THICKENING TIME, RHEOLOGY AND FLUID LOSS OF OILFIELD CEMENT WITH NANO-PARTICLE SIZED ADDITIVES

By

VIGLEIK NICOLAI KJELDAL

Bachelor of Science in Petrochemical Engineering

Telemark Tekniske Skole

Porsgrunn, Norway

1976

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THICKENING TIME, RHEOLOGY AND

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Thesis Approved:

Dr. Geir Hareland

Thesis Advisor

Dr. Mohammed Al Dushaishi

Dr. Prem Bikkina

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Name: VIGLEIK NICOLAI KJELDAL

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Abstract: Cementing around the casing in oil and gas wells provides proper zonal isolation, holds the casing in place, and prevent fluid migration is an important part of the completing process, and well plugging for abandonment. By adding different nanoparticles (NPs) additives of barite or magnetite to heavy cement and bentonite to light cement, we seek to create the perfect cement sheet. This thesis study shows how adding NPs influences cement fluid properties such as thickening time, fluid loss, and rheology. A heavy control case cement formulation using Portland cement class H, barite, hydroxyethyl cellulose (HEC), boric acid, and seawater, was modified with three different concentrations for 1, 3, and 5 % by weight of cement (BWOC) of barite or magnetite NPs. A light control case cement formulation using Portland cement class A, bentonite, HEC and, seawater, was then modified with three different concentrations for 1, 3, and 5 % (BWOC) of bentonite NPs. A consistometer was used to find the cement thickening time, a high-pressure high-temperature (HPHT) fluid loss tester was used to study the cement slurry filtration and a viscometer apparatus was used to find the rheology properties, just after the cement placement. Thickening time and fluid loss were measured at high pressure and high temperature for heavy cement and high pressure and low temperature for light cement. The thickening time increased for all concentrations of NPs, except for the 5% BWOC magnetite NPs. Rheology properties were measured at low pressure and high temperature for heavy cement and low pressure and low temperature for light cement. The shear stress of the heavy cement increased for all concentrations of NPs, while there was an insignificant change for the light cement. Plastic viscosity decreased for all concentrations of NPs, except 1% BWOC magnetite NPs. For all types of NPs, it was observed that fluid loss generally decreased by increasing NP concentrations for both heavy and light cement.

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NOMENCLATURE

Al	Aluminum
API	American Petroleum Institute
Barite	Barium Sulfate, $BaSO_4$
Bc	Bearden of consistency (dimensionless unit)
Bentonite	Nanoclay Montmorillonite, $Al_2H_2O_{12}Si_4$
BWOC	By Weight Of Cement, unit (%)
boric acid	H_3BO_3
Ca	Calcium
CMHEC	carboxymethyl hydroxyethylcellulose
C/W	Cement/water ratio
CV	Coefficient of variation (%)
DI	deionized water
ECD	Equivalent Circulation Density, unit (ppg)
^oC	degrees Celsius, temperature
^oF	degrees Fahrenheit, temperature
Fe	Ferro (Iron)
HEC	Hydroxyethyl Cellulose
HPHT	High Pressure High Temperature
K	consistency index for the Power-law rheology model
Magnetite	Iron oxide, Fe_3O_4
n	Power-law exponent or flow-behavior index
N_2	Nitrogen
NAS	National Academy of Sciences
nm	nano meter
NP	Nanoparticle
PAA	Polyacrylic acid
PEC	Polyelectrolyte-complex
PEI	Polyethyleneimine
ppg	pounds per gallon
psi	pound per square inch
RPM	Revolutions Per Minute, unit (1/min)
SGS	Static Gel Strength
Si	Silisium
C_2S	dicalcium silicate
C_3A	tricalcium aluminate
C_3S	Tri-calcium silicate
C_4AF	tetracalcium aluminoferrite
US	United States

CHAPTER I

INTRODUCTION

1.1 Overview

This study is a part of a project at Oklahoma State University, funded by the National Academy of Sciences (NAS) Gulf Research Programs, and investigates the effect of different NP's on the cement slurry thickening time, rheology, and fluid loss. Casing installation and cementing around the casing is an important step to complete a well properly. Primary cementing is the process of placing cement in the annulus between the casing and the formations exposed to the borehole. Zonal isolation, supporting the walls of the hole, protecting the casing against plastic formations, and corrosive formation fluids are among the benefits of primary well cementing. A squeeze cement job is the other type of cementing that is often used to carry out remedial operations during a workover on the well. Repairing casing failures by squeezing cement through leaking joints or a corrosion hole, as well as sealing off lost circulation zones are among the benefits of squeeze cementing. Cement plugs are sometimes used during drilling as a remedial procedure to seal over the junk in the borehole. If the fishing job could not recover the junk out of the hole, usually a cement plug is placed on the top of the junk and a sidetrack well is drilled to bypass the junk. Placing a cement plug before well abandonment minimizes the risk of leaking hydrocarbons and other underground liquids and gases, as well as groundwater contamination and related threats to public health. This process is required by law to be completed by the operator company.

When designing the cement, it is important to select the right additives so that the cement slurry will set at the right time after the placement. Cement slurry should achieve adequate strength and set quickly enough so that the waiting time will be short. A long waiting time for cement slurry to set means a higher total cost of a well-construction; especially in offshore drilling where the daily rates of drilling rigs are much higher. At the same time, the cement slurry should not set too quickly, to avoid setting before pumping or during pumping. The other important consideration for designing a cementing job is to select the right density; cement slurry has to be heavy enough to withstand the pore pressure, but it should not be too heavy, as that could breakdown the formation and generate fractures. Possible contamination of cement slurry inside the hole by drilling fluid and formation fluids can influence the final cement quality.

The importance of cementing and designing cement slurry requires selecting the right additives at the right concentration to get the correct cement sheath for a specific application. To do so, there is a possibility of selecting between a broad range of additives including, but not limited to, accelerators, retarders, fluid loss control materials, extenders, and weighting agents. In this research, the addition of small amounts of a few types of NPs to cement slurry was studied.

1.2 NP as an additive

The use of NPs in cement is relatively new in the oil industry and most of the possible effects of using NPs in cement slurry are not known. NPs are very small particles (less than 1 micrometer in diameter), and considering the complex nature of cement itself, NPs might physically or chemically react with cement. In this study, three different NPs that consist of barite, magnetite, and bentonite were used as additives in the cement slurry. The heavy cement slurry with a density of 16.9 ppg was formulated by the cement class H, which contained normal barite as a weighting agent. barite and magnetite NPs were used at low concentrations in the slurry by replacing part of the weight of normal barite. The light cement slurry with a density of 13.7 ppg was formulated by using cement class A, and normal bentonite was used as an extender in this slurry. Bentonite NPs were used at low concentrations in this slurry by replacing normal bentonite. This study investigated the effects of adding these NPs to the slurries on the thickening time, rheology, and fluid loss.

1.3 Research objectives

This study had two main objectives. The first objective of this research was to shed light on the unknowns related to the properties of cement slurry containing different types of NPs. The second objective is to investigate the effect of NPs' concentration on cement thickening time, rheology and fluid loss properties.

1.4 Thesis structure

Chapter 2 describes the history of cementing. Well cementing was developed early in the 20^{th} century and improved through the following decades. New additives were introduced, tested, and improved. The purpose of this chapter is to introduce a well-cementing procedure, as well as cement itself and its additives. The use of NPs as an additive started about 20 years ago and many researchers were mostly focused on enhancing one specific property of the cement sheath. In the literature review, several important advancements of the application of well cementing were addressed. Chapter 3 introduces the research materials and describes the research methodology. The purpose of this chapter is to introduce the important experimental analyses that are required to be performed before conducting an actual cementing job. For example, setting time, rheology, and fluid loss are important cement tests that have to be done before the actual cementing job. The required apparatuses and procedures are described, and the final formulations have been created. Chapter 4 presents the experimental results for each specific set of tests. In this chapter, the trends were identified, compared, and analyzed for different NPs at different concentrations. The experimental results were discussed and explained. Anomalies and irregularities in the results have been identified and discussed. Chapter 5 presents the conclusions of this study. Chapter 6 present recommendations for future research in this field.

CHAPTER II

BACKGROUND AND LITERATURE REVIEW

2.1 Background

Since Erle Halliburton started with the cementing of the casing in the oil and gas wells in 1920, different cement experiments have been done and many additives have been tested to get the optimum and desired cement quality, the perfect cement sheath. Figure 2.1 shows an unknown employee from Halliburton sitting in his car and Halliburton's US patent for a well-cementing procedure.

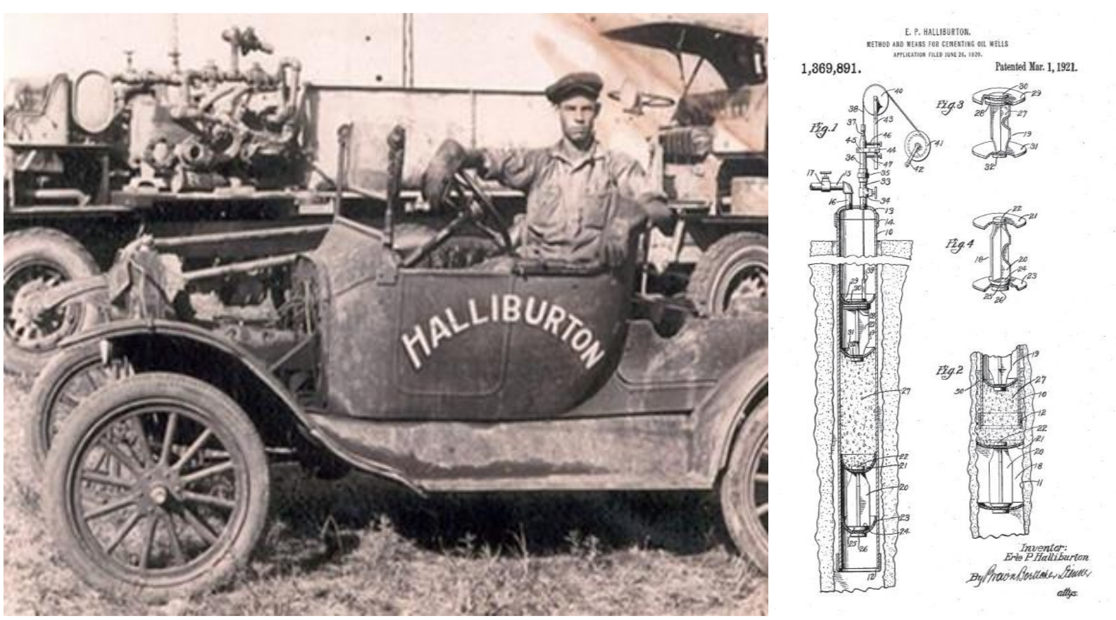

Figure 2.1 An unknown employee from Halliburton in 1920 and the Halliburton well-cementing process from 1921[13]

Cement is used to seal the well from the formation. It is placed in the annulus between the casing and the formation, which is important to prevent leakage from the wellbore into the formation and prevents potential pollution of the groundwater and possible pollution of the air and the environment at the surface. When production from a well is completed, the wellbore is sealed with a cement plug inside the casing close to the formation where the production was collected, then heavy mud is filled up to about 5-6 ft below the surface. The Wellhead is removed, and the casing is cut and welded to prevent leakage in the future. It's desirable that the cementing last “forever”, or as long as possible.

Different classes of cement that are used for well construction are all a form of Portland cement. The name Portland was taken from an island located in the south of Weymouth, England with a limestone mine that was used for the production of Portland cement. To produce Portland cement, limestone and clay are pulverized and roasted at 2600 to 3000°F. The resulting material is called clinker cement. Oxides of Ca, Al, Si, and Fe react at high temperatures to form balls of cement “clinker”. A Portland clinker storage facility is shown in Figure 2.2.

Figure 2.2 Portland clinker storage[14]

After the roasting, the clinker cement is ground to a size specified by the grade of the cement. The final size of the cement particles and the composition of cement have a direct relationship with how much water is required to make a slurry without producing an excess of water at the top of the cement as the cement hardens. Figure 2.3 shows the manufacturing process of Portland cement.

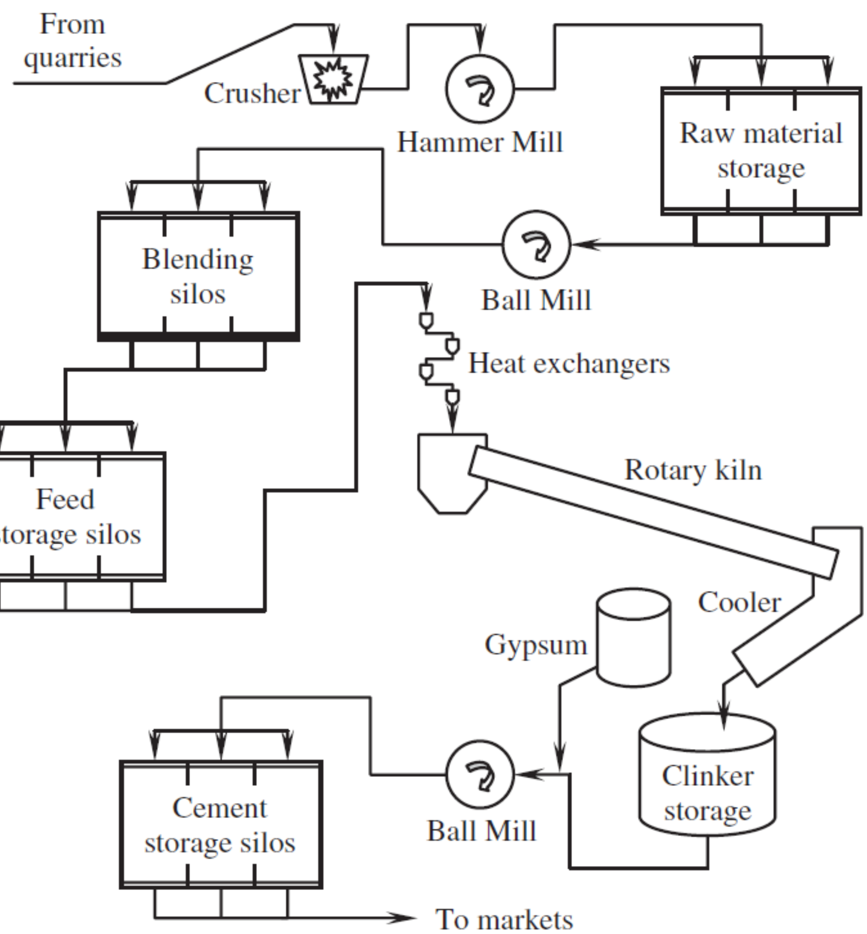

Figure 2.3 Manufacture of Portland cement[15]

By changing the proportion of raw material, the final product will be different in composition and properties. The principal components of Portland cement include tri-calcium silicate (C_3S), dicalcium silicate (C_2S), tetracalcium aluminoferrite (C_4AF), and tricalcium aluminate (C_3A). Different cement classes contain different amounts of these chemicals. The American Petroleum

Institute (API) Specification 10A classifies API cement into 8 classes including A, B, C, D, E, F, G, and H. Table 2.1 shows the typical composition of API cement

Table 2.1 Typical composition of API cement[8]

Typical potential phase composition (wt. %)				
API Class	C_3S	$\beta - C_2S$	C_3A	C_4AF
A	45	27	11	8
B	44	31	5	13
C	53	19	11	9
D	28	49	4	12
E	38	43	4	9
G	50	30	5	12
H	50	30	5	12

The hydration of Portland cement is a complex dissolution and precipitation process in which, the various hydration reactions (regarding C_3S, C_2S, C_3A, C_4AF) proceed simultaneously at different rates. A typical schematic thermogram of Portland cement hydration is shown in Figure. 2.4.

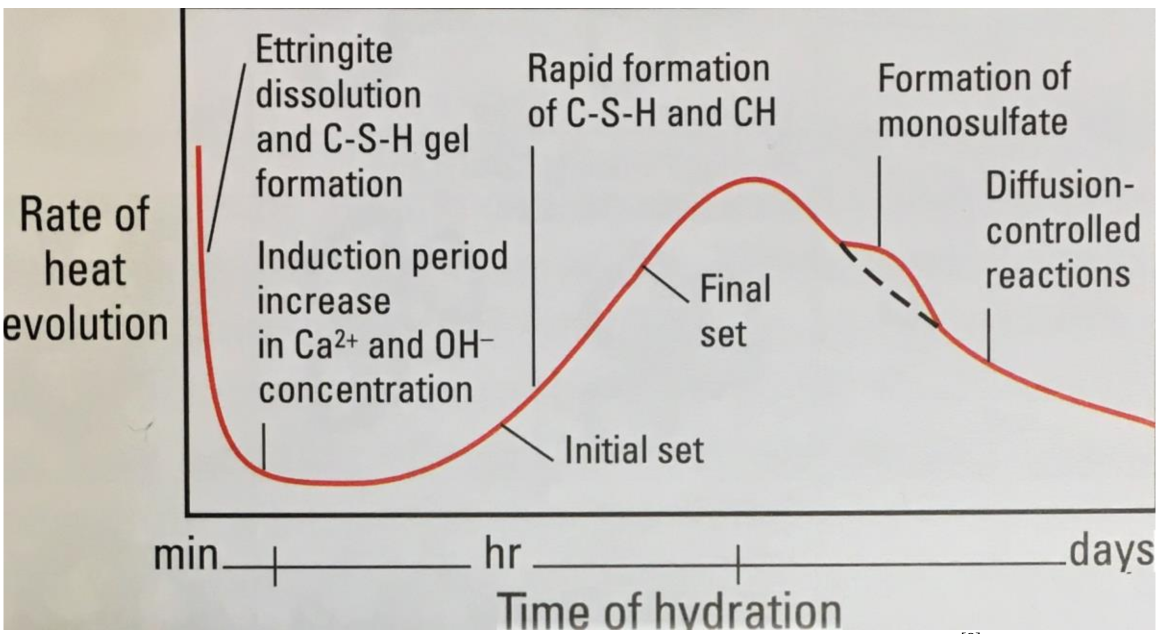

Figure 2.4 A typical schematic thermogram of Portland cement hydration[8]

Table 2.2 shows the API standard cement classes and their application.

Table 2.2 API standard cement classes & their application[8]

API cement class	Application
A	Surface to 6000 ft depth, when special properties are not required
B	Surface to 6000 ft depth, when conditions require moderate to high sulfate resistance
C	Surface to 6000 ft, when conditions require high early strength
D	6000 ft to 10000 ft, under conditions of high temperature and pressure
E	10000 ft to 14000 ft, under the condition of high temperature and pressure
F	10000 ft to 16000 ft, under the condition of extremely high temperature and pressure
G	Surface to 8000+ ft as basic cement. Can be used with retarders or accelerators to cover a wide range of well depth and temperature
H	Surface to 8000+ ft as basic cement. Can be used with retarders or accelerators to cover a wide range of well depth and temperature

Different groups of cement additives can be used in cement to control or achieve the desired cement property. Cement additives can be categorized into the following groups: density control, setting time control, lost circulation, filtration control, viscosity control, and special additives. Additives are dry blended with cement at the service company yard or dispersed in mixing water at the rig site. Table 2.3 shows the cement additives and examples of the additives.

Table 2.3 Cement additives and examples of the additives[8]

Cement Additives	Function	Example
Density control	Weighing agents	Barite, Hematite,
	Extenders	Bentonite, Pozzolan
Setting time control	Accelerators	Calcium chloride, Sodium chloride
	Retarders	Boric acid, HEC
Lost circulation	Mitigate lost circulation	Cellophane, Gilsonite, walnut shells
Filtration control	Mitigate cement filtration	CMHEC, HEC
Viscosity control	Dispersant	Calcium lignosulphonate
Special additives	Antifoam, …	Polypropylene glycol, …

Nano-size particles are defined in size from approximately 1nm to100 nm (1 nm = $1x10^{-9}$ m). The NPs in the oilfield cementing are industrially made. Figure 2.5 shows the comparison of the length scale.

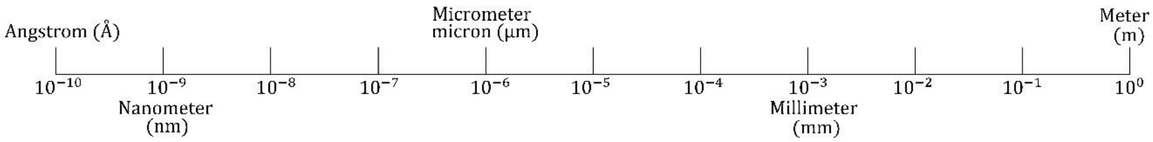

Figure 2.5 Comparison of length scale (Logarithmic scale from 1 meter to 1 Angstrom)

2.2 Literature review

Robinson et al., (1939) published the well cementing and cement test procedures that were the foundation for the "API code for testing cement used in wells" published in 1948. Since that time, many researchers have studied cement additives and practical procedures for testing cement. Most of the technological advancements related to cement additives have focused on improving only one property. Literature related to each property of cement were separated to add more clarity in the sections below.

2.2.1 Thickening time

Thickening time is a measurement of the time during which a cement slurry remains in a fluid state and is capable of being pumped. Thickening time is a function of both temperature and pressure, and these parameters must be estimated for each cement job before additives are selected. Thickening time is assessed under simulated downhole conditions using a consistometer that plots the consistency of a slurry versus time at the anticipated temperature and pressure conditions. The thickening time was ended when the slurry reached 80 Bc for all the tests done in this study. The tests in this study are done using the pressure of 3000 psi and temperature of 160°F for heavy cement, and the pressure of 3000 psi and the temperature of 100°F for light cement, which is considered to be the downhole condition of the wellbore.

Bermudez, M. (2007) studied how sugar as an additive in cement will act as a retarder and how it affected the compressive strength with different concentrations of sugar. The tests were done with Lafarge Type I cement and common granulated table sugar at the temperature of 80°F and the pressure of 200 psi. The result shows that sugar acts as a retarder at low concentrations; while it becomes an accelerator at higher concentrations. It is noticeable that using sugar as an accelerator will shorten the cement thickening time and decreases the cement compressive strength significantly, indicating that hydration did not occur. Figure 2.6 shows the effect of sugar concentration on the cement thickening time.

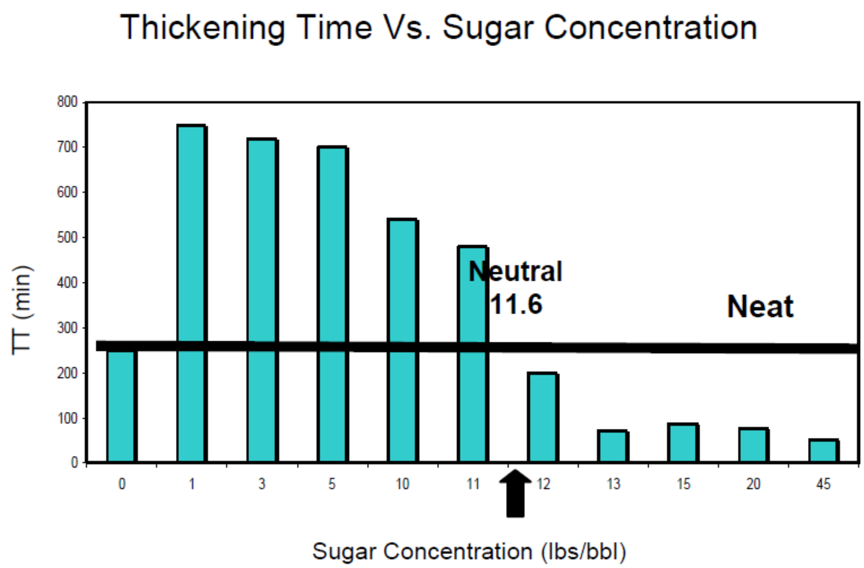

Figure 2.6 Cement thickening time vs sugar concentrations[4]

Umeokafor and Joel, (2010) investigated the cement thickening time with different retarder concentrations. They highlighted the importance of sealing the annulus to obtain zonal isolation and having enough time to place the cement in the well and the importance of having a cement that withstands different operations like stimulation, perforation, production, and intervention during the life of the well. They developed a model equation to predict the thickening time at different concentrations and temperatures for the Dyckerhof Retarded Cement (Retarder/Intensifier). Equation 2.1 shows the model equation developed, and Figure 2.7 shows the actual thickening time versus predicted thickening time.

$$Y = 32.53882954 - 0.15380761X_1 + 12.10530547X_2 + 29.80930457X_3 \qquad 2.1$$

Where:

Y = Thickening time (hr)

X_1 = BHCT (°F)

X_2 = Retarder consentration (%BWOC)

X_3 = Intensifier concentration (%BWOC)

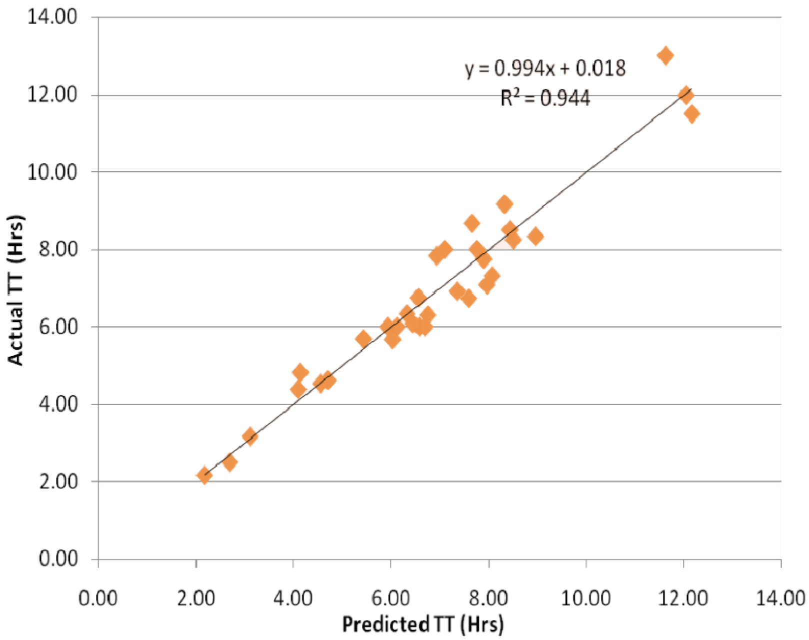

Figure 2.7 Actual thickening time vs. predicted thickening time[11]

Kelessidis et al., (2014) used two additives (latex and micronized silica) in Portland cement class G and studied various properties of the cement including rheology, fluid loss, thickening time, and compressive strength. The control slurry contained 43.8% water BWOC, while the test slurry contained only 31.3% water BWOC. The tests were completed at room temperature and pressure as well as at a higher temperature and pressure. They discovered that adding latex in combination with micronized silica to cement class G will increase the thickening time. Unfortunately, the effect of the addition of each additive as well as the water content was not investigated separately.

Salehi et al., (2016) studied Class F fly ash geopolymer as a replacement to Portland cement class H. They tested the fly ash sheathes for compressive strength, shear bond strength, and durability, as well as the slurry thickening time, and then compared these results to the results of the control sample. The tests were conducted at different temperatures and curing times. They concluded that

The Fly Ash geopolymer increased the strength and that the temperature was a crucial factor for the thickening time. They added a 2% poly carboxymethyl superplasticizer and retarders to all the geopolymer mixtures for temperatures above 175ºF to increase the thickening time. The authors concluded that Class F fly ash geopolymer mixture could replace Portland cement class H specifically for primary cementing in addition to cement plugs for well abandonment. The authors suggested that fly ash can be produced cheaper and has less impact on the environment.

