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## Investigation of the Effect of Nano Zeolite on The Rheological and Mechanical Properties of Heavy Weight Cement Slurry for Drilling Wells in Iranian Southern Oil Field

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### Highlights

- Impact of nano zeolite on rheological and mechanical properties of heavy cement slurry
- The effect of nano zeolite on the compressive strength of heavy cement slurry
- The effect of nano zeolite on the thickening time of heavy cement slurry

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### Abstract

Oil well cementing is a multi-purpose operation, in which cement slurries are prepared by mixing water, cement and various additives and is pumped into the well in order to isolate productive zones, protect the casing pipe, perform remedial operations, controlling drilling fluid lost or abandon the well. Various additives are used to improve the mechanical properties of the slurry, like cement retarders and accelerators which increase and decrease the thickening time of the cement slurry, respectively. Weight-enhancing additives are materials with specific gravity higher than cement, which can weight-up the slurry to overcome the hydrostatic pressure of mud and performing a good cementing job. Improving the mechanical properties of these type of cement slurries has always been an important issue in the discussion of oil wells cementing. In this study, the effects of nano zeolite on heavy-weight oil well cement slurry were investigated in laboratory to improve the rheological and mechanical properties of the cement. In the designed experiments, nano-zeolite was added to the slurry with the amount of 1, 2 and 3% BWOC (By Weight of Cement). The results showed that nano zeolite acts as an additive to reduce the thickening time, increase the plastic viscosity and reduces the yield point of the slurry. So, it should be noted to adjust the pumping time of the cement slurry by using other additives based on the required cementing job timing schedule. The experiments also showed that in general, the addition of nano-zeolite to the cement slurry from 1 to 3% BWOC led to an increase in the free fluid of the cement slurry, but did not show any effect on the control of the fluid loss. Finally, by adding 2% BWOC of nano zeolite, the compressive strength of the cement stone increased and the initial setting time of cement slurry decreased

**Keywords:** Casing pipe, High weight cement slurry, Nano zeolite, Oil well cementing.

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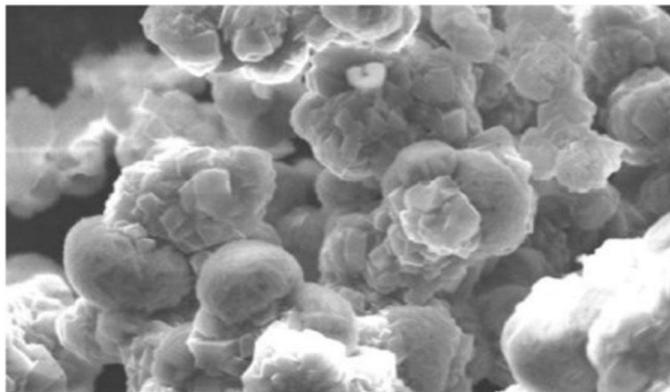
## 1. Introduction

Cementing is one of the important operations in oil and gas well drilling. The quality of cement behind the casing plays a vital role in providing support for the casing and zonal isolation between the casing and the cement and between the cement and the formation during drilling and has a serious impact on secondary cementing, well remedial cementing and well stimulation. Remedial cementing operations will be performed to repair primary-cementing problems or to treat conditions revealed after the wellbore has been produced for a while. The two mostly known categories of remedial cementing are squeeze cementing and cement plugs. Squeezing cement slurry in the weak formations by setting a packer in the well can control the mud lost circulation during drilling a well. Good cementing behind the casing can isolate the productive zones of a well and decrease the risk of kicking formation fluid into the well. However, low quality cement slurry may lead to re-cementing and increase the time and cost of the drilling operation. Quality of the primary cementing job performed, may be characterized by the cement bond quality logs such as CBL/VDL. The composition of a cement slurry should be designed before pumping into the well according to the depth (temperature), formation type and mud weight in the well, and the best cement properties should be achieved by applying appropriate additives in the slurry and it will be assessed by special experimental analysis. Nanotechnology has an improving potential to bring about various changes in several areas of the oil and gas industry, such as exploration, drilling, cementing, operation, EOR, and so on. Nanoparticles, especially silica, iron and aluminum nanoparticles have been widely used to increase the compressive and flexural strength of Portland cements (Shahrudin et al. 1993, Hodne.2007). Increasing the quality and strength of drilling cements has always been of great importance in the oil industry. For this reason, many researches are being done in this direction in the world and in our country. In the cement industry, special additives are used to improve the rheological and mechanical properties of the slurry. Every additive of drilling cement has some side-effects one of these effects is the cement compressive strength alteration. One of the special cement slurries for an oil well is high weight cement. Increasing the strength of these slurries has always been an important topic in the discussion of cementing oil wells. Global demand for excess energy is predicted to increase in the coming decades. The oil industry itself is undergoing a fundamental change, as ordinary oil is on the verge of extinction. The oil and gas industry are beginning to explore challenging areas that show high economic risks as well as technical problems (Ridha et al.2010, Pang et al.2013). Drilling and producing oil from oil wells is becoming a costly task. Researchers around the world are trying to find ways to reduce the cost of each drilling operation, thus trying to optimize the overall cost of the well. Cementing is one of these operations and is an asset of the well structure in the reservoir. Cementing oil and gas wells requires materials that not only meet performance standards but are also quite different from those found in conventional cementing. The search for alternative additives for cementing oil wells has been increasing worldwide. In recent years, many studies have been performed to improve the mechanical and rheological properties of cement used for cementing oil and gas wells using additives or various cement classes. We selected a nano-material as an additive to achieve some of these positive effects in the oil well cement. It is desirable that these alternative materials strengthen the cement sheath resistance, durability, flexibility, Plasticity, cost-effectiveness. The primary objectives of cementing operations are to support and hold the casing pipe, to prevent corrosion by the formation fluids (Abbas et al.2020, Mintova et al.2016). Novriansyah et al. (2016) used Palm Shell Carbon (PSC) nanocomposite to increase the strength of cement for well cementing operations. Cement slurry with high compressive strength and shear bond strength can be a good protector for casing pipes. The results of their researches showed that the addition of PSC can increase the strength of cement slurry compared to samples without PSC. In a study, Soltanian.H (2013) investigated design of ultra-lightweight cement slurries using nanotechnology. Lightweight slurries have lower compressive strength and separation probability and, in this research, with the help of HSL nanoparticles (Hydrophilic fumed silica, Specific Surface Area: 175-225 m<sup>2</sup>/g), microspheres, G-class cement and using particle size distribution method, in addition to lightening cement slurries up to 62 pcf, various lightweight slurries with the expected properties at the actual conditions of the well were

