Effects of Fresh Nano Zeolite on the Physical Properties of Oil Well Cement Slurry at High Temperature*

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Abstract

There are several types of additives that have been used to improve upon the physical properties of cement slurries at high temperatures. The search for unconventional materials for cementing of oil and gas wells at high temperatures has increased worldwide. It is expected that the unconventional materials make oil well cement withstand adverse temperatures, pressures, and other adverse wellbore problems without altering the intended purpose of the cement. This research seeks to evaluate the effects of fresh nano zeolite on the physical properties of oil well cement slurry at Bottomhole Circulating Temperature of 150 ºF (66 ºC). Experiments were conducted on a base cement slurry mixed with varying concentrations of fresh nano zeolite from 1% to 3% by weight of cement (bwoc) to determine the physical properties such as compressive strength, thickening time, rheology, free fluid and fluid loss. The physical properties were determined based on American Petroleum Institute standards for testing cements. Test results showed that compressive strength was improved by the addition of fresh nano zeolite at 2% and 3% bwoc with the exception of 1% bwoc. An increase in fresh nano zeolite concentrations resulted in an increase in the thickening time of the cement slurries and thus exhibited a high retardation effect. Plastic Viscosity (PV) increased from base cement slurry when 1% and 3% bwoc were added. However, the addition of 2% bwoc of fresh nano zeolite resulted in a decreased PV as compared to the base cement slurry. Yield Point (YP) decreased as fresh nano zeolite concentrations were increased. Increase in concentrations of fresh nano zeolites resulted in an increasing trend in PV/YP values for all the cement slurries. The addition of fresh nano zeolite to the base cement slurry did not cause any free fluid separation and also did not improve upon the amount of filtrate lost.

Keywords: Compressive Strength, Fluid Loss, Free Fluid, Thickening Time, Zeolite

1 Introduction

Cementing is considered a critical operation, not only during drilling but for all remaining period of production. Performance of a drilled well in terms of production is dependent on a successful cementing job, which prevents any fluid migration in the well, provides a good seal between the formation and casing, protects casing from corrosion and provides support to it (Calvert *et al.*, 1990). To ensure efficient performance of oil well cement slurry requires that the best cement additives are used. Generally, additives are expected to make the cement withstand adverse temperatures, pressures, and other adverse wellbore problems without altering the intended purpose of the cement.

Currently, there are several types of conventional additives that are available for use at high temperatures during oil well cementing operation. Notwithstanding, there are investigations into other materials that can alternatively perform a similar function as conventional additives at low cost. The use of Supplementary Cementitious Materials (SCMs) has over the last few decades received a lot of attention as materials with the potentials for oil well cementing operations. These SCMs include a large number of industrial and naturally occurring materials such as fly ash, volcanic ash, ground granulated blast furnace slag, silica fume, zeolite, diatomaceous earth, rice husk ash and the like. A number of studies have investigated the use SCMs such as fly ash, silica fume either in conventional Portland cement paste or slurries (Vikan and Justnes, 2003; Zhang and Han, 2000; Sybert and Reick 1990; White *et al*., 1985). Not much attention has been given to the use of fresh nano zeolite as a possible oil well cement additive at both low and high temperatures which according to Ahmadi and Shekarchi (2010), has excellent SCM. The literature on the use of zeolite in oil well cementing indicates the possibility of using zeolite in cementing operations.

Poon *et al*. (1999) studied the hydration rate of natural zeolite blended cement pastes. It was concluded that zeolite is a pozzolanic material with reactivity in between silica fume and fly ash. Zeolite replacement reduced the porosity but at high replacement around 25%, it increased the porosity. Moreover, it was observed that the porosity of the sample had a decreasing trend with the curing age, which can be attributed to the increase in pozzolanic reaction with time. A good correlation was seen between porosity and compressive strength development. Luke *et al*. (2004) demonstrated that zeolite could be an effective foam-cement stability agent. It was revealed that zeolite in cement slurries acts as a good settling agent, friction reducer and better fluid loss control. It was concluded that zeolites having lower mean particle size showed

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improved rate of early compressive strength. Fyten *et al.* (2005) also reported the effectiveness of zeolite in economically reducing Equivalent Circulating Densities (ECD) while keeping the strength values adequate and no free fluid development. Zeolite slurry was observed to be thermally stable and having good bonding abilities with the casing and formation.

There is still little information in open literature regarding the performance of fresh nano zeolite in oil well cement slurry at high temperature. This research seeks to evaluate the effect of fresh nano zeolite on the physical properties of oil well cement slurry at a high temperature. This will help operators to know the performance of fresh nano zeolite as an alternative material or additive for oil and gas well cementing at high temperature environment.

All zeolites are composed of an elementary structure of an aluminosilicate framework which comprises of a tetrahedral arrangement of silicon cations (Si^{4+}) and aluminium cations (Al^{3+}) that are surrounded by four oxygen anions (O^{2-}). Each oxygen ion within the Si-O and Al-O bonds connects two cations and is shared between two tetrahedrons, thus yielding a macromolecular three-dimensional framework of SiO_2 and AlO_2 tetrahedral building blocks. In this arrangement of atoms, each tetrahedron consists of four O atoms surrounding a Si, resulting in a three-dimensional structure of silicate tetrahedra with a Si:O ratio of 1:2 (Armbruster *et al.*, 2001). Some Si^{4+} ions are substituted by Al^{3+} ions, resulting in a net negative charge in the tectosilicate framework (Fig. 1). This charge arises from the difference in formal valency between the $(AlO_4)^{5-}$ and $(SiO_4)^{4-}$ tetrahedrons and is normally located on one of the oxygen anions connected to an aluminium cation. The resulting negative charges are balanced by counterions which are usually alkaline or alkaline earth metals, such as Na^+, K^+ or Ca^{2+} in most cases. Li^+, Mg^{2+}, Sr^{2+} and Ba^{2+} are also found in some zeolites. These ions are found on the external surface of zeolite, bound with the aluminosilicate structure by weaker electrostatic bonds (Armbruster *et al.*, 2001).

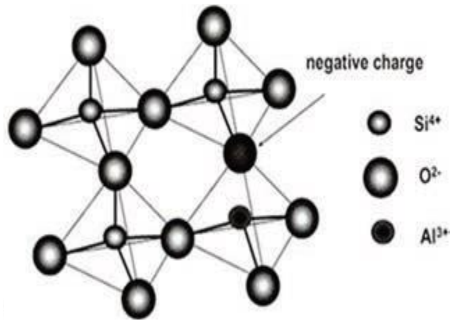

Fig. 1 Structure of Zeolite (Wiyantoko and Rahmah, 2017)

2 Resources and Methods Used

2.1 Materials

API class G cement with high sulphate-resistant and a specific gravity of 3.14 was used in this research. All the cement slurries were prepared using fresh water. Fresh nano zeolite, spherically shaped with an average particle size of 100 nm was obtained from Tema Oil Refinery, Ghana. The microscopic image of the fresh nano zeolite is as presented in Fig. 2. The energy-dispersive X-Ray spectroscopic results revealed that the fresh nano zeolite was mainly comprised of aluminium, carbon and silicon (Fig. 3).