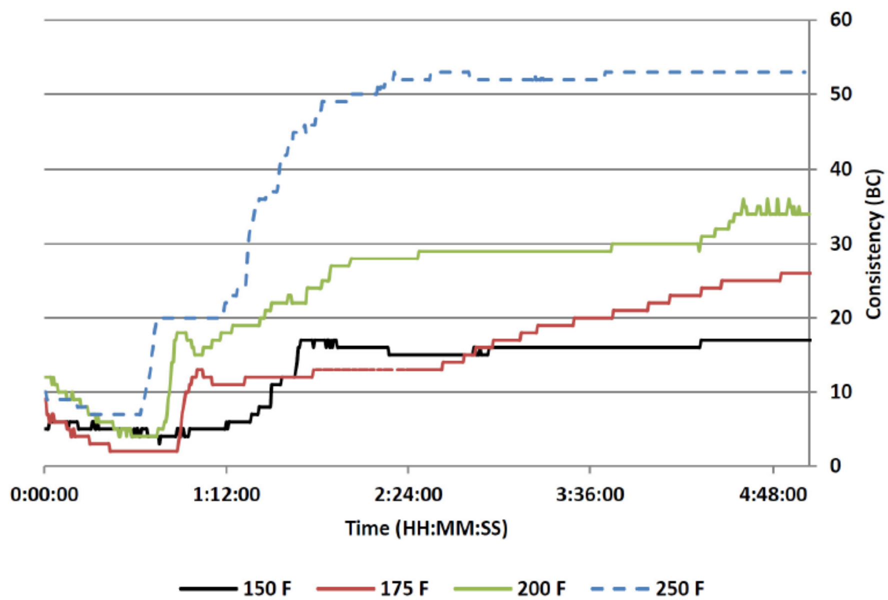

Figure 2.8 Thickening time for Geopolymer slurries at different temperatures[10]

Atashnezhad et al (2017) investigated the effect on fluid loss when adding barite NPs in different concentrations. barite NPs replaced normal barite in the slurry which resulted in a significant reduction of the fluid loss (about 50% decrease in the fluid loss for 3% weight of barite replaced with barite NPs). The barite NPs influence fluid loss and also reduced the thickening time. Figure 2.9 shows the fluid loss effect on thickening time.

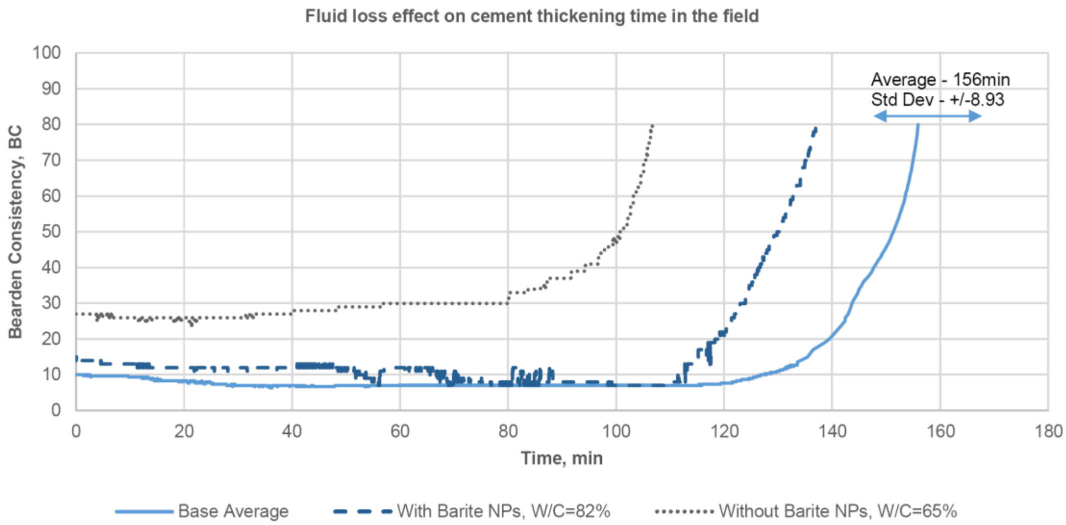

Figure 2.9 The cement thickening time for two different cement fluid loss[3]

Deshpande and Patil (2017) used two different NPs in their experimental study on cement: nano-alumina as an accelerator and halloysite as a tensile strength enhancer. Alumina particles used in the study ranged in size from 200 to 400 nm. The halloysite used had a particle size diameter of 30 to 70 nm and a length of 1 to 1.3 microns. They claimed that by using 0.2 gals/sack of Alumina particles in cement slurry at 80°F, it is possible to reduce setting time by 75%. Their results show that using 1.5% Halloysite NPs in cement increased tensile strength up to 141%.

2.2.2 Rheology

Rheology is the science and study of the deformation and flow of matter. The term is also used to indicate the properties of a given fluid, as in mud rheology. Rheology is an extremely important property of drilling muds, drill-in fluids, workover and completion fluids, cement and specialty fluids, and pills. Mud rheology is measured continually while drilling and adjusted with additives or dilution to meet the needs of the operation. In water-based fluids, water quality plays an important role in how additives perform. Temperature affects the behavior and interactions of the water, clay, polymers, and solids in the mud. Downhole pressure and temperature must be taken into account in evaluating the rheology of oil muds.

Ahmed et al. (2018) used conductive carbon nanomaterials (nano-synthetic graphite) to improve cement sealing properties and integrity. They concluded that using 0.5% BWOC nano-synthetic graphite in cement class H slurry increased the apparent viscosity by 23%, however, it reduced the thickening time slightly. They also observed that the addition of 0.5% BWOC nano-synthetic graphite to the cement class H slurry increased the compressive strength of the cement sheath by more than 20% after 1 day 3 hrs. Figure 2.10 shows the apparent viscosity for control and test samples.

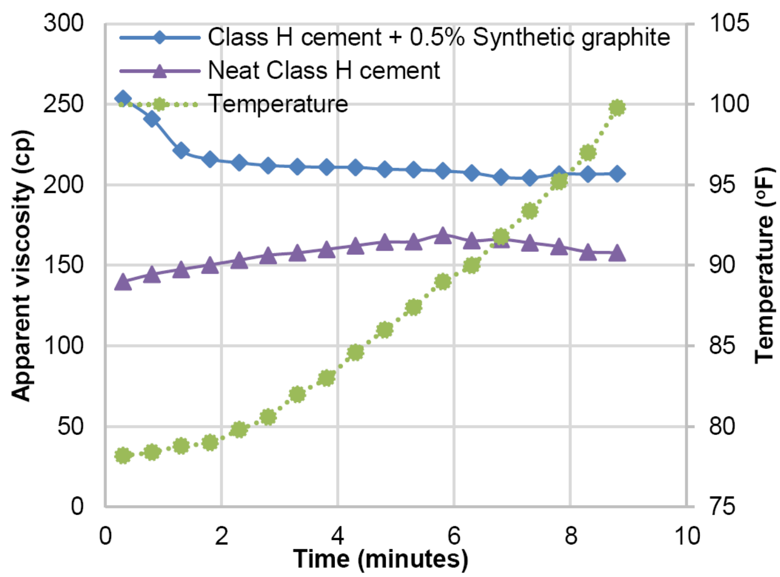

Figure 2.10 Apparent viscosity for control and test samples[1]

Patil et al., (2012) used nano-silica in cement formulations to develop high early strength and to help enhance final compressive strength, and to help control fluid loss. With the correct quantities of nano-silica, it's possible to design cement slurry with low rheology and good mechanical properties and with controllable fluid loss. They concluded that nano-silica improved mechanical properties, especially compressive strength, improved early strength development, and helped the fluid loss control. Nano silica can be used at a wide range of temperatures and can provide flexibility on different design and operation conditions. Nano silica can also easily be combined with other additives to get the best possible and suitable cement slurry with the correct properties.

Vipulanandan et al, (2015) used Iron NPs to reduce electrical resistivity in the cement. They used 0.5% and 1% concentrations of Iron NPs in a 16.5 ppg class H cement slurry. The shear stress at the same shear rates was relatively higher for samples with Iron NPs than the control samples. Figure 2.11 shows the rheology for the control sample and the 1% Fe NPs at 85°F.

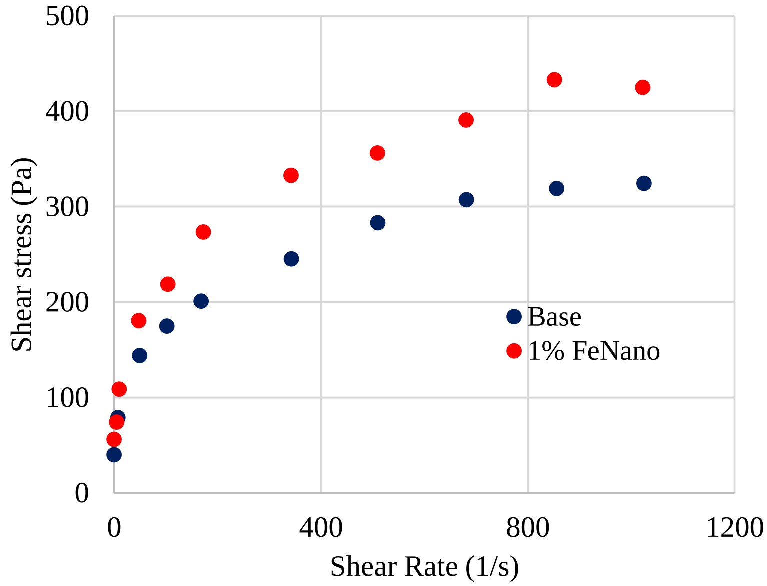

Figure 2.11 Rheology for the control case and test sample containing 1% Fe NPs at 85 °C[12]

Figure 2.12 shows the bulk electrical resistivity development of the cement with various amounts of NanoFe after 7 days.

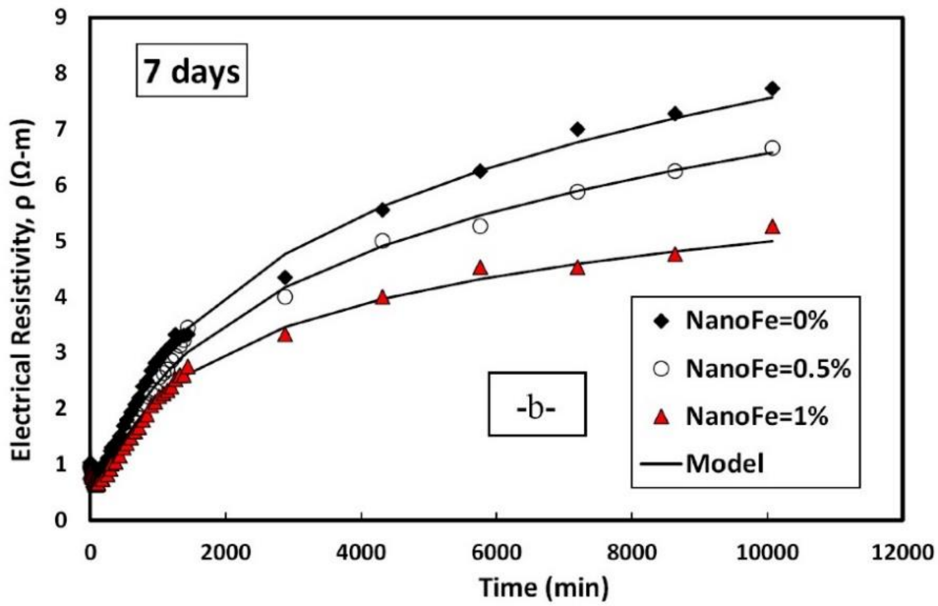

Figure 2.12 Bulk electrical resistivity development of smart cement with various amounts of NanoFe after 7 days[12]

Andersen et al. (2019) introduced polyelectrolyte-complex NPs as a replacement candidate for fluid loss control material such as HEC (Hydroxyethyl Cellulose), CMHEC (carboxymethyl hydroxyethylcellulose), and polyvinyl alcohols that cause undesired high viscosity slurry. They synthesized the polyelectrolyte-complex (PEC) NPs by combining the three polymer components including polyethyleneimine (PEI), polyacrylic acid (PAA), and CMHEC at high mixing speed. The NP size was reported to be 144 nm. Their results show a 20% reduction in plastic viscosity for a given fluid loss. As expected, PEC NPs as well as CMHEC act as a retarder for the cement slurry and increase cement setting time.

Murtaza et al. (2019) investigated the impact of modified nano-clay on the rheology and gel strength with cement class G, and the impact of the cement thickening time. They added small amounts of nano-clay (1 to 2% BWOC) to Portland cement class G and tested the slurry rheological properties at different temperatures. The authors concluded that the addition of nano-clay to the cement class G is a game-changer, especially at high temperatures. Figure 2.13 shows the variation of plastic viscosity with a change in temperature for samples with different concentrations of nano-clay.

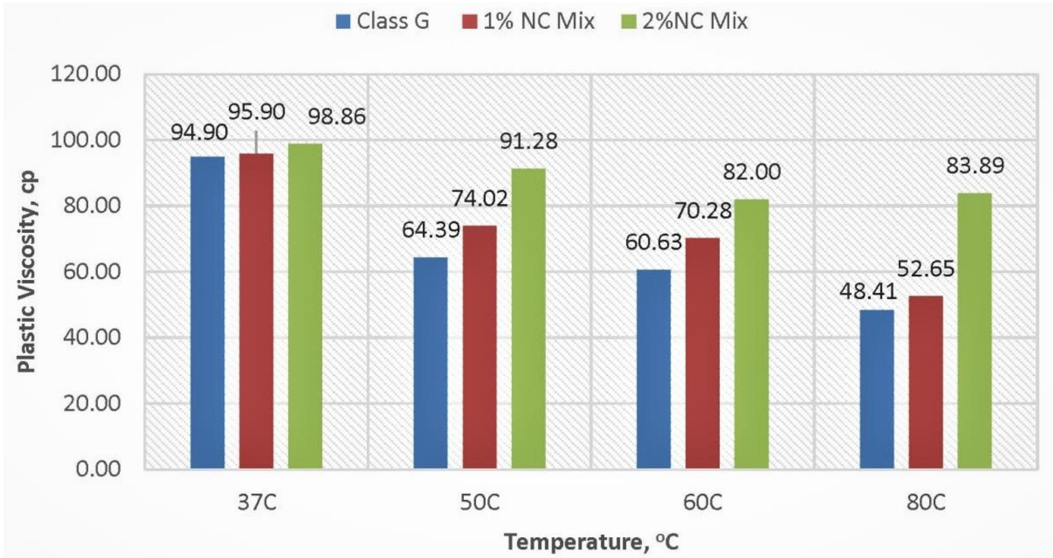

Figure 2.13 Variation of plastic viscosity with change in temperature at different concentration of nano clay[7]

2.2.3 Fluid loss

Fluid loss is the leakage of the liquid phase of drilling fluid, slurry, or treatment fluid containing solid particles into the formation matrix. The resulting buildup of solid material or filter cake may be undesirable, as may the penetration of filtrate through the formation. Fluid-loss additives are used to control the process and avoid potential reservoir damage.

Atashnezhad et al., (2017) performed a study where barite NPs (NPs) reduced the cement fluid loss. Cement fluid loss is important for cement quality. In the oilfield, it is often an advantage to create a cement with the desired density by replacing some of the weight agents with a small number of NPs. The authors tested barite NPs and found that the average fluid loss was decreased by about half by replacing 3% barite with 3% barite NPs. They also did a theoretical field test with a higher area of the filter, due to real field conditions in the wellbore. The fluid loss decrease can prevent many problems such as low cement quality, higher equivalent circulation density (ECD) which fails formation during cementing. However, this field fluid loss also reduced the cement thickening time due to significant fluid loss.

They concluded that barite NPs improved the cement quality by adding small amounts. They also concluded that the water-cement ratio has a strong effect on the cement thickening time. Adding barite NPs indirectly through control of the fluid loss has a strong effect on the cement thickening time. They showed this by performing a theoretical field test with a greater filtration area. The barite NPs can replace other fluid loss agents and weighting agents. The barite NPs can likely plug the filter cake and through that decrease the cement fluid loss. Figure 2.14 shows the Fluid loss for oilfield cement with barite NPs

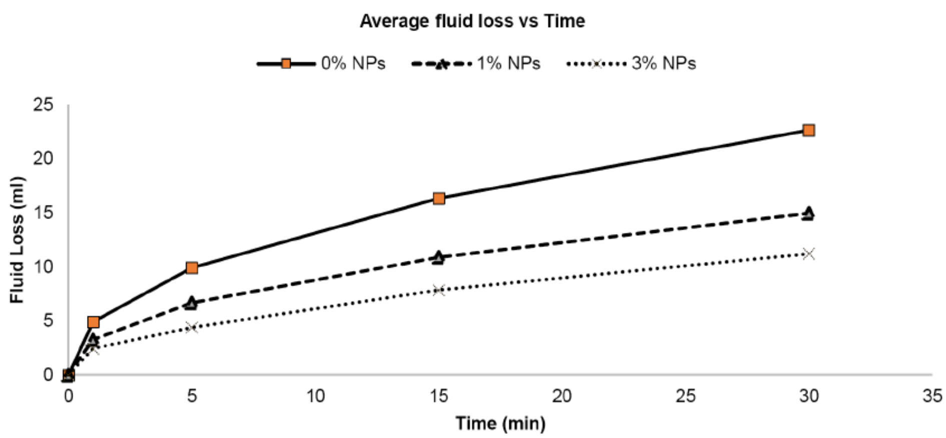

Figure 2.14 Fluid loss for oilfield cement with barite NPs[3]

2.3. Research Gap.

NP as additives is a rather new technology in oil field cements and not much research has been done on this. NP additives improve the cement properties, so this study will look at Portland class H heavy cement with magnetite NP and barite NP additives, and Portland class A light cement with bentonite NP additives. The concentration of NPs is 1%, 3% or 5% (BWOC). This study explores how thickening time, rheology, and fluid loss are affected by using NP additives.

The specific properties of NPs have been studied by many researchers in the last few years, but their effects have not been investigated thoroughly. This thesis will study the effect of magnetite and barite NPs on the thickening time, rheology, and fluid loss on heavy cement, and the effect of bentonite NPs on the thickening time, rheology, and fluid loss on light cement.

2.4. Justification

This research is performed as part of a National Academy of Sciences funded project on the benefits of nano additives in cement and will specifically determine the thickening time by adding NPs for heavy cement in deep wells and light cement in shallow wells and compare the results to a cement control slurry without nano additives. The corresponding effect on rheology and fluid loss with the chosen NP additives and concentrations will also be investigated.

CHAPTER III

METHODOLOGY

This chapter introduces the research materials and describes the research methodology. The purpose of this chapter is to introduce the experimental analyses that typically are required before conducting an actual cementing job. For example, setting time, rheology, and fluid loss are among the cement tests that have to be done before the actual cementing job. The materials, and the selected cement formulations used in this study, and the required apparatuses and procedures are described in the next sections.

3.1. Materials

The main materials for this research were two types of Portland cement class A and class H provided by Halliburton. The additional additives used are listed in table 3.1.

Table 3.1 Materials used in this research

Component	Manufacturer	Purity
Hydroxyethyl Cellulose (HEC)	Eisen Golden Laboratories	83% - 95%
Boric acid	Macron Fine Chemicals	Lab grade
Bentonite	Halliburton	Commercial grade
Bentonite NPs (<80Nm)	Nanoshel LLC	99%
Barite (<38 µm)	Halliburton	Commercial grade
Barite NP (<400 Nm)s	American Elements	99%
Magnetite NPs (50-100 Nm)	Alfa Aesar	97%
Antifoam agent (D-Air 5000)	Halliburton	Commercial grade
Sea salt	Lake Products Company LLC	See table 3.2

The sea salt was artificially made and mixed with 41.953 g of sea salt per liter deionized (DI) water and titrated with 0.1 M NaOH or 0.1 M HCl respectively to get a pH of 8.2. The sea salt composition (ASTM D1141-98) is listed in table 3.2.

Table 3.2 Sea salt (ASTM D1141-98) composition from Manufacturer

Component	**Wt. %**
Sodium Chloride ($NaCl$)	58.49
Magnesium Chloride ($MgCl_2$-$6H_2O$)	26.46
Sodium Sulfate (Na_2SO_4)	9.75
Calcium Chloride ($CaCl_2$)	2.765
Potassium Chloride (KCl)	1.645
Sodium Bicarbonate ($NaHCO_3$)	0.477
Potassium Bromide (KBr)	0.238
Boric acid (H_3BO_3)	0.071
Strontium Chloride ($SrCl_2$-$6H_2O$)	0.095
Sodium Fluoride (NaF)	0.007

3.2 Cement slurry formulation

The formulations were created based on experiences gained from previous research projects and field applications. Bubbles can reduce the mechanical strength of cement sheath and increase the porosity of set cement that is not considered a beneficial property in well cementing. Antifoam agent (D-Air 5000) was used to reduce the number and size of bubbles in the cement. Cement slurry needs to keep water available for complete hydration and crystallization. HEC is a typical fluid loss control additive in cement and has been used in both light and heavy cement formulations. Seawater use was recommended by Halliburton and has traditionally been used for mixing with cement powder and making cement slurry. Seawater can improve slurry stability and its metallic salt builds weak but extensive hydroxide structures through the slurry. This structure building substantially reduces free water.

3.2.1 Light cement formulation

Cement slurry for shallow intervals usually is lighter (less density) when compared to the cement slurries designed for deeper intervals. In this case, Portland cement class A was selected to formulate light cement. Bentonite is considered an extender additive in cement, which means bentonite reduces the cement density by increasing its volume. Interestingly, cement thickening time shortens at higher temperatures, considering the exothermic nature of cement hydration reactions. Boric acid was selected as a retarder additive in cement formulation to adjust the cement thickening time. Initially, a test was performed with 160ºF and 3000 psi and compared with the control formulation to investigate how the retarder boric acid influenced the thickening time as shown in Table 3.3.

Table 3.3 Formulation for light cement boric acid test, 160 ºF, and 3000psi

Component Test #	Cement class A (g)	Seawater (g)	HEC (g)	D-air 5000 (g)	Bentonite (g)	Boric acid (g)
Control	550	478.34	2.75	1.28	66	0
Case 1	550	478.34	2.75	1.28	66	2.75
Case 2	550	478.34	2.75	1.28	66	3.3
Case 3	550	478.34	2.75	1.28	66	3.85
Case 4	550	478.34	2.75	1.28	66	5.5

Secondly, a test was performed with 100ºF and 3000 psi with no boric acid as shown in Table 3.4.

Table 3.4 Formulation for Light cement control without boric acid, 100ºF, and 3000 psi

Component Test #	Cement class A (g)	Seawater (g)	HEC (g)	D-air 5000 (g)	Bentonite (g)	Boric acid (g)
Control	550	478.34	2.75	1.28	66	0

Finally, a test was performed with bentonite NPs without boric acid as shown in Table 3.5.

Table 3.5 Formulation for the Light cement with 1-3-5% bentonite NPs

Component Test #	Cement class A (g)	Seawater (g)	HEC (g)	D-air 5000 (g)	Bentonite (g)	Bentonite NPs (g)
Control	550	478.34	2.75	0.83	66	0
1% BWOC bentonite NPs	550	478.34	2.75	0.83	60.5	5.5
3% BWOC bentonite NPs	550	478.34	2.75	0.83	49.5	16.5
5% BWOC bentonite NPs	550	478.34	2.75	0.83	38.5	27.5

3.2.2 Heavy cement formulation

Cement slurry for the deeper intervals usually is heavier than slurries designed for shallower intervals. The test was done with a temperature of 160ºF and pressure of 3000 psi. Initially, a test was performed to find a proper thickening time for the cement slurry as shown in the formulation in Table 3.6

Table 3.6 Formulation for Heavy Cement, control case boric acid test, 160ºF, and 3000 psi

Component Test #	Cement class H (g)	Seawater (g)	HEC (g)	D-air 5000 (g)	Barite (g)	Boric acid (g)
Control	850	362.68	4.25	1.28	100	0
Case 1	850	362.68	4.25	1.28	100	2.55
Case 2	850	362.68	4.25	1.28	100	3.40
Case 3	850	362.68	4.25	1.28	100	4.25

Secondly, a test was performed to investigate how the barite NPs in different concentrations reacted to the thickening time. The formulation is shown in Table 3.7.

Table 3.7 The Formulation for heavy cement with barite NPs and no boric acid

Component Test #	Cement class H (g)	Seawater (g)	HEC (g)	D-air 5000 (g)	Barite (g)	Barite NPs (g)
Control	850	362.68	4.25	1.28	100	0
1% BWOC barite NPs	850	362.68	4.25	1.28	91.5	8.5
3% BWOC barite NPs	850	362.68	4.25	1.28	74.5	25.5
5% BWOC barite NPs	850	362.68	4.25	1.28	57.5	42.5

This test was repeated for magnetite NPs and no boric acid. The formulation is shown in Table 3.8.

Table 3.8 Formulation for heavy cement with magnetite NPs and no boric acid

Component Test #	Cement class H (g)	Seawater (g)	HEC (g)	D-air 5000 (g)	Barite (g)	Magnetite NP (g)
Control Case	850	362.68	4.25	1.28	100	0
1% BWOC magnetite NPs	850	362.68	4.25	1.28	91.5	8.5
3% BWOC magnetite NPs	850	362.68	4.25	1.28	74.5	25.5
5% BWOC magnetite NPs	850	362.68	4.25	1.28	57.5	42.5

A test was performed to see how the amount of seawater affected the thickening time as shown in Table 3.9.

Table 3.9 Formulation for heavy cement with 1-3-5% barite NP's with higher amount of seawater

Component Test #	Cement Class H (g)	Seawater (g)	HEC (g)	D-air 5000 (g)	Barite (g)	Barite NP (g)	Boric acid (g)
Control Case	850	362.68	4.25	1.28	100	0	3.4
1% BWOC barite NPs	850	375.78	4.25	1.28	91.5	8.5	3.4
3% BWOC barite NPs	850	401.99	4.25	1.28	74.5	25.5	3.4
5% BWOC barite NPs	850	428.19	4.25	1.28	57.5	42.5	3.4

Finally, a formulation for the heavy cement was created showing both barite NPs and magnetite NPs as shown in Table 3.10.

Table 3.10 Formulation for Heavy cement with 1-3-5% barite NPs and magnetite NPs

Component Test #	Cement Class H (g)	Seawater (g)	HEC (g)	D-air 5000 (g)	Barite (g)	Barite NPs (g)	Magnetite NPs (g)	Boric acid (g)
Control Case	850	362.68	4.25	1.28	100	0	0	3.4
1% BWOC barite NP	850	362.68	4.25	1.28	91.5	8.5		3.4
3% BWOC barite NP	850	362.68	4.25	1.28	74.5	25.5		3.4
5% BWOC barite NP	850	362.68	4.25	1.28	57.5	42.5		3.4
1% BWOC magn NP	850	362.68	4.25	1.28	91.5		8.5	3.4
3% BWOC magn NP	850	362.68	4.25	1.28	74.5		25.5	3.4
5% BWOC magn NP	850	362.68	4.25	1.28	57.5		42.5	3.4

3.3. Cement mixing procedure

The API standard 10A procedure mixing is 15 seconds at 4000 RPM ±200 RPM with the fluid components already in place and the solids added in this time frame; followed by 12000 RPM ±500 RPM for an additional 35 seconds. This mixing procedure was followed for all the cement slurries made and reported in this thesis. The cement mixer is shown in Figure 3.1.

Figure 3.1 OFITE Model 20 cement mixer

3.3. Thickening time

The thickening time is the time the cement is pumpable after the cement mixture has been made. The unit for thickening time is Bc (Bearden units of consistency) which is a dimensionless quantity with no direct conversion to more common units of viscosity.

The Chandler Model 7322 HPHT consistometer was used to determine the thickening time. The thickening times were tested with the final control case and the different NPs to determine the final and desired thickening time. The tests were done with a temperature of 160°F and pressure of 3000 psi for heavy cement, and temperatures of 160°F and 100°F at 3000 psi pressure for light cement. The tests were ended when the consistency reached 80 Bc, and the thickening time was recorded. The apparatus is shown in Figure 3.2

Figure 3.2 The Chandler 7322 Consistometer

3.4. Rheology

The rheology is the cement slurry resistance to flow at different shear rates which describes the flow in pipes and the annular space.