designed. The results showed that it is less costly compared to conventional systems and also causes a rapid increase in the compressive strength of cement. The wait-on-cement time for the resumption of operations is reduced to less than 8 hours for a weight of 8.34 PPG. Ahmadi et al. (2017) in a study designed an optimal cement slurry that has rheological properties, compressive strength and very good stability for use in horizontal sections of oil and gas wells using nanotechnology. Optimal cement slurry may be designed by correct adjusting additive dosages and water/cement ratio and selecting suitable cement class. The results of their research showed that 90 pcf cement slurry using nanoparticles has less permeability and this property can minimize the possibility of fluid penetration into the cement rock from formations containing gas or salt water. Also, the amount of free water in all their designed slurries that used nanotechnology and particle size distribution was equal to zero and the slurry stability profile indicated the flat state in its stability curve. In the stability curve, specific gravity of different sections of the cylindrical cement molds are plotted versus the sections sequence. ariq et al. (2020) have investigated the combined effect of nanosilica and nano-clay on the mechanical properties of Class G cement. Class G cement is the basic cement without any added additive in the factory; so it is the most stable and durable type of cement for experimental studies. The results of their study showed that the addition of silica and clay nanoparticles increases the tensile and compressive strength of Class G cement under Compression and temperature rise. Eric Bruni et al. (2021) in a study evaluated the performance of fresh nano zeolite as an additive for shallow oil well cementing operations. The results of their research showed that increasing the concentrations of nano zeolite leads to a decrease in rheological values. Also, the values of plastic viscosity have generally increased with increasing the concentration of fresh nano zeolites. This nanoparticle had no effect on free fluids. Kianfar (2019) worked on the synthesis and application of nanozeolite in a study. The results of his study showed that the most effective and widely used methods for the synthesis of zeolite nanotubes is the use of clear primary solutions and colloidal suspensions. These colloidal suspensions are stable and the dispersed zeolites do not settle for a longer period of time. Colloidal crystals give unparalleled purity to the structures and increase their applications in pharmaceutical, chemical and optoelectronic applications by increasing the contact surface with zeolite. By properly distributing the pore size in the inert matrix, the size of the crystal zeolite can be controlled without gel dependence. In short, advances in nanozeolite science require multidisciplinary skills (synthesis and processing of materials, accurate understanding of their chemical, physical, thermal and optical properties). Ahmad et al. (2020) studied a work on Barite and Hematite as a weighting additive in oil well cement slurry. The results of their study showed that the addition of barite improved the rheological properties of cement slurry and also reduced the plastic viscosity of the slurry. Adding hematite to cement slurry in deep gas wells also controls gas migration through the cement. If gas channeled into the cement sheath, negative hydraulic test (dry test) identify bubble at the surface. Kharat et al. (2019) in a study on the chemical modification of lignite and its properties in fluid Loss controlling of well cement slurries. This study showed that lignite and its analogues can be used due to the presence of lateral hydroxyl groups and can be used as a suitable additive for high density cement slurries that are able to operate in high temperature and pressure conditions. By adding lignite, the compressive strength of psi800-1100, thickening time of 400 minutes, and viscosity of 25 cp, a smooth drop below 130 ml API was achieved. The effect of some nanomaterials such as carbon nanotubes, nanocellulose, nanocomposites, nanoclay, nanozinc oxide in drilling cement has been investigated. The use of nanozeolite in drilling cement has not been clearly identified in research, but there are articles on the effect of this material in improving the strength of construction cements. Because in heavy weight drilling cements (above 130 pcf) there are always problems due to the strength of cement sheath and the initial setting time, it is very important to investigate the effect of this material in high weight cements. In this research, we investigated the use of nanoparticles of grade A, 100 nm nanozeolite powder made by the American company on the properties of high weight cement slurry.

