can be trusted and if this is the right experiment for this mud. Oil based mud is known for low filtrate loss, a way to improve this test and give better indications of which nanoparticle actually improves the mud is a test with higher pressure between the mud and filtrate. A test closer to reality would be to raise the temperature while flowing and to use something different than paper between the mud and the filtrate container. A commercial available option is to use a test which is called HPHT (high pressure high temperature) filter press. It can go up to 177^{o}C, 500psi and has the same time span as the one used in this experiment. The use of ceramic filter would also bring the test condition closer to real life.

4.5.3 Density test
The mud was measured in a 10mL test sample, so a small amount of fluid could influence this measurement. Other sources of error that could dominate the results are cleaning the container before measuring the first time and when the container is full. The sensitivity of this experiment is also strongly dependent on the sensitivity of the weight which was 0,001grams. The result of the density test does not vary much and is consistent with the added nanoparticles and therefore it does not seem that the error sources have had too much influence, but to improve the test a larger container could have been used. With this sensitivity of the test it is hard to say how much the different nanoparticles changes the density of the mud. However it is clear that miscible porous light weight nanoparticles did not change the density significantly. The light weight is due to the very porous structure of the nanoparticles and therefore it takes more space in air than in liquid where it is suspended.

4.5.4 FANN viscosity test
Looking at both the MCR 302 test for the oil mud base case and the FANN test it can be seen that there are some differences when testing at the same shear rate. One of the reasons could be that most of the fluids are thixotropic meaning that once they have experienced shear, it takes some time to go back to their original state. So mixing the fluids in the order 3, 100, 300, 6, 200, 600rpm was convenient but did probably not give the most accurate result. The MCR 302 gave an even increase in the shear rate and so the thixotropic properties of the fluid did not influence these results. The shear could produce a significant source of error and so the MCR 302 or any other rheometer with a higher precision is recommended.

The results of the viscosity tests of the water based mud and the 5 % water wet nanoparticles in Table 2 produced an error. The dial reading indicator went up and down several times in a couple of minutes which indicated that the viscometer could not handle measurements for that fluid. The reason for the fluctuation might be because of the high thixotropic property of the fluid, when the fluid in the container was sheared the viscosity went significantly down and the centrifugal force pushed it away. When the fluid was moved away from the bob and spring new thicker fluid might have gone inside and showed an increase in the dial reading. Due to this only happened on a single fluid with a high percentage of nanoparticles it did not influence the rest of the experiment and therefore not significant.

The calculated viscosity in the 511 to 1022 /s shear rate or 300 to 600 rpm was done as a Bingham fluid. The strong point of using the Bingham method is that the fluids are easily comparable. There might have been a better way to analyze the fluids tested, for example the yield power law which would have given different viscosities for all shear rates and utilized more of the data points. The error between the points would also have been minimized. The purpose of these experiment is however to investigate the influence of the nanoparticles and not to determine the viscosity of the different fluids and therefore the Bingham method is sufficient even though not the most precise results, it gives however a viscosity which is easy to compare to each other.

4.5.5 MCR 302 rheometer tests
For the test done in this rheometer there are fewer error sources. One of the biggest sources might be the settings on the computer when preforming the experiment. This is minimized by the producer by

automatically sett right settings when mounting the different tools and cells. The software is also user friendly and none of the results indicates that the settings were wrong. Something that might have happened is that small amounts of shear was added to the fluids minutes before testing it, for example while using a pipette to add the fluid to the plate. Cleaning is always an issue especially for the tribology tests due to the small parts in the testing cell. The results of the tribological tests might indicate that something is wrong due to the fluctuation of the friction factor and could be due to cleaning. It also could be nothing at all and to ensure this, the test could be done again. For the rheological tests there were large surfaces which were easily cleaned.

4.6 Evaluation of the fluids

Some of the experiments in this report have used too high amount of nanomaterial for commercial use. With a quick calculation this can be seen, when the nanoparticles are stored dry it has a density from 50 g/L as the lowest (most of the nanoparticles have a higher dry storage density). A large drill rig has total storage capacity of 3000m^3 or 3000 000 liter, let us say a quarter of this will be mixed on board to 1 weight % nanofluid and the mud weight is 1,2 SG (which is low). Then we need 9000kg nanoparticles which is 180 000L or 180 m^3. Let us also say that the same ship has a capacity of storing 7500 sacks dry material then 180m^3 equals to around 6300 sacks if one sack is about 1 cubic foot. This example followed the large drill ship Stena DrillMax ICE capacity and it shows that even at 1wt % nanofluid the dry storage is not large enough to have both the low weight nanoparticles and the rest of the drilling muds dry material. This calls for other solutions for mixing the nanomaterial than mixing it onboard a drilling vessel. When the nanoparticles are mixed from the dry-form into a fluid, the bulk volume of the fluid does not change much due to the nano size and the porosity of the dry particles. One option is to mix the nanomaterials to a fluid with a very high content of nanoparticles at the service company and then bring and store it for use on the rig. Or mix the total fluid before it arrives at the rig. A reduction in the amount of nanomaterial used is beneficial and if it is going to be practical to use it should at least have a weight percent below 1.

The economy is also dependent on the weight percent of the nanomaterial in the fluid. If the weight percent is too high the economic benefit of the nanofluid can outweigh the cost of the nanomaterial. For this to become economically beneficial the nanomaterial should be made on an industry scale, some of the material used in the experiments in this report is already on such a level. However not all the nanoparticles are on such a level yet especially if the nanoparticles require surface treatment. It all depends on how good the nanofluid is to give economic benefits to the mud. The better the fluid is the more it is needed in the industry and the more the price will go down.

For the oil based mud the results are influenced by significant error sources and might be untrustworthy. The LPLT filtrate test does not seem suitable for such fluids due to the inconsistent results. The water based mud results shows a significantly higher fluids loss which economically means that an improvement in this base case gives more fluids or money saved. With an improved oil based mud there are not that much fluid to be saved, still the oil based mud are usually used to drill the reservoir part of the well. An improvement in the oil based mud like the ease of cleaning and fluid loss can improve the skin factor and thus an improved recovery. This means better improvement economically speaking than the water based mud can help with the lost mud.

The overall results shown in the above chapters indicate that the porous particles give a higher filtrate loss than the compacted does when the particles are miscible. The immiscible particles indicate that they give better filtrate loss. The down side about them is that they are difficult to mix, gives a thicker mud cake and decreases the density. The results in Table 2 and Table 4 also show that the density varies insignificantly with the added nanoparticles when they are miscible.

It was shown in Riveland 2012 [2] that aggregation was one of the main challenges that the previous published studies and that manipulation of the zeta potential may be critical to overcome this challenge. It is therefore thought that the aggregation could play a major part in the experiments in this report as well. If the aggregation would disappear the nanoparticles would truly be particles in the nano size and not aggregations in macro size.

At the University of Calgary almost the same experiments have been done, with an invert emulsion similar to the oil based mud used in this report. They found that Iron based nanoparticles gave a reduction of 91,5 % which is a significant reduction compared to the reduction in this report. However they reduced the fluid loss from 11 mL to 0,93 mL where the best reduction in this report shows a reduction from 0,8 to 0,5 mL which is a 62,5 % reduction. One difference in the two experiments is that the fluid they used was without lost circulation material but in the fluid used in this report was with. Maen Hussein and his colleagues in Calgary also tried with LCM and got a reduction of 10% [16].

The porous particles from Aerosil did not show any improvement in the filter loss rate in Table 2 or Table 4. A technical information sheet from Aerosil states that “The fractal structure of the fumed silica aggregate is the basis of the microporous matrix within coting sizing layer. Its sponge-like structure with well-defined pores and channels provides the capillary action needed to quickly transport the ink vehicle away from the paper surface and prevent spreading along the fibers.”[24] These fumed or porous particles from Aerosil are used in the paper business and this citation may explain why the porous particles do not give favorable results. Compacted particles however have been tested by 3 other studies mentioned previously in this report and they also show indications of reducing the filtrate loss rate in Table 4.

5. Further work

Nanotechnology in the drilling engineering and petroleum business is new hence there have not been done a significantly amount of work. In 2010, 2011 and 2012 there were published over 300 papers with the key word nanofluid and before 2005 less than 25 papers a year was published with that key word [8]. This report did not find a nanofluid that meet the expectations that was hoped for, but it gives indications of what does not work and where further work is needed to explore the potential of nanoparticles in drilling fluid.

The rheology tests in this report show that there are indications that nanoparticles can be used to enhance the rheology. More specifically the particles have lowered the viscosity of the drilling fluid and increased the yield point. A study to understand how the nano scale particles interact with the complexity of the drilling fluid is needed. Other work that also should be done is an in-depth study of the drilling fluid to design the shape and surface property of a nanoparticle. The drilling fluid should have a reasonably high yield point and at the same time have low viscosity from the point the shear stress breaks the yield point and to high shear rate. A rheometer with good precision and the ability to measure flow point and viscosity at low shear rates such as the MCR 302 should be used.

The filtration tests in this report shows where there is a need for further work and which particles that should not be used. From the data presented in this report it is not clear what particles that are good for oil based mud, however this report shows that for oil based drilling fluid with already good filtration properties should be tested in another test apparatus then the LPLT filter press. For the water based drilling fluid the report indicates that a porous nanoparticle does not show favorable results to reduce the filtration. It also shows that there is more volume to be reduced for the water based mud than the oil based. The report also indicates that the smaller particles influences the filtration volume more than larger ones. It suggests that the surface of the nanoparticles is modified in such a way that they do not aggregate together and stay inert to the rest of the fluid. A study of other water based nanomaterial is therefore recommended, preferably compacted nanoparticles and an even PSD all the way from the smallest nano particles to the barite sizes.

A study that should be done after a nanofluid has been found with a filtrate test is a return permeability test. That is to first flow the mud through a core and then measure the return permeability. This is a good test to see if the nanoparticles get stuck in the formation or how easy it is to clean the wellbore.

This report did not do many tests with regards to the tribology changes with added nanoparticles, but the tests that were made and previous reports suggests that nanoparticles may have a good effect to reduce the friction factor and scar depth. Reduction in friction can make it possible to drill a significantly longer well and so a study to find out which nanoparticles that can do this is highly recommended. The same procedure as is written in this report can be applied to find steel against steel or steel against rock friction factor. To do a study of steel against rock the small steel blocks in this report has to be changed with smooth and similar rock blocks. It might be a challenge, but to see if the nanoparticles give a different result for rock versus steel than for steel versus steel is interesting.