Rheology tests of the cement slurries were modeled with the Bingham Plastic and the Power-law rheological models. These models can approximate the pseudoplastic behavior of the cement slurries. A Fann 35A Viscometer was used together with an OFITE heating cup in the procedure. The rotor dimensions are radius r_1=1.7245 cm for the bob, and r_2= 1.8415 for the rotor, and height 3.8 cm. The rotational speed is selected from 3, 6, 100, 200, 300, and 600 RPM. The purpose of this test is to determine the cement slurry rheology properties. The temperature of the slurry was set to 150°F for heavy cement and 100°F for light cement. The Bingham Plastic and Power-law formulas used are presented as Equations 3.1- 3.7.

$$\tau = 1 * \text{Fann reading} x 1.065 \quad \text{unit (lb/100 ft}^2\text{)} \qquad 3.1$$

Equation 3.1 is the formula used to find the shear stress of slurry from the reading of the FANN 35A.

$$\dot{\gamma} = 1.703 \times \mathrm{N} + 479 \frac{\tau_y}{\mu_p}\left(\frac{3.174}{1.7245^2} - 1\right) \quad \text{unit (1/s)} \qquad 3.2$$

Equation 3.2 is the formula used to find the Bingham Plastic shear rate of the slurry.

$$\mu_p = \frac{300}{\mathrm{N}_2 - \mathrm{N}_1}(\theta_{\mathrm{N2}} - \theta_{\mathrm{N1}}) \quad \text{or} \quad \mu_p = \theta_{600} - \theta_{300} \quad \text{unit (cP)} \qquad 3.3$$

Equation 3.3 is the formula used to find the Bingham Plastic viscosity of the slurry.

$$\tau_y = \theta_{\mathrm{N1}} - \mu_p \frac{\mathrm{N}_1}{300} \quad \text{or} \quad \tau_y = \theta_{300} - \mu_p \quad \text{unit (lb/100 ft}^2\text{)} \qquad 3.4$$

Equation 3.4 is the formula used to find the Bingham plastic yield point of the slurry.

$$\dot{\gamma} = 0.2094\mathrm{N} \frac{\frac{1}{r^{\frac{2}{n}}}}{n\left[\frac{1}{r_1^{\frac{2}{n}}} - \frac{1}{r_2^{\frac{2}{n}}}\right]} \quad \text{unit (1/s)} \qquad 3.5$$

Equation 3.5 is the formula used to find the Power-law shear rate of the slurry.

$$n = \frac{\log\left(\frac{\theta_{N_2}}{\theta_{N_1}}\right)}{\log\left(\frac{N_2}{N_1}\right)} \qquad 3.6$$

Equation 3.6 is the formula used to find the n value for the Power-law model

$$K = \frac{510\ \theta_{300}}{511^n} \quad \text{unit (equivalent cp) unit (dynes-s}^n\text{/cm}^2\text{)} \qquad 3.7$$

Equation 3.7 is the formulas used to find the K value for the Power-law model

The cement was mixed according to the formulations and poured into the heating cup. The heater was turned on and the desired temperature was reached. Next, the cup was moved up to the level over the bob and rotor, and the test started. The rheological properties were measured at each specific temperature. The measurement of temperature was done for both the heating cup and slurry. The tests were running for 1 minute at each speed, then the reading was recorded. The Bingham Plastic and the Power-law rheological models were used to approximate the pseudoplastic behavior of the cement slurries. The Shear Stress was calculated for the different RPMs, 3, 6, 100, 200, 300, 600 RPM. The plastic viscosity and the yield point were calculated for the Bingham plastic model, and n and K-value were calculated for the Power-law model and recorded for each sample. The apparatus with the sample cup is shown in Figure 3.3

Figure 3.3 Fann 35 A The Viscometer with an OFITE heating cup

3.5. Fluid loss

The fluid loss is the leakage of the liquid phase from the cement slurry into the formation matrix. An OFITE HPHT fluid loss tester was used with a quantitative filter paper (2-5 microns) to find the cement slurries' fluid loss for both heavy and light cement. The test cup was mounted with a screen mesh (#170-18) and the standard FANN (2-5 microns) paper filter in the bottom of the test cup, then sealed with rubber O-rings on both sides to prevent leaking and then closed. The cement samples were made and poured into the test cell up to ¼ inch from the top, mesh filter was mounted with rubber O-rings on both sides and then closed. High-pressure nitrogen (N_2) was connected to the top (upstream) and bottom (downstream) of the test cell. All the valves were closed and the heater turned on. Initially, the temperature was measured in the wall of the sample cup, then it was measured in the sample slurry. The sample temperature was allowed to raise to 100°F for light cement or 150°F for heavy cement. After the front pressure (upstream) pressure was raised and adjusted to 600 psi and the back pressure (downstream) was raised and adjusted to 100 psi, the subsequent differential pressure on the slurry is then 500 psi. The test was started and a timer was turned on. Samples of fluid were collected every minute through the test by opening the valve in the bottom of the cell and the accumulated amount of fluid was set for about 10-15 minutes to clear, then it was recorded. The apparatus is shown in Figure 3.4.

Figure 3.4 OFITE HP/HT fluid loss instrument

CHAPTER IV

RESULTS AND DISCUSSIONS

4.1. Testing of cement thickening time

The first phase of testing on the light cement was performed to determine the right concentration of the retarder additive at 160°F. It was decided not to use any retarder for tests being done on light cement at 100°F. The consistency tests on the light cement slurries were done at 3000 psi and temperatures of 100°F and 160°F for the light cement. The thickening time tests for all of the heavy cement formulations were evaluated at 3000 psi and 160°F temperature. Thickening time was determined when cement consistency reached 80 Bc. Figure 4.1 shows the cement after its consistency has reached 80 Bc.

Figure 4.1 The cement after it has reached 80 Bc in the consistometer

4.1.1 Heavy cement, boric acids effect on the thickening time

A set of consistency tests were performed with the heavy control case containing different concentrations of boric acid. The cement slurry formulation was previously shown in Table 3.6. The results are presented in Table 4.1 and Figure 4.2 and 4.3.

Table 4.1 Summarized results for the heavy cement control case. boric acid concentration

Boric acid			
Heavy Cement	Thickening Time		Change
160ºF, 3000psi	(HH:MM)	Min	from CC
CC, 0.0% boric acid	1:07	67	
CC, 0.3% boric acid	2:09	129	93%
CC, 0.4% boric acid	3:22	202	201%
CC, 0.5% boric acid	4:34	274	309%

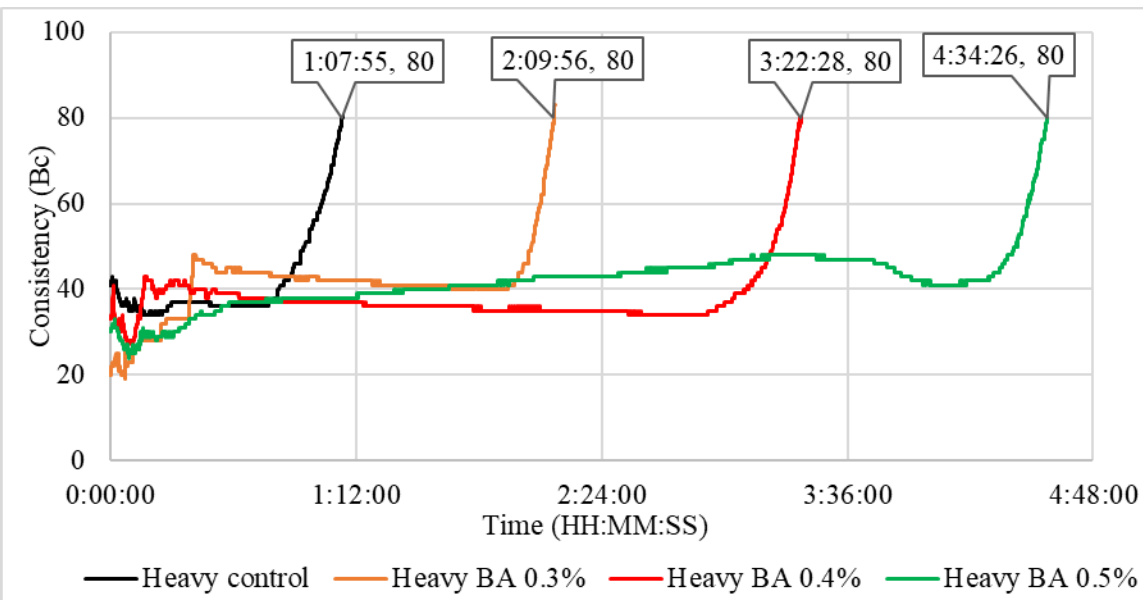

Figure 4.2 Heavy cement control case boric acid tests

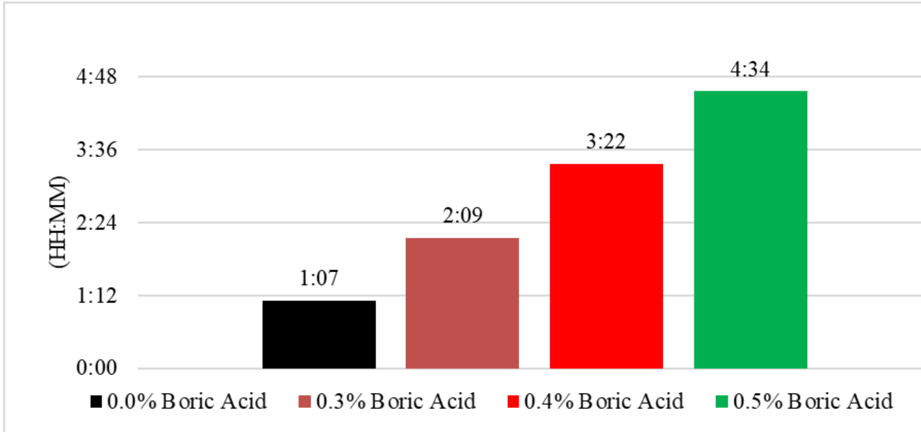

Figure 4.3 Heavy cement control case boric acid tests

The 0.4% BWOC boric acid gives a thickening time of 202 minutes, which is desired for heavy cement in a deep well. Figure 4.4 shows the incremental percentage of thickening time with increasing boric acid concentrations compared to the control case.

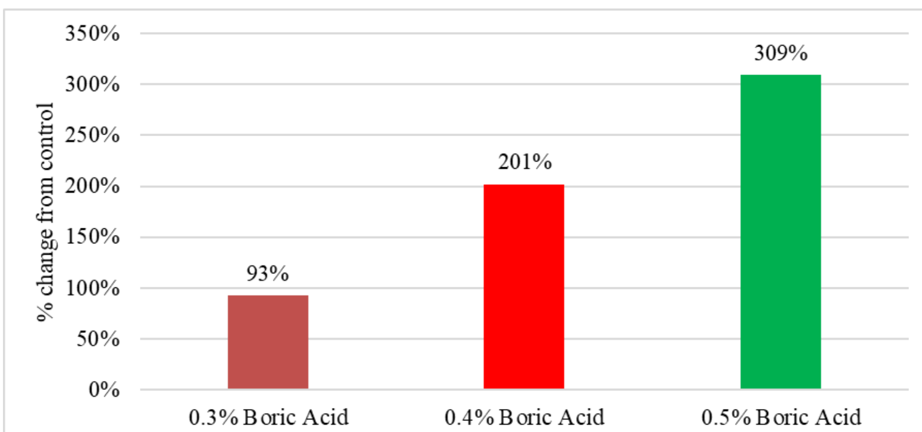

Figure 4.4 Heavy cement control case boric acid tests. The thickening time change

4.1.2 Heavy cement, barite NPs effect on the thickening time (no boric acid)

A set of tests were performed using heavy cement containing barite NPs without boric acid. The cement slurry formulation has been previously shown in Table 3.7 and the results are presented in Table 4.2, Figures 4.5 and 4.6

Table 4.2 Summarized results for heavy cement with barite NPs without boric acid

No boric acid			
Heavy Cement	Thickening Time		Change
160 ºF, 3000psi	(HH:MM)	Min	from CC
Control case	1:07	67	
1% barite NPs	1:10	70	4%
3% barite NPs	1:14	74	10%
5% barite NPs	1:19	79	18%

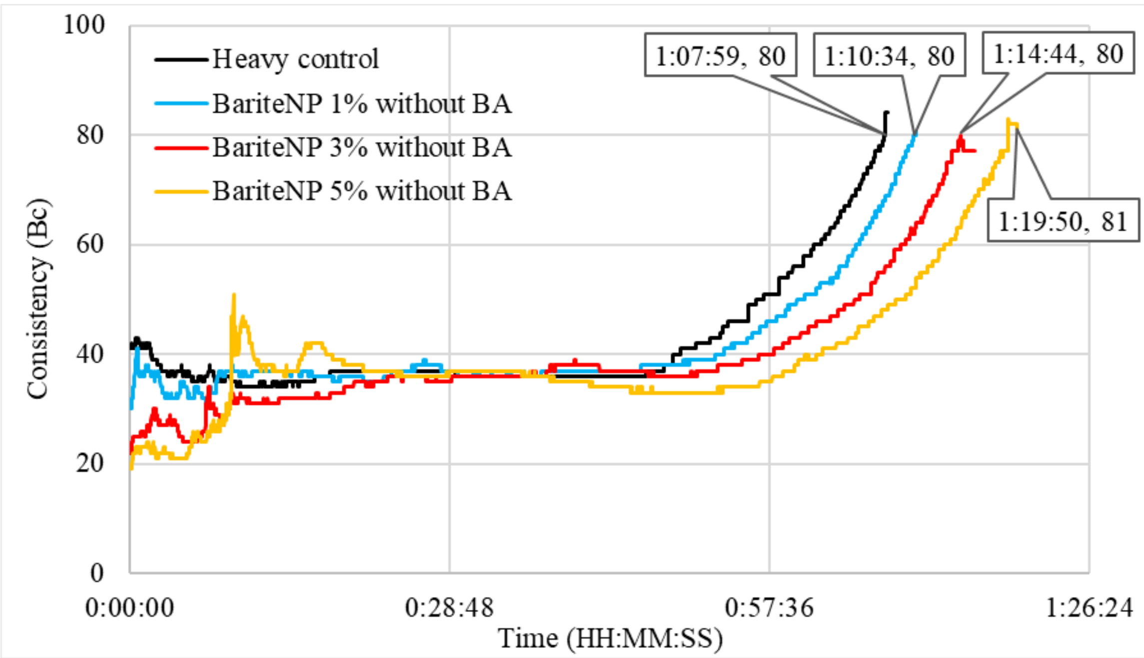

Figure 4.5 Heavy cement with barite NPs without boric acid

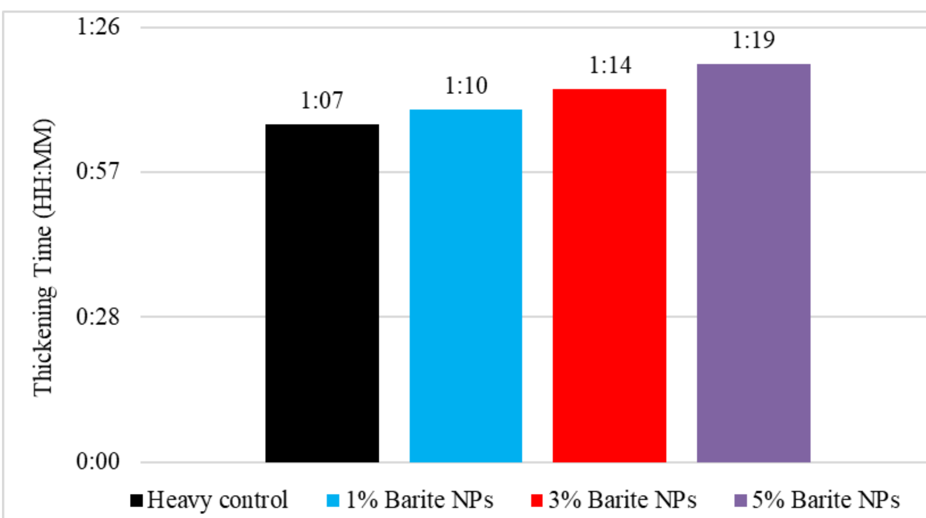

Figure 4.6 Heavy cement thickening time with barite NPs and no boric acid

These tests were performed to see how the barite NPs concentration affects the thickening time. Three different concentrations of barite NPs (1%, 3%, and 5% BWOC) in heavy cement were tested without boric acid and the results suggest a little increase in the thickening time with a higher concentration of barite NPs. The 5% barite NPs had the highest change with an 18% higher thickening time than the control case. The difference in thickening time from the control case is presented in Figure 4.7

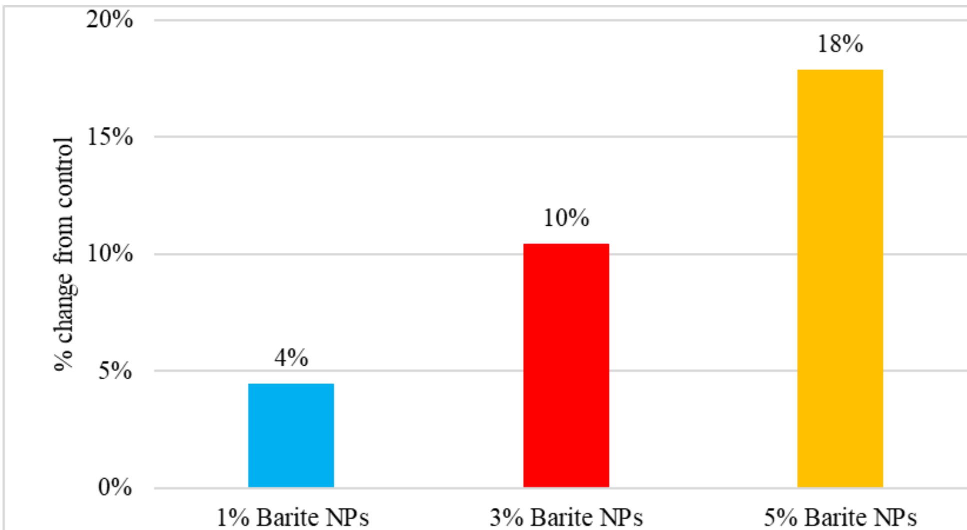

Figure 4.7 Heavy cement with barite NPs and no boric acid showing the thickening time change from the control case

4.1.3. Heavy cement with barite NPs and higher amount of seawater

One consistency test was performed with the heavy control case and barite NPs with an increasing amount of seawater, and with the formulation previously shown in Table 3.9, and the results are shown in Table 4.3 and Figures 4.8 and 4.9.

Table 4.3 Summarized results for the heavy control case with barite NPs with a higher amount of seawater in the NPs than the control case.

0.4% BWOC boric acid			
Heavy Cement	Thickening time		Change
160ºF, 3000psi	(HH:MM)	Min	from CC
Control Case	3:22	202	
1% barite NPs	5:11	311	54%
3% barite NPs	4:08	248	23%
5% barite NPs	4:13	253	25%

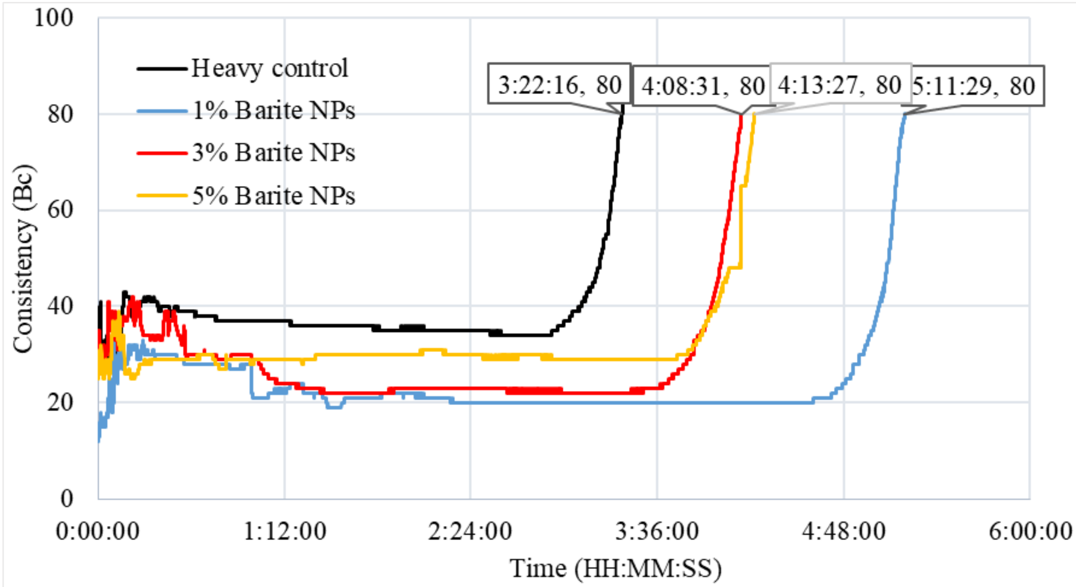

Figure 4.8 Heavy control case with barite NPs with a higher amount of seawater in the NPs than the control case.

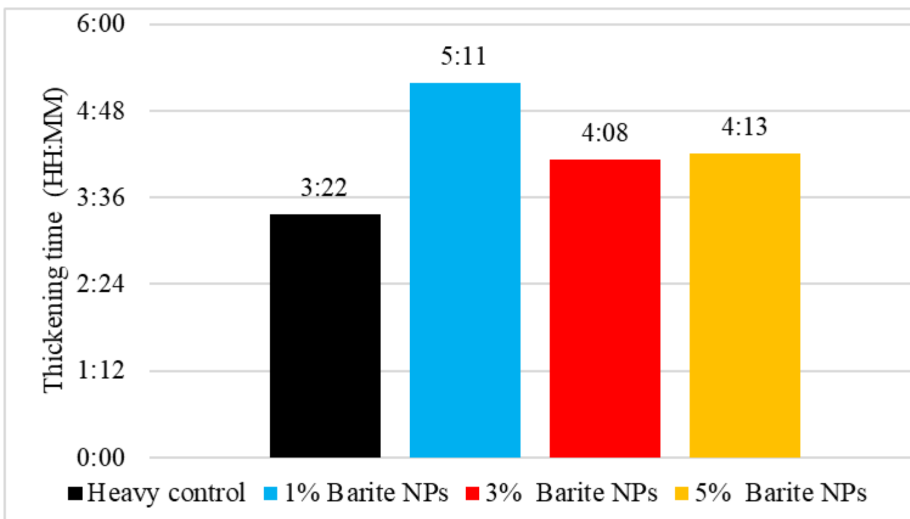

Figure 4.9 Heavy control case with barite NPs with a higher amount of seawater in the NPs than the control case

The tests were done with a higher amount of seawater in the barite NPs compared to the control case. The thickening time was higher for all the test samples compared to the control case. The test sample containing 1% BWOC barite NPs had a 54% higher thickening time than the control case, while the 3% and 5% BWOC barite NPs had a 23%, and a 25 % higher thickening time than the control case. This series of tests confirmed that an increasing amount of seawater will result in a longer thickening time. The difference from the control case is shown in Figure 4.10

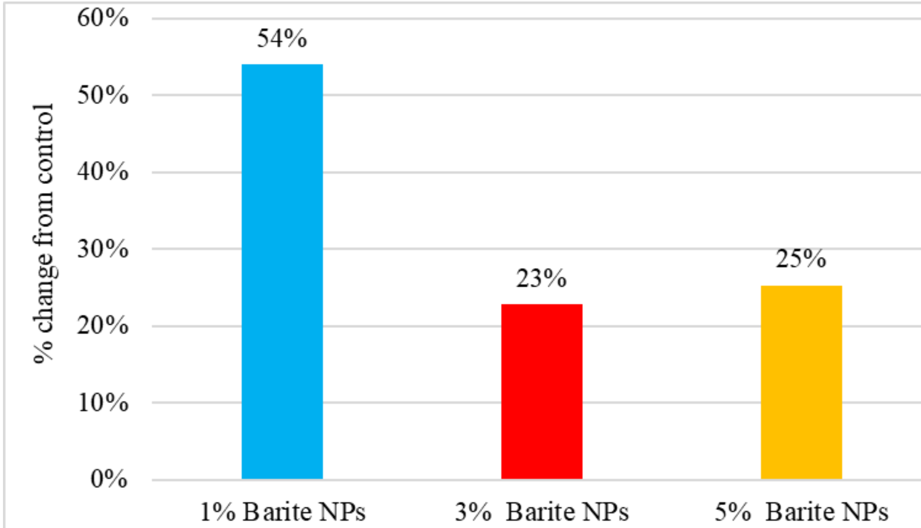

Figure 4.10 Heavy cement with barite NPs changes from the control case, with a higher amount of seawater in the NPs than the control case.

4.1.4 Heavy cement with barite NPs and the same amount of seawater as in the control case

One consistency test was performed with the heavy cement control case and barite NPs with the same amount of seawater as in the control case with the formulation previously shown in Table 3.10, and the results are shown in Table 4.4 and Figures 4.11 and 4.12.

Table 4.4 Summarized result for heavy cement with barite NPs, but now with the same amount of seawater in the NPs as in the control case.

Heavy Cement	Thickening time		Change
160˚F, 3000psi	(HH:MM)	Min	from CC
Control Case	4:01	241	
1% barite NPs	5:20	298	23.7%
3% barite NPs	5:11	311	29.0%
5% barite NPs	4:09	249	3.3%

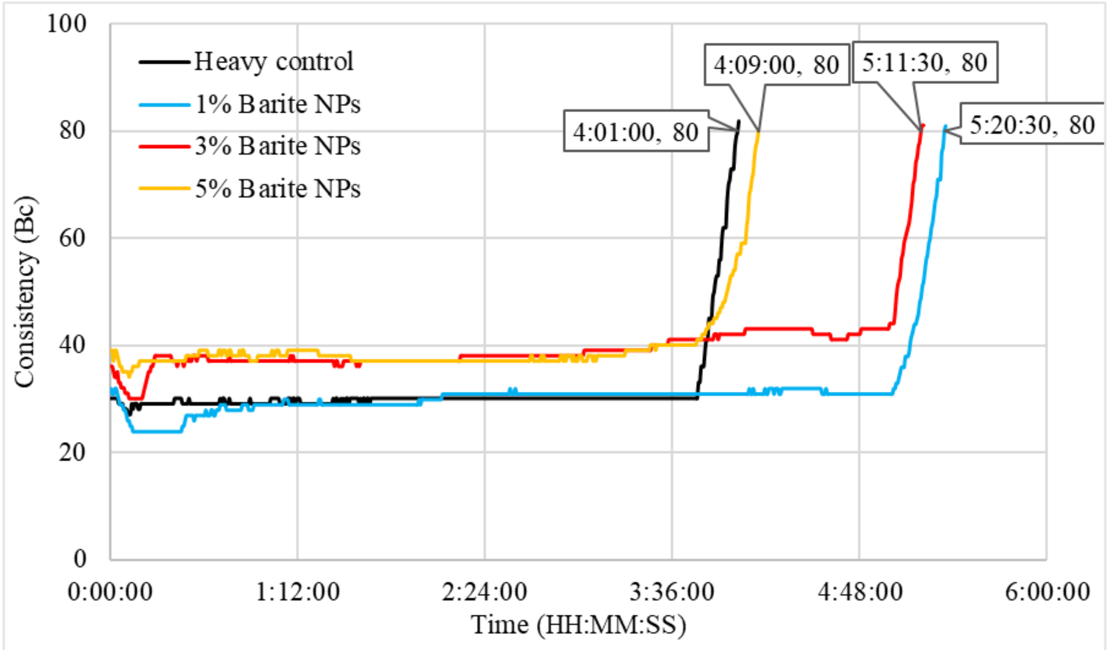

Figure 4.11 Heavy control case with barite NPs with the same amount of seawater in the NPs as in the control case.