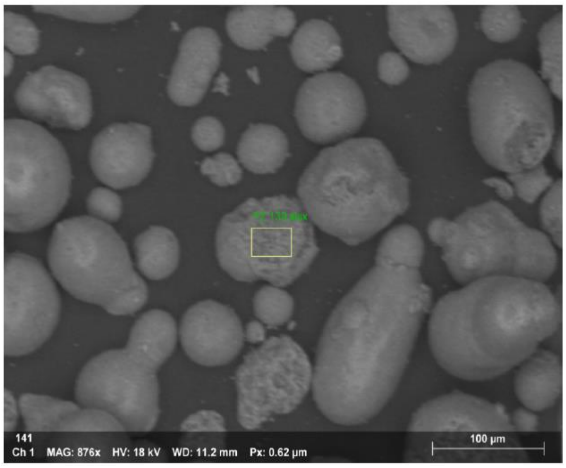

Fig. 2 Fresh Zeolite Scanning Electron Microscope Image (Amarfio, 2020)

2.2 Experimental Design

Experiments were conducted with class G cement slurry mixed with different concentrations of fresh nano zeolite (FNZ) at Bottom Hole Circulating Temperature (BHCT) 150 ºF (66 ºC) and Bottom Hole Static Temperature of 190 ºF (88 ºC). The cement slurry preparation was carried out by closely following American Petroleum Institute (API) Specification 10A.

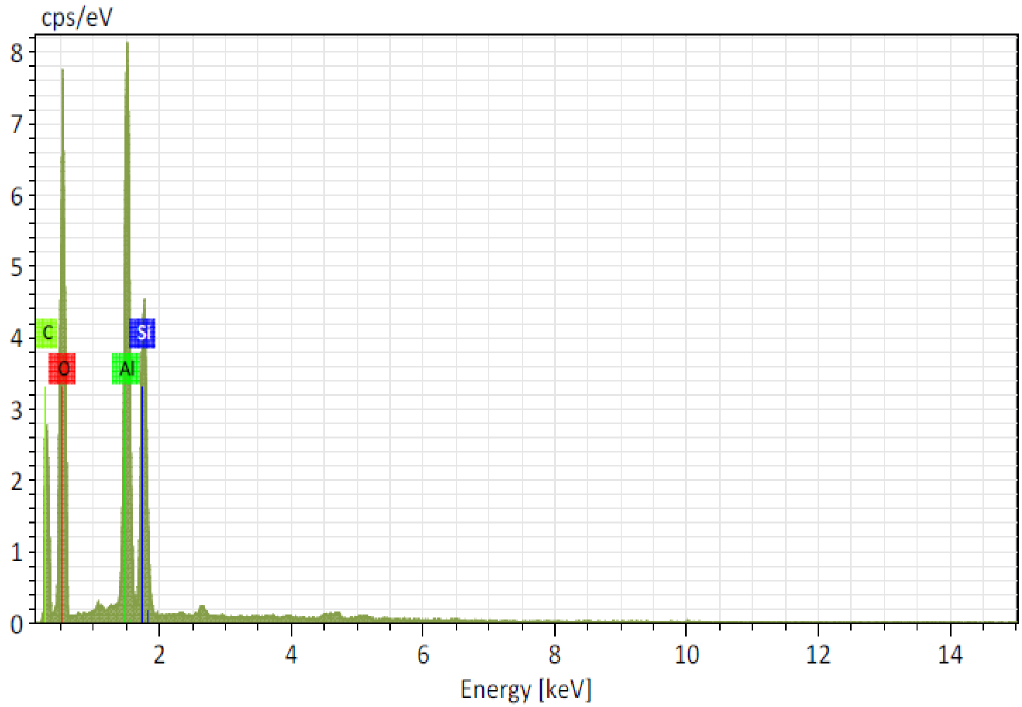

Fig. 3 Energy-Dispersive X-Ray Spectroscopy Plot for Fresh Zeolite (Amarfio, 2020)

The physical properties such as compressive strength, thickening time, free fluid, fluid loss and rheology were determined by closely following API Specification 10A and API Recommended Practice 10B (Anon, 1997; 2013). The experiment was conducted using test conditions in Table 1 and slurry composition shown in Table 2.

Table 1 Experimental Conditions

Test Condition	Units	Test
BHST	°F (ºC)	190 (88)
BHCT	°F (ºC)	150 (66)
BHP	psi (MPa)	1 000 (6.9)
Heat Up Time	min	53

Table 2 Slurry Compositions

Slurries	Description	Water Cement Ratio (%)
S-0	Class G Cement	44
S-1	Class G +1% bwoc FNZ	44
S-2	Class G + 2% bwoc FNZ	44
S-3	Class G + 3% bwoc FNZ	44

2.3 Compressive Strength Test

Compressive strength is one of the properties used to test the reliability of cementing and is the ability of a material to withstand deformation when load is applied, hence, the higher the compressive strength, the lower the porosity and increased durability (Broni-Bediako *et al.*, 2015a). Insufficient compressive strength will more likely lead to casing failures more likely, and eventually shorten significantly, the efficiency and productivity of the well over time (Huwel *et al.*, 2014). There are two common methods for determining the compressive strength of a cement slurry; non-destructive and destructive. The non-destructive method uses an Ultrasonic Cement Analyser (UCA) and the destructive method which is an Unconfined Compressive Strength (UCS) works by applying load to four square inch cement cubes to determine the compressive strength of cement. This study employed the use of non-destructive method to determine of the compressive strength. The UCA passes ultrasonic signals through a cement sample and measures the transit time. As cement begins to build compressive strength; the transit time decreases. Through the use of mathematical algorithms, the transit time is then converted into an approximate value for the compressive strength in pound per square inch (psi) (Huwel *et al.*, 2014). The compressive strength test was conducted at 190 ºF (88 ºC) and 3 000 psi (20.68 MPa) for 12 and 24 hours for cement slurry mixed with different concentrations of fresh nano zeolite.

2.4 Thickening Time Test

The thickening time of cement slurry determines the length of time cement slurry remains in pumpable state for wellbore temperature and pressure (Abbas *et al*., 2014). The thickening time test was performed in a High-Pressure High-Temperature (HPHT) Consistometer that is usually rated at pressure up to 30 000 psi (206.8 MPa) and temperatures up to 400 ºF (204 ºC). The cement slurry was mixed according to API procedures and then placed in a slurry cup into the consistometer for testing. The testing pressure and temperature were controlled to simulate the conditions the slurry will encounter in the well. The test concluded when the slurry reaches a consistency considered unpumpable in the well.

