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Experimental Investigation of Nano-Boric Acid Effects as Retarding Agent on Physical/Chemical Properties of Cement Slurries for High-Pressure High-Temperature Oil and Gas Wells

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Article

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Abstract

In oil/gas well-cementing operations, high strength and optimum thickening time are required to ensure the long-term integrity of wells. In High-Pressure, High-Temperature (HPHT) wells, cement hydration happens faster due to temperature effects. Furthermore, most of the mentioned wells are located in the deep subsurface, so cement must remain in its fluid shape for more time by using retarding agents in HPHT wells. This paper presents an experimental study for evaluating the effect of adding nano-boric acid in cement slurry properties. Cement slurries were prepared by adding acid boric (1% wt./wt.), and nano- acid boric (0.5%, 1% and 1.5% wt./wt.). The slurries were aged at 290˚ F temperature and 3000 psi pressure and then rheological, strength, thickening time were measured after conditioning slurries at 190° F and atmospheric pressure as per API 10-B standard. Based on results, nano-boric acid powder as a retarding agent is more effective than conventional micro-acid boric particles in various concentrations. nano-boric acid increases compressive strength and thickening time which can improve cementing operations in drilling HPHT wells. Mechanisms of nano-boric acid for retardation effect on cement slurries are Nucleation and Adsorption. The goal of this research is applying nano-boric acid as a retarder agent, and strength improver of cement slurry in HPHT oil and gas wells.

1. Introduction

Oil and gas wells faced different problems during drilling, completion, and production stages [1-3]. One of the most challenging problems is designing a cementing operation. Growing demand for drilling HPHT wells requires enhanced slurry properties to conquer the HPHT challenges [4]. The cement slurry is a combination of cement particles, water, and specialty chemical agents, pumped down into the well and placed in the annular space (between the casing and the wellbore) in the wellbore. Obtaining the needed properties of cement is much more complicated at HPHT conditions than usual wells due to the high temperatures and the high length of the well. In these types of wells, cement slurry must have the flowing ability (pumpability) in a long period for complete displacing of the mud. Also, the compressive

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strength of cement under the HPHT condition has to be in a proper value to provide long-term integrity, and higher compressive strength to provide less crack-generation in the cement sheath. Having optimum short-time properties of cement- such as thickening time, filtrate loss, rheological properties, -and long-term requirements -such as mechanical integrity and thermal stability- facilitate effective zonal isolation. Adequate compressive strength and strong bonding of set cement to the wellbore walls are very crucial in perfect drilling operation [5]. Also, the proper cementing operation requires high mud removal from the formation, and casing surface while pumping cement [6].

Cement design is always performed for each well according to available downhole conditions data and will be validated with running laboratory tests. Standard cement slurries for oil/gas wells are prepared with cement Class H or Class G without any retarder, which can be pumped up to 8,000 feet where the bottom-hole circulation temperature (BHCT) is not more than 125°F [7]. Retarders of cement mixtures are usually chosen based on the type of cement, BHCT, and its effect on slurry rheology and fluid loss parameters and cost issues [8].

Mechanical properties such as compressive strength are dependent on the cement hydration rate and value. In fact, compressive strength only starts to develop once the slurry has begun to hydrate. In other words, some extent of hydration is needed to form compressive strength. The hydration rate of cement decreases significantly when the temperature is reduced. The slow hydration rate of cement results in longer setting times and slower strength development [8].

Due to the reasons mentioned above, retarding agents are commonly used to slow down the hydration rate of cement. Retarders are classified based on their chemistry, application, etc. Typical retarders can be classified as below [9]:

- **Salts**

Salt water with concentrations of more than 20% BWOC (by weight of cement) has a retarding effect on cement.

- **Organophosphates**

Organophosphates, are the most potent cement retarders but hardly used in well-cementing operations because of their sensitivity to find optimum concentration for operational usage. Organophosphate retarders are the best choice in ultra-high-temperature wells.

- **Inorganic compounds**

Inorganic compounds, such are borax, boric acid, and its sodium salt are commonly used as a retarder agent for high-temperature retarders at BHCT of 300°F and higher. At higher temperatures, the borate power as a retarder decreases. Borates show a synergistic effect with other retarders, such as lignosulfonates. Also, Lignosulfonate is typically applied in low-temperature wells as a retarder agent [10].