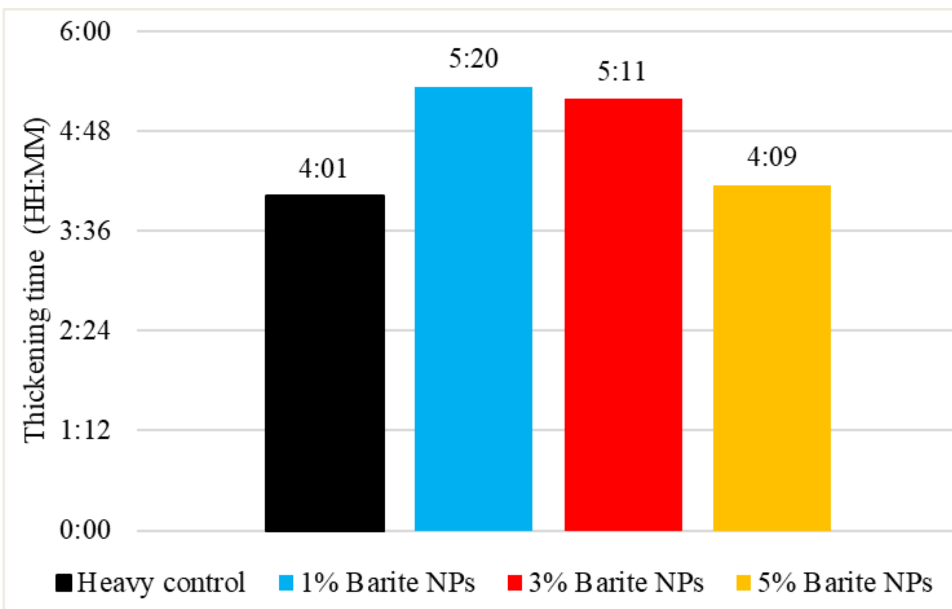

Figure 4.12. Heavy control case with barite NPs, but now with the same amount of seawater in the NPs as in the final control case.

The thickening times were higher than the control case for all the barite NPs, and it was highest for 3% barite NPs (29% higher than the control case). The difference from the control case is shown in Figure 4.13.

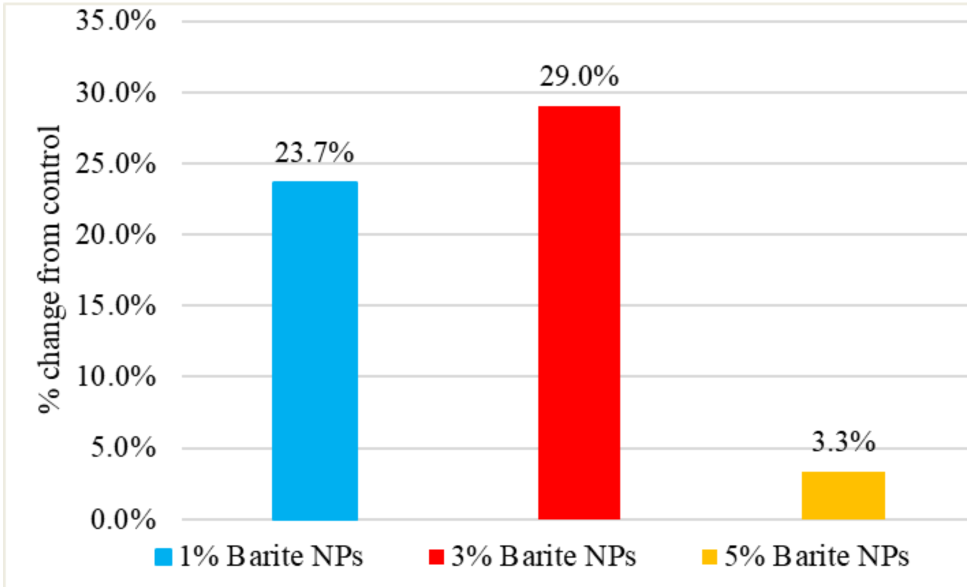

Figure 4.13 Heavy cement with barite NPs change from the control case, but now with the same amount of seawater in the NPs as in the final control case.

4.1.5 Heavy cement, magnetite NPs effect on the thickening time (no boric acid)

One consistency test was performed with the heavy control case and magnetite NPs without boric acid, as previously shown in Table 3.8. The results are shown in Table 4.5 and Figures 4.14 and 4.15.

Table 4.5 Summarized result for heavy cement with magnetite NPs, without boric acid

No boric acid			
Heavy Cement	Thickening Time		Change
160°F, 3000 psi	(HH:MM)	Min	from CC
Control case	1:07	67	
1% magnetite NPs	1:06	66	-1%
3% magnetite NPs	1:01	61	-9%
5% magnetite NPs	0:53	53	-21%

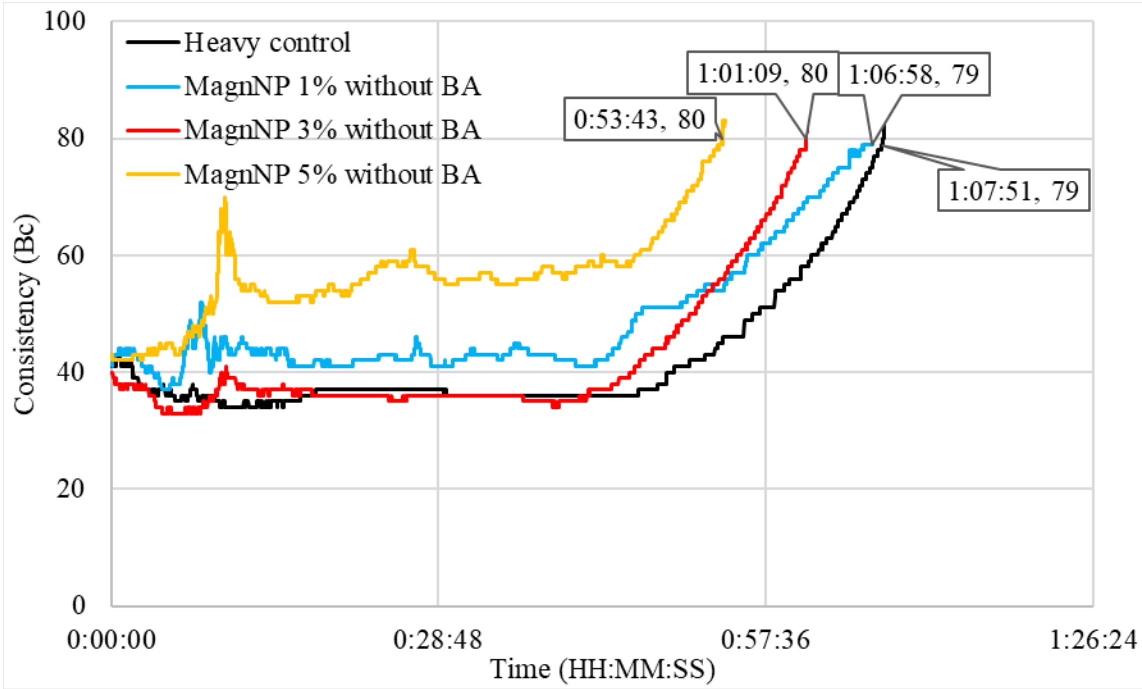

Figure 4.14 Heavy cement with magnetite NPs without boric acid

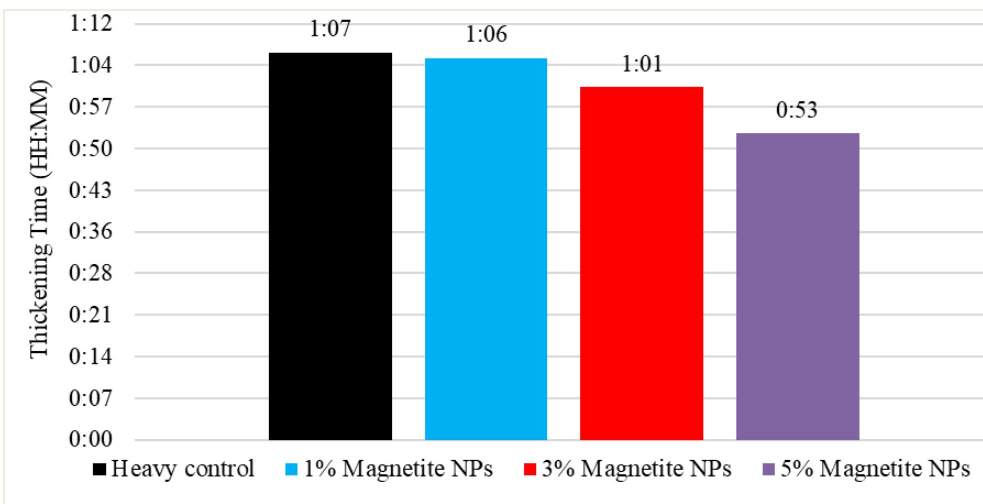

Figure 4.15 Heavy cement thickening time with magnetite NPs without boric acid

All the magnetite NPs have a little lower thickening time than the control case. The lowest was for 5% magnetite NPs with 21% lower thickening time than the control case. This result indicates that magnetite NPs reduce the cement thickening time. The change from the control case is shown in Figure 4.16.

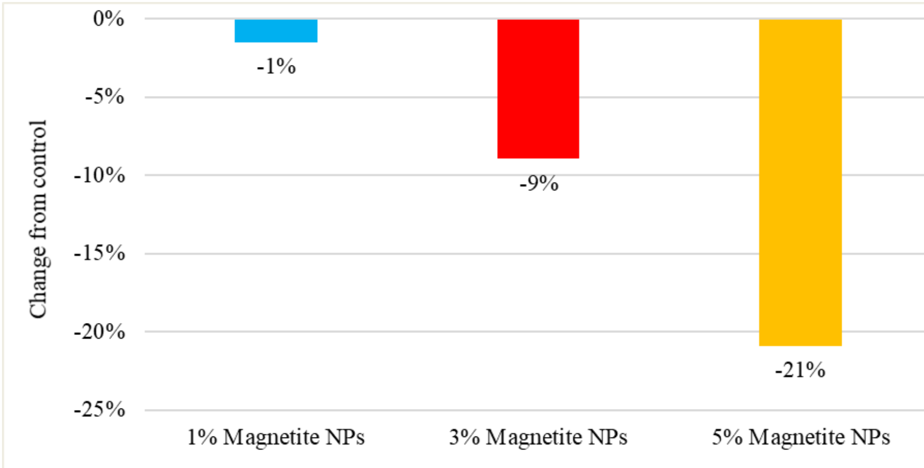

Figure 4.16 Heavy cement thickening time change from the control case, with magnetite NPs without boric acid

4.1.6 Heavy cement with magnetite NPs

One consistency test was performed with the heavy control case and magnetite NPs. The formulation is previously shown in Table 3.10, and the results are shown in Table 4.6 and Figures 4.17 and 4.18.

Table 4.6 Summarized result for heavy cement with magnetite NPs

Heavy Cement	Thickening time		Change
160˚F, 3000 psi	(HH:MM)	Min	from CC
Control Case	4:01	241	
1% magnetite NPs	4:37	277	14.9%
3% magnetite NPs	4:13	253	5.0%
5% magnetite NPs	3:29	209	-13.3%

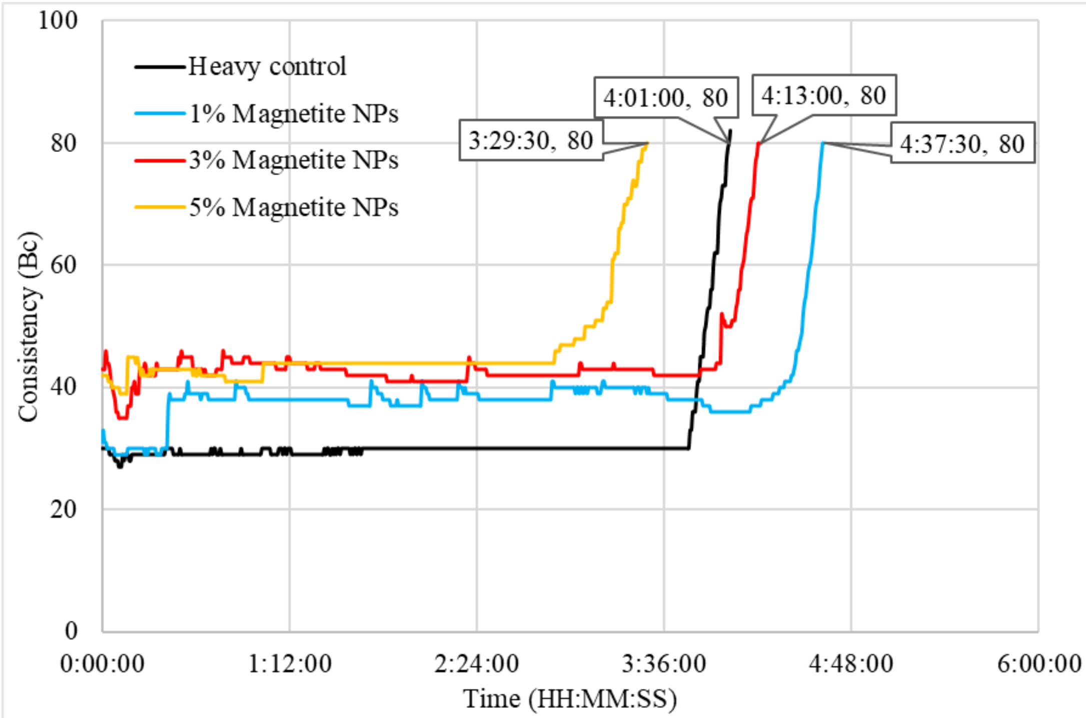

Figure 4.17 Heavy magnetite NPs with the same amount of seawater in the NPs as in the control case.

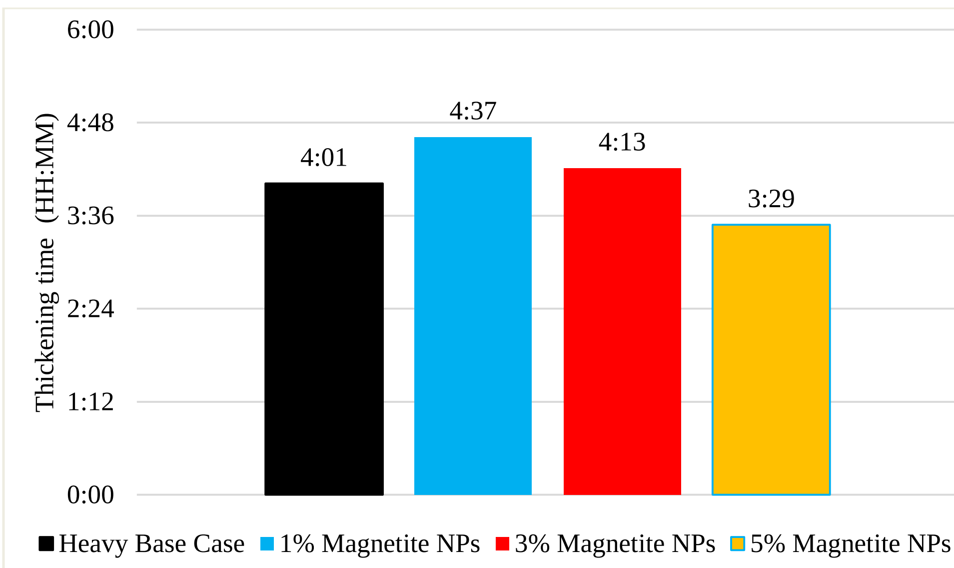

Figure 4.18 Heavy magnetite NPs with the same amount of seawater in the NPs as in the control case.

The thickening time was 14.9% higher than the control case for 1% BWOC magnetite NPs and 3% BWOC magnetite NPs had a 5% higher thickening time, while the 5% BWOC magnetite NPs had a 13.3% lower thickening time than the control case. Adding magnetite NPs will change the thickening time in the slurry, both higher and lower depending on the concentrations. The difference from the control case is shown in Figure 4.19.

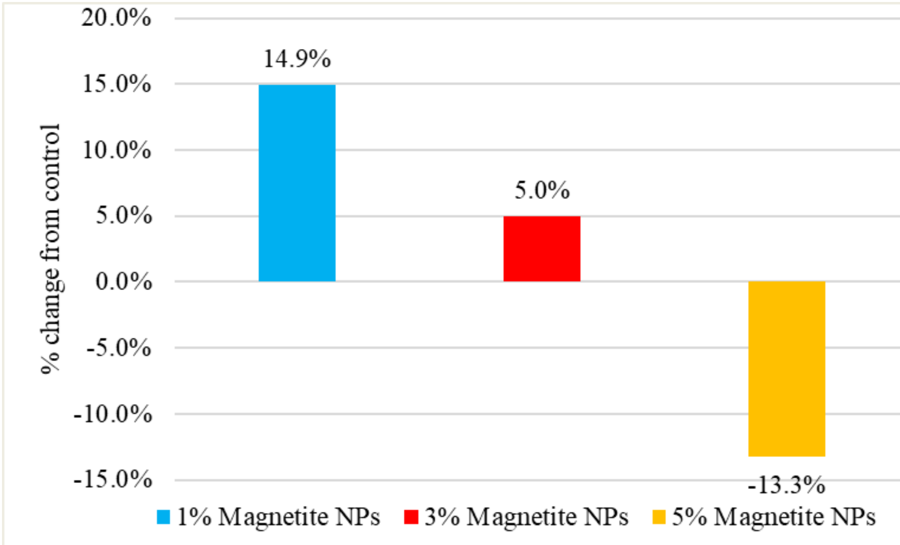

Figure 4.19 Heavy magnetite NPs, change from the control case

4.1.7 Light cement boric acid effect on thickening time at temperature 160°F and pressure 3000 psi

Several concentrations of boric acid were tested at the temperature 160°F with the formulation previously shown in Table 3.3, and the results are shown in Table 4.7 and Figure 4.20 and 4.21.

Table 4.7 Summarized results for the light cement control case. Boric acid concentration

Boric acid			
Light Cement	Thickening time		Change
160°F, 3000 psi	(HH:MM)	min	from 0.0%
CC, 0.0% boric acid	1:06	66	
CC, 0.5% boric acid	2:21	141	114%
CC, 0.6% boric acid	3:47	227	244%
CC, 0.7% boric acid	5:42	345	423%
CC, 1.0% boric acid	N/A	N/A	

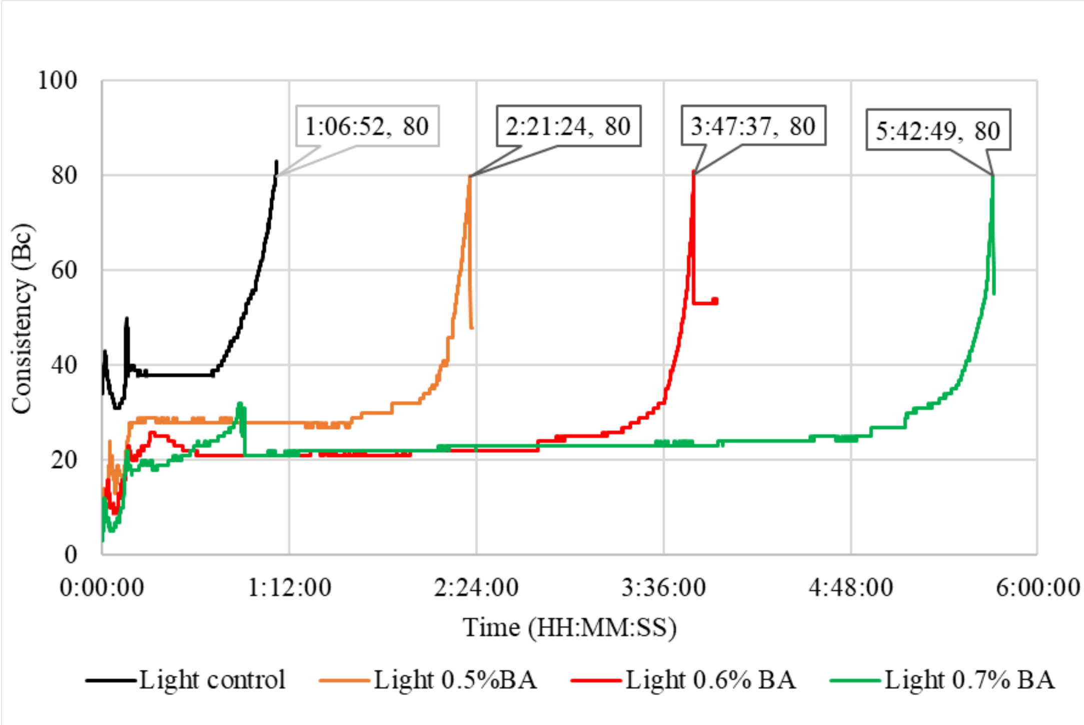

Figure 4.20 Light cement boric acid tests, 160°F and 3000 psi

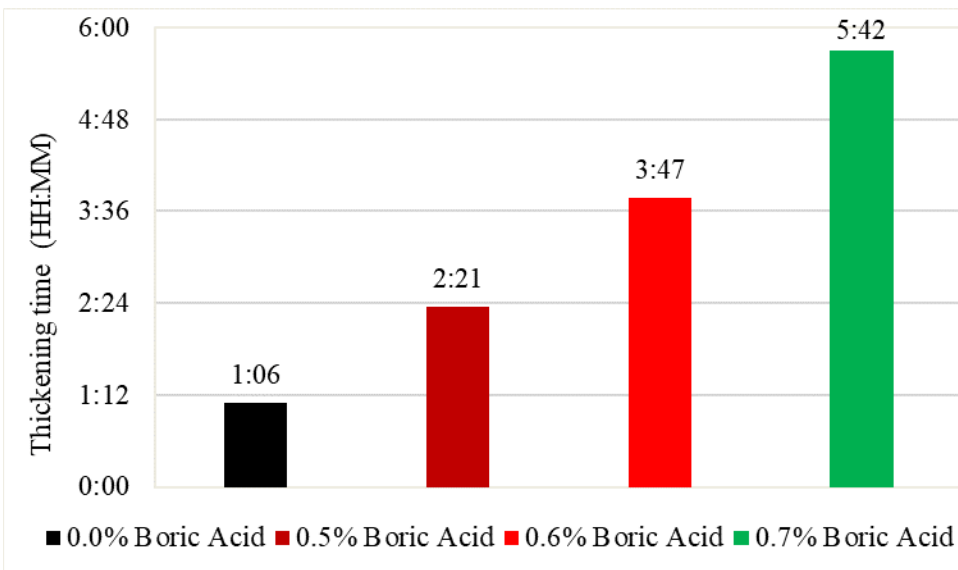

Figure 4.21 Light cement boric acid test, 160°F, and 3000 psi

The thickening time for light cement increased heavily with increasing boric acid concentrations when the temperature was 160°F and the pressure was 3000 psi, as can be shown in Figure 4.22.

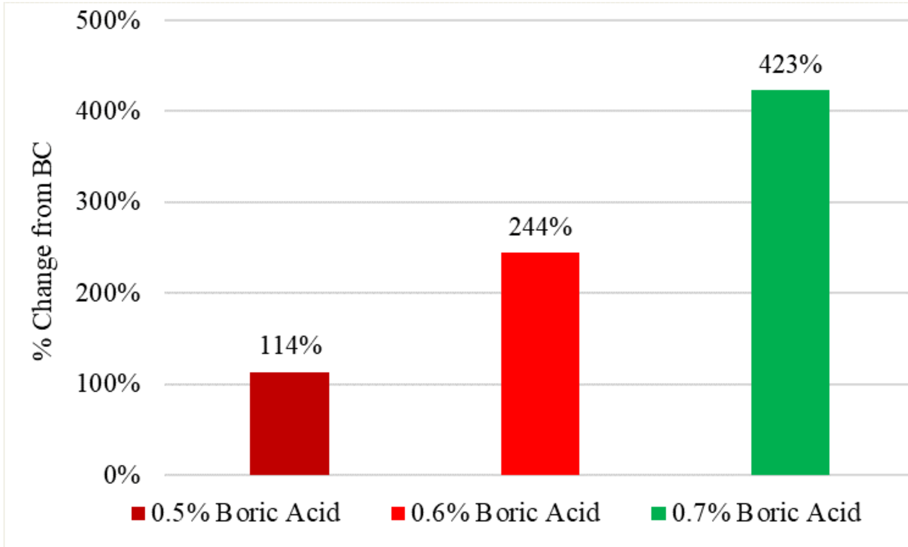

Figure 4.22 The light cement control boric acid tests. The thickening time change at 160°F

4.1.8 Light cement boric acid effect on thickening time at temperature 100°F and pressure 3000 psi

A lower temperature of 100°F was selected for the light cement considering it is supposed to be used in shallow wells. A series of new tests were conducted for the control case at 100°F and 3000 psi without boric acid. The tests were done to find the average control case thickening time as shown in the formulation in Table 3.4 and the results are shown in Table 4.8 and Figure 4.23 and 4.24.

Table 4.8 Light cement average control thickening time.

Light Cement	Thickening time			
100˚F, 3000psi	(HH:MM)	Min		
Light control 1	3:07	187	Mean	180
Light control 2	3:01	181	Std. dev	7.05
Light control 3	3:00	180	CV (%)	4%
Light control 4	2:50	170		
Average Control	3:00	180		

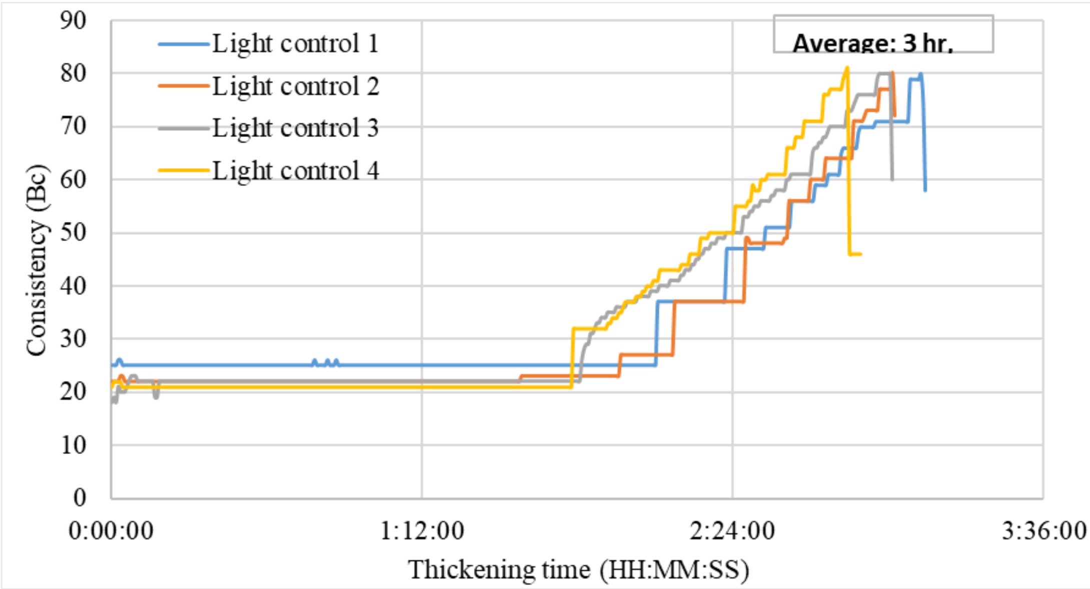

Figure 4.23 Light cement control without boric acid at 100°F and 3000 psi

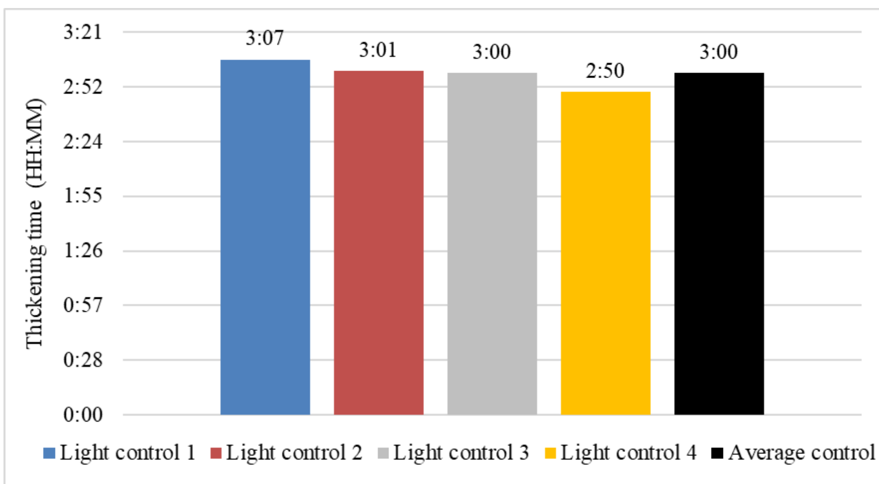

Figure 4.24 Light cement control case without boric acid at 100°F and 3000 psi

The average thickening time for a light cement control case with 100°F and pressure 3000 psi is 3 hours. This is enough time to mix and place the cement in the well. No boric acid is then necessary when using light cement at 100°F.