## 2. Introduction of nanozeolite

The structure of nanozeolite is divided into two parts: melting columns, which include rings that expand with heat and areas of the inner column, which shrink in heat. In the structure of a natural nanozeolite, water and cations can be reversed or replaced with other cations. Most nanozeolite materials are white. However, some of them, which contain small amounts of iron, appear pale yellow or reddish brown. In general, the sediment type of a particular zeolite is more stable than volcanic acid because it usually contains more Si. In general, nanozeolite is characterized by the following characteristics: high degree of hydration during discharge, low density and large empty space in the crystal structure. (Muzzammil.et al .2018)



**Figure 1**

Nanozeolite particles in SEM

**Table 1**  
Specifications of Nanozeolite used.

<b>SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub></b>	40
<b>Bulk Density</b>	1.18 gr/cc
<b>Specific Surface Area</b>	300 m <sup>3</sup> /gr
<b>Color</b>	Whitish Gray
<b>Appearance</b>	Powder
<b>Crystal Size</b>	0.5 μm
<b>Manufacturer</b>	Nanoshell-USA



**Figure 2**

Nanozeolite particles

### 3. Research methods

In this study, experiments were performed with Class G cement slurry mixed with weighting additive along with different concentrations of nanozeolite at a temperature of 150 F. Cement slurry was prepared in accordance with API 10A specifications. Samples were labeled as S-0, S-1, S-2 and S-3 depending on different concentrations of nanozeolite (1%, 2% and 3% BWOC). We used Hidense as a weighting agent to weight up cement slurry. Because Hidense is a chemically neutral additive, it is used in the slurry as the form of cement-dry-blended. Then the physical properties of cement such as compressive strength, thickening time, free fluid, fluid loss and rheology were determined by strictly observing API 10A and API 10B.

#### 3.1. Preparation of cement slurries

First, we have to make a cement slurry without nanozeolite and measure its physical properties to be the basis for evaluating the desired additive and then we will compare the properties of other slurries containing nanozeolite with this slurry. These experiments were performed under the test conditions presented in Table 2 and with the slurry compositions expressed in Tables 3-6.

**Table 2**  
Test conditions

<b>Temperature (F)</b>	150
<b>Pressure (psi)</b>	1000
<b>Slurry weight (pcf)</b>	145

**Table 3**  
S-0 slurry compositions

<b>Ingredient</b>	<b>Percentage of additives (%)</b>	<b>Laboratory number of materials</b>
Class G cement of Abadeh company	-	611.4 gr
Weighting agent (HIDENSE)	63.7 BWOC*†	389.60 gr
Salt	35 BWOW**‡	102.21gr
Dispersant (CFR-8)	0.4 BWOC	2.45 gr
Distilled water	-	288.6 cc

**Table 4**  
S-1 slurry compositions

<b>Ingredient</b>	<b>Percentage of additives (%)</b>	<b>Laboratory number of materials</b>
Class G cement of Abadeh company	-	611.4 gr
Weighting agent (HIDENSE)	63.7 BWOC	389.6 gr
‡FNZ*	1	6.11 gr
Salt	35 BWOW	101.2 gr

† By weight of cement

‡ By weight of water

§ FRESH NANO ZEOLITE

Dispersant (CFR-8)	0.4 BWOC	2.45 gr
Distilled water	-	283.7 cc

**Table 5**  
S-2 slurry compositions

Ingredient	Percentage of additives (%)	Laboratory number of materials
Class G cement of Abadeh company	-	610.7 gr
Weighting agent (HIDENSE)	63.7 BWOC	389.2 gr
FNZ*	2	12.21 gr
Salt	35 BWOW	101 gr
Dispersant (CFR-8)	0.4 BWOC	2.44 gr
Distilled water	-	278.9 cc

**Table 6**  
S-3 slurry compositions

Ingredient	Percentage of additives (%)	Laboratory number of materials
Class G cement of Abadeh company	-	610 gr
Weighting agent (HIDENSE)	63.7 BWOC	388.7 gr
FNZ*	3	18.3 gr
Salt	35 BWOW	100.9 gr
Dispersant (CFR-8)	0.4 BWOC	2.44 gr
Distilled water	-	274 cc