2.5 Free Fluid Test

Free fluid test is intended to help determine the quantity of free fluid that will gather on the top of cement slurry between the time it is placed and the time it gels and sets up (Joel, 2009). Excessive free fluid in cement slurry can cause problems with water pockets, channelling, sedimentation, zonal isolation, and the like (Baig, 2017). In this research, a 250 mL graduated cylinder was used to determine free fluid contents in the cement slurry. The slurry was prepared and preconditioned for 30 minutes in an Atmospheric Consistometer which consists of a rotating cylindrical slurry container, equipped with an essentially stationary paddle assembly, in a temperature-controlled liquid bath. The preconditioned slurry was then transferred to the 250 ml graduated cylinder and allowed to set for 2 hours after covering the top of the graduated cylinder. The slurry was then examined for any free fluid on the top of the cement column. This free fluid was decanted and measured to determine the percent of free fluid based on the 250 ml volume. The free fluid was determined using Equation (1) (Broni-Bediako and Naatu, 2021; Anon, 1997).

$$\text{Free Fluid} = \frac{\text{ml of fluid x 100}}{250} \tag{1}$$

2.6 Rheology Test

Rheology of cement slurries is of great importance for the design, construction and quality of primary cementing. It is critical for proper displacement of drilling mud and calculating the frictional pressures. Incomplete removal of drilling mud can cause poor cement bonding, zone communication and ineffective stimulation treatment (Broni-Bediako *et al.*, 2015b). The rheology of the cement slurries was measured using Fann Viscometer. The slurries were conditioned at 150 ºF (66 ºC) using Atmospheric Consistometer. Viscosity readings at 3 rpm, 6 rpm, 100 rpm, 200 rpm, 300 rpm and 600 rpm were observed and recorded. The values from the direct readings were used to compute the plastic viscosity (μ_p) in centipoise (cP) and the yield point (τ_o) in pounds per 100 square feet (lb/100ft^2) respectively using Equations (2) and (3) (Broni-Bediako and Amorin, 2018).

$$\mu_p = 1.5(\theta_{300} - \theta_{100}) \tag{2}$$

$$\tau_o = \theta_{300} - \mu_p \tag{3}$$

where θ_{300} is 300 rpm dial reading and θ_{100} is 100 rpm dial reading.

2.7 Fluid Loss Test

Fluid loss test determines the relative effectiveness of a cement slurry to retain its water phase or to lose a portion of its water phase as a filtrate to the formation (Anon, 2020a). Excessive fluid loss to permeable zones can cause a number of problems, such as insufficient mud displacement, high viscosity, unwanted change of set-time, and lack of final compressive strength (Anon, 2020b). Stirred and non-stirred fluid loss equipment can be used for fluid loss measurement at desired temperature and pressure conditions. Usually tests at temperatures less than or equal to 190 °F (88 ºC) may be performed using a non-stirred fluid loss equipment. This study employed the use of non-stirred (static) fluid loss tester which according to Anon (2020a) provides a reliable means of determining the fluid loss characteristics of an oil well cement slurry. The fluid loss test was conducted at 150 ºF (66 ºC) and 1 000 psi (6.9 MPa) pressure per API standards. After conditioning the slurry at the Bottomhole Circulating Temperature (BHCT) for thirty (30) minutes, the slurry was placed in the fluid cell and a differential pressure of 1 000 psi (6.9 MPa) was applied across the 325-mesh (45μm) screen for about thirty minutes. API fluid loss was calculated using Equation 4 (Broni-Bediako and Naatu, 2021; Anon, 1997).

$$\text{Calculated API Fluid Loss} = \frac{10.954 \text{ x } Q_t}{\sqrt{t}} \tag{4}$$

where, Q_t is the volume (ml (cc)) of filtrate collected at the time t (min) of the "blowout".

3 Results and Discussion

3.1 Compressive Strength Analysis

Compressive strength is one of the properties used to test reliability of cementing and is the ability of a material to withstand deformation when a load is applied (Falode *et al.*, 2013). Early compressive strength development is encouraged so as to make a strong bond with walls after placement. This enables drilling operations to resume within the shortest possible time. The sample charts obtained from the UCA test are presented in Figs. 4 to 7.

3.1.1 Compressive Strength at 12 and 24 Hours

Fig. 8 shows compressive strength development after 12 and 24 hours. It was observed that the 1% addition of fresh nano zeolites (S-1) to the base cement slurry decreased the compressive strength significantly for both 12 and 24 hour curing periods by 23.5% and 14.3% respectively.