6. Conclusion

- This study found a reduction of 22,5 % fluid loss in WBM and a reduction from 0,8 to 0,5 mL of fluid loss in OBM, but the study failed to find a superior nanoparticle that enhanced the drilling fluids in all its properties. It was found that the particles should be miscible with the base fluid, to avoid giving higher viscosity, thicker filter cake and lower density. It is also suggested that if the nanoparticles are to be used by the industry the content should be less than 1wt%.
- The further work that needs to be done is: To explore different surface treatments and compact particles to find inert nanoparticles with little agglomeration and a wide spread in the PSD. Test the permeability and reverse permeability through a core. Ball on plate tests with both steel on steel and steel on rock surfaces to optimize friction and wear on the steel.
- Challenges has been identified, the greatest challenge in this study was with the oil based drilling fluid and the low filtrate rate from the test. The solution suggested is to do a different experiment when dealing with oil based drilling fluid with good filtration properties.

7. Nomenclature

API	-	American Petroleum Institute
CMC	-	Carboxymethylcellulose
HEC	-	Hydroxyethylcellulose
HPHT	-	High Pressure High Temperature
LCM	-	Lost Circulation Material
LPLT	-	Low Pressure Low Temperature
OBM	-	Oil Based Mud
OW	-	Oil Wet
PAC	-	Polyanionic cellulose
PSD	-	Particle Size Distribution
ROP	-	Rate of Penetration
Sil	-	S-69 Silane
T120 3li	-	3- (trimethoxysilyl) propyl methacrylate
WBM	-	Water Based Mud
WW	-	Water Wet

8. References

1. Md. Amanulah, Mohammad K Al-Arfaj and AL-Abdullatif. 2011. “Preliminary Test Results of Nano-based Drilling Fluids for Oil and Gas Field Application” SPE/IADC 139534. SPE Drilling conference and Exhibition, Amsterdam, The Nederlands, 1-3 March 2011.
2. Frode Riveland 2012. “Application of Nanotechnology in Drilling & Completions”. Specialization Project, NTNU, Norway.
3. Wikipedia 2012. Nanotechnology, (17 December revision) http://en.wikipedia.org/wiki/Nanotechnology (accessed 17.12.2012)
4. Serrano E, Rus G and Martinez JG. 2009 Nanotechnology for sustainable energy. Renew Sust Energy Rev 2009; 13 (December 9): 2373-84
5. Smalley, R.E. and Yakobsonb, B.I 1998. The Future of the Fullerenes. Solid State Communications, 1998, 107(11), 597-606
6. Zhou, W., Heiney, P.A. and Fan, H. et al. 2005. Single-Walled Carbon Nanotube-Templated Crystallization of H_2SO_4: Direct Evidence for Protonation. J. of the American Chemical Soc., 2005, 127, 1640-1641.
7. R. Kelsall, I. Hamley, and M. Geoghegan.2005. “Nanoscale Science and Technology," John Wiley & Sons Ltd, 2005.
8. J. He.2012.Potentials of Nanofluids for Drilling. SBBU- Center for Drilling and wells for Improved Recovery. (Not yet published)
9. Kostic M, Golubovic M, Hull JR, Choi SUS, One-Step Method for the production of Nanofluids, US Patent Number: US 7,718,033 B1.
10. Paul McElfresh, Marodi Wood and Daniel Ector. 2012. Stabilizing Nanoparticle Dispersions in High Salinity, High Temperature Downhole Environment. Paper SPE 154758 presented at the SPE Internationoal Oilfield Nanotechnology Conference held in Noordwijk, The Netherlands, 12-14 June 2012.
11. Meghan Riley et al. « Wellbore Stability in Unconventional Shale – The Design of a Nano-particle Fluid» SPE 153729 SPE Oil and Gas India Conference and Exhibition held in Mumbai, India, 28-30 March 2012
12. Wikipedia Steric effects (5.08.2012 revision) http://en.wikipedia.org/wiki/Steric_effects#Steric_hindrance (accessed 10.12.2012)
13. Schlumberger glossary, Bentonite http://www.glossary.oilfield.slb.com/Display.cfm?Term=bentonite (accessed 06.11.2012)
14. Todd Gilmore; Effective Slimhole Cementing in a Challanging Environment – A Gulf of Thailand Case History, SPE Asia Pasific Oil and Gas Conference and Exhibition, Jakarta, Indonesia, 17-19 April, 2001.
15. Al-Bezali T.M. et. Al., «Factors Controlling the Membrane Efficiency of Shale when Interacting With Water-Based and Oil Based Muds” SPE 100735-MS2006
16. Maen Hussein “Enhancing Drilling Fluid Performance Using Nanoparticles” Presentation given at the ACAMP Conventional Engery Seminar, Calgary, September 2012.
17. Sushant Agarwal, Tran X. Phuoc, Yee Soong et al. “Nanoparticle-stabilized invert drilling fluids for deep-hole drilling for oil and gas” The Canadian Jurnal of Chemical Engineering 9999 1-9 http://onlinelibrary.wiley.com/doi/10.1002/cjce.21768/pdf

18. Peng, De-Xing. «Size effects of SiO2 nanoparticles as oil.» *Industrial Lubrication and Tribology*, Number 2 Volume 62 2010: 111-120
19. YU He-long, XU Yi, SHI Pei-jing et al.2007. ”Tribological properties and lubricating mechanisms of Cu nanoparticles in lubricant” Science press, www.Sciencedirect.com Accepted 17. December 2007.
20. Pål Skalle «Drilling fluid Engineering» 2011 Pål Skalle & Ventus Publishing. ISBN 978-87-7681-929-3
21. Jamal J. Azar, G. Robello Samuel. 2007. Drilling Engineering. Oklahoma, PennWell Corporation.
22. D.Vader, H.Wyss. “Introduction to Rheology” http://weitzlab.seas.harvard.edu/links/tutorials/introductiontorheology2.pdf (accessed 04.06.2013)
23. Martin Chaplin. “Hydrocolloid rheology”. http://www.lsbu.ac.uk/water/hyrhe.html (accessed 04.06.2013)
24. Evonik Industries “Paper Sizing with AERODISP fumed Silica Dispersions, Technical Information” https://www.aerosil.com/www2/uploads_all/text/TI_1322_AE_e_Sep08.pdf (accessed 04.06.2013)

9. Appendix A

This appendix shows figures that tables 2-4 is based on and the thixotropic test of the OBM.

9.1 Oil based mud figures

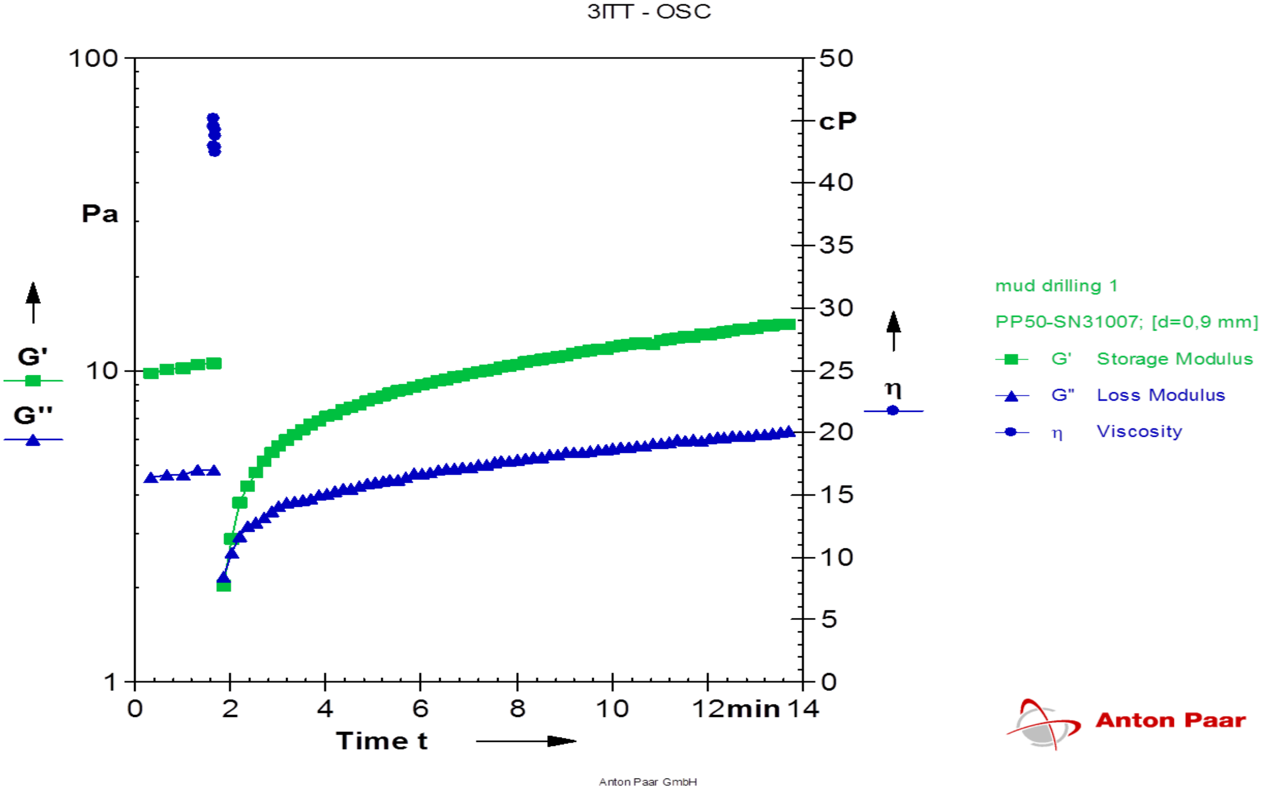

Figure 32 Thixotropy test with OBM. In the first part the fluid is resting, second it is sheared third it is restoring. It takes about 10 min before the fluid is restored.