The goal of this study is to modifying the hydration of cement particles by adding nano-boric material to be more customized for HPHT environments. More specifically, reaching higher long-term compressive strength as well as higher thickening time for cement slurries are the purpose of this study. In prevoius researches, increasing comressive strength is achieved by using different materials such as cellulose nanocrystal [11], altough increasing thickening time to make sure of cement displacemnt into the deep wellbore is still a crucial issue.

The scope of this case study is to evaluate the effect of the selected nano-material in rheological behaviour, HPHT filtration, compressive strength and thickening time as well as finding the true mechanism of applied nano-boric agent on morphology changing and retardation of cement.

Limitation of this research is investigating only on laboratory scale and not performing field study.

2. Materials and methods

2.1. Material Explanation

Boric acid, as H_3BO_3 or $B(OH)_3$, is a very weak acid (Figure 1). Boric acid is a triprotic acid used as a retarder agent for cementing in high-temperature environments (Figure 2). Nano-boric acid has the same retarding effect on cement thickening time and may be more effective due to having smaller particle sizing and more straightforward/more frequent chemical reactivity. Nano-boric acid particles used in this article to validate this hypothesis, had particle size distribution mainly between 10 and 80 nm and 98.5% purity (Figure 3). Both boric acid and nano-boric acid were bought and not synthesized.

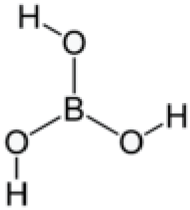

Figure 1. Acid boric molecule

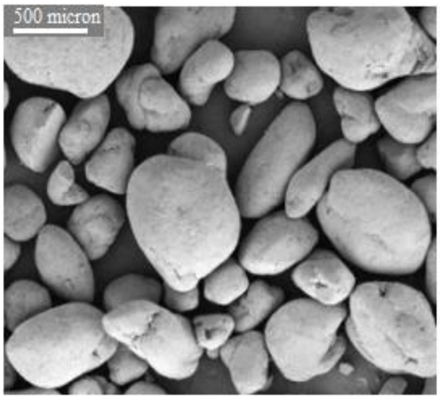

Figure 2. Boric acid SEM image (500 microns)

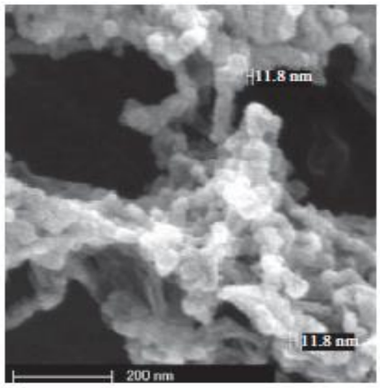

Figure 3. Nano-acid boric SEM image (200nm)

Retarders have different mechanisms for slowing down the hydration reactions of cement slurries. The retarding mechanism is based on the most critical proposed theories: adsorption, nucleation, precipitation, complexation, and dissolution-precipitation [10]. In Table 1, retarding mechanisms are summarized:

Table 1. Retarder Mechanism

Item	Retarding Theory	Mechanism	Reference
1	Adsorption theory	• Retarder adsorption on the surface area of new components called C-S-H which are produced by hydration of C_3S and C_2S • Minimizing contact with water	[12]
2	Precipitation	• Reacting with calcium and hydroxyl ions • Forming an inhibitive semi-permeable layer on the cement particles	[13]
3	Nucleation	• Reduction in hydration rate by adsorbing on cement nuclei	[14]
4	Complexation	• Chelating calcium ions • Reducing the concentration of available calcium ions and inhibiting the formation of cement hydrates	[15]
5	Dissolution-precipitation (mainly applicable for organic phosphonic acid-based retarders)	• Dissolution of calcium ions • Precipitation a layer of water-insoluble calcium phosphonate as a diffusion obstacle to water	[16]

It is hard to diagnose the exact mechanism of each retarder and maybe a few of the mentioned items can be detected in a retarding process of cement particles.

2.2. Methodology

2.2.1. Sample preparation

The cement design used in the experimental tests contained several additives that are typically used in cementing oil wells under the HPHT conditions such as retarders, fluid loss agents, and defoamers. Blank cement slurry formulation is given in Table 2.