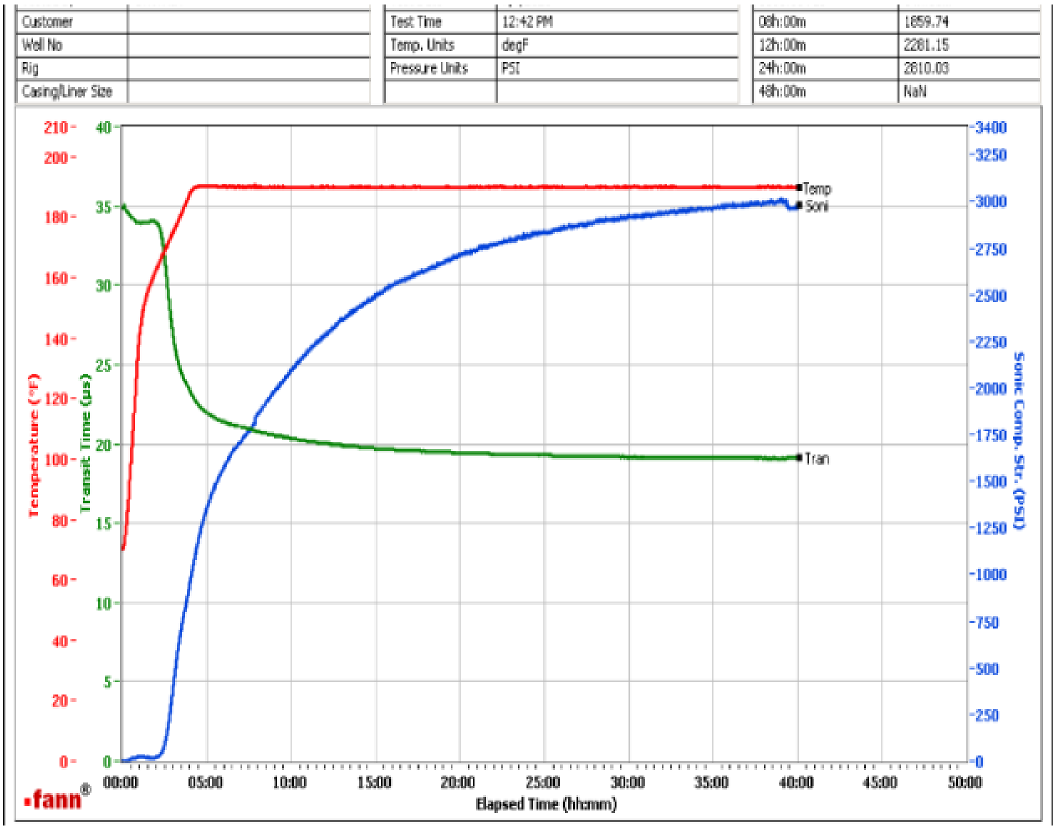

Fig. 4 Compressive Strength Development for S-0

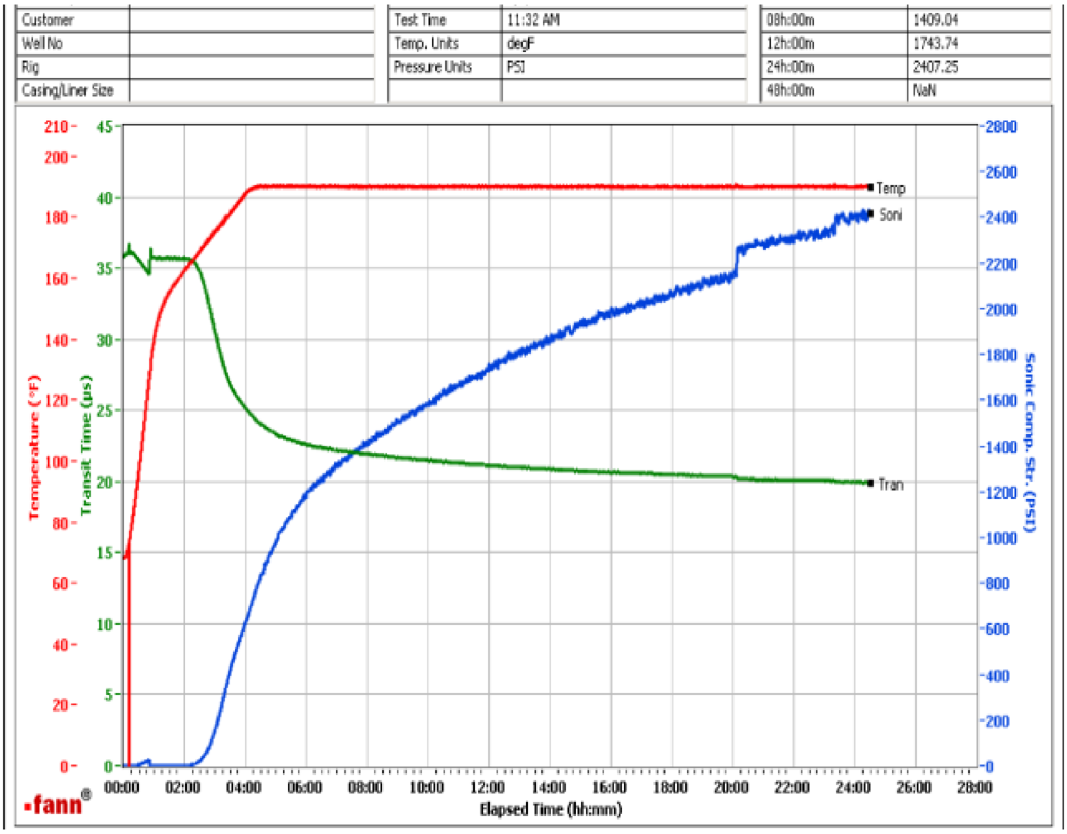

Fig. 5 Compressive Strength Development for S-1

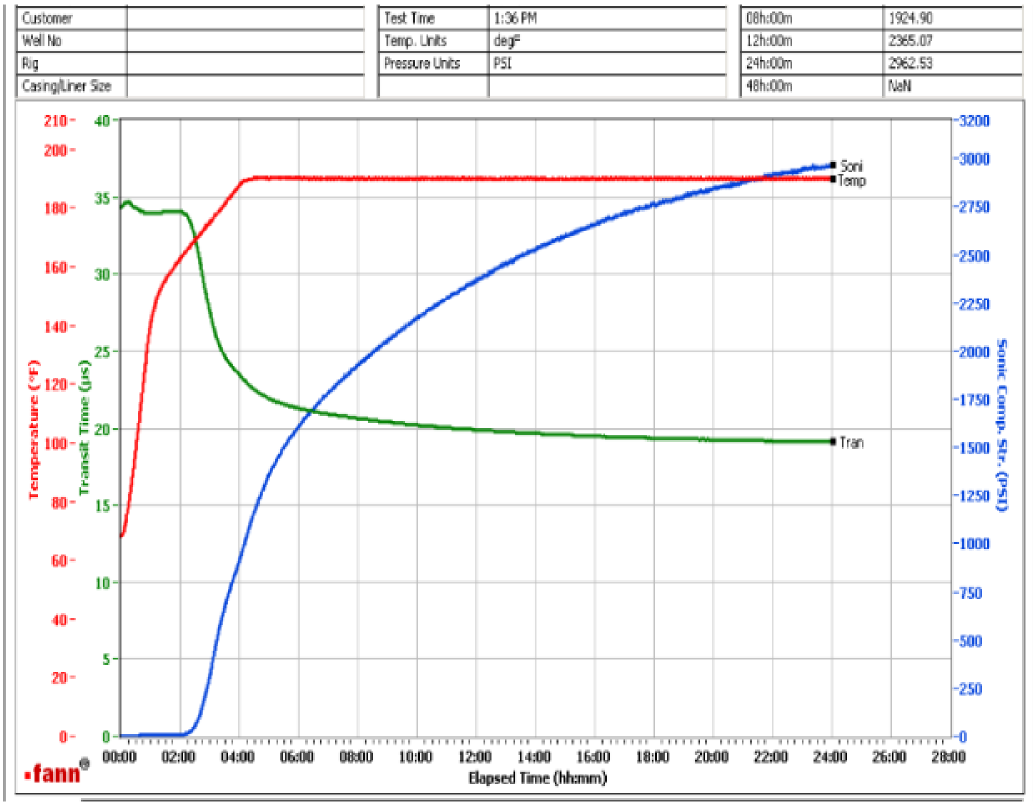

Fig. 6 Compressive Strength Development for S-2

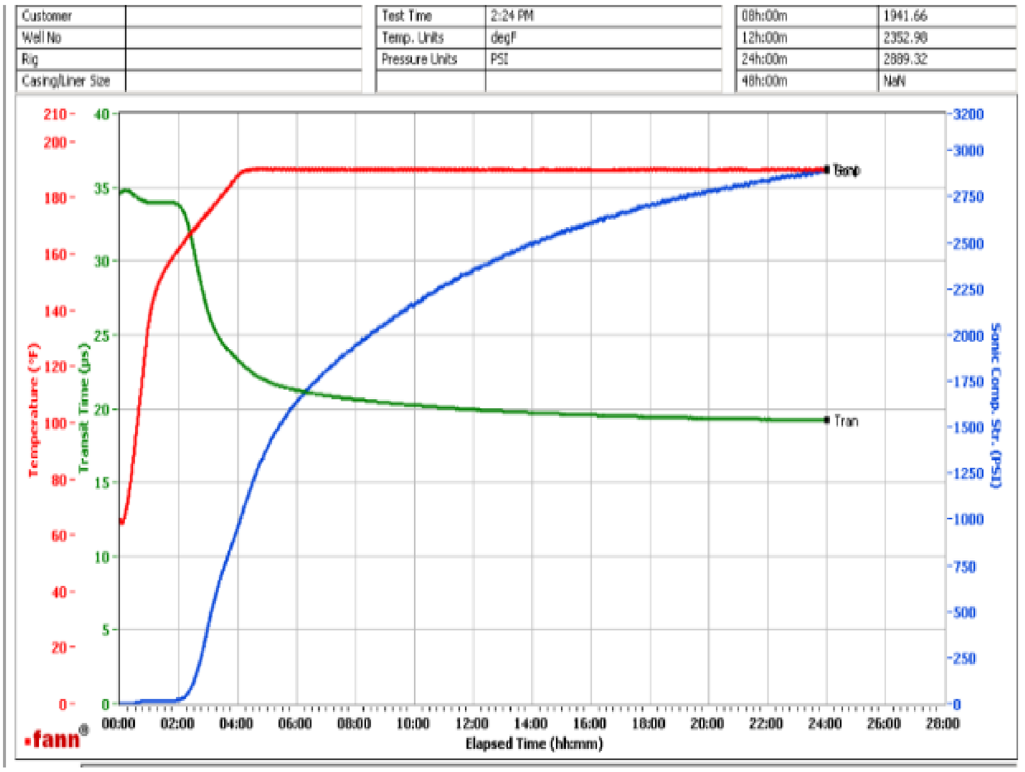

Fig. 7 Compressive Strength Development for S-3

However, the compressive strength increased with an addition of fresh nano zeolite concentration of 2% and 3% bwoc compared with the base cement slurry (S-0). At 2% bwoc, the compressive strength improved by 3.7% and 5.4% respectively for both 12 and 24 hours while at 3% bwoc, the compressive strength improved by 2.8% and 3.2% for the 12 and 24 hours respectively. These observations show that, only S-2 and S-3 had a compressive strength higher than the base cement slurry (S-0).