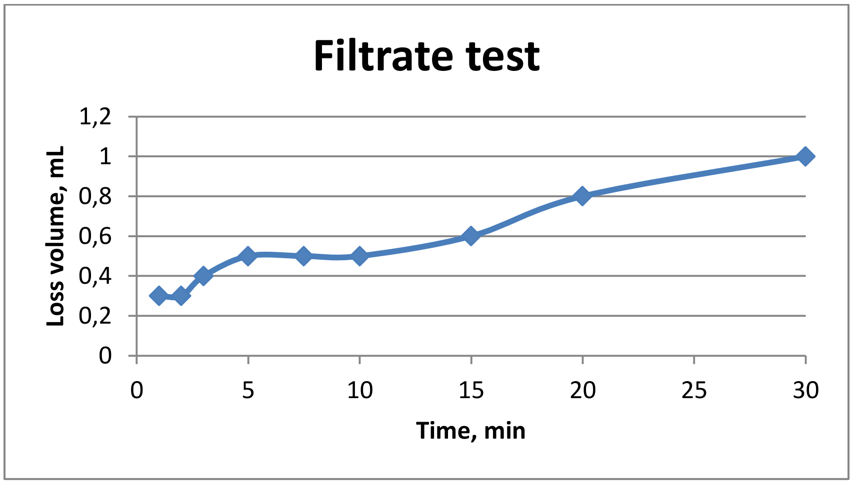

Figure 33 Filtrate test with OBM and 2% R972V

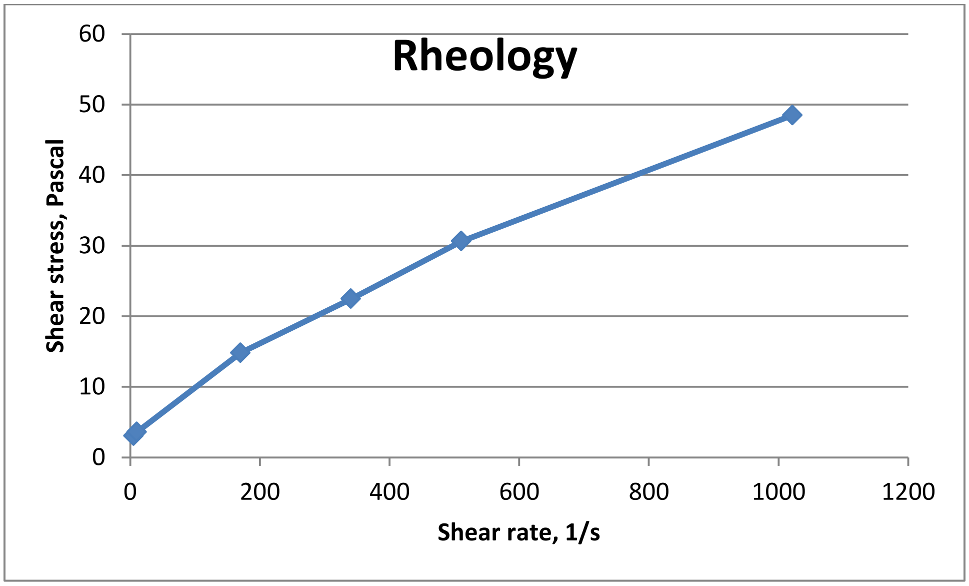

Figure 34 Rheology test with OBM and 2 % R972V

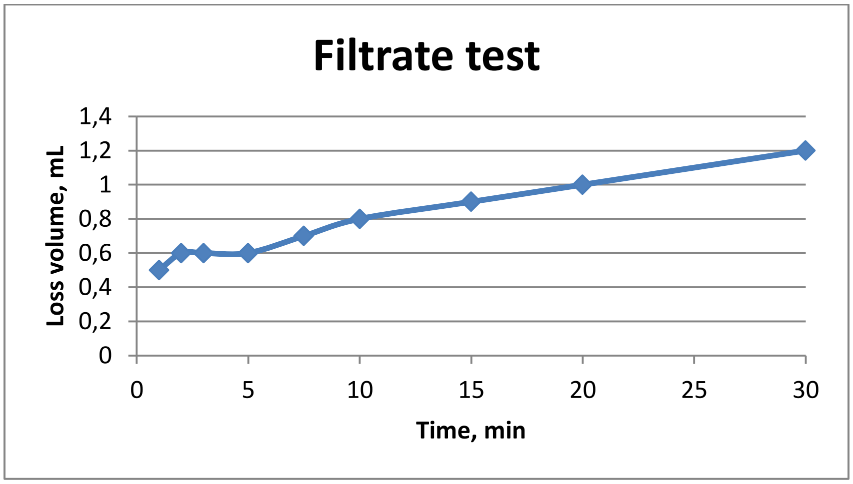

Figure 35 Filtrate test with OBM and 1% R972V

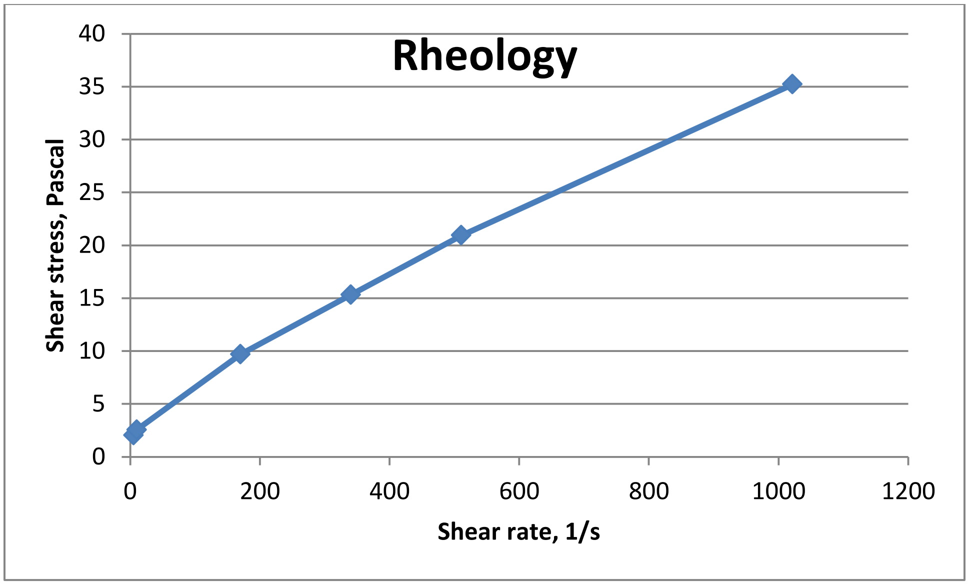

Figure 36 Rheology test with OBM and 1% R972V

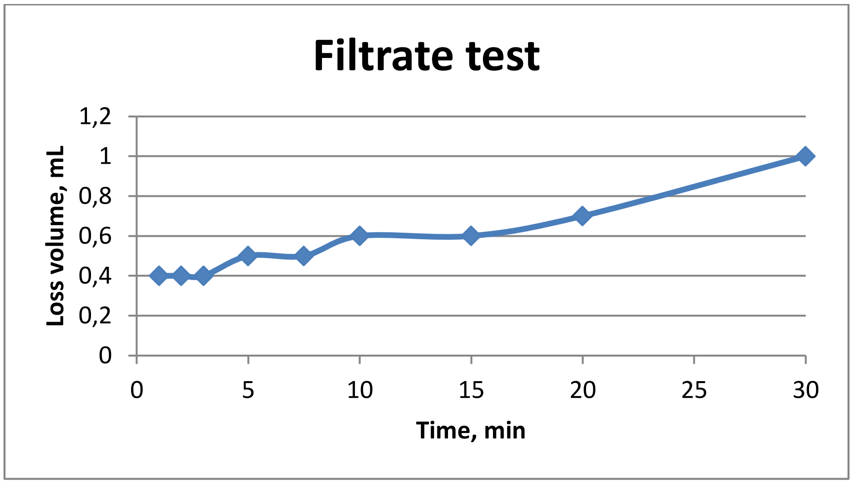

Figure 37 Filtrate test with OBM and 0,5 % R972V

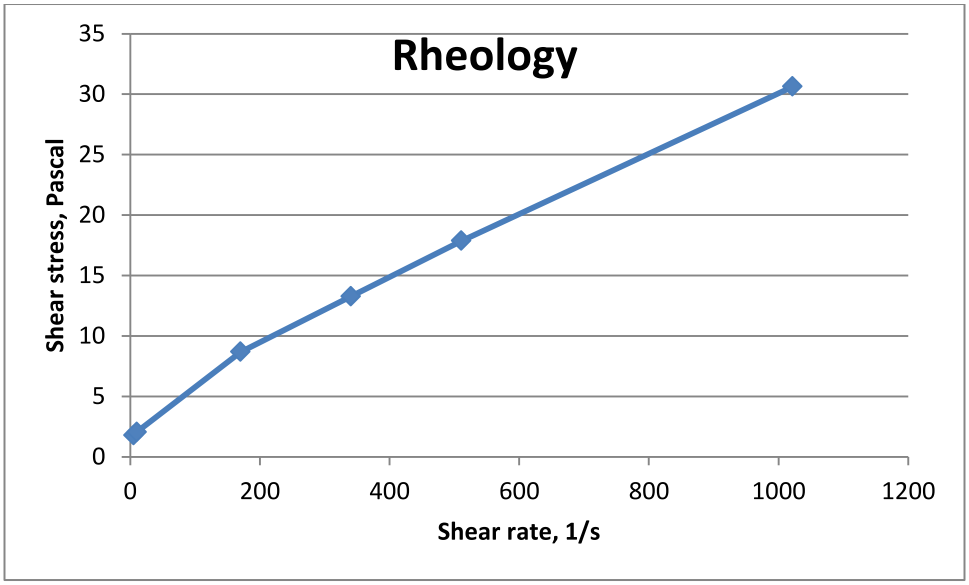

Figure 38 Rheology test with OBM and 0,5% R972V

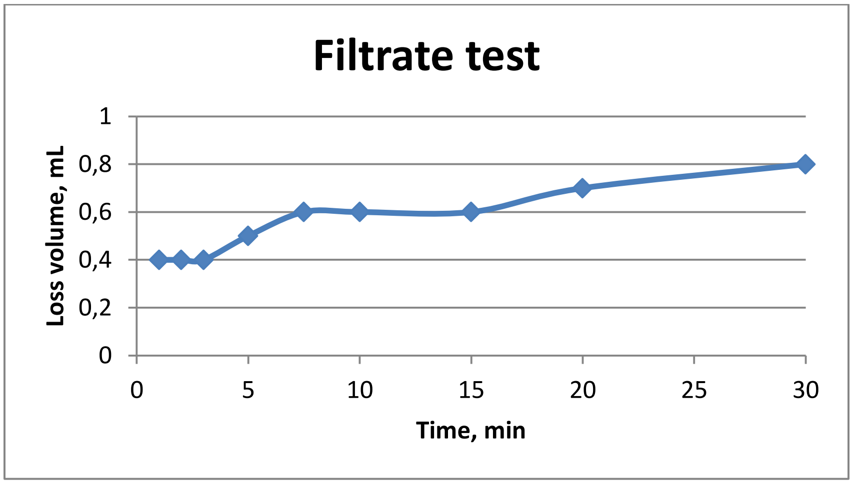

Figure 39 Filtrate test with OBM and 0,1% R972V