The cement slurries were prepared and was conditioned in an atmospheric consistometer at 190˚ Fahrenheit temperature for 20 minutes [17]. Two methods of mixing are exist based on API instructions. The first method is dry mixing, in which all the additives are blended in cement (with the dry method). The second method is wet mixing, in which all the additives are mixed in water before being blended with cement [17]. Wet mixing causes a better dispersion of nanoparticles in cement slurry. In addition, it lowers blending time. That means the slurry samples can be prepared earlier compared to the dry mix. Plus, The operational equipment can adjust themselves with this method easily. In this paper, therefore, wet mixing was used. In fact, all components (except weighting agent and cement) mixed in water before blending with cement.

Firstly, cement, tap water, and additives were first weighed. Liquid and dry additives were mixed in fresh water at a low speed (4000 RPM) and then dry-blended (cement and weighting agent) were added to the mixture within 10 seconds. Then to reaching the uniform slurry, a high-speed mixture was run at a speed of 12000 RPM for 30 seconds. Finally, acid boric BWOC (1%) and nano- acid boric BWOC (0.5%, 1%, and 1.5%) were added to each cement slurry and then conditioned in an atmospheric consistometer at 190˚ F for 20 minutes. It is worth-mentioning that 190˚ F was the maximum accessible temperature based on operational limitations.

Table 2. Blank mud formulation (common operational formulation) used in tests

Additives	Function	Laboratory value
Water	Strength agent	260 cc
Cement G	-	803 gr
Retarder	Standard retarder	1 gr
Dispersant	Dispersant	23 gr
Defoamer	Anti-foaming agent	0.05 gr
Salt	Weighting agent	45 gr
FLC	Fluid loss control agent	60 gr
Hi-Dense	Weighting agent (S.G.=5.2)	240 gr

It should be mentioned that in this study, the broader range of nano-boric concentrations is not considered due to the high cost of nano-materials which are not applicable because of operational limitations.

Flowchart of experiments is presented in Figure 4.

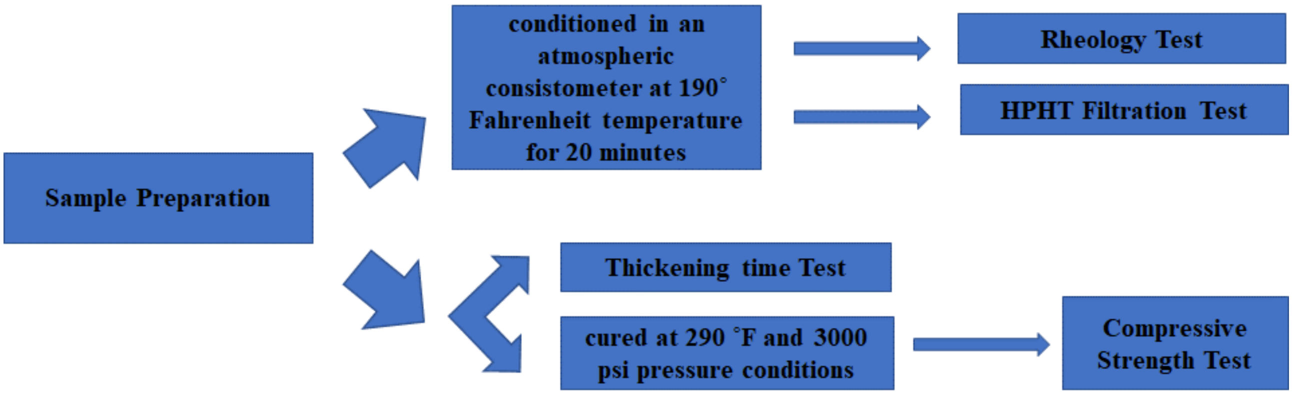

Figure 4. Flowchart of experiments

2.2.2. Experimental equipment

The tests for this article were performed using an HP/HT consistometer, atmospheric consistometer, ultrasonic cement analyzer, static fluid-loss apparatus, and viscometer.

2.2.3. Test procedure

The five mentioned cement slurries were prepared and tested with the ultra-sonic cement analyzer (UCA) apparatus, viscometer, and compressive strength (crushing) tester under high temperature (290 °F) and high-pressure (3000 Psi) for 24 and once in 48 hours. The type of used cement was class G due to common usage in oil/gas cementing operations. Also, the thickening time of the samples was determined using an HPHT consistometer.

1) Rheology test

Cement rheological properties are essential parameters that ensure pumpability of slurry and cement performance in cleaning mud cake on wellbore surface, etc. Using a rotational viscometer, plastic viscosity, yield point, and 10-second/10-minute gel strength were measured in downhole temperature conditions based on API standards. Firstly, the conditioned slurry was poured into a preheated cylinder at 190°F degrees. Then rotational viscometer was run in different shear rates as API standards. 10-second/10-minute gel strength were measured after keeping slurry fixed for 10 second and 10 minutes, respectively and then rotating it with 3 RPM.