#### 4. Evaluation of the results of slurry thickening time

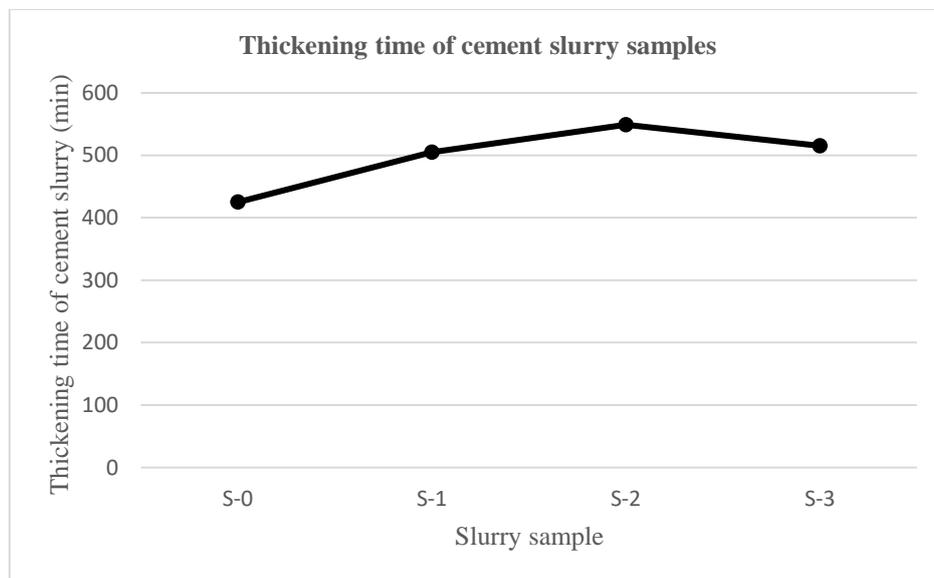
The resulting slurries are transferred to the cement Consistometer at a pressure of 1000 psi and a temperature of 150 F to achieve thickening time of the slurry. This test indicates how long the cement slurry can be pumped on the conditions encountered by the slurry in the well. This test is completed when the cement slurry reaches a consistency of 100 BC. It will be suitable if the cement slurry stays pumpable for a desired time and gel up when the flow stops at the appropriate time interval. Table 7 summarizes the results of the thickening time of all cement slurry samples.

**Table 7**  
consistency of cement slurry samples HPHT Consistometer

Slurry sample	heat up time (minutes)	start (BC)	30 BC	40 BC	50 BC	70 BC	100 BC
			hh:mm				
S-0	40	15	6:00	6:15	6:31	7:05	7:14
S-1	40	13	6:50	7:38	8:02	8:25	7:14
S-2	40	14	6:38	6:47	7:24	9:09	n/a

S-3	40	15	6:54	8:00	8:26	8:35	n/a
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It can be seen from Table 7 that the initial viscosity of the cement slurry decreases with increasing concentration of fresh nanozeolite compared to the base cement slurry. There was no significant difference in the initial viscosities of all samples mixed with fresh nanozeolite from 1 % BWOC to 3 % BWOC. Increasing the concentration of fresh nanozeolite at 150 F increased the setting time of all cement slurries. These results show that nanozeolite has a retarding effect in cement slurry and is therefore not suitable for shallow well cementing operations because operators must be waited a long time on the cement to get hard. At the end of the test, the thickening time of 70 BC was considered as the point of cement slurry that becomes non-pumpable. Figure 3 shows the results obtained from the thickening time of the slurries at 70 BC.



**Figure 3**  
thickening time of cement slurries

**4.1. Evaluation of slurry rheology results**

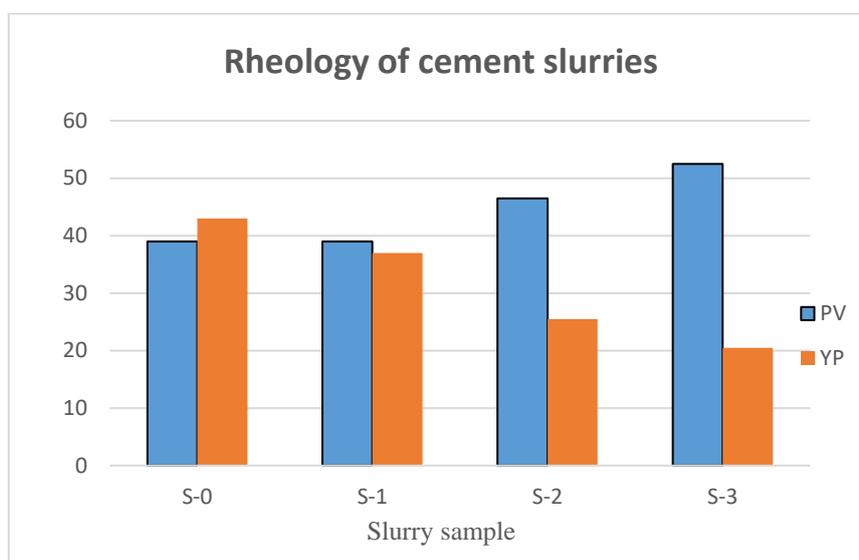
Rheological properties are obtained from direct readings using a rotary viscometer and converted to viscosity plastic (PV) and yield point (YP). The rheology of cement slurries is crucial in the proper displacing of drilling mud to ensure a good cementing operation. This property also has an effect on settling and frictional pressure of the cement slurry. The prepared slurries were cured at 150 F for 30 minutes using an atmospheric Consistometer, then was quickly poured into the Consistometer cup until the specified line and dial readings were recorded at the specified speeds. Table 8 presents the rheological properties of cement slurries mixed with different concentrations of fresh nanozeolite at 150 F and atmospheric pressure.