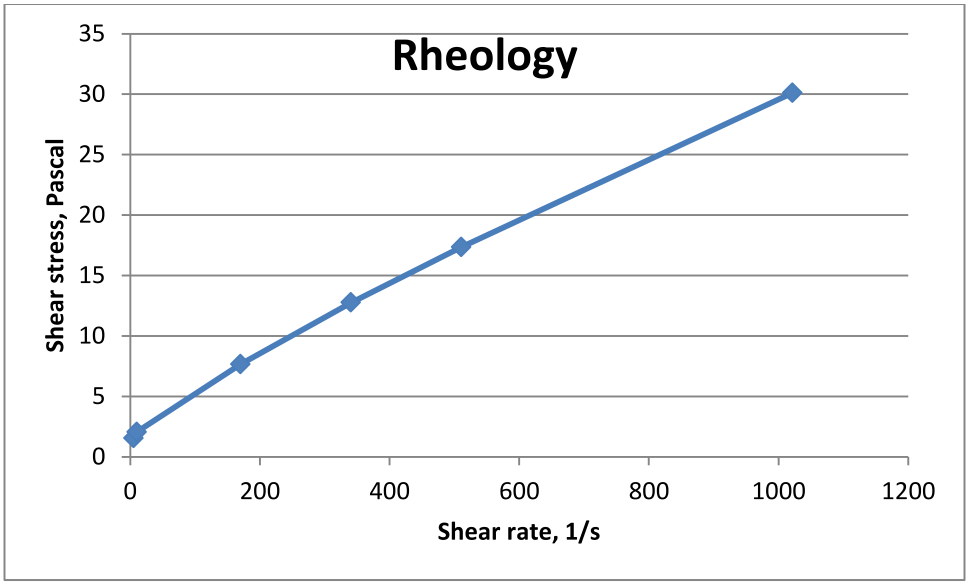

Figure 40 Rheology test with OBM and 0,1% R972V

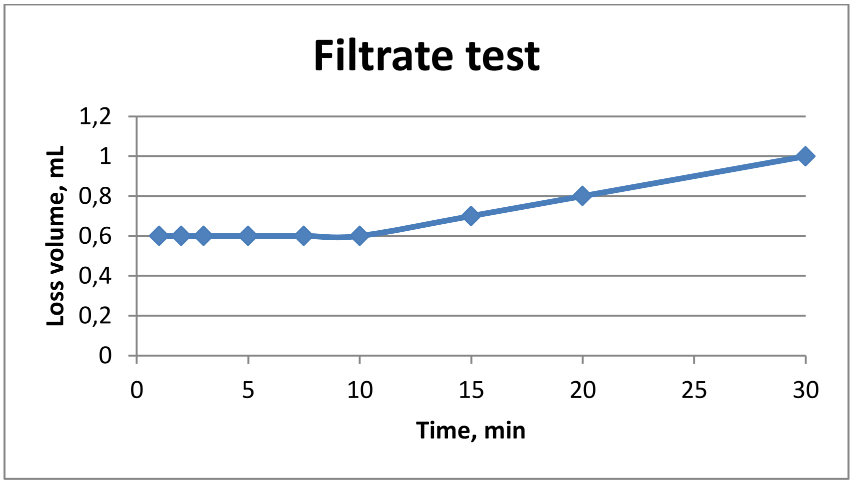

Figure 41 Filtrate test with OBM and 1% T120 3li

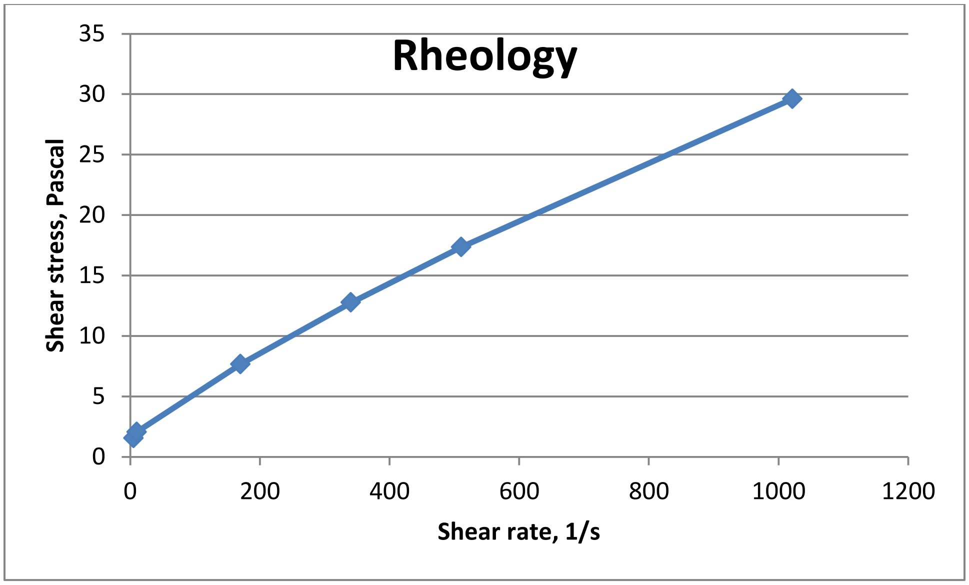

Figure 42 Rheology test with OBM and 1% T120 3li

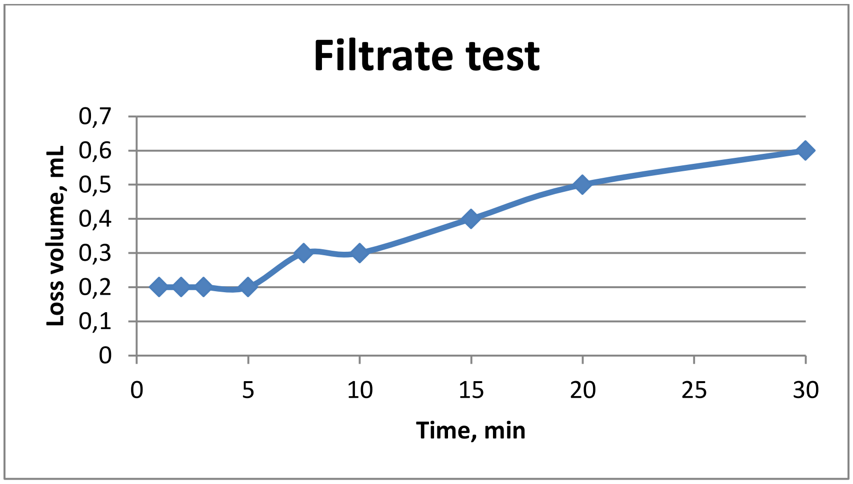

Figure 43 Filtrate test with OBM and 0,1 % T120 3li

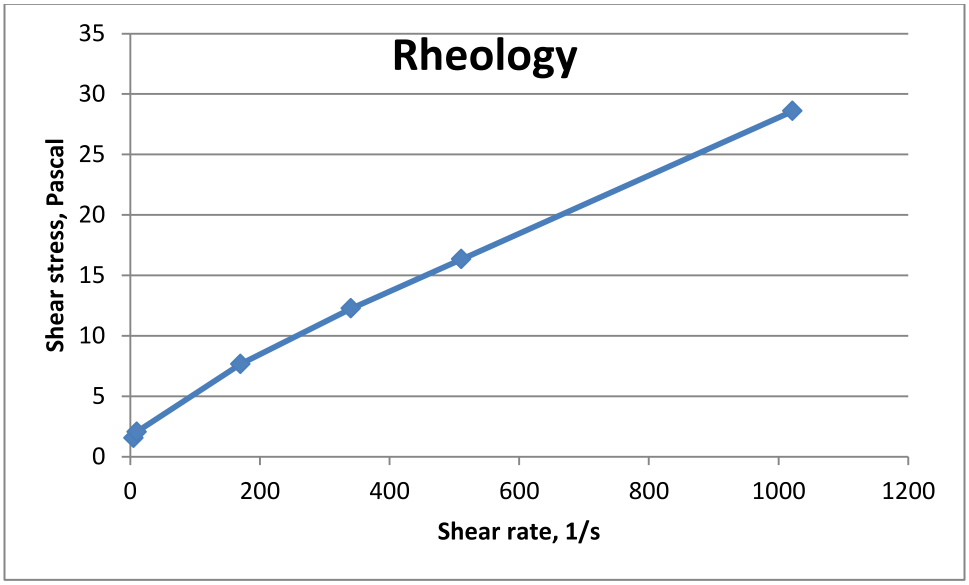

Figure 44 Rheology test with 0,1 % T120 3li

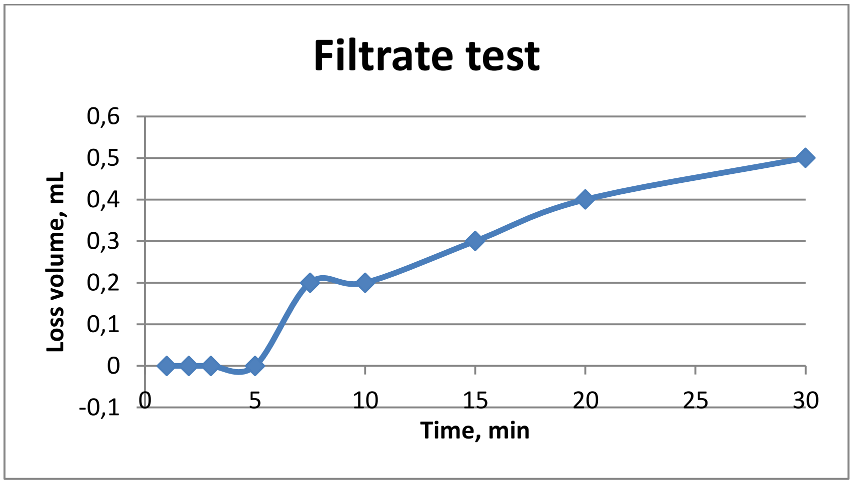

Figure 45 Filtrate test with OBM and 1% sil

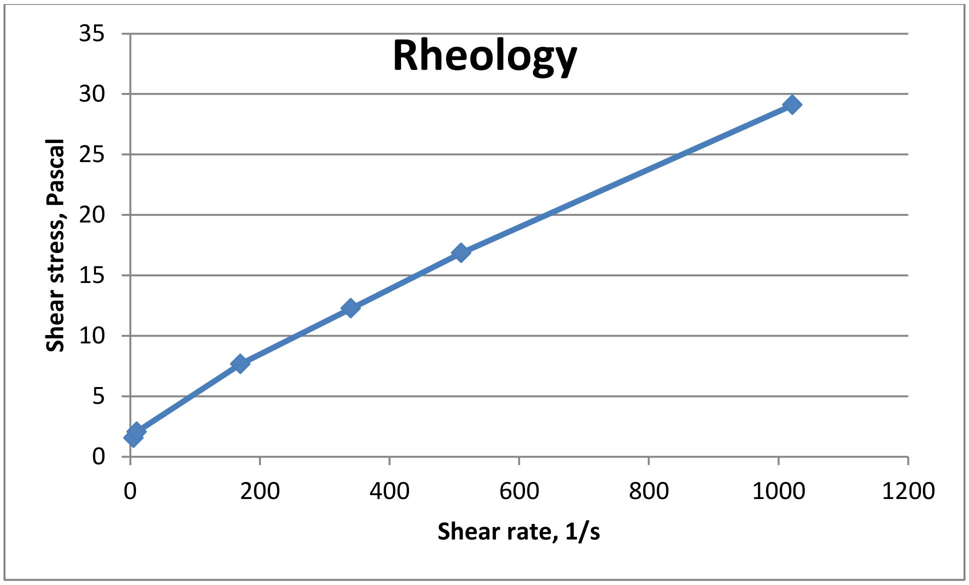

Figure 46 Rheology test with OBM and 1 % sil