4.1.9 Light cement with bentonite NPs, round 1

One test of consistency was performed with the light cement control case and bentonite NPs, temperature 100°F, and 3000 psi, as shown in the formulation previously shown in Table 3.5 and the summarized results in Table 4.9 and Figures 4.25 and 4.26.

Table 4.9 Round 1, Summarized results for light cement with bentonite NPs at 100°F and 3000 psi

Round 1			
Light Cement	Thickening time		Change
100˚F, 3000psi	(HH:MM)	Min	from CC
Control case	3:00	180	
1% bentonite NPs1	3:05	185	3.06%
3% bentonite NPs1	2:57	177	-1.39%
5% bentonite NPs1	3:13	193	7.52%

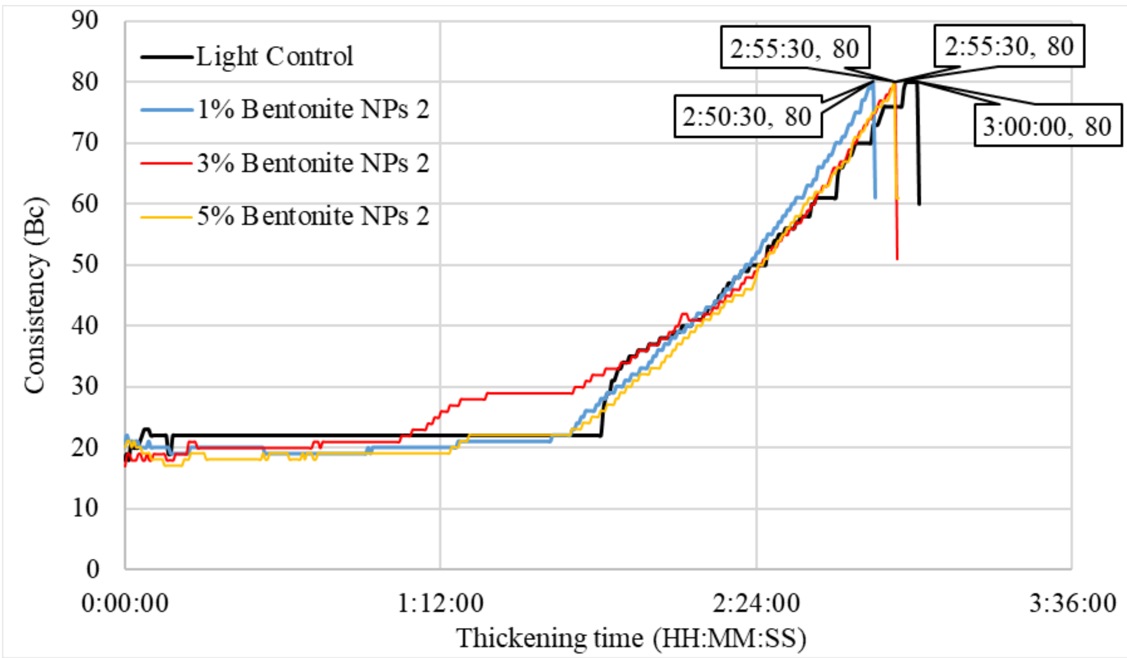

Figure 4.25 Round 1, Light cement with bentonite NPs at temperature 100°F and pressure 3000 psi

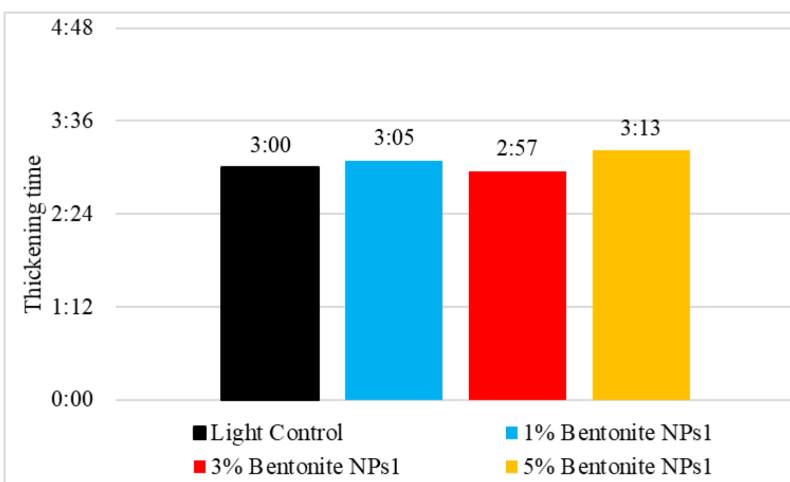

Figure 4.26 Round 1, Light cement with bentonite NPs at temperature 100°F and pressure 3000 psi

The thickening time was slightly higher than the control case for 1% and 5% BWOC bentonite NPs while 3% BWOC bentonite NPs had a slightly lower thickening time than the control case. The difference is shown in Figure 4.27.

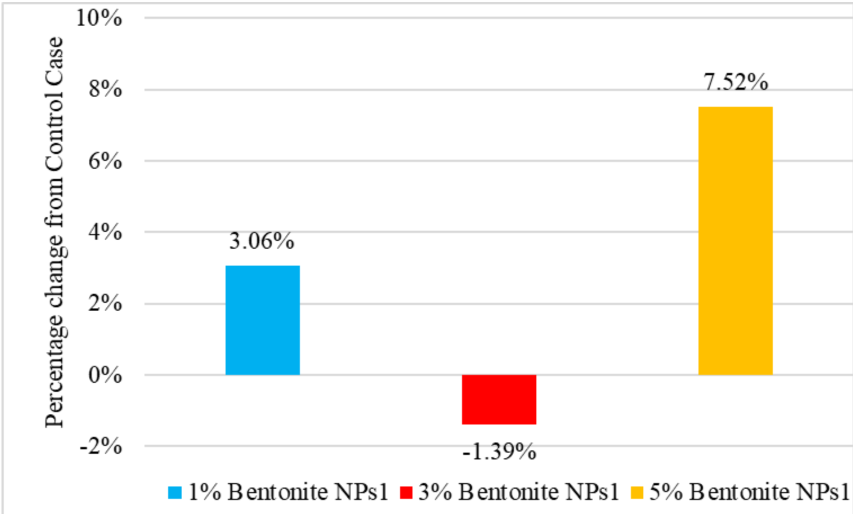

Figure 4.27 Round 1, Light cement change from the control case

4.1.10 Light cement with bentonite NPs, round 2

The thickening time for the light cement slurry containing normal bentonite as well as bentonite NPs was measured at 100°F and 3000 psi. The slurry formulation for each case was presented in the previous chapter Table 3.5. Table 4.10 summarized the thickening time results for this series of tests. Figures 4.28 and 4.29 present the test data and compare the final results.

Table 4.10 Round 2, Summarized results for light cement with bentonite NPs at 100°F and 3000 psi

Round 2			
Light Cement	Thickening time		Change
100˚F, 3000psi	(HH:MM)	Min	from C
Control Case	3:00	180	
1% bentonite NPs 2	2:50	170	-5.29%
3% bentonite NPs 2	2:55	177	-1.39%
5% bentonite NPs 2	2:55	177	-1.39%

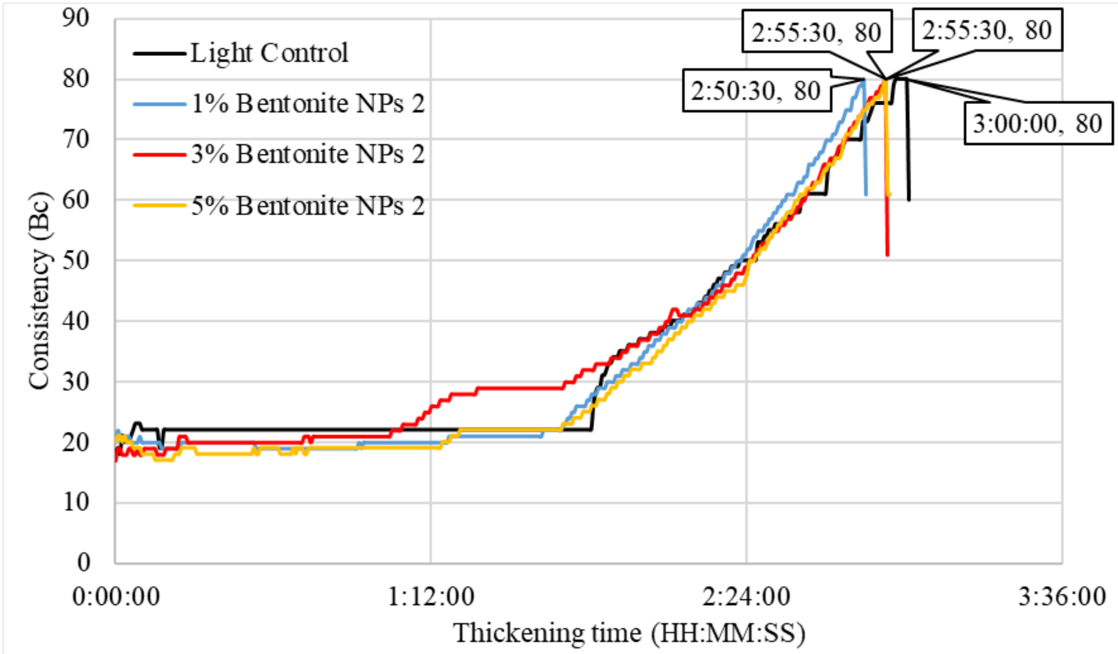

Figure 4.28 Round 2 Light cement with bentonite NPs at temperature 100°F and pressure 3000 psi

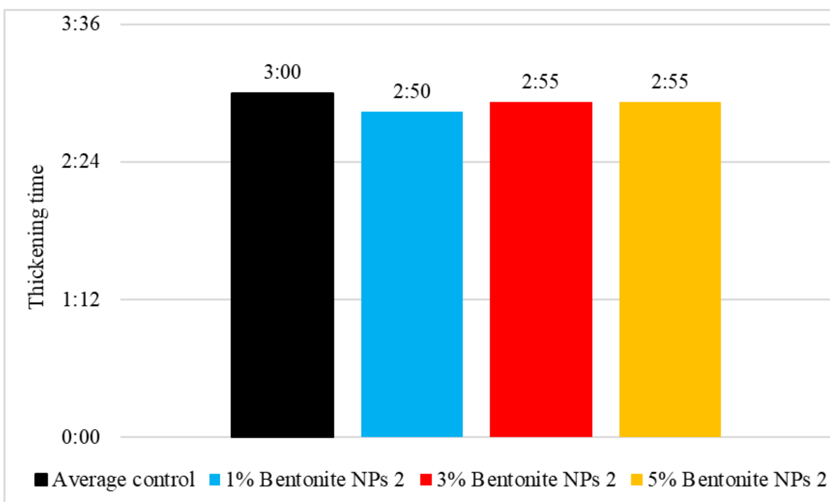

Figure 4.29 Round 2, Light cement with bentonite NPs at temperature 100°F and pressure 3000 psi

The thickening time was lower than the control case for all the bentonite NPs, 5.29% lower for 1%, 1.39% lower for 3%, and 5% BWOC bentonite NPs. The difference is shown in Figure 4.30.

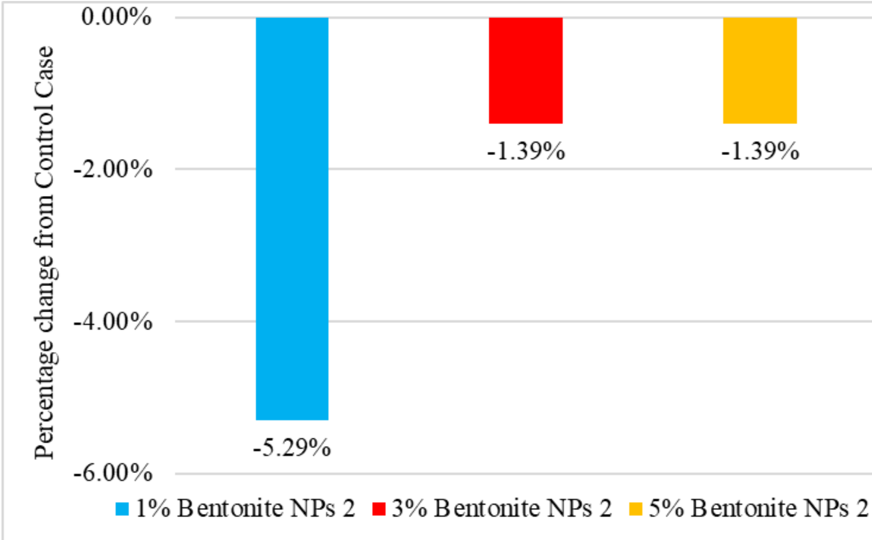

Figure 4.30 Round 2, Light cement change from the control case

4.1.11 Light cement with bentonite NPs, combined thickening time from round 1 and round 2

The results were repeated twice in rounds 1 and 2 and then combined as shown in Table 4.11 and Figure 4.31.

Table 4.11 Combined result for round 1 and round 2

	Round 1		**Round 2**		**Average**		
Light Cement	Thickening time		Thickening time		Thickening time		Change
100˚F, 3000psi	(HH:MM)	Min	(HH:MM)	Min	(HH:MM)	Min	from BC
Control Case	3:00	179.5	3:00	180	3:00	180	
1% bentonite NPs	3:05	185	2:50	170	2:57	178	-1.11%
3% bentonite NPs	2:57	177	2:55	0:00	2:56	177	-1.39%
5% bentonite NPs	3:13	193	2:55	0:00	3:04	185	3.06%

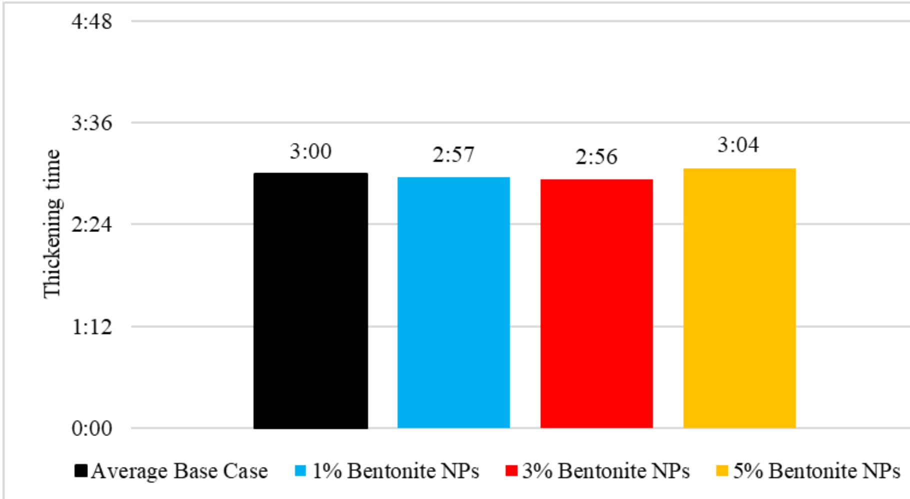

Figure 4.31 The combined average thickening time for bentonite NPs

The thickening time was almost the same for all the tests. The percent change from the control case for all the bentonite NPs was 1.11% lower for 1%, 1.39% lower for 3% BWOC bentonite NPs and 3.06% higher for 5% BWOC bentonite NPs as shown in Figure 4.32.

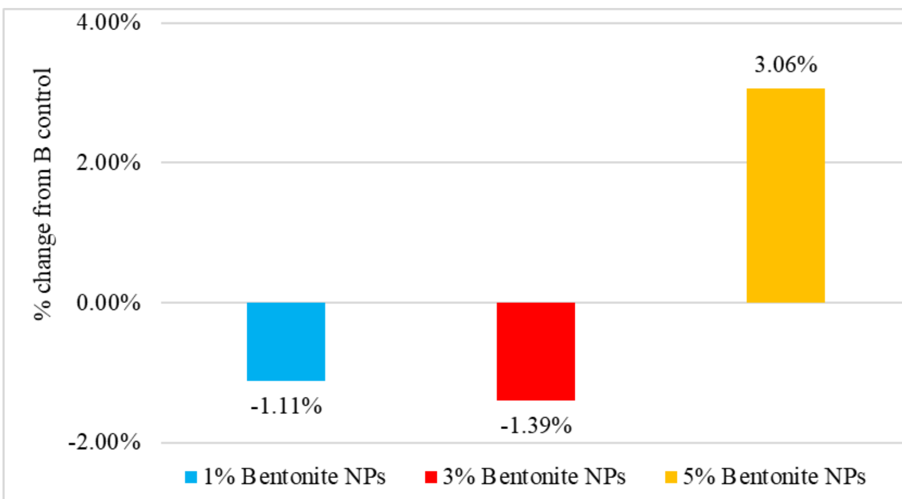

Figure 4.32 The combined change from the control case

4.2. Rheology

The rheology tests were performed with the temperature of 150°F for heavy cement, and the tests were started when the temperatures in the slurry reached 150°F. Two different ways of measuring the temperature were performed for light cement. Test 1 was started when the wall temperature of the slurry cup reached 100°F, and test 2 was started when the temperature within the slurry had reached 100°F. The readings were recorded after running for 1 minute at each speed, 600, 300, 200, 100, 6, and 3 RPM respectively. The formulation as previously shown in Table 3.10 is used for all the heavy tests, and Table 3.5 for all the light tests.

4.2.1 Heavy cement rheology results for the control case and magnetite NPs

The rheological properties of all the heavy cement slurries were measured and evaluated. The Bingham plastic and the Power-law are the models were used to approximate the pseudoplastic behavior of the cement. The temperature was measured in the slurry, and the tests were started when the temperature reached 150°F. The results are shown in Figure 4.33 and 4.34. Test details are shown in Appendix A.

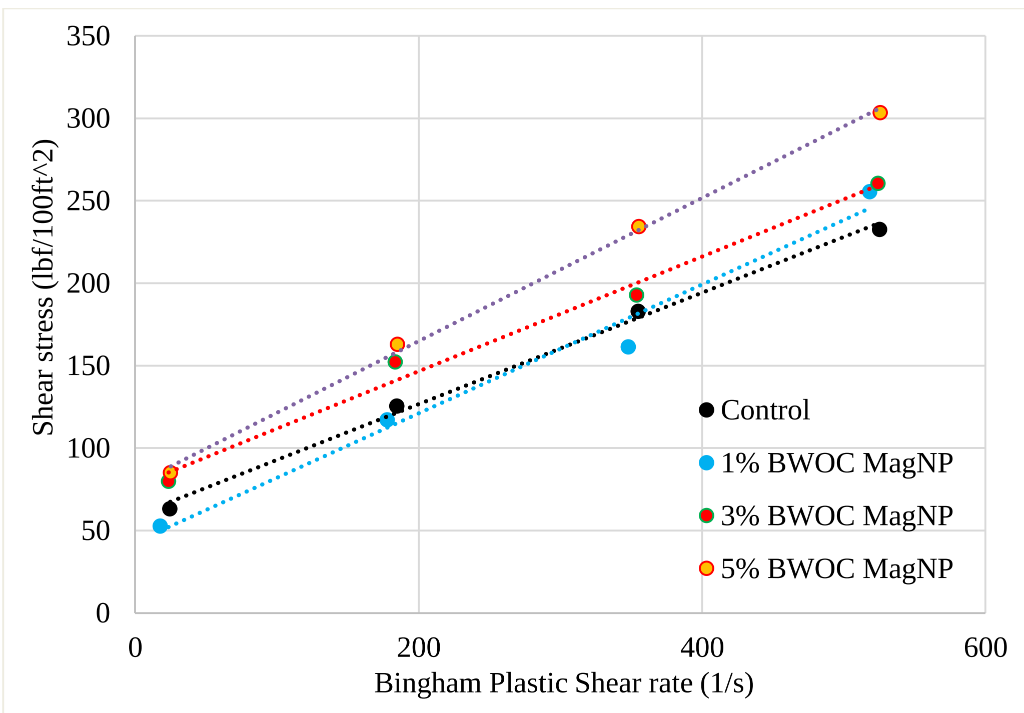

Figure 4.33 Shear stress of heavy cement with magnetite NPs and Bingham Plastic shear rate

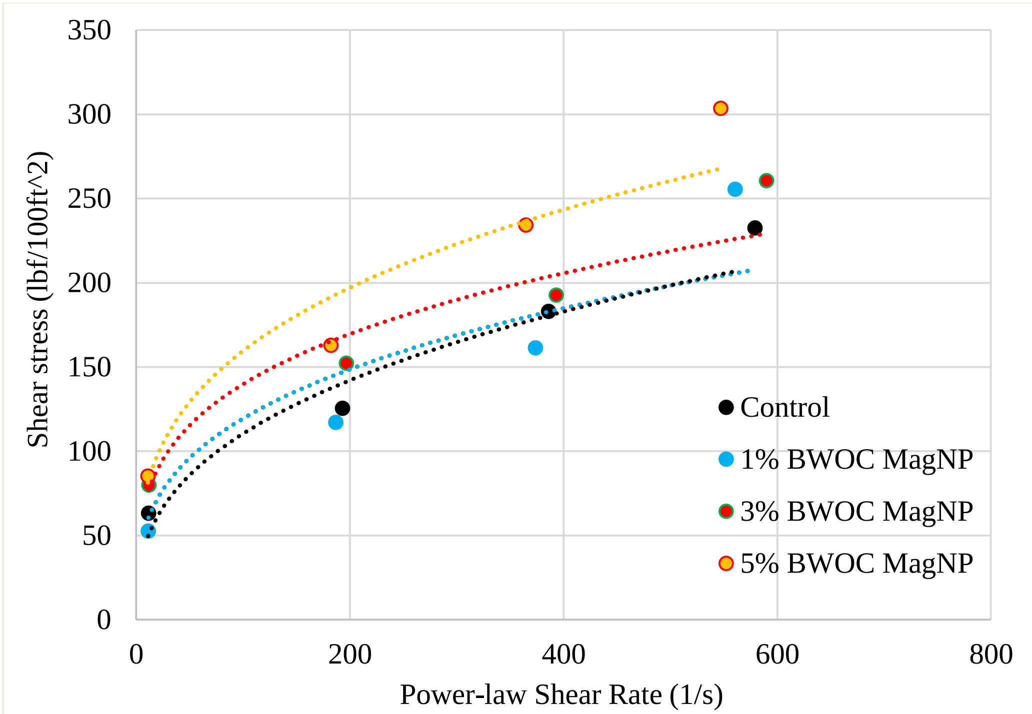

Figure 4.34 Shear stress of heavy cement with magnetite NPs and Power-law shear rate

The shear stress is highest for 5% BWOC magnetite NPs for any given shear rate and it is the lowest for the control case. The reason is the increased surface area of the NPs. The NPs have a higher water requirement, and even the small amount of NPs changes the slurry properties.

4.2.2 Heavy cement rheology results with barite NPs

The temperature was measured in the slurry, and the tests were started when the temperature reached 150°F. The results are shown in Figures 4.35 and 4.36. Test details are shown in the tables in Appendix B.

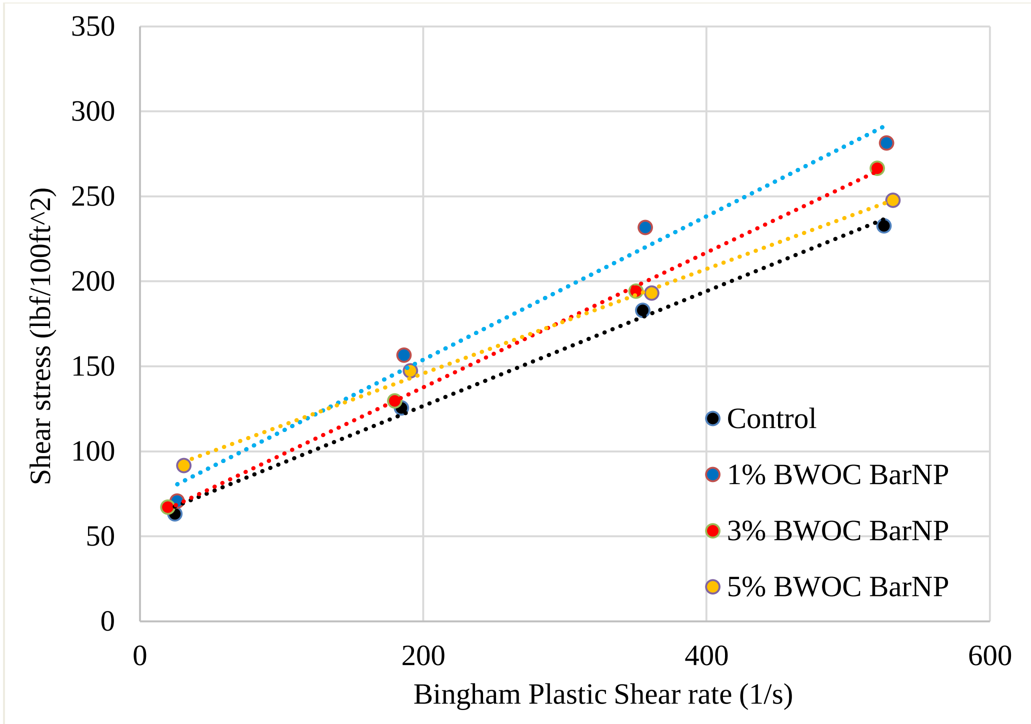

Figure 4.35 Shear stress of heavy cement with barite NPs and Bingham Plastic shear rate

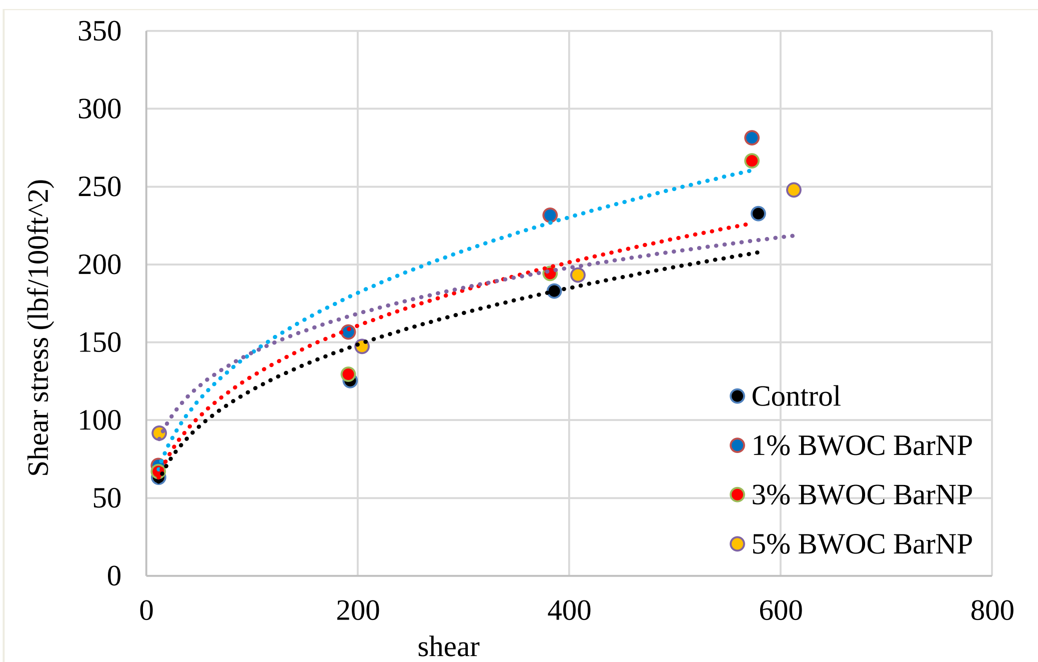

Figure 4.36 Shear stress of heavy cement with barite NPs and Power-law shear rate

The shear stress is highest for 1% BWOC barite NPs and lowest for the control case. A potential reason for higher shear stress for the NP slurries is the higher particle surface area of NPs. More surface area would in general have a higher water requirement. Test details are shown in Appendix B.

4.2.3 Summarized results for Bingham plastics viscosity and yield point

All the heavy cement rheology tests for the Bingham plastic model are combined and the results are shown in Table 4.12 and the plastic viscosity results are shown in Figures 4.37 and 4.38. The yield point results are shown in Figures 4.39 and 4.40.