**Table 8**  
Rheology of slurries in rotational viscometer

Experimental factors	Dial reading (θ)			
	S-0	S-1	S-2	S-3
rpm600	135	122	112	95
rpm300	82	76	72	73

rpm200	70	64	54	53
rpm100	56	50	41	38
rpm6	24	19	17	17
rpm3	16	14	12	11
PV (cP)	39	39	46.5	52.5
YP (lb./100ft <sup>2</sup> )	43	37	25.5	20.5
PV/YP	0.9	1.1	1.8	2.6

The values of plastic viscosity and yield point of the slurries can be checked in a diagram shown in Figure 4 which shows that adding nanozeolite to the heavy weight cement slurry increases the plastic viscosity and decreases the yield point. The lower the YP values, the smoother the cement slurry and the tendency for it to settle. The results showed that the calculated YP values of all cement slurries could be pumped at 150 F because all yield values were higher than 15 lb/100ft<sup>2</sup>.



**Figure 4**

Rheology diagram of cement slurries

#### 4.2. Evaluation of slurry free fluid results

When the cement is gelling, the free liquid separates from the cement slurry and is placed on top of the cement column or in small confined annulus if the well is deviated. This free fluid can create channels when moving toward the top of the cement, weakening the cement bond and also damaging the wall if trapped water is located between the casing wall. Therefore, it is expected that for an ideal cementing operation, the free fluid must be as low as possible and close to zero if necessary. The results of free fluid of all cement slurries at 150 F are presented in Table 9. Table 9 shows that in 1%BWOC (S-1), the percentage of free fluid recorded was 1.56 cc. However, increasing the concentration of nanozeolite by 2 %BWOC and 3 %BWOC (S-2 and S-3) leads to a decreasing trend. In general, the addition of nanozeolite from 1 %BWOC to 3 %BWOC to the base cement slurry increased the free fluid of the cement slurry.

**Table 9**  
Free liquid of cement slurries at a temperature of 150 F

SLURRY SAMPLE	FREE LIQUID (%)	MEASURED VOLUME (ML)
S-0	0.8	2
S-1	1.56	3.9
S-2	1.08	2.7
S-3	0.92	2.3

**4.3. Evaluation of slurry fluid loss results**

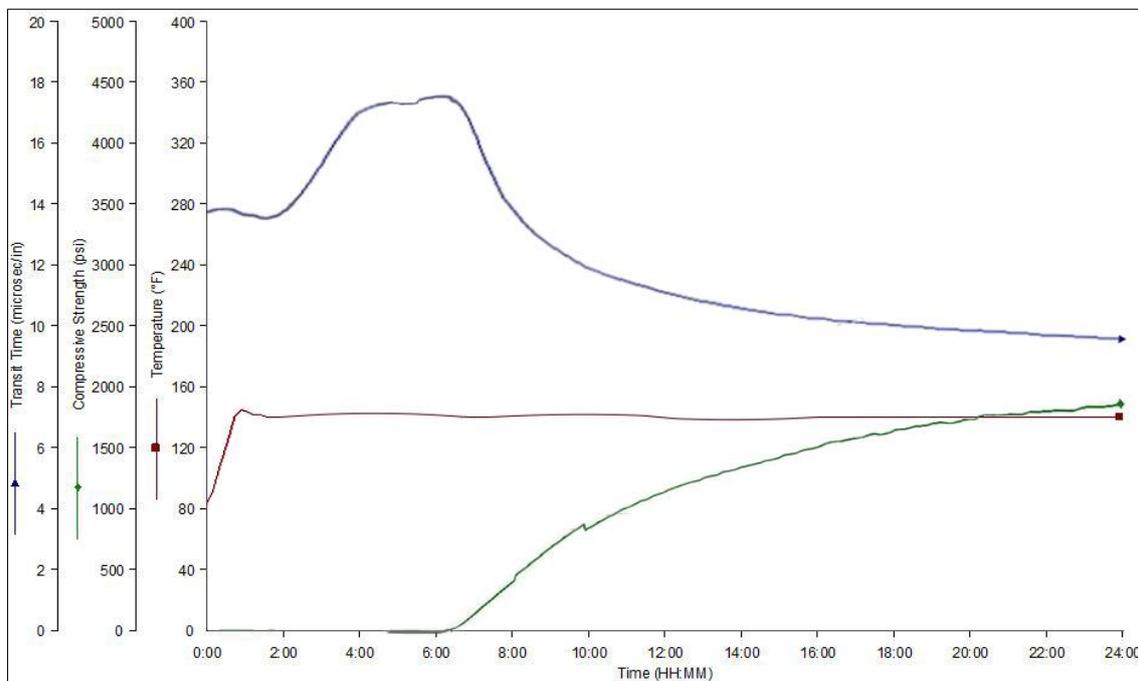
Table 10 clearly shows that with the introduction of nano-zeolite in the base cement slurry, a large volume of the slurry aqueous phase is lost. This amount of fluid loss was observed within seconds of starting the experiment. This indicates that nano-zeolite does not exhibit the properties of a fluid loss controller additive.