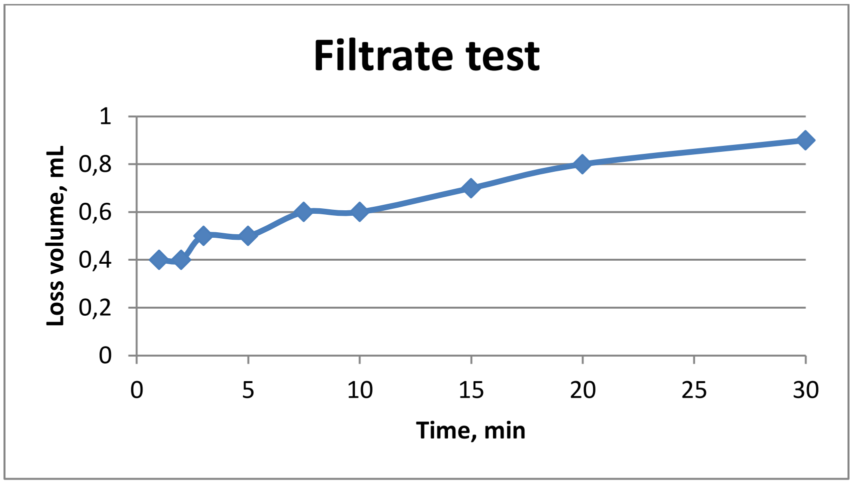

Figure 47 Filtrate test with OBM and 0,5 % sil

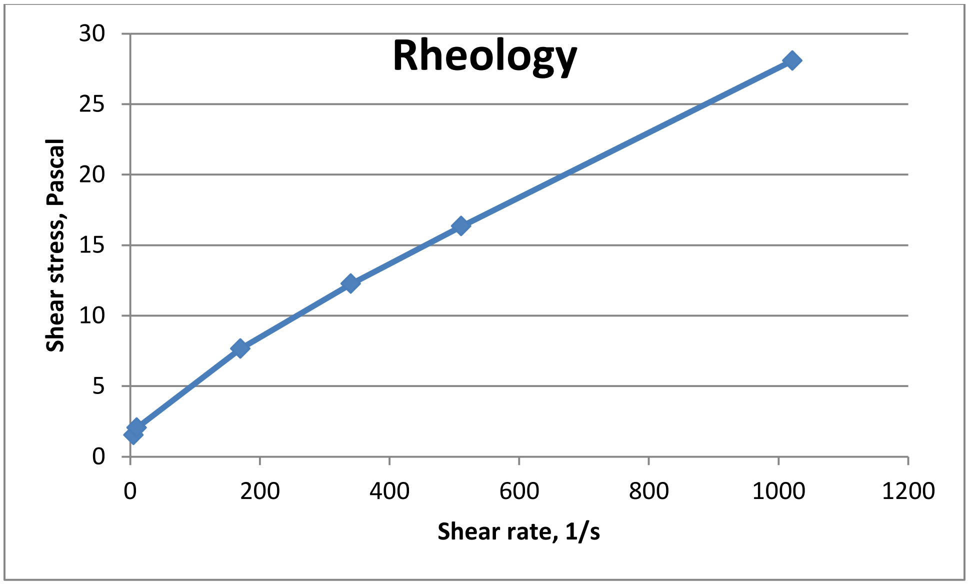

Figure 48 filtrate test with OBM and 0,5 % sil

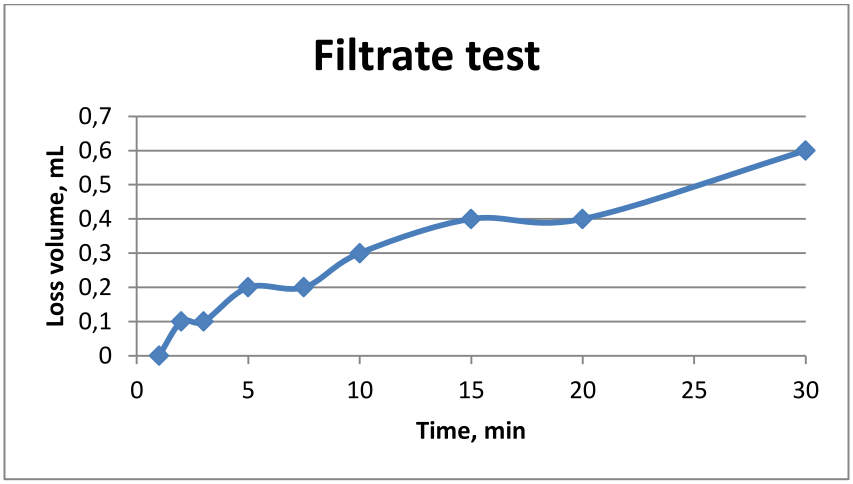

Figure 49 Filtrate test with OBM and 0,1 %sil

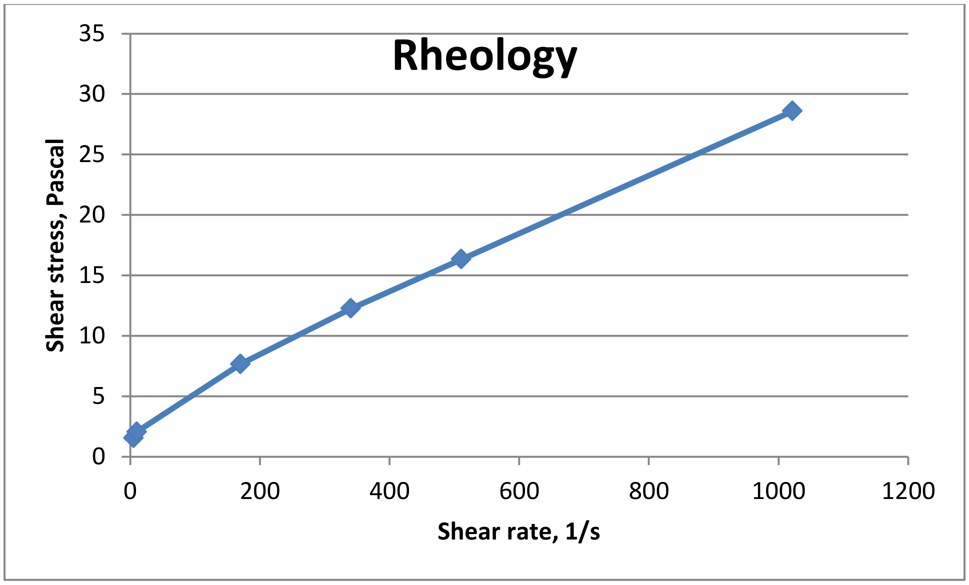

Figure 50 Rheology test with OBM and0,1 % sil

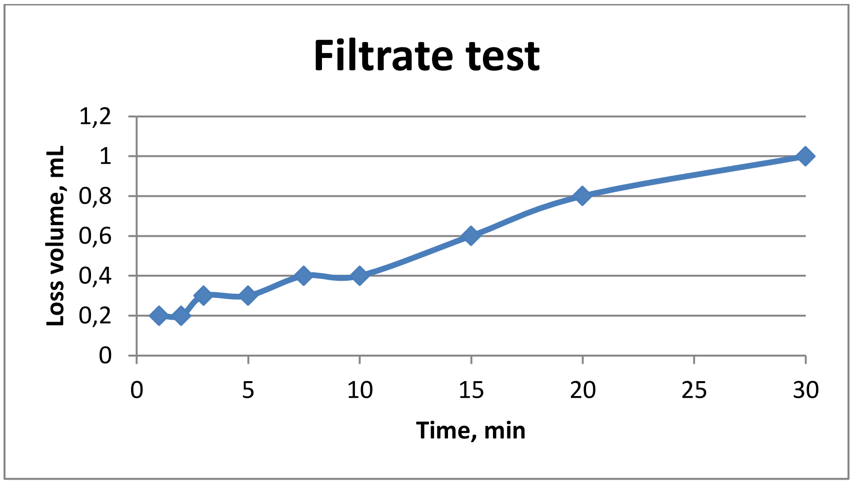

Figure 51 Filtrate test with OBM and 1% R812S

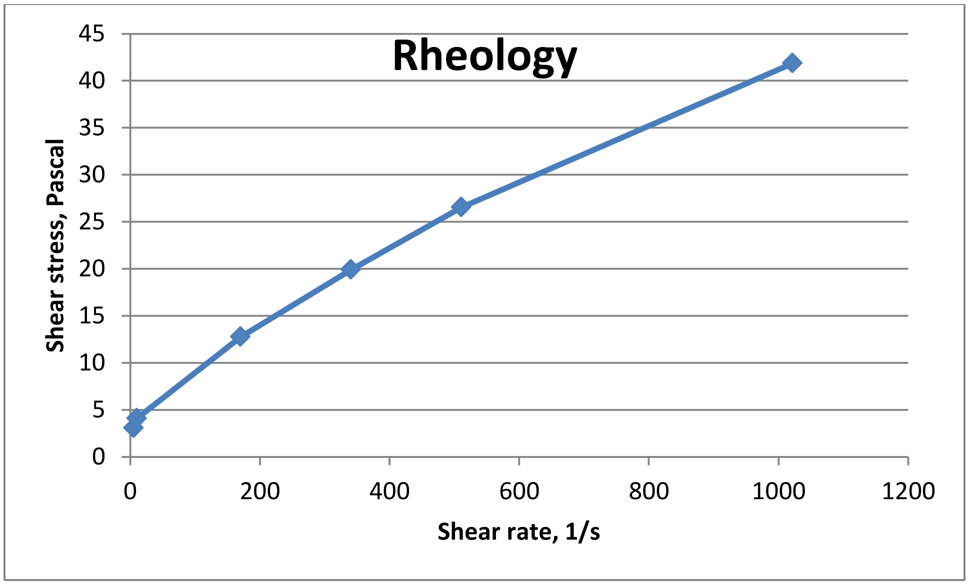

Figure 52 Rheology test with OBM and 1 % R812S

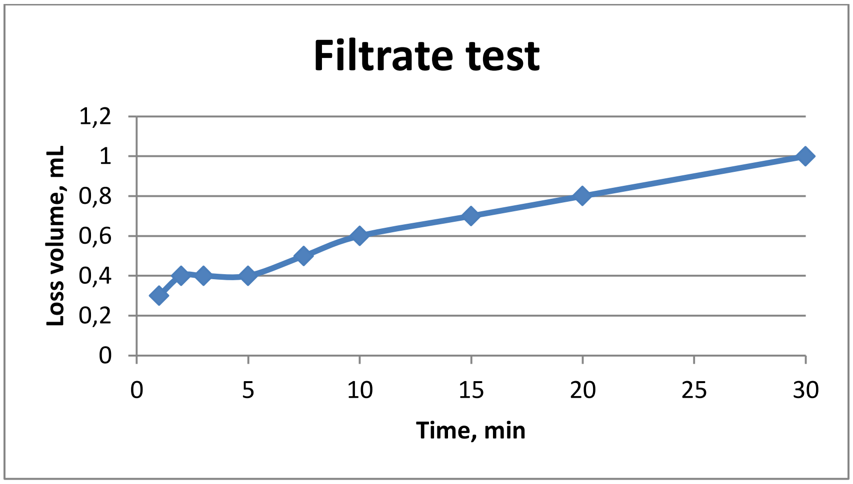