Table 4.12 Summarized result for heavy cement Bingham plastics viscosity and yield point

	Bingham plastic 150˚F			
Component	PV	Change	Yp	Change
Test #	(cp)	from control	(lbf/100ft^2)	from control
Control	150.9		67.5	
1% BWOC magnetite NPs	194.4	29%	45.6	-32%
3% BWOC magnetite NPs	173.1	15%	71.5	6%
5% BWOC magnetite NPs	195.0	29%	90.0	33%
1% BWOC barite NPs	175.9	17%	88.4	31%
3% BWOC barite NPs	193.0	28%	57.3	-15%
5% BWOC barite NPs	141.5	-6%	91.2	35%

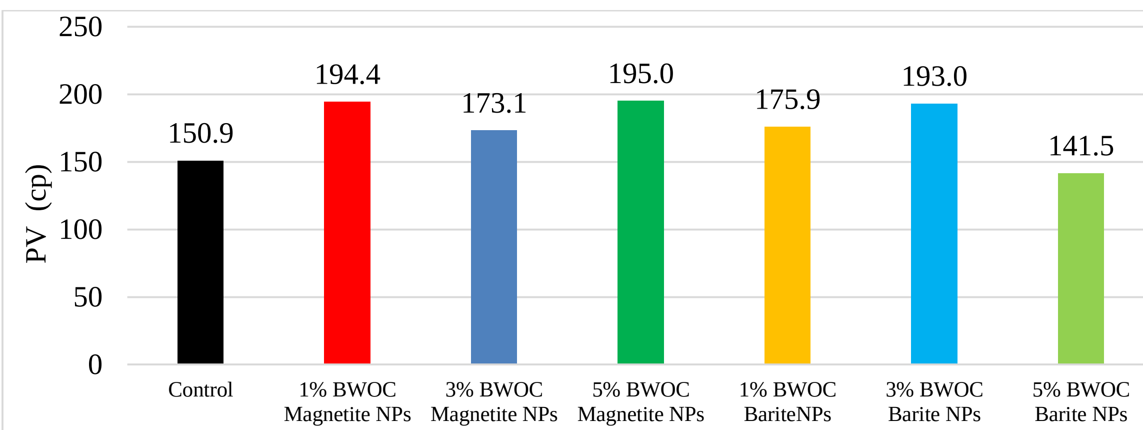

Figure 4.37 The plastic viscosities for heavy cement with magnetite and barite NPs

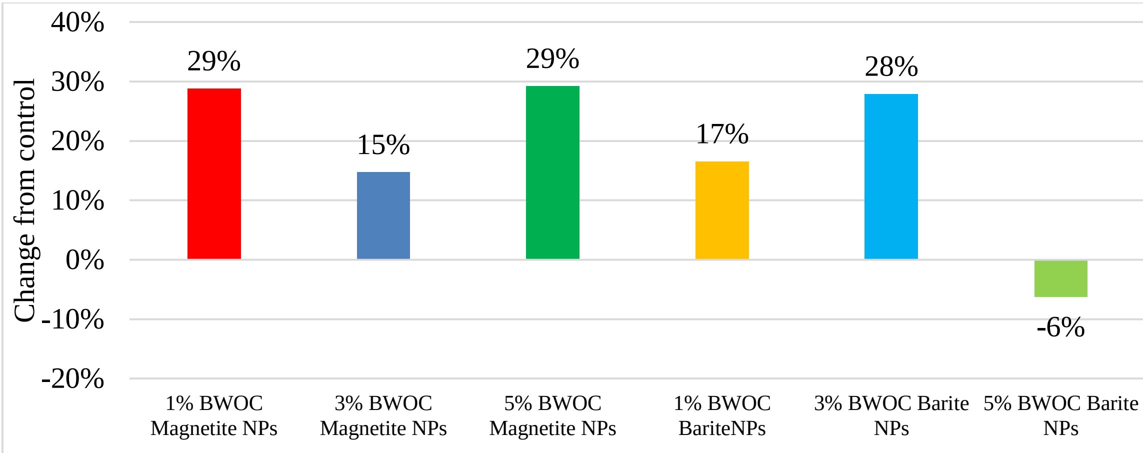

Figure 4.38 The plastic viscosities of heavy cement with magnetite and barite NPs change from the control case

The plastic viscosity of heavy cement changes with the amount of NPs in the formulation. All concentrations of NPs have a higher plastic viscosity than the control case except for 5% BWOC barite NPs which had a 6% lower plastic viscosity than the control case.

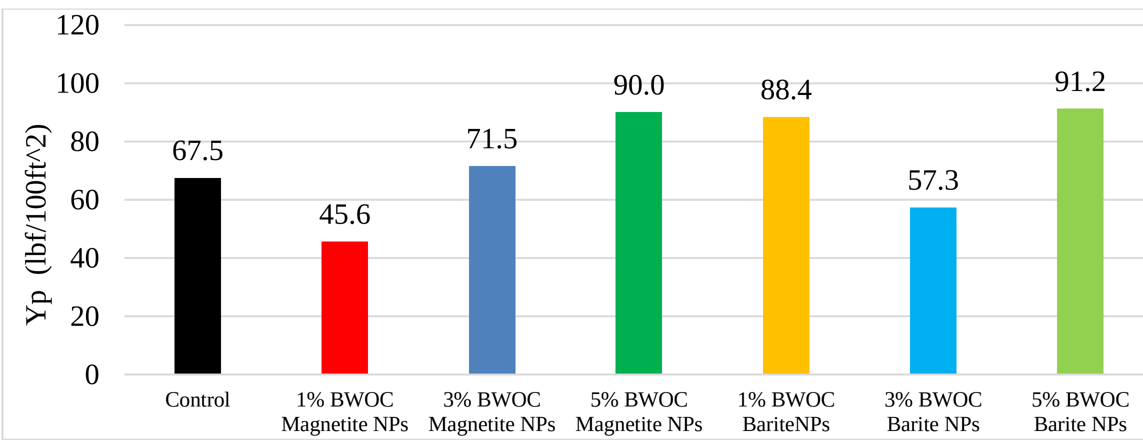

Figure 4.39 The yield points for heavy cement with magnetite and barite NPs

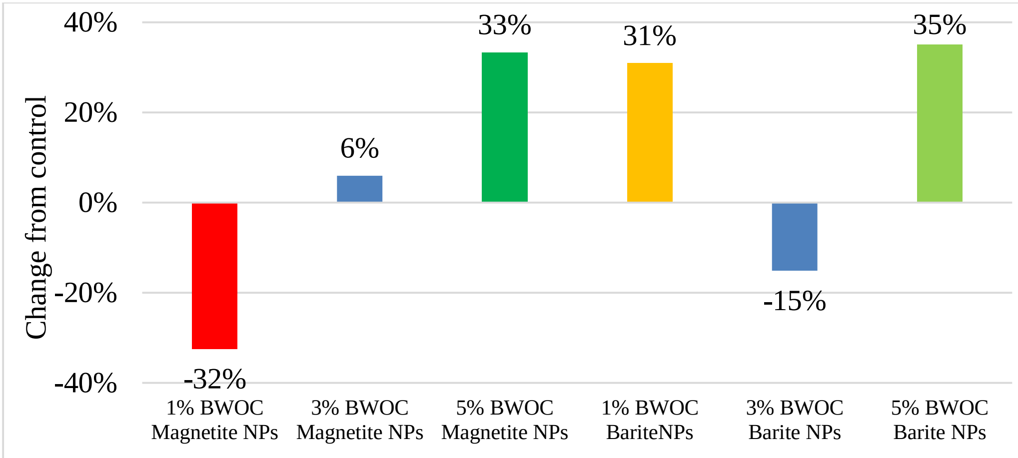

Figure 4.40 The yield points of heavy cement with magnetite and barite NPs change from the control case

The yield point of heavy cement is higher than the control case for 3% and 5% BWOC magnetite NPs and 1% and 5% BWOC barite NPs, while it`s lower for 1% BWOC magnetite NPs and 3% BWOC barite NPs.

4.2.4 Summarized results for power-law K-value

All the heavy cement rheology tests for the Power-law shear rate model are combined and the results are shown in Table 4.13 and the K-value results are shown in Figures 4.41 and 4.42

Table 4.13 Summarized results for heavy cement with the Power-law model's K-value

	Power-law model, 150˚F	
Component	K	Change
Test #	(dyne-s^n/cm^2)	from control
Control	139.8	
1% BWOC magnetite NPs	98.8	-29%
3% BWOC magnetite NPs	189.5	36%
5% BWOC magnetite NPs	191.8	37%
1% BWOC barite NPs	149.4	7%
3% BWOC barite NPs	141.5	1%
5% BWOC barite NPs	242.8	74%

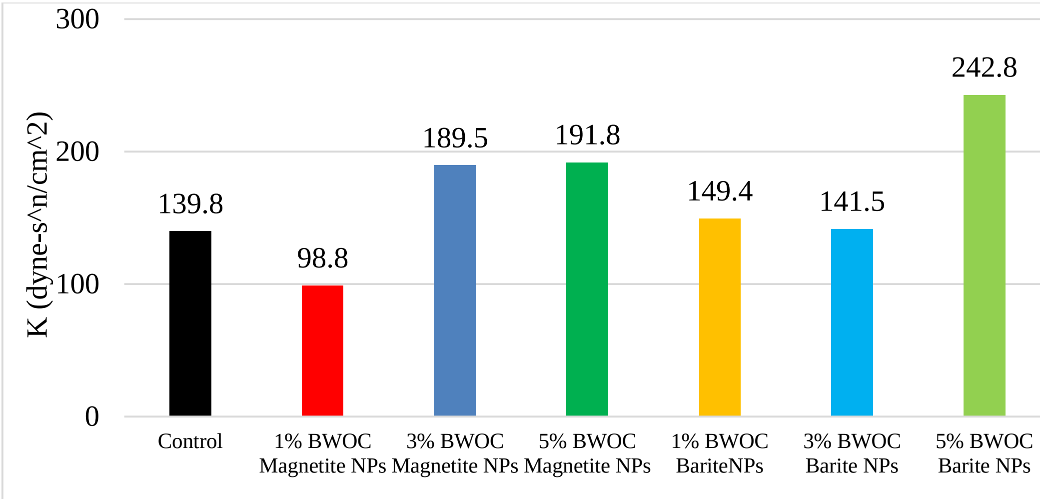

Figure 4.41 The K-value for heavy cement with magnetite and barite NPs

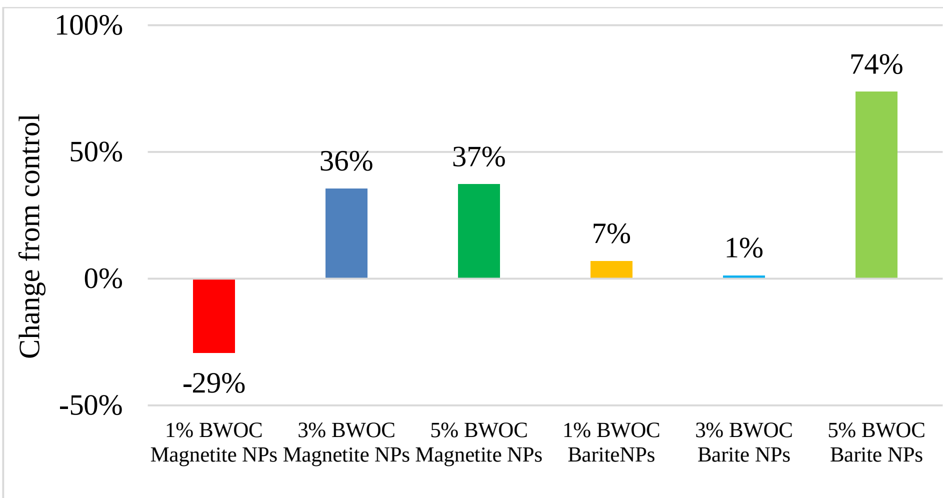

Figure 4. 42 The K-value for heavy cement with magnetite and barite NPs

The K-value of heavy cement with the Power-law model shear rate is 74% higher than the control case for 5% BWOC barite NPs and 29% lower for 1% BWOC magnetite NPs. The 3% and 5% BWOC magnetite NPs have 36% and 37%, respectively higher K-value than the control case, while 1% and 3% barite NPs have a slightly higher K-value than the control case.

4.2.5 Light cement rheology results

The cement rheology for all of the light cement cases has been evaluated. The Bingham plastic and the Power-law shear rates are the models used to approximate the pseudoplastic behavior of the cement. Two tests were performed with two different temperatures, wall temperature 100°F, test 1, and slurry temperature 100°F, test 2.

<u>Test 1, wall temperature 100°F:</u>

Figures 4.43 and 4.44 shows the result for shear stress for light cement with bentonite NPs with wall temperature 100°F in the test sample cup.

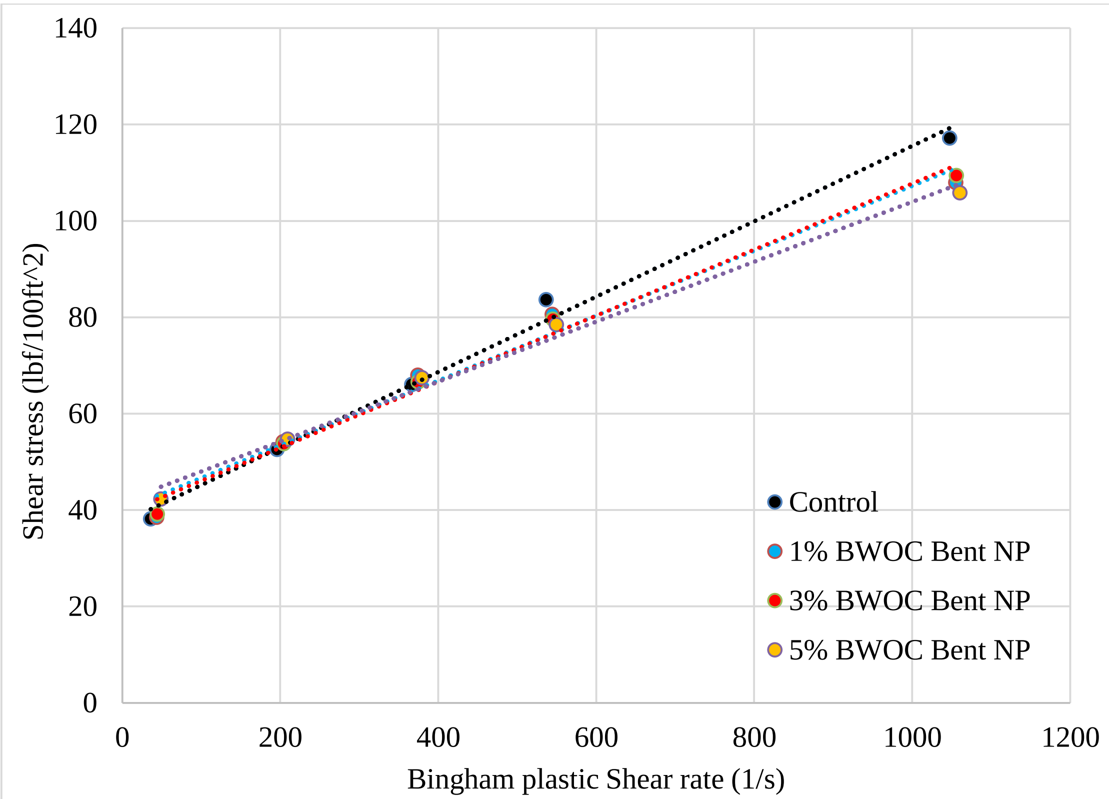

Figure 4.43 Shear stress of light cement with bentonite NPs and Bingham Plastic shear rate

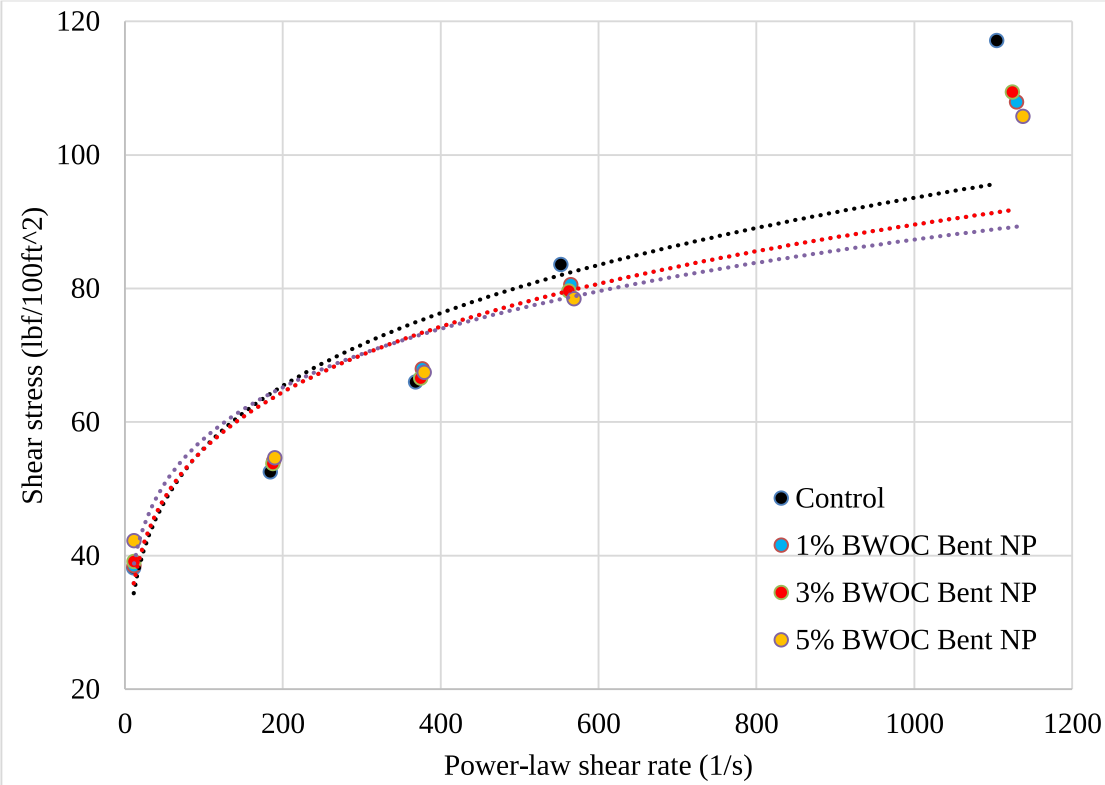

Figure 4.44 Shear stress of light cement with bentonite NPs and Power-law shear rate

The shear stress is highest for the control case and lowest for the bentonite NPs. Bentonite NPs reduce slurries shear stress. The test details are shown in the tables in Appendix C.

Test 2, slurry temperature 100°F:

Figures 4.45 and 4.46 show the result for shear stress for light cement with bentonite NPs with slurry temperature 100°F.

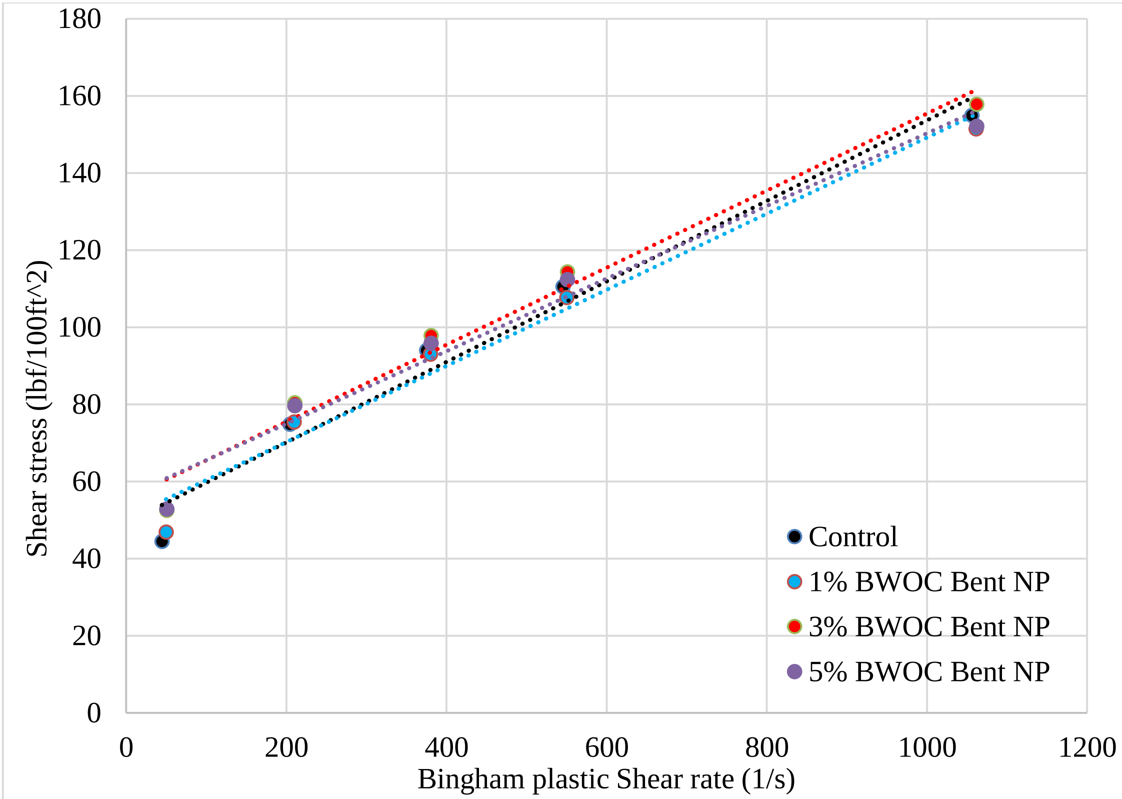

Figure 4.45 Shear stress of light cement with bentonite NPs and Bingham Plastic shear rate

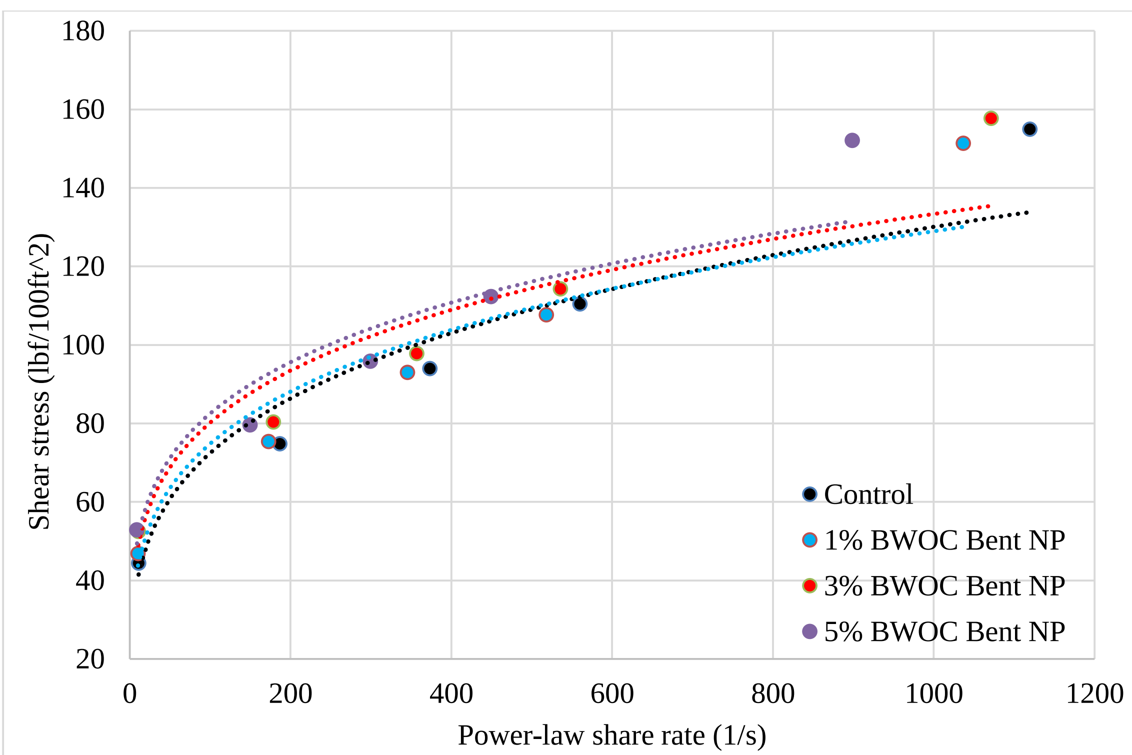

Figure 4.46 Shear stress of light cement with bentonite NPs and Power-law shear rate

The shear stress is highest for 3% BWOC bentonite NPs and lowest for the 1% BWOC bentonite NPs. The shear stress for the light cement with bentonite NPs when the temperature 100°F is measured in the slurry shows very little difference between the control case and the NPs. Test details are shown in the tables in Appendix D.

4.2.6 Summarized results for Light cement viscosity and yield point with the Bingham plastic model.

The summarized light cement viscosity and yield point with the Bingham plastic model are shown in Table 4.14.

Table 4.14 Summarized result for light cement Bingham plastics viscosity and yield point

	Bingham plastic model							
Component	Wall temp 100˚F				Slurry temp 100˚F			
Test #	PV	Change	Yp	Change	PV	Change	Yp	Change
	(cP)	from control	(lbf/100ft^2)	from control	(cP)	from control	(lbf/100ft^2)	from control
Control	43.8		34.8		50.3		53.5	
1% BWOC bentonite NPs	37.3	-14.9%	38.4	10.6%	45.5	-9.5%	55.7	4.0%
3% BWOC bentonite NPs	36.4	-16.9%	38.4	10.4%	47.8	-5.0%	59.6	11.4%
5% BWOC bentonite NPs	33.5	-23.4%	40.2	15.6%	46.0	-8.5%	59.5	11.2%

The plastic viscosity results are shown in Figures 4.47 and the difference from the control case is shown in Figure 4.48.

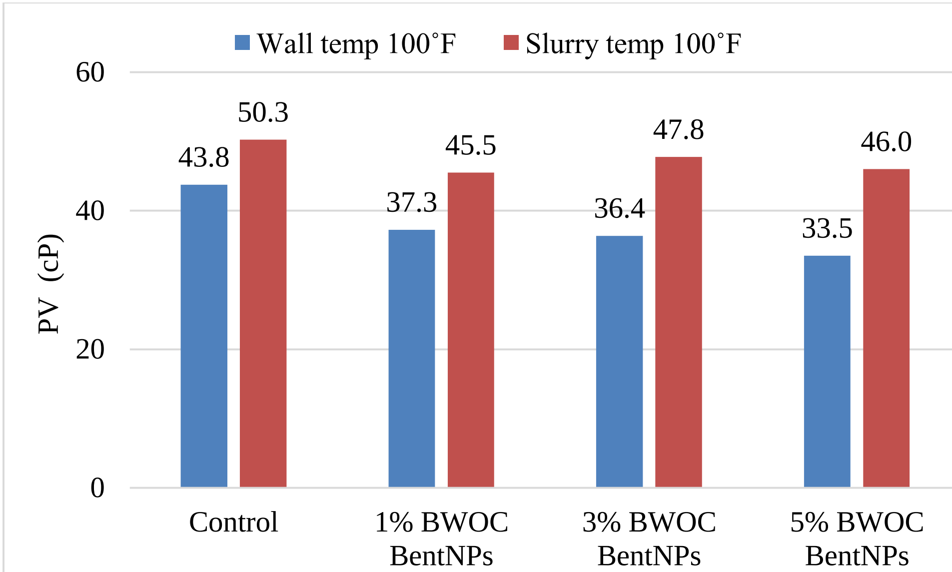

Figure 4.47 The plastic viscosities for light cement with bentonite NPs

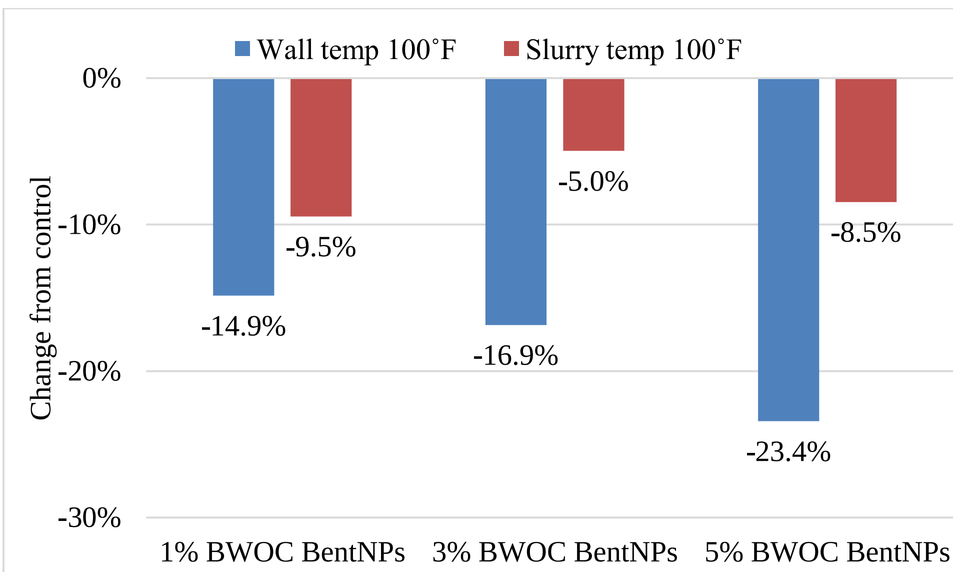

Figure 4.48 The plastic viscosities for light cement with bentonite NPs change from the control case

All the bentonite NPs have lower plastic viscosity than the control case. The difference in the measuring of the temperature between the wall and slurry in the sample cup is high, and the measure in the slurry is more correct for these results.