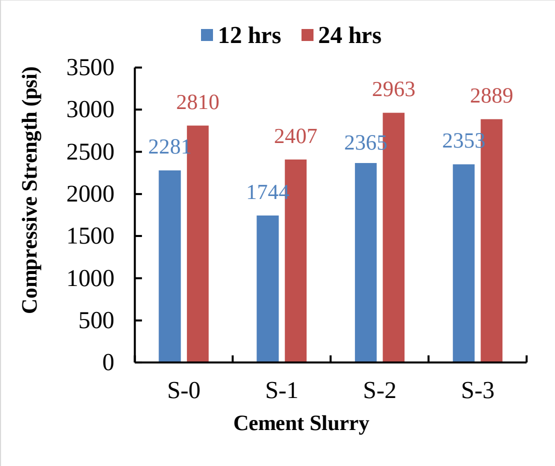

Fig. 8 Compressive Strength of Cement Slurry Mixed with Different Concentrations of Fresh Nano Zeolites at 150 ºF (66 ºC) for 12 and 24 Hours

All the cement samples attained compressive strength greater than 2 000 psi (13.8 MPa) with the exception of S-1 which attained compressive strength of 1 744 psi (12 MPa) after a 12-hour curing period. According to Murtaza *et al.* (2020), 2 000 psi (13.8 MPa) compressive strength development is the minimum compressive strength required before performing any perforation or stimulation job. Generally, the compressive strength of the base cement slurry increased after more than 1% bwoc with the highest strength obtained at 2% bwoc. There was a compressive strength improvement on the base cement slurry after a fresh nano zeolite concentration of more than 1% bwoc was added.

3.1.2 Compressive Strength Achievement Time

Table 3 shows the time needed to achieve a compressive strength of 50, 100, 500 and 1 000 psi for all the cement slurries. From Table 3, as the concentration of the base cement slurry was increased by 1% bwoc and 2% bwoc of fresh nano zeolite, compressive strength development was delayed. In essence, less time was required to attain compressive strength of 50 psi (0.34 MPa), 100 psi (0.69 MPa), 500 psi (3.4 MPa) and 1 000 psi (6.9 MPa) in the base cement slurry as compared to S-1 and S-2. Compressive strength development for fresh nano zeolite concentration of 3% bwoc showed a reverse trend compared to 1% bwoc and 2% bwoc. At high temperatures, it will be ideal to design cement slurry with 3% bwoc of fresh nano zeolite to attain early compressive strength necessary for drilling operations to proceed on time and avoid unnecessary delays on wait on cement time and rig cost.

Table 3 Compressive Strength Achievement Time

Slurry Identity	50 psi (0.34 MPa)	100 psi (0.69 MPa)	500 psi (3.4 MPa)	1000 psi (6.9 MPa)
	(hh:mm)	(hh:mm)	(hh:mm)	(hh:mm)
S-0	2:22	2:31	3:08	4:05
S-1	2:41	2:51	3:40	5:03
S-2	2:29	2:38	3:14	4:10
S-3	2:16	2:27	3:06	4:05

4.1.3 Transition Period between 50 psi (0.34 MPa) and 500 psi (3.4 MPa)

The transition period between developing a compressive strength of 50 psi (0.34 MPa) and 500 psi (3.4 MPa) is very important in cementing operation. It should be as short as possible to reduce the time needed to wait on cement for cement slurry to harden before the next section of a wellbore is drilled. Fig. 9 shows the transition periods from 50 psi (0.34 MPa) to 500 psi (3.4 MPa) for all the cement slurries. It was observed that cement slurry S-2 had the shortest transition time (45 minutes) as compared with all the cement slurries (S-0, S-1 and S-3) which attained a transition time of 46, 59 and 50 minutes respectively.

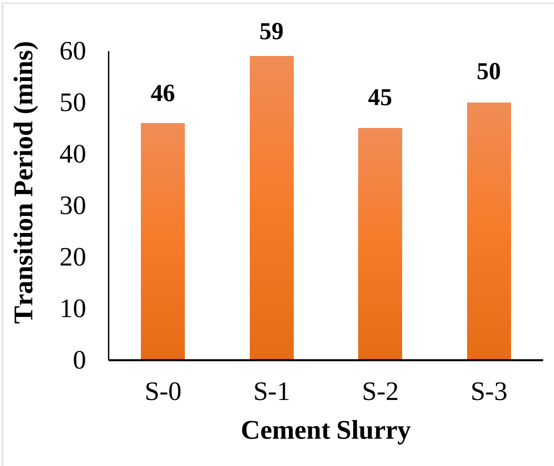

Fig. 9 Transition Period from 50 psi to 500 psi at 150 ºF (66 ºC)

3.2 Thickening Time Analysis

The thickening time test using HPHT Consistometer was conducted at 150 ºF (66 ºC) and 1 000 psi (6.9 MPa) for cement slurry mixed with different concentrations of fresh nano zeolite. The sample charts obtained from the thickening time test are presented in Figs. 10 to 13. Table 4 shows the summary of the thickening time results for all the cement slurries.