Figure 53 Filtrate test with OBM and 0,5% R812S

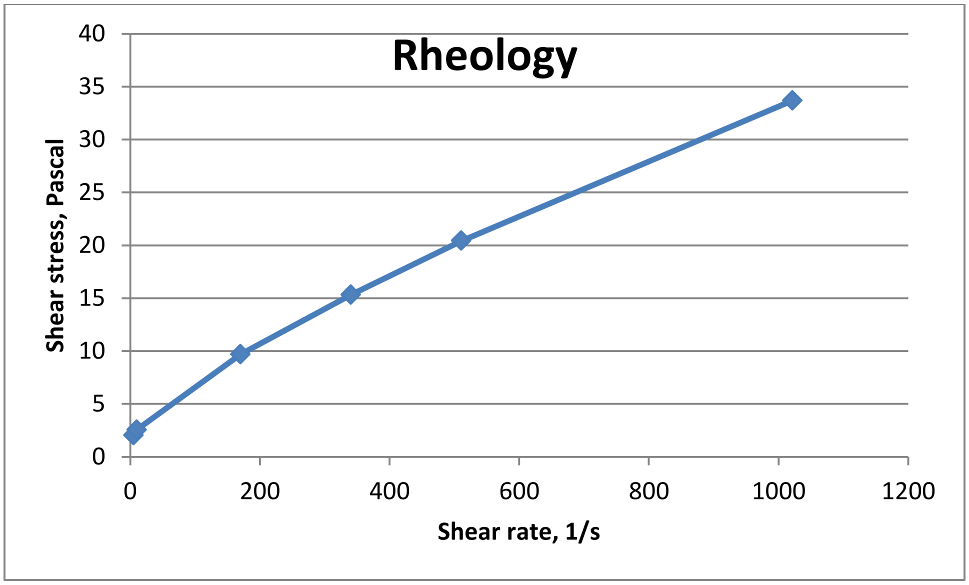

Figure 54 Rheology test with OBM and 0,5 % R812S

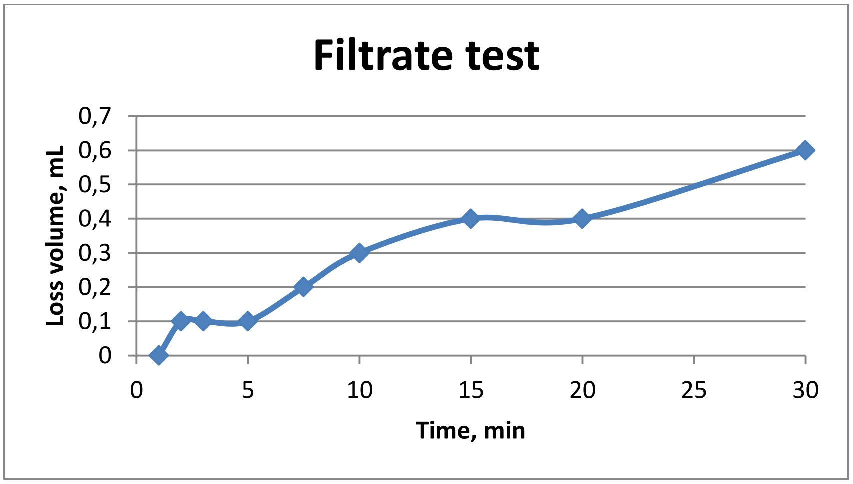

Figure 55 Filtrate test with OBM and 0,1 % R812S

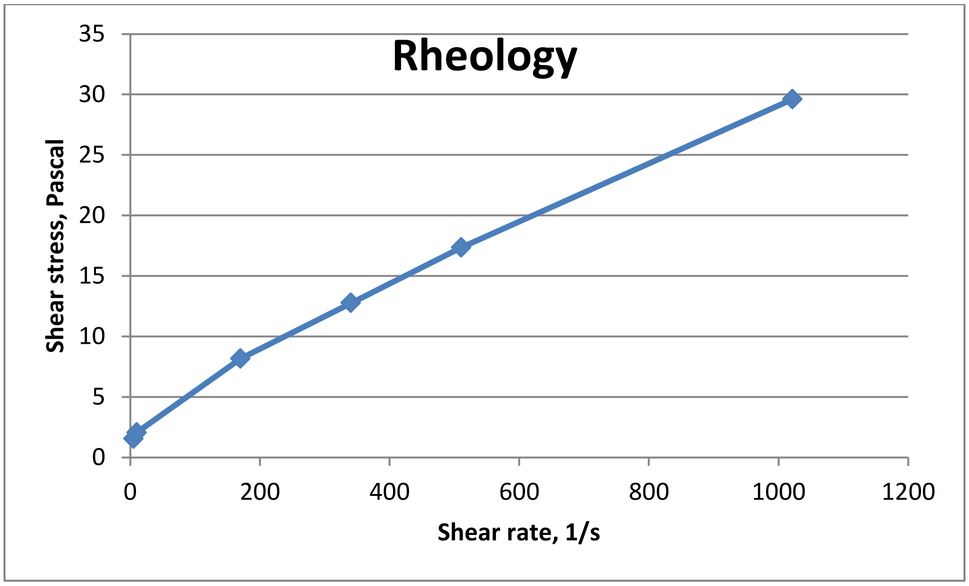

Figure 56 Rheology test with OBM and 0,1 R812S

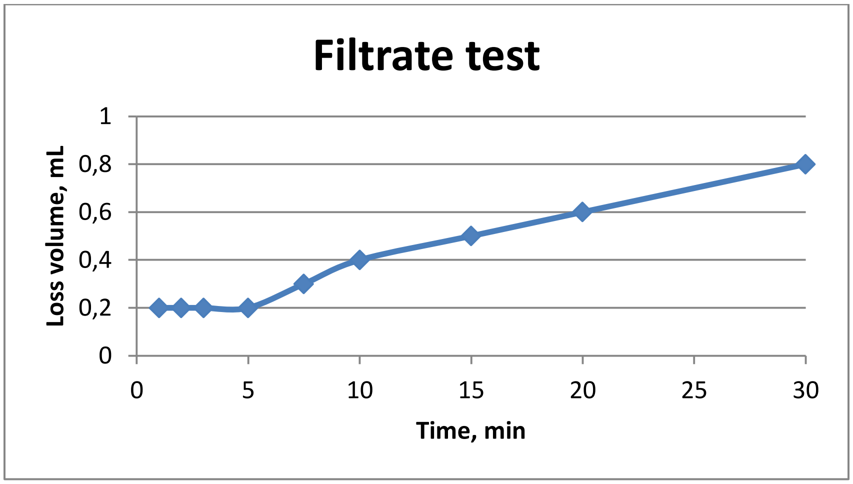

Figure 57 Filtrate test with OBM and 1% R972

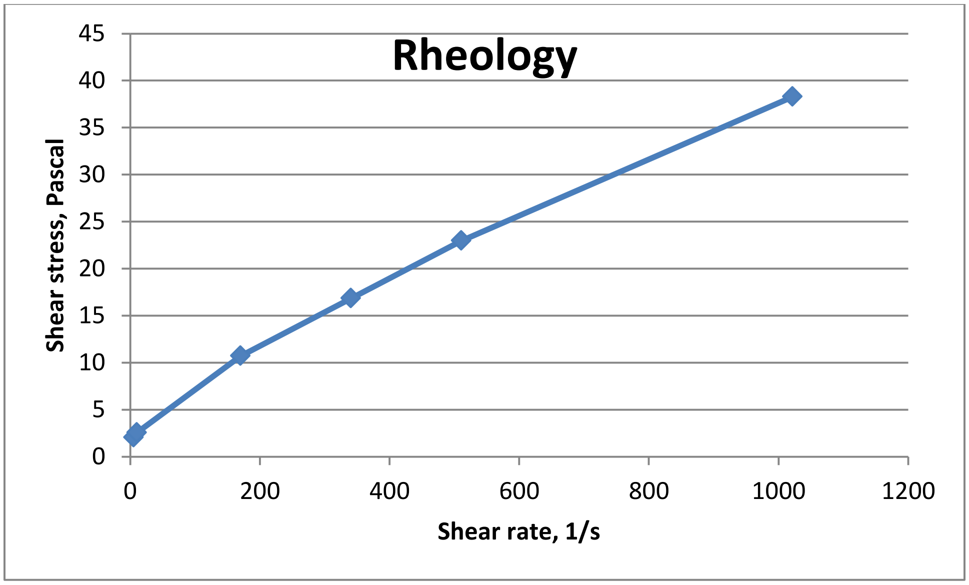

Figure 58 Rheology test with OBM and 1% R972

9.2 Water based mud figures

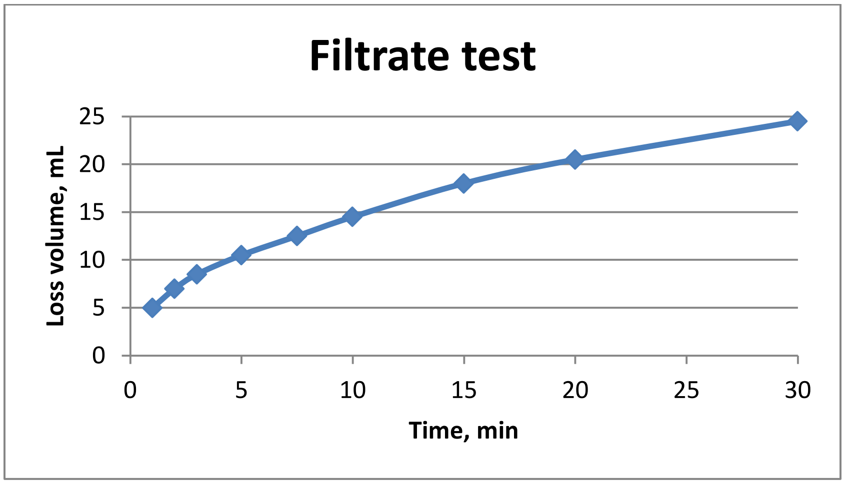

Figure 59 Filtrate test with WBM and 1 % 100nm ww particles