The yield point results are shown in Figures 4.49 and the difference from the control case is shown in Figure 4.50.

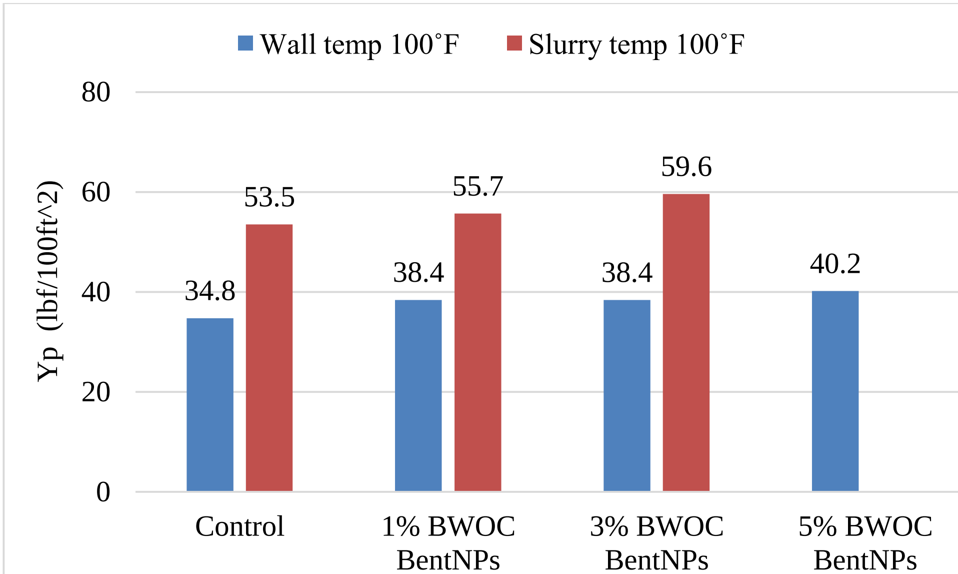

Figure 4.49 The yield points for light cement with bentonite NPs

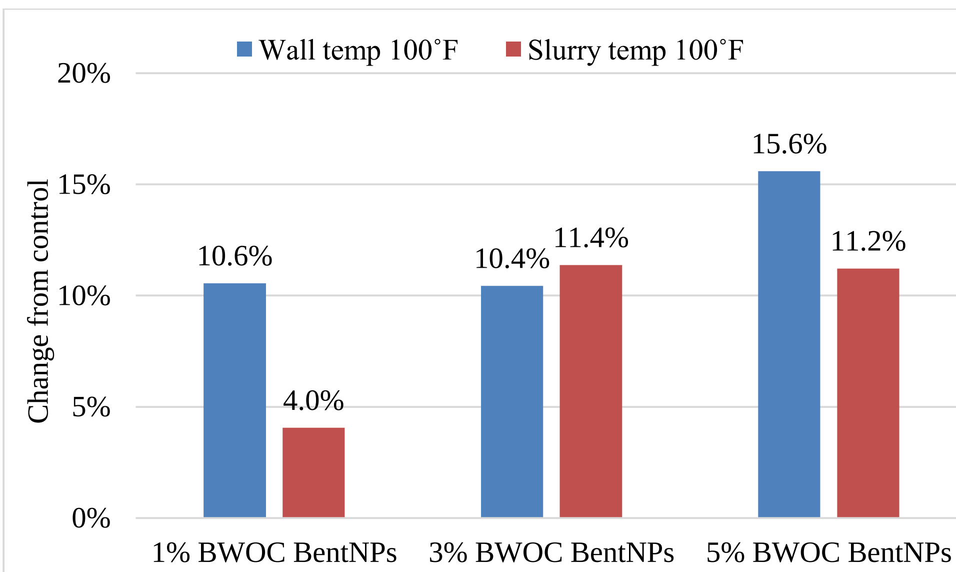

Figure 4.50 The yield points for light cement with bentonite NPs change from the control case

The yield point of the light cement with different temperatures are higher for wall temperature 100°F than for slurry temperature 100°F. There is a small difference in the yield points from the control case for both tests. 5% BWOC bentonite NPs have the highest difference from the control case for wall temperature 100°F, with a 15.6% higher yield point than the control case.

4.2.7 Summarized results for Light cement Power-laws K-value.

The summarized light cement K-value with the Power-law shear rate model are shown in Table 4.15.

Table 4.15 Summarized result for light cement Power-law K-value

	Power-law model			
Component	Wall temp 100˚F		Slurry temp 100˚F	
Test #	K	Change	K	Change
	(dyne-s^n/cm^2)	from control	(dyne-s^n/cm^2)	from control
Control	24.6		42.0	
1% BWOC bentonite NPs	35.0	42.4%	45.7	8.8%
3% BWOC bentonite NPs	32.2	31.0%	52.4	24.8%
5% BWOC bentonite NPs	37.8	53.7%	56.7	35.1%

The K-value results are shown in Figures 4.51 and the difference from the control case is shown in Figure 4.52.

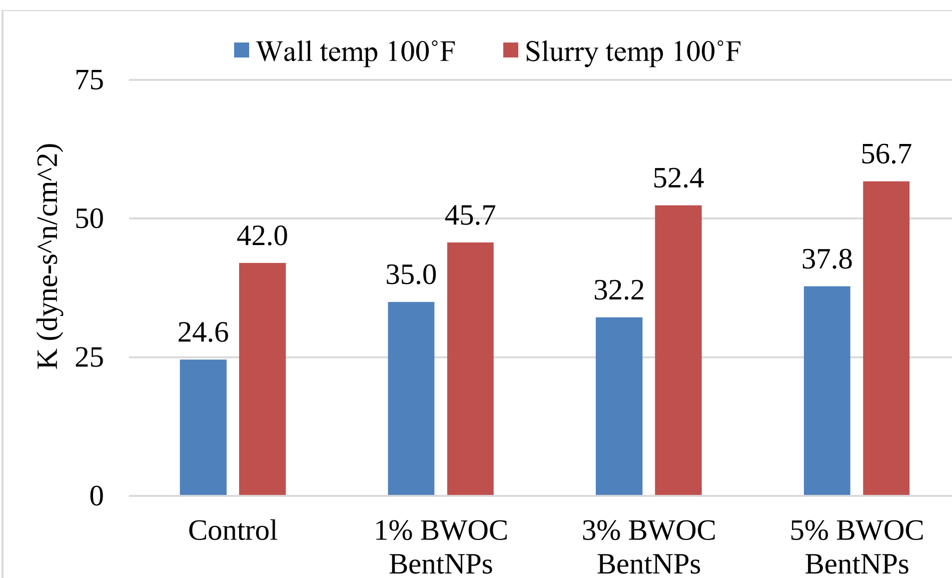

Figure 4.51 The K-value for light cement with bentonite NPs

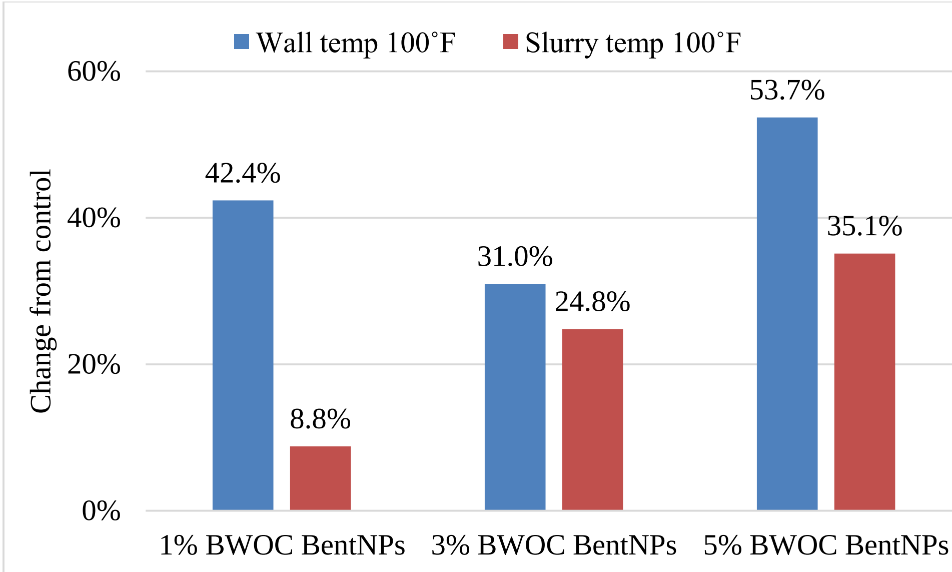

Figure 4.52 The K-value for light cement with bentonite NPs change from the control case

The K-value is higher than the control case for temperature measured in the wall of the sample cup than in the slurry. It's the highest for 5% and 1% BWOC bentonite NPs.

4.3. Fluid loss

The fluid loss tests were performed at 150°F for heavy cement and 100°F for light cement. The pressure was 500 psi. 600 psi downstream and 100 psi upstream. The temperatures were measured in two different ways for light cement, as wall temperature and as a slurry temperature. The formulation as previously shown in Table 3.10 is used for all the heavy tests, and Table 3.5 for all the light tests.

4.3.1 Heavy cement fluid loss with a control case and magnetite NPs

The results for fluid loss for heavy cement control cases and magnetite NPs are shown in Figure 4.53.

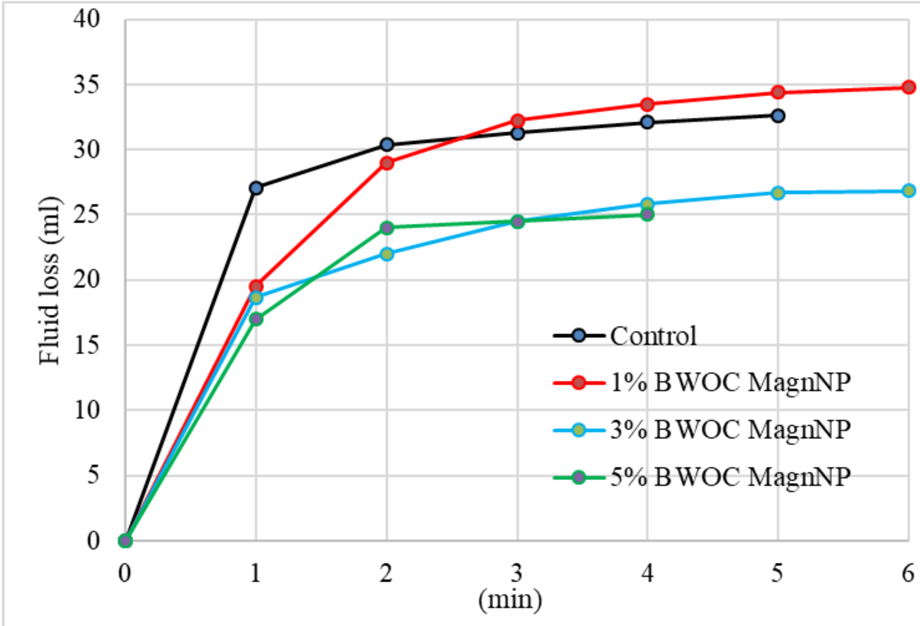

Figure 4.53 Heavy cement with magnetite NPs fluid loss

The highest fluid loss is 1% BWOC magnetite NPs and the lowest is 5% BWOC magnetite NPs. Test result details are shown in Appendix E.

4.3.2 Heavy cement fluid loss with barite NPs

The results for fluid loss for heavy cement control cases and barite NPs are shown in Figure 4.54.

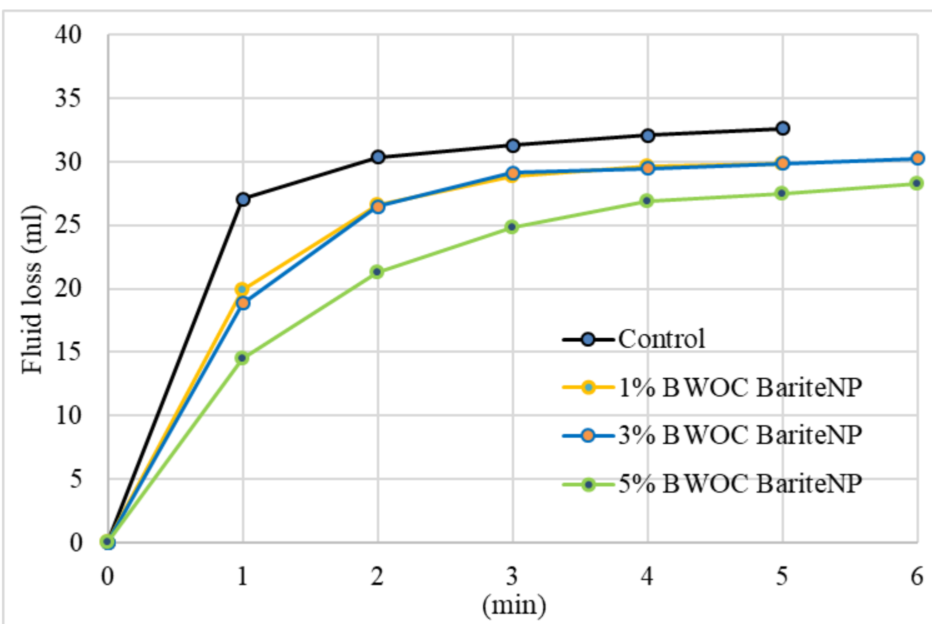

Figure 4.54 Heavy cement with barite NPs fluid loss

The highest fluid loss is the control case and the lowest is 5% BWOC barite NPs. All concentrations with barite NPs reduce fluid loss. The reason is the NPs increase the surface area of the cement and NPs with a higher water requirement. Test details are shown in Appendix F

4.3.3 Summary of heavy cement fluid loss with magnetite and barite NPs

The summarized results of fluid loss for heavy cement with magnetite and barite NPs are shown in Table 4.16.

Table 4.16 Summary of heavy cement with magnetite and barite NPs fluid loss

	Slurry temperature 150°F	
	Total fluid loss (ml)	Percentage change from control case
Control Case	32.6	
1% BWOC magnetite NPs	34.8	6.75%
3% BWOC magnetite NPs	26.8	-17.79%
5% BWOC magnetite NPs	25.0	-23.31%
1% BWOC barite NPs	29.9	-8.28%
3% BWOC barite NPs	30.3	-7.06%
5% BWOC barite NPs	28.3	-13.19%

The summarized results from the heavy fluid loss with magnetite and barite NPs are shown in Figure 4.55.

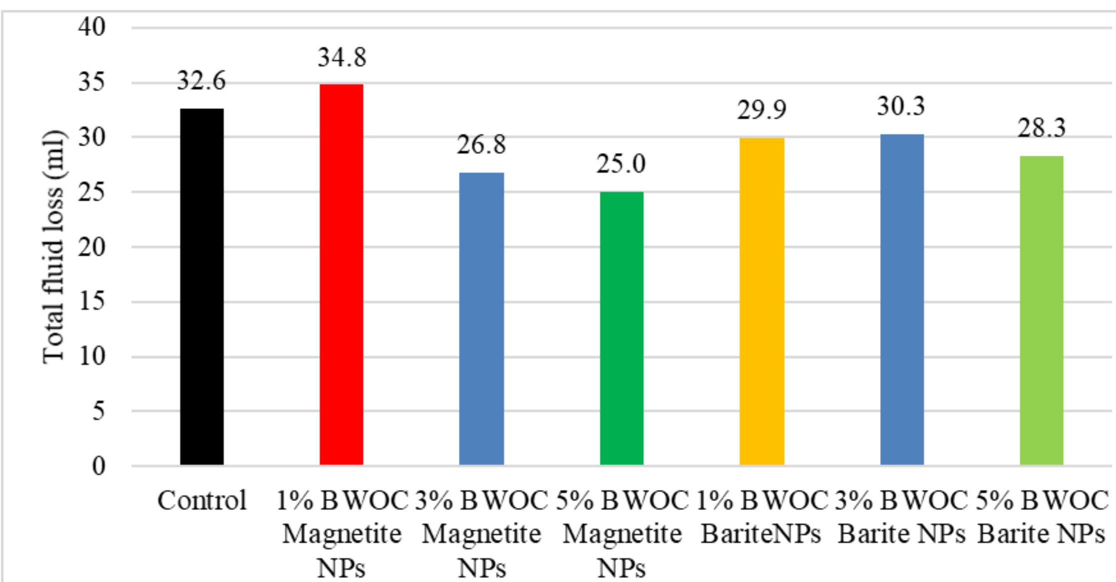

Figure 4.55 The total fluid loss for heavy cement with magnetite and barite NPs

The differences in the fluid loss for magnetite and barite NPs compared to the control case are shown in Figure 4.56.

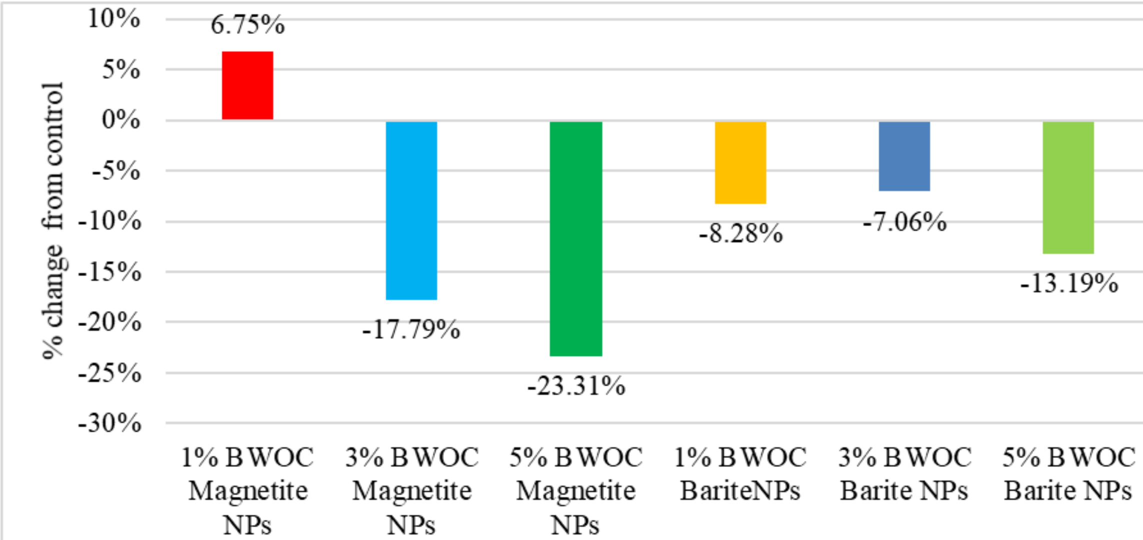

Figure 4.56 The total fluid loss for heavy cement with magnetite and barite NPs change from the control case

All the fluid loss results for heavy cement were lower than the control case, except for 1% BWOC magnetite NPs. The reason is a higher surface area of the NPs with a higher water requirement.

4.3.4 Light cement fluid loss with bentonite NPs and wall temperature 100°F

The result for fluid loss for light cement control case and bentonite NPs with a wall temperature of 100°F in the slurry cup is shown in Figure 4.57.

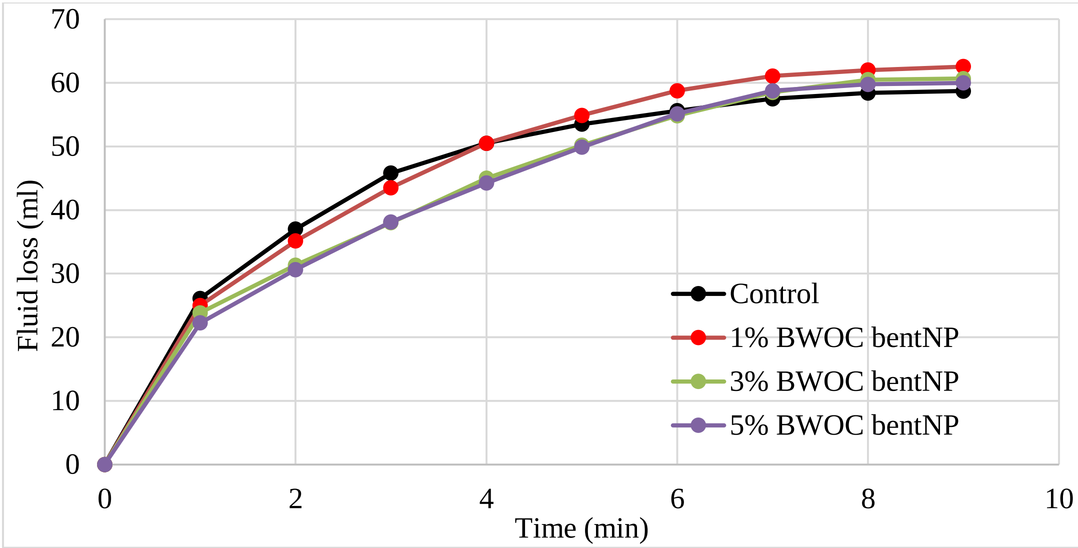

Figure 4.57 Light cement with bentonite NPs fluid loss with wall temperature 100°F

The highest fluid loss is 1% BWOC bentonite NP and the lowest in the control case. All the NPs slurries had a higher fluid loss than the control case. The reason is the higher surface area of the NPs and the NPs higher water requirement. Test details are shown in Appendix G

4.3.5 Light cement fluid loss with bentonite NPs and slurry temperature 100°F

The result for fluid loss for light cement control case and bentonite NPs with a temperature of the slurry at 100°F is shown in Figure 4.58.

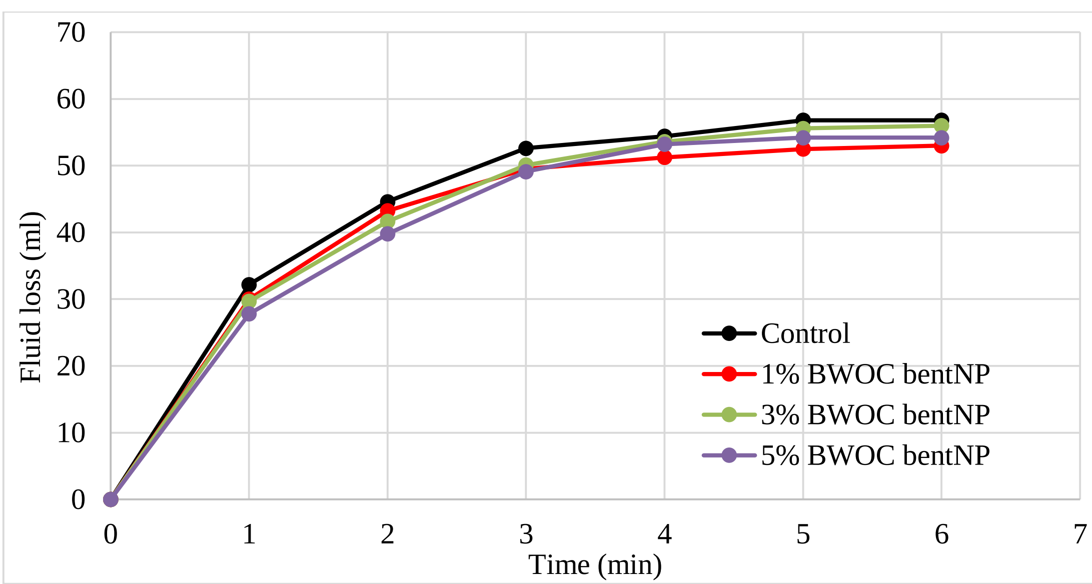

Figure 4.58 Light cement with bentonite NPs fluid loss with slurry temperature 100°F

The highest fluid loss is the control case and 1% BWOC bentonite NPs have the lowest fluid loss. All the NPs cement slurries had a lower fluid loss when the temperatures were measured in the slurry. The reason is the higher surface area of the NPs with a higher water requirement. Test details are shown in the tables in Appendix H.

4.3.6 Summary of light cement fluid loss with bentonite NPs

The summarized results for fluid loss with two different measurements of the slurry temperature are shown in Table 4.17.

Table 4.17 Summary of light cement with bentonite NPs fluid loss

	Wall temperature 100ºF		Slurry temperature 100 ºF	
	Total fluid loss (ml)	Change	Total fluid loss (ml)	Change
Control	59.5		56.8	
1% BWOC bentonite NPs	60.2	1.12%	53.0	-6.69%
3% BWOC bentonite NPs	60.7	1.96%	56.3	-0.88%
5% BWOC bentonite NPs	60.0	0.84%	54.2	-4.58%

The summarized results from the light cement fluid loss with bentonite NPs are shown in Figure 4.59.

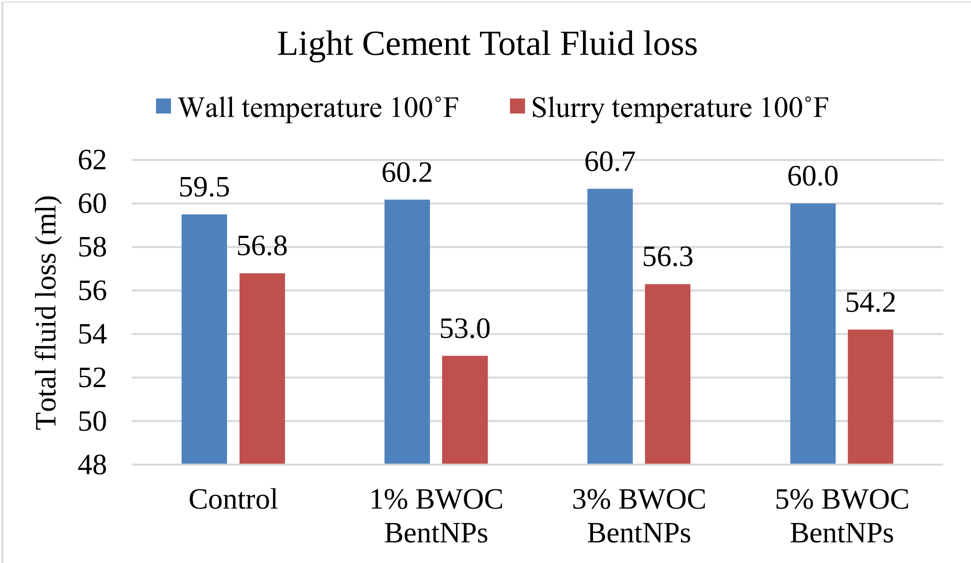

Figure 4.59 The total fluid loss for light cement with bentonite NPs

The differences in the fluid loss for bentonite NPs compared to the control case are shown in Figure 4.60

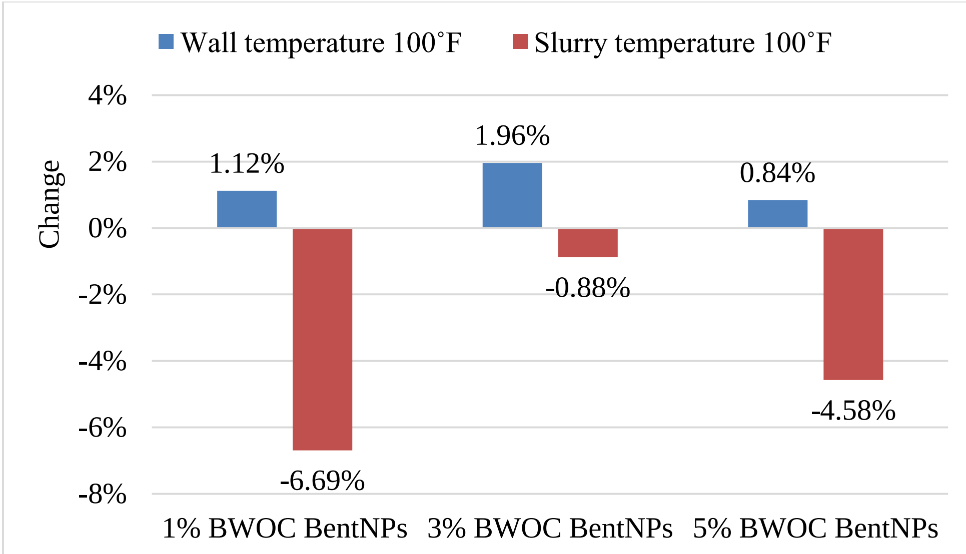

Figure 4.60. The total fluid loss for light cement with bentonite NPs change from the control case

The fluid loss measured is higher when using the wall temperature of the slurry cup 100°F compared to using the slurry temperature of 100°F. The slurry temperature measurement shows that all slurries with bentonite NPs had a lower fluid loss than the control case.