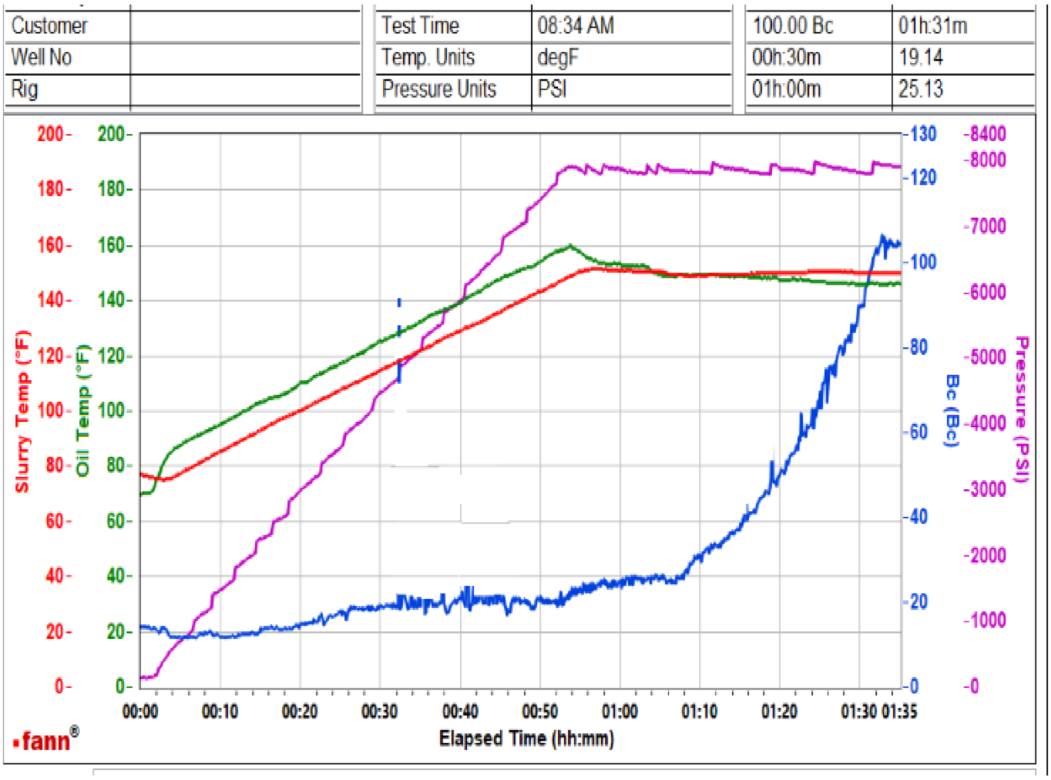

Fig. 10 Thickening Time Chart for S-0

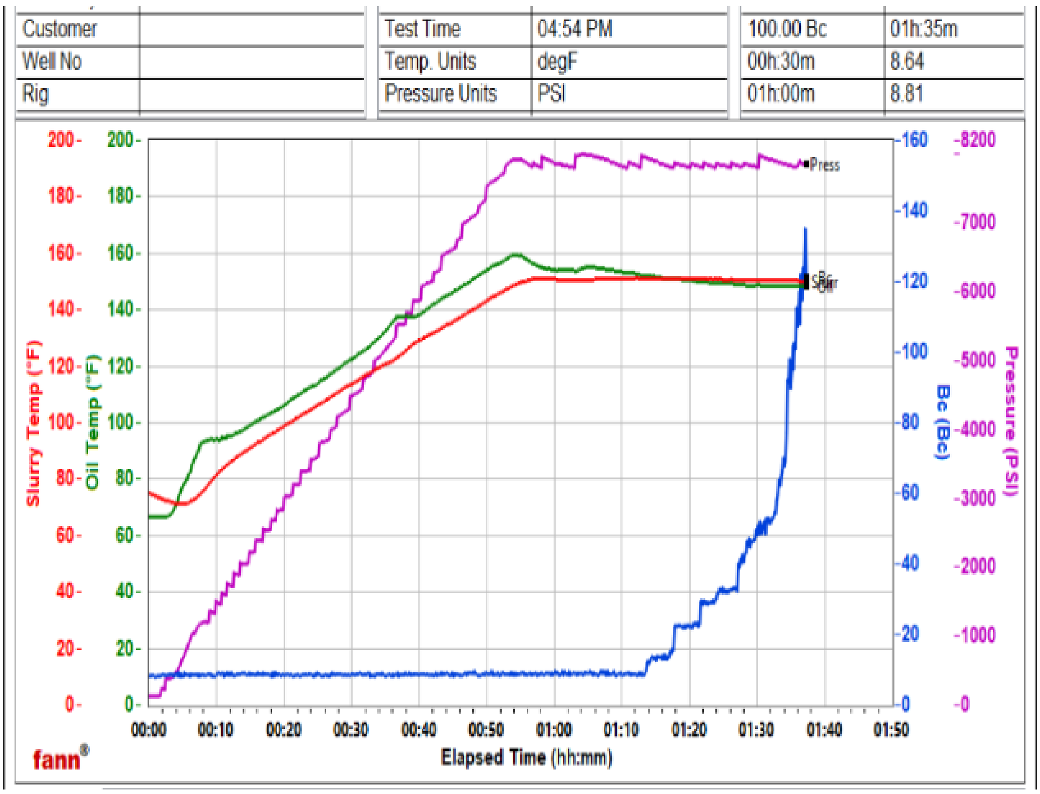

Fig. 11 Thickening Time Chart for S-1

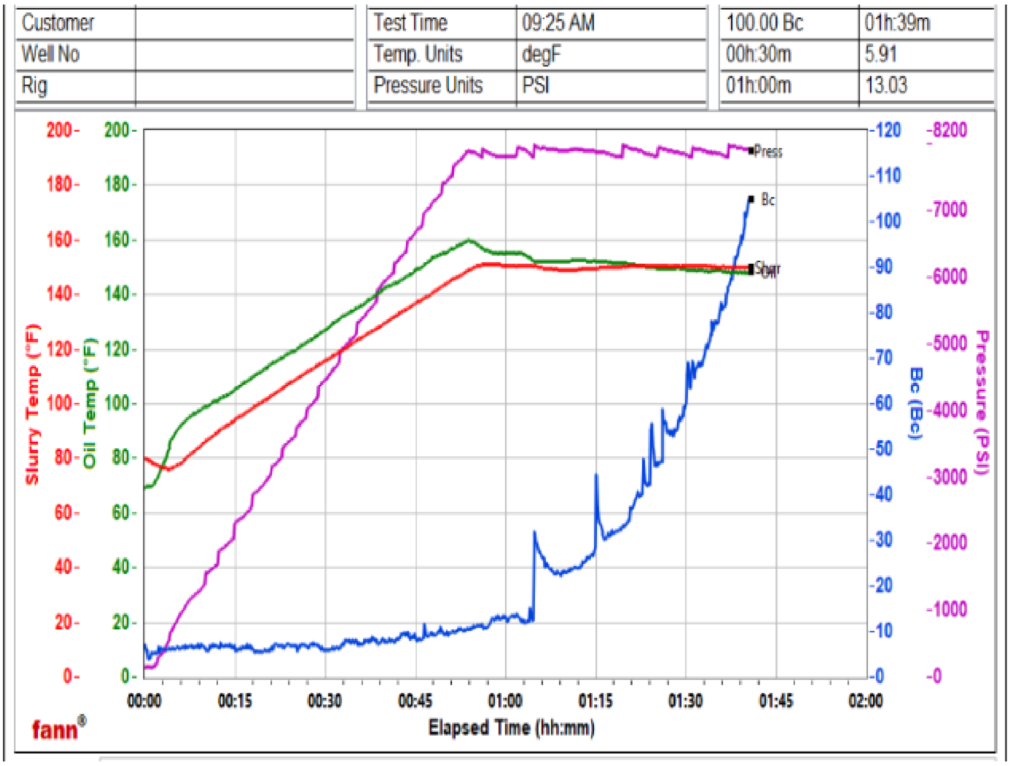

Fig. 12 Thickening Time Chart for S-2

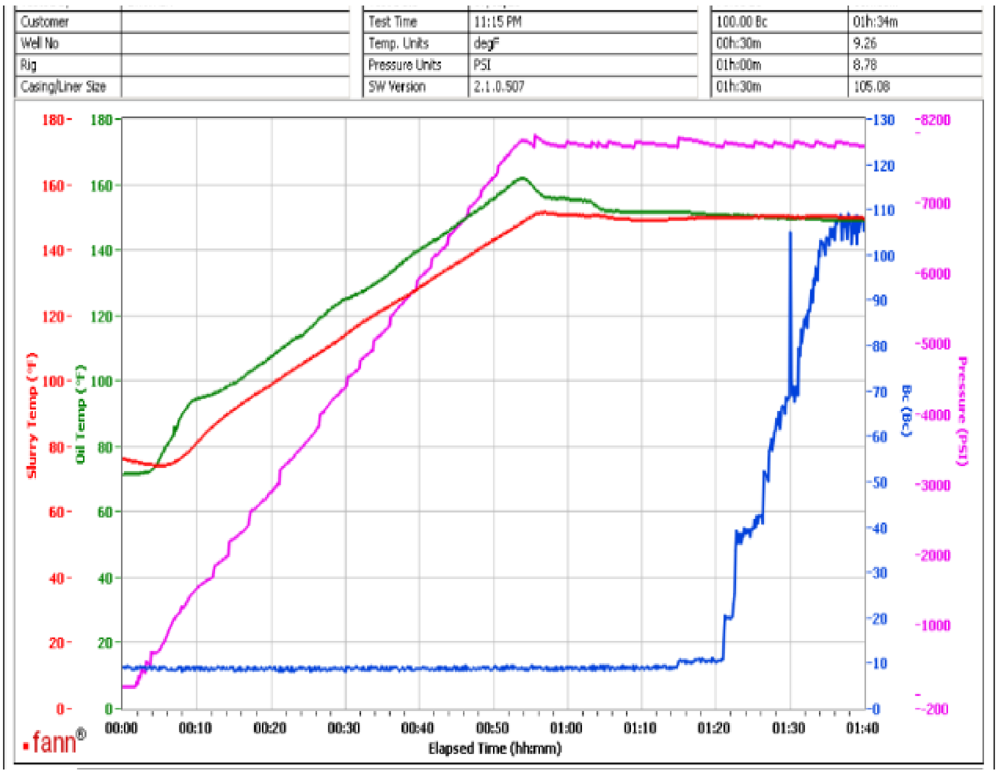

Fig. 13 Thickening Time Chart for S-3

Results of the laboratory thickening time is an indication of the length of time that cement slurry would remain pumpable (Broni-Bediako *et al.*, 2016; Joel 2009). In essence, if the cement slurry remains liquid over a period deemed fit for purpose and functions as a solid when it stops flowing, in a reasonable time, it will be suitable for a good job.

The consistency at the start of a thickening time test indicates the viscosity at the start of the test. It could be observed from Table 4 that, the initial viscosities decreased for all concentrations of fresh nano zeolites compared with the base cement slurry. The viscosities at S-1 and S-3 recorded the same values.

Table 4 Summary of Thickening Time Test for all the Cement Slurries at 150 ºF (66 ºC)

Slurry	Heating Time(min)	Start Bc	30 Bc	40 Bc	50 Bc	70 Bc	100 Bc
		(hh:mm)					
S-0	53	14	1:09	1:16	1:20	1:25	1:31
S-1	53	8	1:24	1:27	1:30	1:34	1:35
S-2	53	6	1:04	1:15	1:24	1:33	1:39
S-3	53	8	1:22	1:25	1:26	1:30	1:34

The end of thickening time test was considered to be 70 Bearden units of consistency (Bc). At 70 Bc, cement slurry starts to set (Alp and Akin, 2013) and it is considered unpumpable (Broni-Bediako and Amorin, 2018; Bett, 2010). At 70 Bc, it was observed that increasing the fresh nano zeolite concentration increases the thickening time at 150 ºF (66 ºC). However, the retardation effect of fresh nano zeolite was not significant compared with the base cement slurry at 70 Bc. According to Broni-Bediako *et al.* (2016), retarders inhibit hydration and delay setting allowing sufficient time for slurry placement in deep and hot wells. In deep and high temperature wells, it is expected that cement withstands the conditions until placement then thickening can set in. The results in Table 4 shows that the fresh nano zeolite has a retardation effect which makes it very appropriate for deep and high temperature wells in order to allow for more time placement and proper setting.

3.3 Rheological Analysis