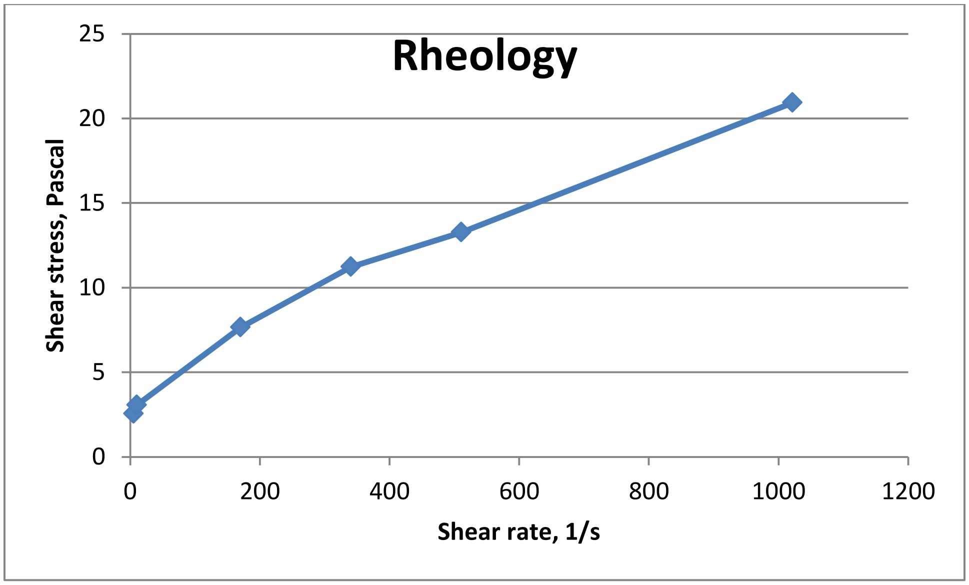

Figure 60 Rheology test with WBM and 1 % 100nm ww particles

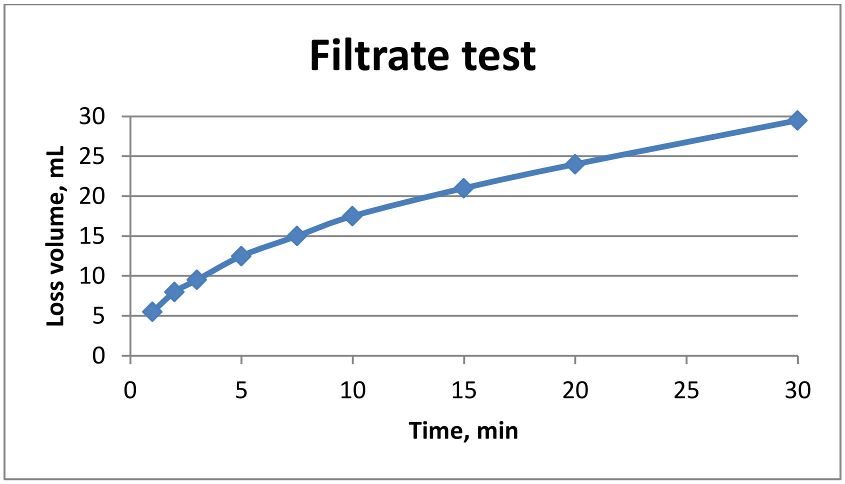

Figure 61 Filtrate test with WBM and 1% 7nm ww particles

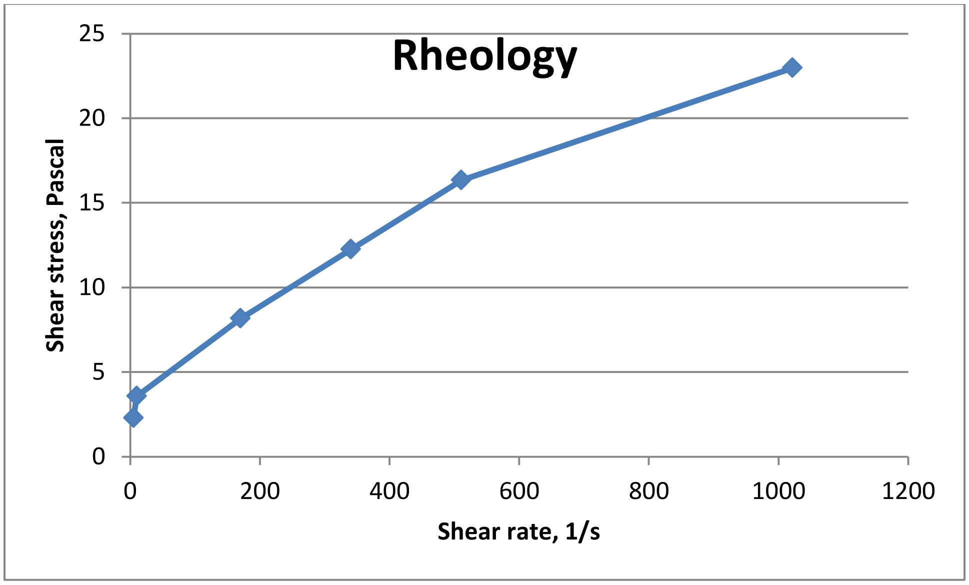

Figure 62 Rheology test with WBM and 1% 7nm ww particles

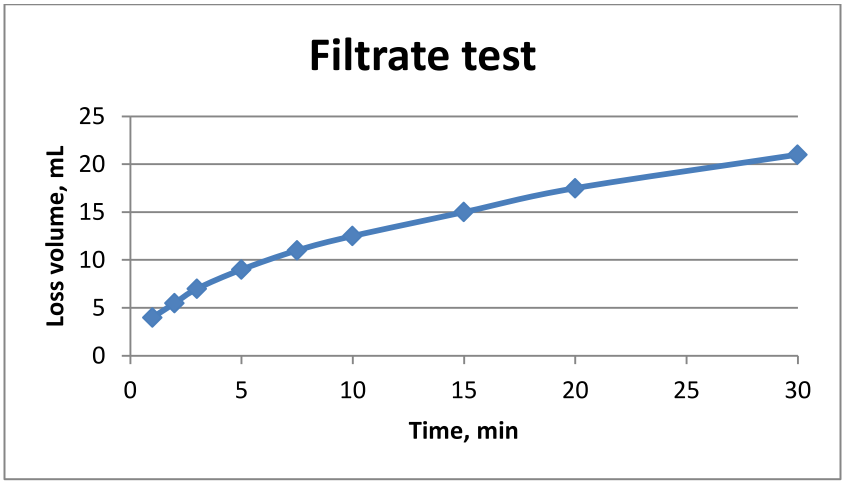

Figure 63 Filtrate test with WBM and 1% ow 16nm particles

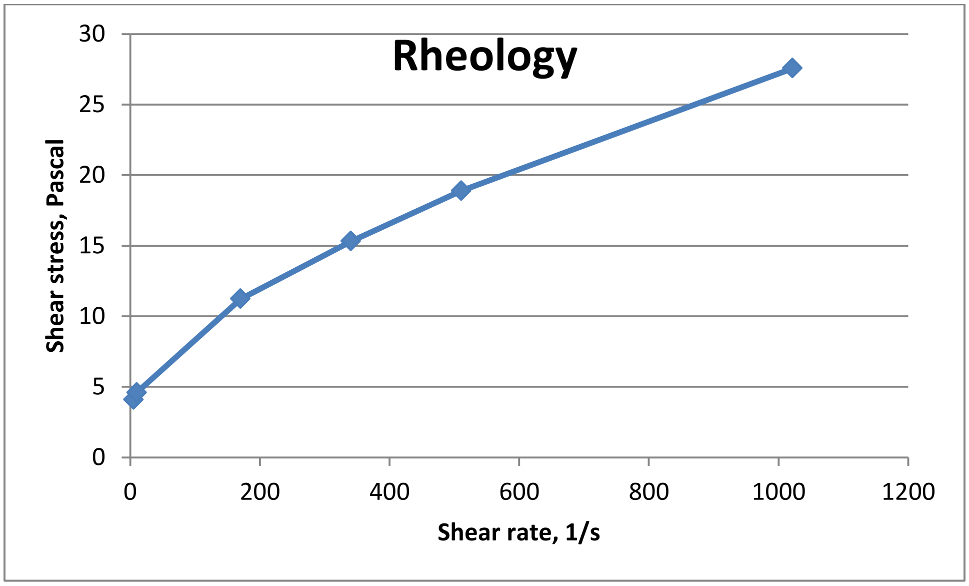

Figure 64 Rheology test with WBM and 1% ow 16nm particles

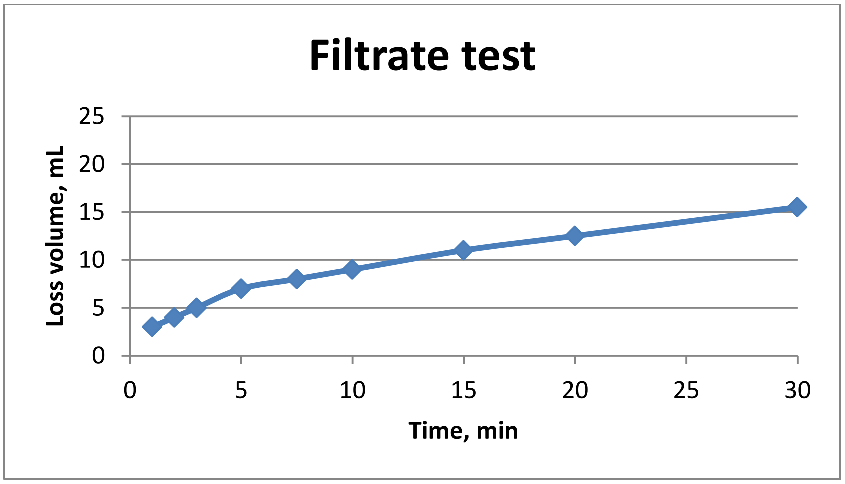

Figure 65 Filtrate test with WBM and 5% ow 7nm particles

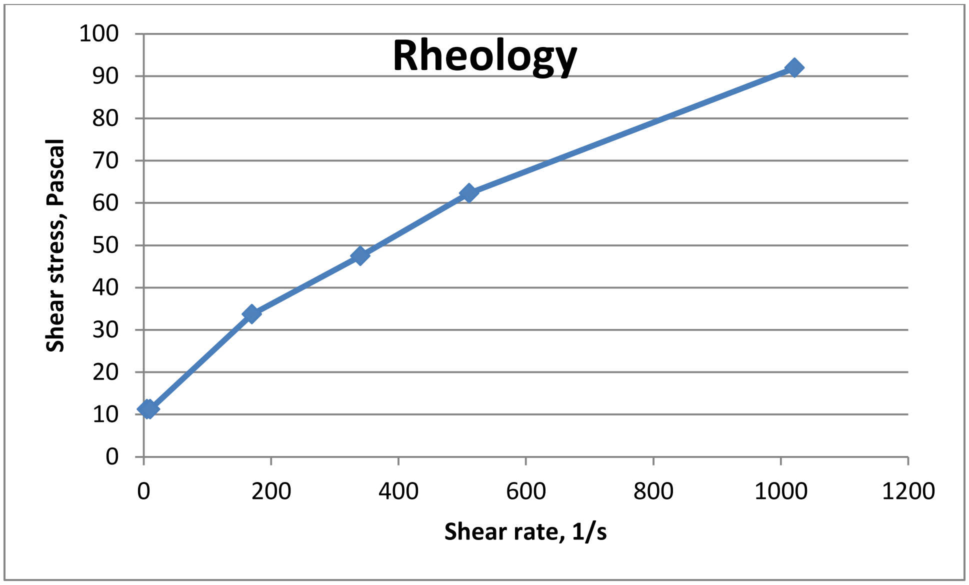