**Table 10**  
fluid loss of cement slurries at 150 F

SLURRY SAMPLE	FLUID LOSS (CC/30MIN)	TEST DURATION (SEC)	MEASURED VOLUME (ML)
S-0	696	45	55
S-1	581	90	65
S-2	-	-	-
S-3	-	-	-

**4.4. Evaluation of compressive strength results**

compressive strength analysis is crucial to determine the integrity and long-term bearing capacity of cement. Insufficient development of compressive strength may lead to casing damages and consequently reduce well life. Sample graphs obtained from the UCA test are presented in Figure 5-8.



**Figure 5**

S-0 slurry compressive strength diagram

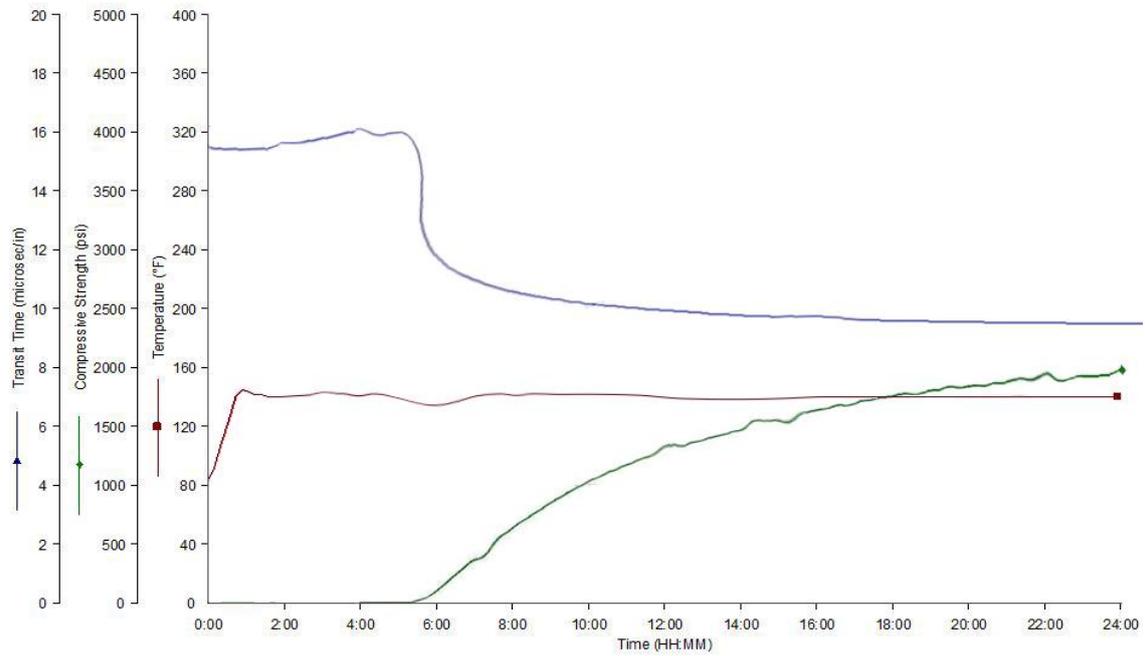


Figure 6

S-1 slurry compressive strength diagram

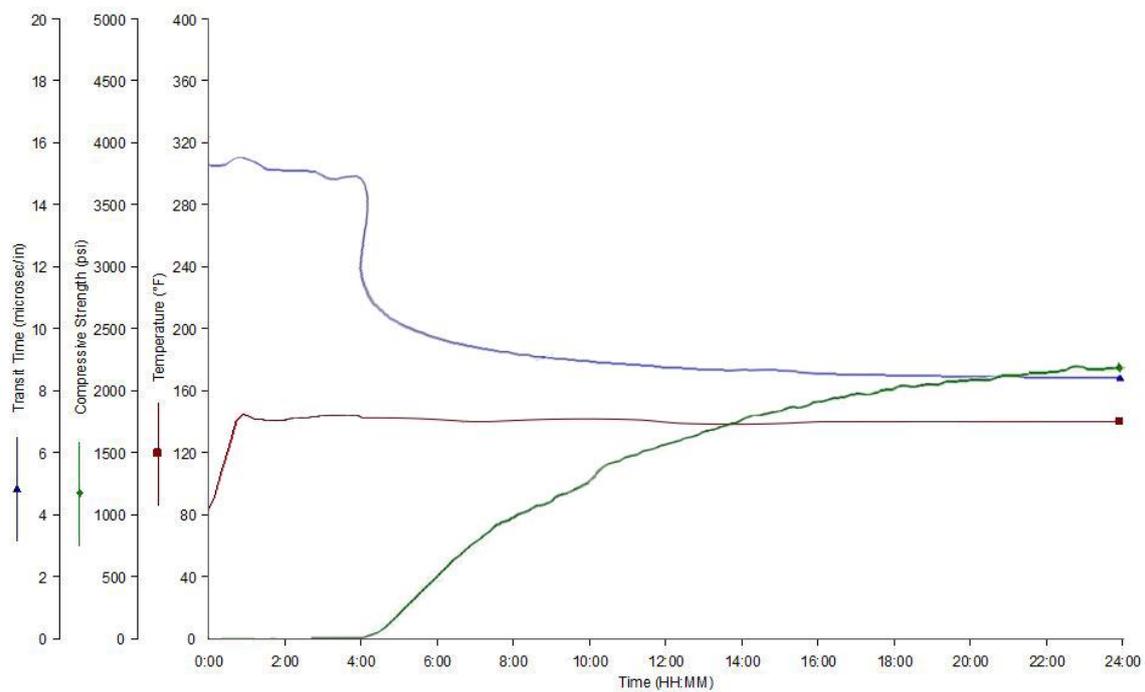
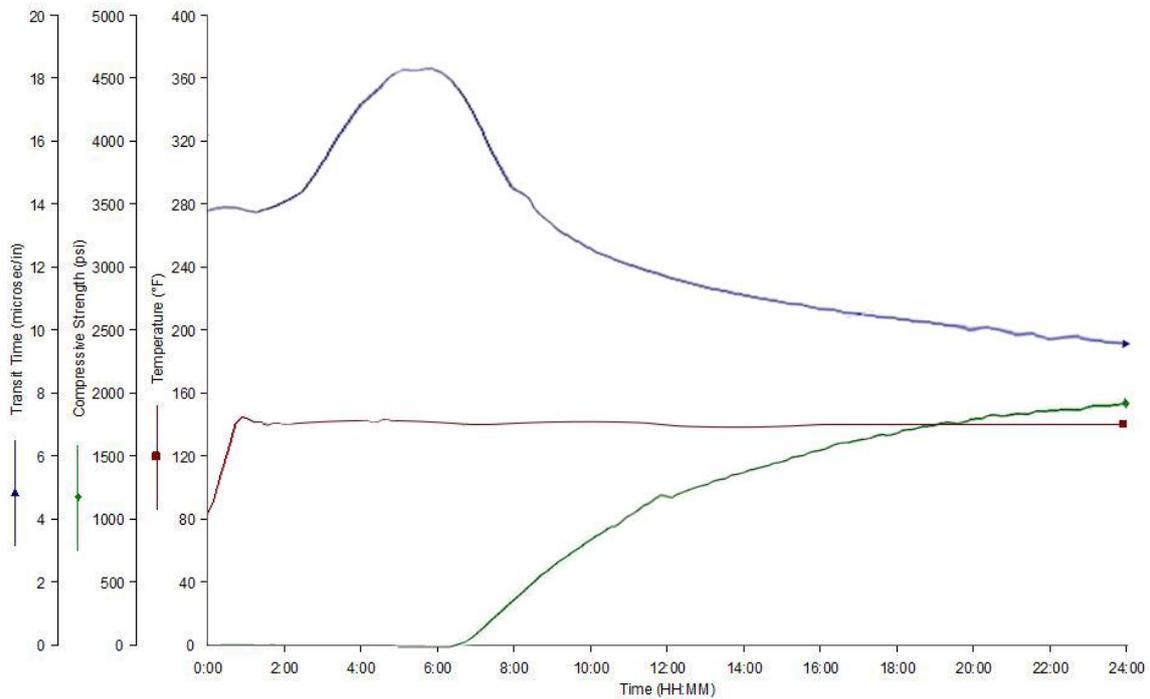