According to Sharhriar (2011), the fundamental knowledge of oil well cement slurry rheology is necessary to evaluate the ability to remove mud and optimise slurry placement. Incomplete mud removal can result in poor cement bonding, zone communication and stimulation treatment (Bannister, 2011). Rheological properties of oil well cement are expected to give characterisation of the final slurry product and assist in forecasting the end use behaviour of the physical properties of the slurry during pumping and after pumping. In this study, flow properties such as plastic viscosity, yield point and apparent viscosities were measured at a BHCT of 150 ℉ (66 ℃). Table 5 indicates the calculated values of plastic viscosity, and yield point from the test conducted.

3.3.1 PV and YP

From Table 5, plastic viscosity was observed to have recorded an increase in value from base cement slurry (S-0), when fresh nano zeolite at a concentration of 1% bwoc and 3% bwoc were added to the base cement slurry. However, the addition of 2% bwoc of fresh nano zeolite resulted in a decreased plastic viscosity value as compared with the base cement slurry (S-0). Generally, all the cement slurries were pumpable. According to Broni-Bediako *et al.*, (2015a) and Abbas *et al.* (2014), plastic viscosity values above 100 cP will require very high pumping pressure for proper placement and subsequent setting. The addition of fresh nano zeolite to the base cement slurry did not change or alter plastic viscosity values significantly and therefore do not require high pumping pressures at 150 ºF (66 ºC).

Table 5 Rheological Properties of Cement Slurries Mixed with Different Concentrations of Fresh Nano Zeolite at 150 ºF (66 ºC)

Test Parameters	S-0	S-1	S-2	S-3
	Dial Readings in Centipoise (cP)			
600 rpm	150	138	116	144
300 rpm	127	109	94	108
200 rpm	113.5	96	83	93.5
100 rpm	97	78.5	66.5	76
6 rpm	22.5	23.5	18.5	25.5
3 rpm	16.5	18.5	15	19.5
PV (cP)	***45***	***45.75***	***41.25***	***48***
YP (lb./100ft^2)	***82***	***63.25***	***52.75***	***60***
PV/YP	***0.55***	***0.72***	***0.78***	***0.80***

Yield point is the measure of the initial resistance to flow (Broni-Bediako and Amorin, 2019; Dankwa *et al.*, 2018; Abdou *et al.*, 2016; Yunita *et al.*, 2016). It also gives an indication of the carrying capacity of the cement slurry (Murtaza *et al.*, 2020). Increase in concentrations of fresh nano zeolites influenced the yield point values as a decrease in values were observed. Addition of fresh nano zeolite concentration of 1% bwoc decreased the yield point by 22% compared with the base cement slurry (S-0). There was a further drop in yield point value of the cement slurry by 36.6% when a concentration 2% bwoc of fresh nano zeolite was added to the base cement slurry. Generally, all the yield point values decreased with increase in the concentration of fresh nano zeolites at 150 ºF (66 ºC). Lower yield point values indicate that the slurry gets thinner and particle settling effect may set in.