Figure 66 Rheology test with WBM and 5% ow 7nm particles

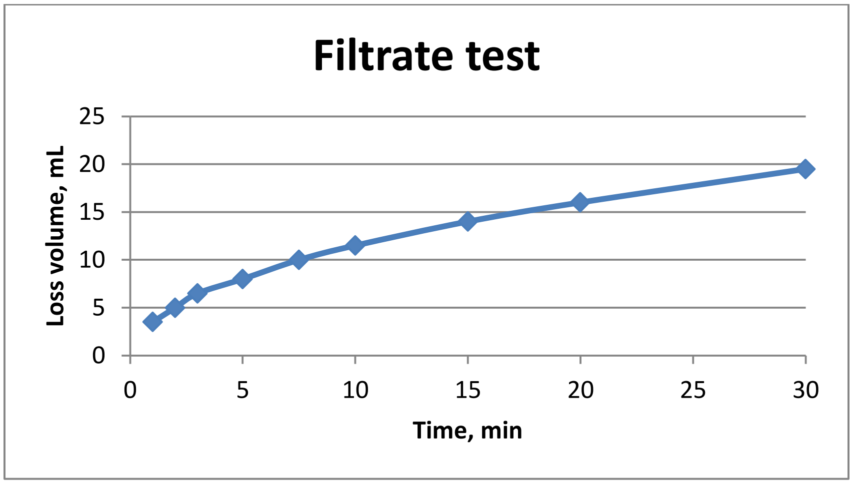

Figure 67 Filtrate test with WBM and 1% ow 7nm particles

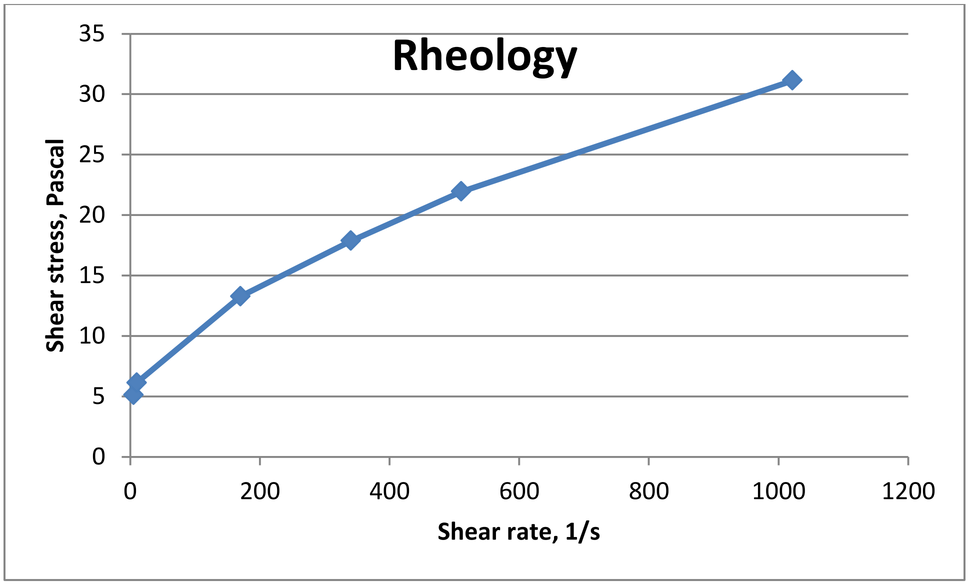

Figure 68 Rheology test with WBM and 1% ow 7nm particles

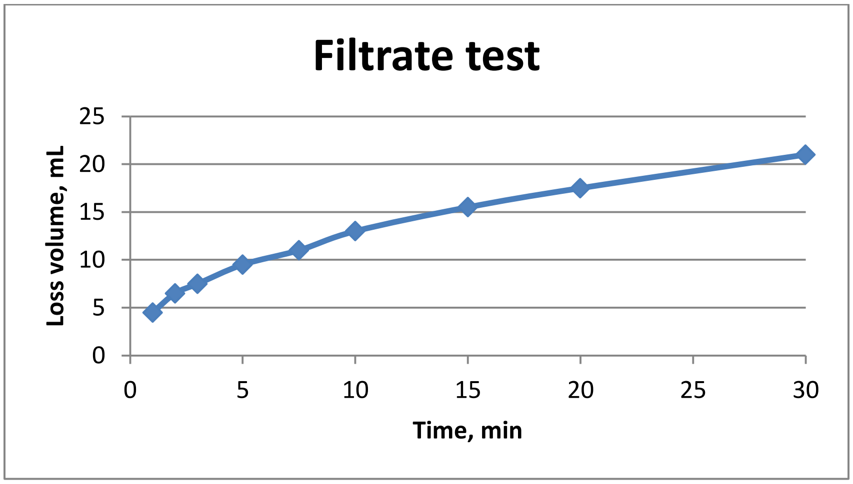

Figure 69 Filtrate test with WBM and 1% PMMA

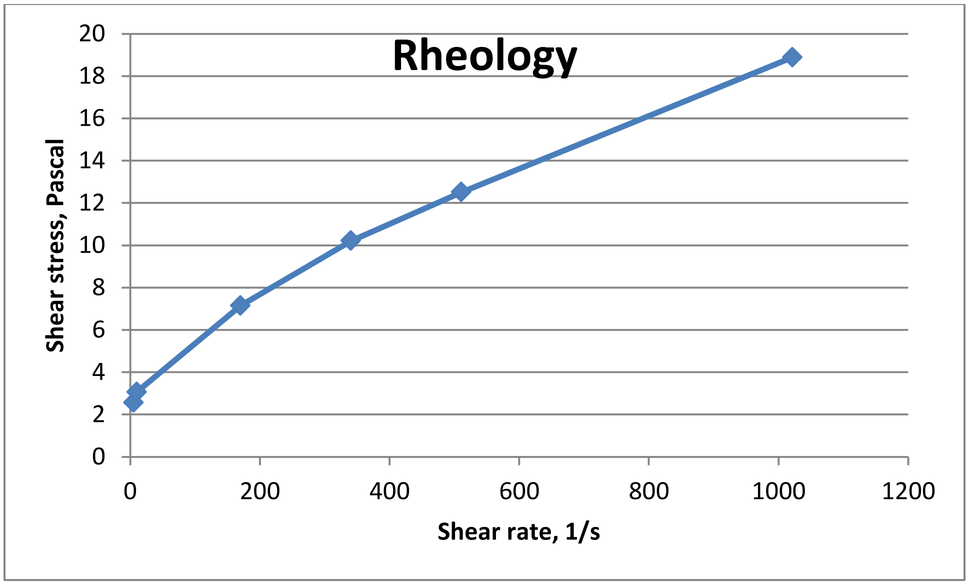

Figure 70 Rheology test with WBM and 1 %PMMA

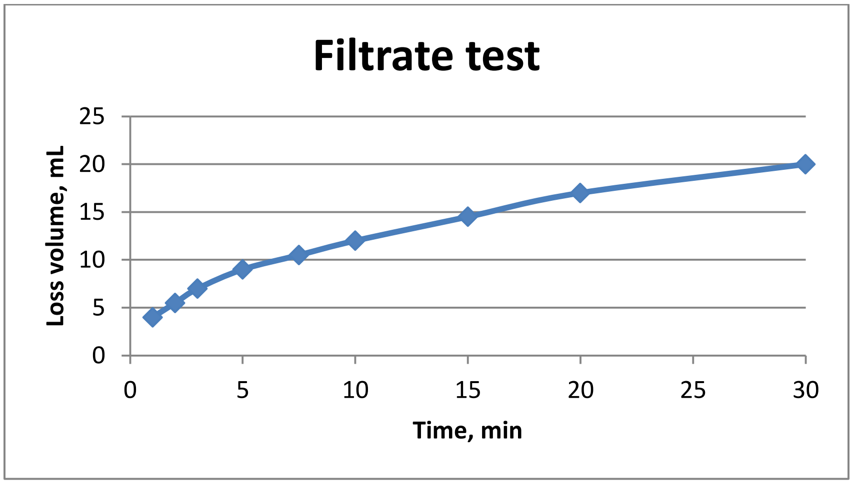

Figure 71 Filtrate test with WBM and 2% PMMA

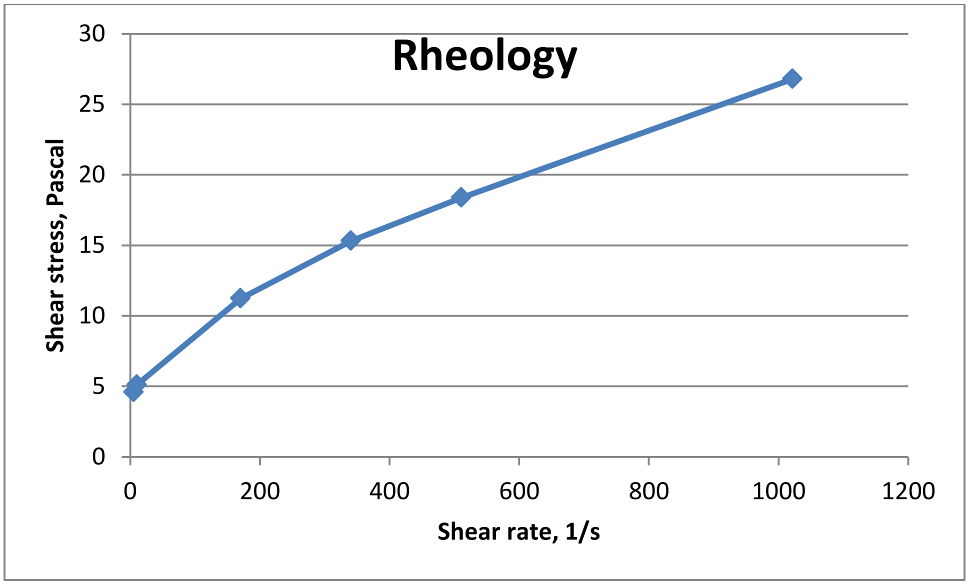

Figure 72 Rheology test with WBM and 2% PMMA