Figure 7

S-2 slurry compressive strength diagram



**Figure 8**

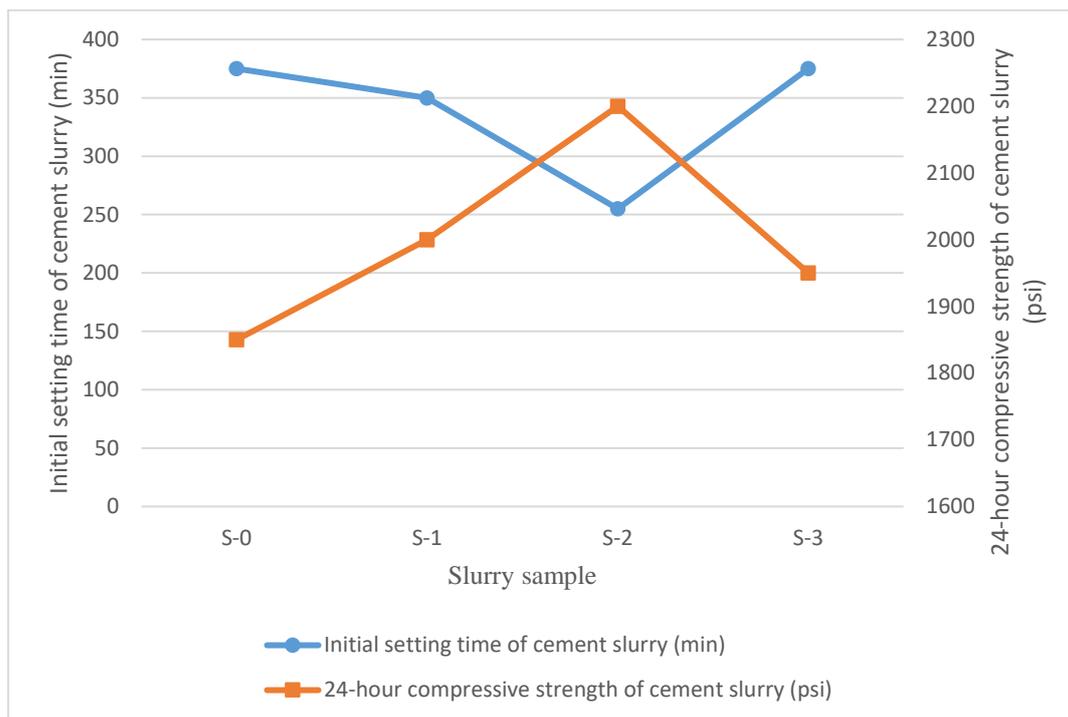
S-3 slurry compressive strength diagram

In order to compare the compressive strength of the slurries with each other, the initial setting times and 24-hour strength were considered. The initial setting value is the initial time required to reach the cement slurry strength of 50 psi. According to the UCA diagrams for the slurries, the initial setting time and the 24-hour compressive strength of the cement samples were gathered in Table-11.

**Table 11**

Comparison of initial setting time and 24-hour compressive strength of the slurries

SLURRY SAMPLE	Initial setting time of cement slurry (min)	24-hour compressive strength of cement slurry (psi)
S-0	375	1850
S-1	350	2000
S-2	255	2200
S-3	375	1950



**Figure 9**

Initial setting time and 24-hour compressive strength of slurries

The highest compressive strength of Class G heavy weight cement rock containing nanozeolite (2200 psi) is related to S-2 slurry in which the amount of nanozeolite was 2 %BWOC. Cement with higher compressive strength generally has a lower porosity and enhanced durability. Also, the shortest initial setting time of Class G heavy weight cement containing nanozeolite was obtained for S-2 slurry. The time needed to reach a specific compressive strength in UCA analyzing curve is an essential characteristic for reducing the Wait on Cement (WOC) time and gas channeling through cement sheath.

## 5. Conclusion

In this study, the effect of fresh nanozeolite particles on the rheological-mechanical properties of high weight oil well cement slurry was investigated. For this purpose, fresh nanozeolite were prepared and various tests including rheological properties, initial setting time, thickening time, free fluid, fluid loss and compressive strength of cement were performed. The obtained results were compared to each other and then the effects of this additive were well defined and the best amount of use in high-weight cement slurry was determined, the results are as follows:

- 1-Increasing the concentration of fresh nanozeolite at 150 F, increased the thickening time of all cement slurries. This indicates that fresh nanozeolite has a retarding effect on cement slurry.
- 2- Increasing the concentration of fresh nanozeolite reduces the rheological values, which is a characteristic of dispersive additives.
- 3- Plastic viscosity in general showed an increasing trend with increasing concentration of fresh nanozeolite. Plastic viscosity of all cement slurries was below 100 cp and therefore pumpable.
- 4- Fresh nanozeolite has no effect on fluid loss of the cement slurry.
- 5- In general, adding fresh nano zeolite from 1% BWOC to 3% BWOC to the base cement slurry increases the free fluid of the cement slurry.

6- The highest compressive strength of cement stone was related to the slurry containing 2 %BWOC nanozeolite. Also, the shortest initial setting time of Class G heavy weight cement slurry containing nano-zeolite was obtained for the same slurry.

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