2) HPHT Filtration test

The fluid should be placed at 200°F temperature in an atmospheric consistometer for 30 minutes; then the fluid is poured into a HPHT cell. Pressure is applied from the top in the HPHT filter press, and filtration volume will be read after 30 minutes.

3) Thickening time

Thickening time is the measured time that slurry can be handled without risk of stuck pipe or other risks in downhole conditions [10]. The end of the thickening time is considered to be 50 or 70 BC (Bourdon consistency units as a dimensionless quantity) for most cementing operations.

4) Compressive strength test

An important role in determining the quality of cement is based on its compressive strength properties and its competence to withstand stresses. There are two ways for measuring compressive strength: A) the destructive (crushing) method and B) the non-destructive method [18]. In the non-destructive method, an ultrasonic cement analyzer (UCA) should be used for measuring compressive strength development under HPHT conditions. The cement slurry must be placed in the autoclave unit of the UCA with applying scheduled downhole temperature and pressure conditions. The UCA transfers sonic waves through the slurry cement to determine the evolution of compressive strength. As the compressive strength develops with time, the acoustic signal passes through the sample faster and shows that relationship between the development of compressive strength and time.

Another laboratory evaluation method for measuring the compressive strength of a set cement under HPHT conditions is the crushing strength test. The crushing compressive strength of the blocks cements was performed by crushing 2-in square cubes. The cement slurry was mixed and poured into the cube molds and cured at 290° F and 3000 psi pressure (based on condition of a common HPHT well) for 24 hours or more in the HPHT cell, as per API specifications [17]. At the final stage, the cubes were extracted from the molds and crushed in a uniaxial compression-testing machine to measure compressive strength.

3. Results and Discussion

3.1. Rheology and HPHT Fluid Loss parameters

Acid boric nano-particles do not cause any unwanted effects on other slurry properties. HPHT fluid loss at 200°F for cement slurry having 1% boric acid was 48.5 mL, whereas cement slurries containing nano-boric acid retarder provided 48 to 46.5 ml fluid loss. Used retarder agents did not affect much on HPHT filtration properties; Although as it is shown in Table 3, adding more concentration of boric acid slightly reduced filtration volume, and it can be explained by changes in hydration of cement particles and its reaction with water molecules. Filtration measurements are presented in Table 5.

Table 3. HPHT filtration test results (1000 psi and 200°F)

Sample No.	Sample formulation	HPHT Fl volume (cc)/30 min
1	Blank Sample	53
2	Blank Sample + 1% boric acid	48.5
3	Blank Sample +0.5% nano- boric acid	48
4	Blank Sample +1% nano- boric acid	47
5	Blank Sample +1.5% nano- boric acid	46.5

Rheological measurements were performed immediately after mixing the slurry. The slurry was conditioned at atmospheric consistometer (190°F for 20 minutes). Rheological measurements (performed at 190°F) are shown in Table 4.

Table 4. The properties of rheological parameters

Sample No.	Temperature (°F)	PV	YP	10 Sec Gel	10 Min Gel
1	190	135	19	2.5	79
2	190	133	20	2	73
3	190	136	19	2	71
4	190	138	20	2	69
5	190	139	21	2	67

As shown in Table 4, adding retarders decreased 10-minutes gel strength due to slowing down the hydration reactions of cement particles and slightly increased plastic viscosity of slurries due to more friction force between uniform particles of nano-retarder and cement particles. Also, 10 seconds gel strength and yield point remained without significant changes due to not changing in early thickening time and electrochemical forces, respectively.

3.2. Thickening time and Compressive strength

The thickening time results obtained for various sample concentrations in the cement slurry. The thickening time tests were performed using an HPHT consistometer at 290°F and 3000 psi. The thickening time for the blank cement sample (without retarder) happened to be 100 minutes, but it was

3:05 (hr:mm) for 1% BWOC of boric acid and 5:09 (hr:mm) for 1% BWOC of nano-boric acid. With increasing temperature, the hydration rate of cement increases and thickening time will be decreased.