CHAPTER V

CONCLUSIONS

The conclusions from this study are based on three different types of testing, cement thickening time, rheology, and fluid loss.

The main conclusions from this study on the cement thickening time were;

- A small amount of boric acid (0.4% BWOC) was needed for the desired thickening time of approximately three hours for the heavy cement.
- For the heavy cement, the barite and magnetite NPs had minor effects on the thickening time.
- For the heavy cement, the thickening time slightly increased with barite NP concentration in heavy cement.
- For the heavy cement, the thickening time slightly decreased with magnetite NP in heavy cement.
- For the light cement with a control case of 227 minutes setting time and no boric acid adding bentonite NPs increased the thickening time to approximately 273 minutes with 5% BWOC bentonite NPs.

The main conclusions for this study on the cement rheology were;

- The Bingham Plastic rheology model should be used for cement slurries since the cement slurries have a yield value.
- The Power Law model should not be used to describe cement slurry rheology since it does not include a yield value in the model.
- For the heavy cement with barite and magnetite NPs, the shear stress increased for all the NPs concentrations.
- For the heavy cement, the plastic viscosity was higher than the control case for 5% BWOC magnetite and 1 and 3% barite NPs, while it was lower for 1 and 3% magnetite and 5% barite NPs.
- For the light cement with bentonite NPs, the shear stress and plastic viscosity were almost identical to the control case.

The main conclusions for this study on cement fluid loss were;

- For the heavy cement with magnetite and barite NPs, the fluid loss of the slurry was from 5 to 22% lower than the control case for all the NP concentrations.
- For the light cement with bentonite NPs, the fluid loss was from 4 to 6% lower than the control case for all the concentrations.

CHAPTER VI

FUTURE RECOMMENDATIONS

Future recommendations for studying cement slurries with NP additives and other additives are listed below.

Use of other NP additives;

- Evaluate different types of NPs that inherit different properties that could improve the cement quality and prevent a specific problem or enhance a specific property. Suggested NPs are titanium oxide NPs, zinc oxide NPs (ZnO), graphite nanotubes, Portland cement class A and H NPs.

Other new additives;

- Evaluate and test different weighting agents or extenders
- Evaluate and test of different retarders
- Evaluate and test of different fluid loss controllers
- Evaluate and test of different rheology controllers

Thermodynamic conditions;

- Study the cement property sensitivities at different temperatures.
- Study the cement property sensitivities at different pressures.

The C/W ratio;

- Evaluate different C/W ratios for the different mixtures by applying the “Dime” water requirement test, adopted by some oil service companies, to measure the required amount of water for all of the cement additives to compose new water requirements for different cement mixtures.
- Invent a new and accurate procedure more accurate than the “Dime” test for measuring the required amount of water for each cement additive.

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APPENDICES

APPENDIX A

Appendix A shows the rheology calculations for the shear stress, and shear rate, plastic viscosity and yield point with the Bingham plastic model, and the shear rate, n, and K-value with the Power-law model, for the heavy cement control case and magnetite NPs.

Table A.1 Heavy cement control case

Control	Reading		Bingham Plastic			Power-law			
	Mean	Shear stress	Shear rate	PV	Yp	Shear rate	n	K-value	
RPM	Fann 35A	(lbf/100ft^2)	(1/s)	(cp)	(lbf/100ft^2)	(1/s)	0.33283	eq (cp)	(dyne-s^n/cm^2)
600	286.0	304.6		150.9	67.5	1158.1		13976	139.8
300	218.4	232.6	525.3			579.1			
200	171.8	183.0	355.0			386.0			
100	117.8	125.5	184.7			193.0			
6	59.4	63.3	24.6			11.6			
3	37.0	39.4	19.5			5.8			
3 (10m)	47.2								

Table A.2 Heavy cement with 1% BWOC magnetite NPs

1% BWOC	Reading		Bingham Plastic			Power-law			
magnetite NPs	Mean	Shear stress	Shear rate	PV	Yp	Shear rate	n	K-value	
RPM	Fann 35A	(lbf/100ft^2)	(1/s)	(cp)	(lbf/100ft^2)	(1/s)	0.40354	eq (cp)	(dyne-s^n/cm^2)
600	267.0	284.4	1029.4	194.4	45.6	1121.1		9881	98.8
300	240.0	255.6	518.5			560.5			
200	151.5	161.3	348.2			373.7			
100	110.0	117.2	177.9			186.8			
6	49.5	52.7	17.8			11.2			
3	38.5	41.0	12.7			5.6			
3 (10m)	36.0								

Table A.3 Heavy cement with 3% BWOC magnetite NPs

3% BWOC	Reading		Bingham Plastic			Power-law			
magnetite NPs	Mean	Shear stress	Shear rate	PV	Yp	Shear rate	n	K-value	
RPM	Fann 35A	(lbf/100ft^2)	(1/s)	(cp)	(lbf/100ft^2)	(1/s)	0.30225	eq (cp)	(dyne-s^n/cm^2)
600	257.0	273.7		173.1	71.5	1179.8		18946	189.5
300	244.7	260.6	524.2			589.9			
200	181.0	192.8	353.9			393.3			
100	143.0	152.3	183.6			196.6			
6	75.0	79.9	23.5			11.8			
3	52.3	55.7	18.4			5.9			
3 (10m)	56.0								

Table A.4 Heavy cement with 5% BWOC magnetite NPs

5% BWOC	Reading		Bingham Plastic			Power-law			
magnetite NPs	Mean	Shear stress	Shear rate	PV	Yp	Shear rate	n	K-value	
RPM	Fann 35A	(lbf/100ft^2)	(1/s)	(cp)	(lbf/100ft^2)	(1/s)	0.32476	eq (cp)	(dyne-s^n/cm^2)
600				195.0	90.0	1094.1		19179	191.8
300	285.0	303.5	525.8			547.1			
200	220.0	234.3	355.5			364.7			
100	153.0	162.9	185.2			182.4			
6	80.0	85.2	25.1			10.9			
3	60.0	63.9	20.0			5.5			
3 (10m)	75.0								

APPENDIX B

Appendix B shows the rheology calculations for the shear stress, Bingham Plastic and Power-law shear rate, plastic viscosity, and yield point for heavy cement with barite NPs

Table B.1 Heavy cement with 1% BWOC barite NPs

1% BWOC	Reading		Bingham Plastic			Power-law			
barite NPs	Mean	Shear stress	Shear rate	PV	Yp	Shear rate	n	K-value	
RPM	Fann 35A	(lbf/100ft^2)	(1/s)	(cp)	(lbf/100ft^2)	(1/s)	0.35268	eq (cp)	(dyne-s^n/cm^2)
600			1038.0	175.9	88	1146.1		14941	149.4
300	264.3	281.4	527.1			573.1			
200	217.5	231.6	356.8			382.0			
100	147.0	156.6	186.5			191.0			
6	66.5	70.8	26.4			11.5			
3	50.3	53.5	21.3			5.7			
3 (10m)	53.5								

Table B.2 Heavy cement with 3% BWOC barite NPs

3% BWOC	Reading		Bingham			Power-law			
barite NPs	Mean	Shear stress	Shear rate	PV	Yp	Shear rate	n	K-value	
RPM	Fann 35A	(lbf/100ft^2)	(1/s)	(cp)	(lbf/100ft^2)	(1/s)	0.35267	eq (cp)	(dyne-s^n/cm^2)
600			1031.4	193.0	57.3	1146.1		14155	141.5
300	250.3	266.6	520.5			573.1			
200	182.3	194.2	350.2			382.0			
100	121.7	129.6	179.9			191.0			
6	63.0	67.1	19.8			11.5			
3	57.7	61.4	14.7			5.7			
3 (10m)	59.7								

Table B.3 Heavy cement with 5% BWOC barite NPs

5% BWOC	Reading		Bingham			Power-law			
barite NPs	Mean	Shear stress	Shear rate	PV	Yp	Shear rate	n	K-value	
RPM	Fann 35A	(lbf/100ft^2)	(1/s)	(cp)	(lbf/100ft^2)	(1/s)	0.25441	eq (cp)	(dyne-s^n/cm^2)
600			1042.6	141.5	91.2	1225.1		24280	242.8
300	232.7	247.8	531.7			612.5			
200	181.3	193.1	361.4			408.4			
100	138.3	147.3	191.1			204.2			
6	86.0	91.6	31.0			12.3			
3	67.7	72.1	25.9			6.1			
3 (10m)	75.7								

APPENDIX C

Appendix C shows the rheology calculations for the shear stress, and shear rate, plastic viscosity and yield point with the Bingham plastic model and shear rate, n and K value for the Power-law model, for control case and bentonite NPs with the wall temperature (Test 1)

Table C.1 Light cement control case

Control	Reading		Bingham plastic			Power-law			
	Mean	Shear stress	Shear rate	PV	Yp	Shear rate	n	K-value	
RPM		(lbf/100ft^2)	(1/s)	(cp)	(lbf/100ft^2)	(1/s)	0.44754	eq (cp)	(dyne-s^n/cm^2)
600	110	117	1047	43.8	34.8	1104		2457	24.6
300	79	84	536			552			
200	62	66	366			368			
100	49	53	196			184			
6	36	38	36			11			
3	30	32	31			6			
3 (10m)	32	34							

Table C.2 Light cement with 1% BWOC bentonite NPs

1% BWOC bentonite NPs	Reading		Bingham plastic			Power-law			
	Mean	Shear stress	Shear rate	PV	Yp	Shear rate	n	K-value	
RPM		(lbf/100ft^2)	(1/s)	(cp)	(lbf/100ft^2)	(1/s)	0.38502	eq (cp)	(dyne-s^n/cm^2)
600	101	108	1055	37.3	38.4	1129		3497	35.0
300	76	81	544			565			
200	64	68	374			376			
100	51	54	204			188			
6	36	39	43			11			
3	26	28	38			6			
3 (10m)	25								

Table C.3 Light cement with 3% BWOC bentonite NPs

3% BWOC bentonite NPs	Reading		Bingham plastic			Power-law			
	Mean	Shear stress	Shear rate	PV	Yp	Shear rate	n	K-value	
RPM		(lbf/100ft^2)	(1/s)	(cp)	(lbf/100ft^2)	(1/s)	0.39644	eq (cp)	(dyne-s^n/cm^2)
600	103	109	1056	36.4	38.4	1124		3217	32.2
300	75	80	545			562			
200	63	67	375			375			
100	51	54	204			187			
6	37	39	44			11			
3	28	30	39			6			
3 (10m)	30								

Table C.4 Light cement with 5% BWOC bentonite NPs

5% BWOC bentonite NPs	Reading		Bingham plastic			Power-law			
	Mean	Shear stress	Shear rate	PV	Yp	Shear rate	n	K-value	
RPM		(lbf/100ft^2)	(1/s)	(cp)	(lbf/100ft^2)	(1/s)	0.36843	eq (cp)	(dyne-s^n/cm^2)
600	99	106	1060	33.5	40.2	1138		3775	37.8
300	74	78	550			569			
200	63	67	379			379			
100	51	55	209			190			
6	40	42	49			11			
3	31	33	44			6			
3 (10m)	30								

APPENDIX D

Appendix D shows the rheology calculations for the shear stress, and plastic viscosity and yield point with the Bingham plastic model and shear rate, n and K value, for the Power-law model for control case and bentonite NPs with the slurry temperature (Test 2)

Table D.1 Light cement control case

Control	Reading		Bingham plastic			Power-law			
	Mean	Shear stress	Shear rate	PV	Yp	Shear rate	n	K-value	
RPM		(lbf/100ft^2)	(1/s)	(cp)	(lbf/100ft^2)	(1/s)	0.40637	eq (cp)	(dyne-s^n/cm^2)
600	146	155	1056	50.3	53.5	1120		4197	42.0
300	104	110	545			560			
200	88	94	375			373			
100	70	75	205			187			
6	42	44	45			11			
3	32	34	39			6			
3 (10m)	33								

Table D.2 Light cement with 1% BWOC bentonite NPs

1% BWOC bentonite NPs	Reading		Bingham plastic			Power-law			
	Mean	Shear stress	Shear rate	PV	Yp	Shear rate	n	K-value	
RPM		(lbf/100ft^2)	(1/s)	(cp)	(lbf/100ft^2)	(1/s)	0.38882	eq (cp)	(dyne-s^n/cm^2)
600	142	151	1061	45.5	55.7	1037		4566	45.7
300	101	108	550			518			
200	87	93	380			346			
100	71	75	210			173			
6	44	47	50			10			
3	34	36	45			5			
3 (10m)	32								

Table D.3 Light cement with 3% BWOC bentonite NPs

3% BWOC bentonite NPs	Reading		Bingham plastic			Power-law			
	Mean	Shear stress	Shear rate	PV	Yp	Shear rate	n	K-value	
RPM		(lbf/100ft^2)	(1/s)	(cp)	(lbf/100ft^2)	(1/s)	0.37628	eq (cp)	(dyne-s^n/cm^2)
600	148	158	1062	47.8	59.6	1072		5238	52.4
300	107	114	551			536			
200	92	98	381			357			
100	76	80	211			179			
6	49	53	50			11			
3	36	38	45			5			
3 (10m)	34								

Table D.4 Light cement with 5% BWOC bentonite NPs

5% BWOC bentonite NPs	Reading		Bingham plastic			Power-law			
	Mean	Shear stress	Shear rate	PV	Yp	Shear rate	n	K-value	
RPM		(lbf/100ft^2)	(1/s)	(cp)	(lbf/100ft^2)	(1/s)	0.36077	eq (cp)	(dyne-s^n/cm^2)
600	143	152	1062	46.0	59.5	899		5672	56.7
300	106	112	551			449			
200	90	96	381			300			
100	75	80	211			150			
6	50	53	50			9			
3	38	40	45			4			
3 (10m)	35								

APPENDIX E

Appendix E shows the result of heavy cement with magnetite NPs fluid loss

Table E.1 Fluid loss result for the heavy cement control case

Heavy	Control	Control	Control	Control	Control	Control	Control	
Date	5/13/2019	7/12/2019	7/15/2019	7/15/2019	7/17/2019	7/24/2019	9/2/2020	MEAN
Temp	150°F	150°F	150°F	150°F	150°F	150°F	150°F	150
Min	ml	ml	ml	ml	ml	ml	ml	ml
0	0	0	0	0	0	0	0	0.0
1	24	25	27	32	30	24.5	27	27.1
2	29	27	28.5	32.5	32.5	31	32	30.4
3	30	28	29	33.5	33	31.5	34	31.3
4	30.5	30	29.5	34	33.5	32	35	32.1
5	30.5	32	30	34	33.5	32.5	36	32.6

Table E.2 Heavy cement with 1% BWOC magnetite NPs fluid loss

Heavy	1% BWOC magnetite NPs	1% BWOC magnetite NPs	1% BWOC magnetite NPs	1% BWOC magnetite NPs	
Date	4/18/2019	5/7/2019	7/22/2019	8/5/2019	MEAN
Temp	150°F	150°F	150°F	150°F	150
Min	ml	ml	ml	ml	ml
0	0	0	0	0	0.0
1	15	19	20	24	19.5
2	20	30	31	35	29.0
3	22.5	35	36	35.5	32.3
4	25	36	37	36	33.5
5	26	37	38	36.5	34.4
6	26	37.5	38	37.5	34.8

Table E.3 Heavy cement with 3% BWOC magnetite NPs fluid loss

Heavy	3% BWOC magnetite NPs	3% BWOC magnetite NPs	3% BWOC magnetite NPs	
Date	5/22/2019	7/22/2019	7/23/2019	MEAN
Temp	150ºF	150ºF	150ºF	150ºF
Min	ml	ml	ml	ml
0	0	0	0	0.0
1	19	19	18	18.7
2	22	23	21	22.0
3	24.5	25.5	23.5	24.5
4	26	27	24.5	25.8
5	26.5	28	25.5	26.7
6	27	28	25.5	26.8

Table E.4 Heavy cement with 5% BWOC magnetite NPs fluid loss

Heavy	5% BWOC magnetite NPs	
Date	7/24/2019	MEAN
Temp	150ºF	150
Min	ml	ml
0	0	0
1	17	17
2	24	24
3	24.5	24.5
4	25	25

APPENDIX F

Appendix F shows the result for heavy cement with barite NPs fluid loss

Table F.1 Heavy cement with 1% BWOC barite NPs fluid loss

Heavy	1% BWOC barite NPs	1% BWOC barite NPs	1% BWOC barite NPs	1% BWOC barite NPs	
Date	5/8/2019	8/7/2019	7/26/2019	7/29/2019	MEAN
Temp	130ºF	150ºF	150ºF	150ºF	150
Min	ml	ml	ml	ml	ml
0	0	0	0	0	0.0
1	20	20.5	18	21	19.9
2	23.5	29	24	30	26.6
3	26	32	27	30.5	28.9
4	26.5	32.5	28.5	31	29.6
5	26.5	33	28.5	31.5	29.9

Table F.2 Heavy cement with 3% BWOC barite NPs fluid loss

Heavy	3% BWOC barite NPs	3% BWOC barite NPs	3% BWOC barite NPs	3% BWOC barite NPs	
Date	5/15/2019	7/26/2019	7/29/2019	8/5/2019	MEAN
Temp	150ºF	150ºF	150ºF	150ºF	150
Min	ml	ml	ml	ml	ml
0	0	0	0	0	0.0
1	17	18.5	22	18	18.9
2	23	27	27	29	26.5
3	28	28.5	29.5	30.5	29.1
4	28.5	29	29.5	31	29.5
5	29.5	29	29.5	31.5	29.9
6	30	29	29.5	32.5	30.3

Table F.3 Heavy cement with 5% BWOC barite NPs fluid loss

Heavy	5% BWOC barite NPs	5% BWOC barite NPs	5% BWOC barite NPs	5% BWOC barite NPs	
Date	5/16/2019	7/31/2019	8/1/2019	8/5/2019	MEAN
Temp	165ºF	150ºF	150ºF	150ºF	150
Min	ml	ml	ml	ml	ml
0	0	0	0	0	0.0
1	16	15	15.5	11.5	14.5
2	21	21.5	20.5	22	21.3
3	24	27	23.5	25	24.9
4	29	29	24	25.5	26.9
5	30	29.5	24.5	26	27.5
6	30.5	30.5	26	26	28.3

APPENDIX G

Appendix G shows the detailed result for a light cement control case and bentonite NPs fluid loss with wall temperature.

Table G.1 Light cement control case fluid loss, wall temperature 100°F

Light	Control	Control	Control	Control	Control	Mean
Date	9/29/2019	2/11/2020	2/11/2020	2/11/2020	2/11/2020	
Temp	Wall temp 100ºF	Wall temp 100ºF	Wall temp 100ºF	Wall temp 100ºF	Wall temp 100ºF	
Min	ml	ml	ml	ml	ml	ml
0	0	0	0	0	0	0
1	20.5	18	33	28	31	26.1
2	30	28	45	41	41	37.0
3	37.5	35	55	51.5	50	45.8
4	43.5	41	60	55	53	50.5
5	48.5	46	61	57	55	53.5
6	53.5	50	62.5	57	55	55.6
7	58	55	62.5	57	55	57.5
8	58.5	59	62.5	57	55	58.4
9	59	60	62.5	57	55	58.7

Table G.2 Light cement with 1% BWOC bentonite NPs fluid loss, wall temperature 100°F

Light	1% BWOC bentonite NPs	1%BWOC bentonite NPs	1%BWOC bentonite NPs	1%BWOC bentonite NPs	1%BWOC bentonite NPs	1%BWOC bentonite NPs	1%BWOC bentonite NPs	1%BWOC bentonite NPs	Mean
Date	1/10/2020	1/15/2020	1/16/2020	2/10/2020	2/10/2020	2/10/2020	2/10/2020	2/10/2020	
Temp	Wall temp 100ºF	Wall temp 100ºF	Wall temp 100ºF	Wall temp 100ºF	Wall temp 100ºF	Wall temp 100ºF	Wall temp 100ºF	Wall temp 100ºF	
Min	ml	ml	ml	ml	ml	ml	ml	ml	ml
0	0	0	0	0	0	0	0	0	0.0
1	23	21	21	25	25	26	29	30	25.0
2	31	30	29	33	35	39	42	42	35.1
3	37	36	37	41	44	49	52	52	43.5
4	43	42	43	48	51	58	60	59	50.5
5	47	48	48	54	58	63	61	60	54.9
6	51	52	54	59	62	66	63	63	58.8
7	55	56	58.5	63	63	67	63	63	61.1
8	57	60	59	64	63	67	63	63	62.0
9	60	60.5	60	64	63	67	63	63	62.6

Table G.3 Light cement with 3% BWOC bentonite NPs fluid loss, wall temperature 100°F

Light	3% BWOC bentonite NPs	3% BWOC bentonite NPs	3% BWOC bentonite NPs	Mean
Date	9/26/2019	1/11/2020	1/15/2020	
Temp, f	Wall temp 100℉	Wall temp 100℉	Wall temp 100℉	
Min	ml	ml	ml	ml
0	0	0	0	0.0
1	18.5	25	28	23.8
2	27	36	31	31.3
3	33	43	38	38.0
4	39	51	45	45.0
5	43	57.5	50	50.2
6	48.5	61.5	54.5	54.8
7	54.5	62	59	58.5
8	58	63.5	60	60.5
9	58	63.5	60.5	60.7

Table G.4 Light cement with 5% BWOC bentonite NPs fluid loss, wall temperature 100°F

Light	5% BWOC bentonite NPs	5% BWOC bentonite NPs	5% BWOC bentonite NPs	5% BWOC bentonite NPs	Mean
Date	9/27/2019	1/13/2020	1/15/2020	1/16/2020	
Temp	Wall temp 100℉	Wall temp 100℉	Wall temp 100℉	Wall temp 100℉	
Min	ml	ml	ml	ml	ml
0	0	0	0	0	0.0
1	20	24	21	24	22.3
2	28.5	30	30	34	30.6
3	35.5	37	38	42	38.1
4	42	42	44	49	44.3
5	47.5	47	50	55	49.9
6	53	52	55	60.5	55.1
7	57.5	52.5	60	65	58.8
8	58	53	61	67	59.8
9	59	53	61	67	60.0

APPENDIX H

Appendix H shows the result for light cement control case and bentonite NPs fluid loss with slurry temperature.

Table H.1 Light cement control case fluid loss, slurry temperature 100°F

Light	Control	Control	Control	Control	Control	Mean
Date	2/11/2020	2/11/2020	2/12/2020	2/14/2020	2/18/2020	
Temp	Slurry temp 100℉	Slurry temp 100℉	Slurry temp 100℉	Slurry temp 100℉	Slurry temp 100℉	
Min	ml	ml	ml	ml	ml	ml
0	0	0	0	0	0	0.0
1	34	34	31	30	32	32.2
2	45	46	41	45	46	44.6
3	49	55	50	53	56	52.6
4	50	58	53	54	57	54.4
5	52	61	55	56	60	56.8
6	52	61	55	56	60	56.8

Table H.2 Light cement with 1% BWOC bentonite NPs fluid loss, slurry temperature 100°F

Light	1%BWOC bentonite NPs	1%BWOC bentonite NPs	1%BWOC bentonite NPs	1%BWOC bentonite NPs	Mean
Date	2/10/2020	2/10/2020	2/14/2020	2/18/2020	
Temp	Slurry temp 100℉	Slurry temp 100℉	Slurry temp 100 ℉	Slurry temp 100℉	
Min	ml	ml	ml	ml	ml
0	0	0	0	0	0.0
1	32	34	28	26	30.0
2	46	47	41	39	43.3
3	52	50	49	47	49.5
4	54	53	50	48	51.3
5	54	53	52	51	52.5
6	54	53	54	51	53.0

Table H.3 Light cement with 3% BWOC bentonite NPs fluid loss, slurry temperature 100°F

Light	3%BWOC bentonite NPs	3%BWOC bentonite NPs	3%BWOC bentonite NPs	3%BWOC bentonite NPs	3%BWOC bentonite NPs	3%BWOC bentonite NPs	Mean
Date	2/12/2020	2/12/2020	1/13/2020	1/14/2020	1/18/2020	6/8/2020	
Temp	Slurry temp 100℉	Slurry temp 100℉	Slurry temp 100℉	Slurry temp 100℉	Slurry temp 100℉	Slurry temp 100℉	
Min	ml	ml	ml	ml	ml		ml
0	0	0	0	0	0	0	0.0
1	34	30	31	28	29	26	29.7
2	46	43	44	40	41	36	41.7
3	51.5	52	51	50	52	44	50.1
4	53	54.5	51	53	60	50	53.6
5	53	54.5	51.5	55.5	63	56	55.6
6	53	54.5	51.5	55.5	63	58.5	56.0
7	53	54.5	51.5	55.5	63	60.5	56.3

Table H.4 Light cement with 5% BWOC bentonite NPs fluid loss, slurry temperature 100°F

Light	5% BWOC bentonite NPs	5% BWOC bentonite NPs	5% BWOC bentonite NPs	5% BWOC bentonite NPs	5% BWOC bentonite NPs	Mean
Date	2/13/2020	2/13/2020	2/14/2020	2/18/2020	2/19/2020	
Temp	Slurry temp 100℉	Slurry temp 100℉	Slurry temp 100℉	Slurry temp 100℉	Slurry temp 100℉	
Min	ml	ml	ml	ml	ml	ml
0	0	0	0	0	0	0.0
1	29	28	31	23	28	27.8
2	42	40	44	31	42	39.8
3	51	50.5	46	45	53	49.1
4	54.5	55.5	48	54	54	53.2
5	54.5	55.5	48	57	56	54.2
6	54.5	55.5	48	57	56	54.2

VITA

Vigleik Nicolai Kjeldal

Candidate for the Degree of

Master of Science

Thesis: THICKENING TIME, RHEOLOGY AND FLUID LOSS OF OILFIELD CEMENT WITH NANO-PARTICLE SIZED ADDITIVES

Major Field: Petroleum Engineering

Biographical:

Education:

Completed the requirements for the Master of Science in Petroleum Engineering at Oklahoma State University, Stillwater, Oklahoma in December, 2020.

Completed the requirements for the Bachelor of Science in Petroleum Chemistry at Telemark Tekniske Skole, Norway in 1976.

Experience:
Worked as a high school teacher in Norway 18 years teaching mathematics, physics, chemistry, computer science, and English

Worked as a cofounder starting a company in Norway and the US, based on a drilling optimization program.

Started several companies in Norway related to my ranch/farm

Worked for the County's sewage plant in Norway for 3 years

Worked in a plant producing leisure boats in Norway for 3 years

Lieutenant for the National Military Guard in Norway for 22 years

Professional Memberships:
Member of SPE and AADE