3.3.2 PV/YP Ratio

The PV/YP ratio mainly determines the carrying capacity of the drilling fluid. PV/YP ≥ 1.3 displays a good carrying capacity behaviour of the drilling fluid which results in improved wellbore cleaning performance. This ratio (PV/YP) was used to study the carrying capacity of the cement slurry. A cement slurry with good carrying capacity avoids settling of cement particles and therefore results in homogeneous placement of cement slurry around casing without providing sag effect. A heterogeneous placement with varying density profile along the length of the cement column results in many wellbore issues such as fluid migration, free water separation and fracturing (Broni-Bediako and Naatu, 2021). In some studies, cement slurries with acceptable and best carrying capacities are suggested to lie in the range from 1 to 2 ratio as safe window (Elkatatny *et al.*, 2020). Fig. 14 displays the PV/YP ratios for all the cement slurries. Increase in the concentrations of fresh nano zeolite resulted in an increasing pattern or trend in PV/YP values for all the samples (S-1 to S-3). Cement slurry mixed with fresh nano zeolite of 3% bwoc recorded the highest PV/YP ratio followed by 2% bwoc and then 1% bwoc. None of the cement slurries proved to have good carrying capacity since all of them did not fall within the acceptable range at a temperature of 150 °F (66 °C) recommended by Elkatatny *et al.* (2020).

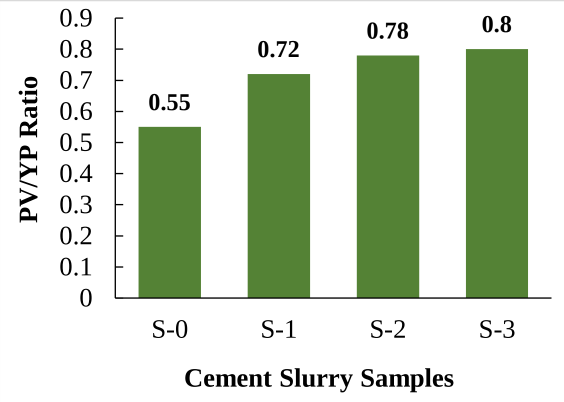

Fig. 14 PV/YP Ratio for Different Concentration of Zeolite at 150 °F (66 °C)

3.3.3 Shear Stress- Shear Rate Curve

Fig. 15 indicates the shear stress-shear rate curves of the various cement slurries mixed with different concentrations of fresh nano zeolite. It was observed that the addition of fresh nano zeolite in the base cement slurry reduced the shear stresses for shear rates of 100 rpm (170 s^{-1}) to 600 rpm (1021 s^{-1}) (Table 5). Generally, additives with retarding characteristics exhibit a decrease in rheological values as the concentration of the additive are increased. Test results as indicated in Fig. 15 showed that increasing the fresh nano zeolite concentrations resulted in decrease in rheological values. It was observed that the fresh nano zeolite in addition to affecting the thickening of the cement slurries also exhibited dispersing tendencies.

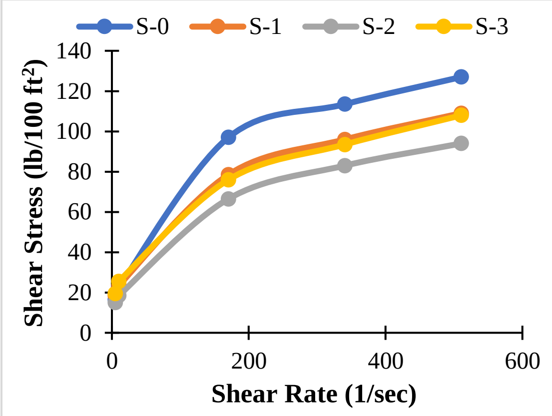

Fig. 15 Shear Stress-Shear Rate Curve for Various Concentrations of Fresh Nano Zeolite-based Cement Slurries at 150 °F (66 °C)

3.4 Free Fluid Analysis

The intention of free fluid test is to help determine the quantity of free fluid that will gather on top of cement slurry between the time is placed and the time it gels and sets up (Otaraku *et al.*, 2019; Joel, 2009). Results obtained from the study showed fresh nano zeolites not causing any free water separation at 150 °F (66 °C).

3.5 Fluid Loss Analysis

Cement fluid loss is an important characteristic that determines the quantity of fluid that is lost to the formation when exposed to a positive differential pressure. This usually occurs when cementing across permeable zones. Fluid loss of the cement should be controlled to prevent early dehydration of the slurry causing an artificial premature hardening (Darbe *et al.*, 2008). The effect of the fresh nano zeolites on fluid loss is summarised in Table 6.

Table 6 Fluid Loss at 150 °F (66 °C)

Slurry	Measured Volume (ml)	Test Time (secs)	Calculated Fluid Loss (ml/30min)
S-0	55	20	1 044
S-1	-	-	-
S-2	-	-	-
S-3	-	-	-

The base cement slurry (S-0) after being subjected to test conditions had a very high fluid loss value of about 1 044 ml for a recorded test time of 20 seconds. At S-1, S-2 and S-3, the tests blew dry within few seconds of being subjected to test conditions. The results indicate that fresh nano zeolite does not have fluid loss control characteristics at a temperature of 150 °F (66 °C).

4 Conclusions and Recommendation

From the research, it could be concluded that:

(i) Compressive strength was improved by the addition of fresh nano zeolite at 2% and 3% bwoc except for 1% bwoc.

(ii) Fresh nano zeolite concentration of 1% bwoc recorded the highest time to reach the set compressive strengths of 50 psi (0.34 MPa), 100 psi (0.69 MPa) and 500 psi (3.4 MPa). However, fresh nano zeolite concentration of 3% bwoc recorded the least time to reach the respective compressive strengths.

(iii) Increase in the concentration of fresh nano zeolite resulted in an increase in thickening time of all the cement slurries at 150 °F (66 °C). Hence the fresh nano zeolite has a retardation effect and thus suitable for high temperature wells.

(iv) Plastic viscosity increased from base cement slurry (S-0) when 1% and 3% bwoc were added. However, the addition of fresh nano zeolite of 2% bwoc resulted in a decreased plastic viscosity value as compared to the base cement slurry.

(v) Yield point values decreased significantly as fresh nano zeolite concentrations were increased at 150 °F (66 °C). Lower yield point values meant that the slurry gets thinner and particle settling effect may set in thereby preventing homogenous placement.

(vi) Increase in the concentrations of fresh nano zeolites resulted in an increasing trend in PV/YP values for all the cement slurries (S-1 to S-3). However, none of the cement slurries proved to have a good carrying capacity since all slurries did not fall within the acceptable range recommended by Murtaza *et al*. (2011).

(vii) The introduction of fresh nano zeolite to the base cement slurry did not cause any free fluid separation at 150 °F (66 °C).

(viii) The introduction of fresh nano zeolite did not improve upon the amount of filtrate lost in the cement slurry and therefore do not exhibit the characteristics of fluid loss additives or agent.

From the results and the conclusion drawn from the study, it is recommended that work be done on the combined effects of fresh nano zeolites and conventional additives on oil well cement slurries at high temperatures.

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