The cement slurry starts to develop its compressive strength with time under HPHT conditions. The transition time between gaining a compressive strength of 50 psi and 500 psi is so crucial. It needs to be as small as possible to prevent extended WOC prior to the restart of drilling operation. In ultrasonic cement analyzer tests, rapid development of strength due to fast hydration at early ages for the blank sample was observed. For all mixes, this phase time approximately happens at the exact times. However, the 500-psi compressive strength, is achieved in 1% acid boric mix in 4:26 (hr:mm) as compared to 6:58 (hr:mm) for sample 5, and this is a significant improvement. Thickening time, 50 psi and 500 psi strength time of cement slurries are presented in Table 5 and also thickening times are compared schematically in Figure 5.

Table 5. Thickening time, 50 psi and 500-psi strength of cement slurries (different concentration of retarders)

Sample No.	Formulation + Retarder (BWOC %)	Unit	Thickening time (70 Bc)	50 psi-strength	500 psi-strength
1	Blank Sample	hr:mm	01:40	01:51	02:09
2	Blank Sample + 1% boric acid	hr:mm	03:05	03:30	04:26
3	Blank Sample +0.5% nano- boric acid	hr:mm	04:11	04:52	05:17
4	Blank Sample +1% nano- boric acid	hr:mm	05:09	05:41	06:12
5	Blank Sample +1.5% nano- boric acid	hr:mm	06:01	06:30	06:58

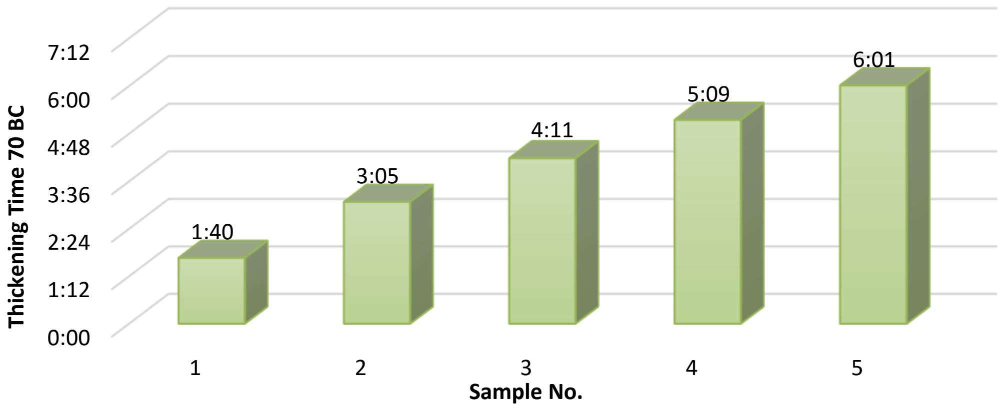

Figure 5. Comparison of thickening time for different cement slurries (different type and concentration of retarders)

The compressive strength of the slurries after 24 & 48 hours under HPHT conditions is shown in Table 6.

Table 6. Average compressive strength of cement aged for 24 and 48 hours at 3000 psi and 200°F

Sample No.	Sample formulation	Time	Average compressive strength (destructive) (psi)
1	Blank Sample	24 hr	1907
		48 hr	2050
2	Blank Sample + 1% boric acid	24 hr	2045
		48 hr	2159
3	Blank Sample +0.5% nano- boric acid	24 hr	2150
		48 hr	2290
4	Blank Sample +1% nano- boric acid	24 hr	2305
		48 hr	2489
5	Blank Sample +1.5% nano- boric acid	24 hr	2399
		48 hr	2501

Increasing nano-boric acid improved compressive strength. 1.5% BWOC nano-boric acid cement slurry provided the highest average compressive strength after 24 and 48 hours. Effect of Nano-boric acid and boric acid on compressive Strength of cement cured for 24 and 48 hours at 3000 psi and 200°F are given in Table 6. As it is demonstrated, nano-acid boric provides better strength in comparison with boric acid (in equal BWOC concentration.)

3.3. Chemistry Slurry Explanation

Enhancing properties of cement by nano-acid boric material can be explained by forming a more uniform microstructure with less porosity. Smaller and broader particle sizes of boric acid enhance the retardation effect on cement slurries. The mechanism of nano-boric acid for improving the properties of cement blocks, is its wide particle size and also its capability to fulfill the initial area between particles of the cement, which cause significantly lower porosity/permeability in cement slurry.

The SEM image of cement set samples (one including nano-boric acid as retarder and one without it) shows that some empty spaces between the cement particles in the cement set without retarder (Figure 6). But, nano-acid boric particles reduce the porosity of the cement structure by helping with constant hydration of cement particles (Figure 6) which makes the compressive strength of the cement higher compared to the blank case (formulation #1).

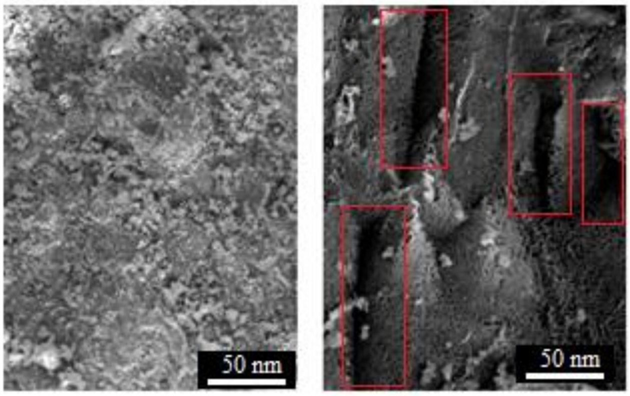

Figure 6. Cement set without retarder (in right) with highlighted unfilled pores and cement set (with 1% nano-boric acid) (in left) SEM images (50-nm scale)

Calcium hydroxide is the hydration product of cement that forms C-S-H, which is the main factor that develops strength gain of cementitious materials [19]. C-S-H primarily grows on the top layer of cement grains. The addition of nano-boric acid particles based on Nucleation and Adsorption mechanisms causes to change in hydration time and thickening quality of the cement slurry as it performs as nucleation places for C-S-H. This results in slowing down the cement water adsorption and modifying the microstructure of C-S-H for forming a more vital and more intact cement. Based on adsorption theory, the retarder becomes adsorbed on the surface of the hydration products, inhibiting its contact with water and turn it to a hydrophobic surface [12]. Also, Nucleation theory suggests that the retarder reduce the rate of cement hydration by adsorbing on cement particles' nuclei [14]. In fact, the retarder prevents the growth of the formed highly reactive nano-nuclei of cement by fulfilling their reactive areas.

In this paper, based on test results, adding nano-boric acid changed cement hydaration, decreased porosity of set cement (Figure 6) and improved resistance of cement compressive strength under HPHT conditions. More specifically, nano-boric acid improved physical resistance of set cement by changing microstructures of cement in nano and micro scale (Figure 6). Plugging micro-pores and nano-pores by adding nano-boric acid particles can be named as main factor of low-degradation of cement compressive strength. Also, an increase in the thickening time was obvious in cement slurry. As conclusion, nano-boric acid is applicable for cementing operations in HPHT environments such as depp wells.

4. Conclusion

- The drilling industry moving toward using multi-functional additives in drilling and cementing fluids, so more effective multi-functioning retarding agents can play an important role in cementing operations.
- This study is aimed at obtaining the nano-boric acid effects in oil well cement slurry designs. Three different concentrations of nano-boric acid (0.5, 1, and 1.5% wt./wt.) and one concentration of boric acid (1% wt./wt.) were used as retarding agents. The effect of different concentrations of nano-boric acid on thickening time and strength development was evaluated.
- Cement slurry was prepared using four different concentrations of boric acid/nano-boric acid as retarders. Thickening time tests were performed in an HP/HT consistometer at 200°F and 3000 psi. Also, Compressive strength was measured using a UCA at 290°F and 3,000 psi.
- As the concentration of nano-boric acid was increased, the long-term compressive properties improved.
- 1.5% BWOC nano-boric acid cement slurry provided the highest average compressive strength after 24 and 48 hours.
- Applying 1% of nano-boric acid improved the thickening time for 85 minutes (approximately 210 % increase comparing blank sample). Also, adding 1% nano-boric acid to cement slurry improved 24-hr average compressive strength from 1907 psi to 2305 psi (398 psi improvemnet which is more than 20% increase compared to blank sample).
- Smaller size and having a high surface area make nano-acid boric particles capable of improving the properties of cement slurries. Mechanisms of nano-boric acid for retardation effect on cement slurries are Nucleation and Adsorption, simultaneously.

Nomenclature

API	American petroleum institute
BWOC	By weight of cement
BHCT	Bottom hole circulation temperature
CSH	Calcium silicate hydrate
HPHT	High pressure, high temperature
PV	Plastic viscosity
RPM	Rotation per minute
UCA	Ultrasonic cement analyzer
WOC	Weight on cement
YP	Yield point

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