

# Revolutionizing Oil and Gas Well Cement: Harnessing the Power of Nano Silica for Enhanced Mechanical Properties and Performance Optimization

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**Abstract:** In recent times, numerous oil and gas wells have experienced diverse failures ranging from downhole plug malfunctions to compromised well bore integrity, resulting in damage to aquifers, loss of assets, and posing significant economic and environmental hazards. The inclusion of the nano-silica into oil and gas well cement emerges as a promising solution, demonstrating a positive influence on enhancing mechanical properties such as compressive strength and durability. This research will provide a comparative analysis of cement slurry (15.8 ppg) performance, examining scenarios with and without the addition of nano-silica under downhole reservoir conditions. The experimental results reveal a substantial improvement in cement mechanical properties. when 3% by weight of cement (BWOC) nano-silica is introduced, however any percentage below 3 % had a negative impact on the cement sample compressive strength. Notably, this enhancement is attributed to accelerated cement hydration, ultimately leading to increased compressive strength and improved overall performance.

**Keywords:** Nano-Silica, Well Cement, Enhancing Performance in Oil and Gas.

## 1. Introduction

In the oil and gas industry, cement is an essential component in the well construction phase. Adequate cement design is significant so as to ensure the integrity of the well and to prevent any migration from the formation during the drilling phase, completion phase and production phase. Moreover, it prevents any migrations of the undesired formation fluids covered by cemented casing.

Additionally, the cement is used for the well plugging & abandonment applications. It also secures the safety of personnel and equipment and eliminates any negative environmental impact. In order to achieve these objectives, cement must have few low terms characteristics to ensure adequate wellbore integrity; it should be impermeable to gases and downhole fluids, and withstand the downhole conditions including mechanical loads, temperature, and chemical degradation.

Cementation of the casing within the drilled hole provides a primary and essential zonal isolation and effectively separates the porous formations from the tight formations (Hemant Ladva 2013) Some wellbore problems which are caused by poor cementation for zonal isolation include but not limited to mud infiltration to the cement sheath, micro-

annulus, gas and fluid migration through the annular space and also chemical gas and water fluid reactive with cement [8].

Thus, just using API class-G cement slurry for cementing is not sufficient even for some primary cementing jobs. The set cement sheath is in a position to transfer the load. This means there should be at least a minuscule amount of interface materials (e.g. cement particle) between casing and cement because only then there can be stress transfer between casing and cement. An oil well cement slurry absolutely needs at least one solidified film surrounding all the casing OD. With the increase in the depths and pressures of the drilled wells in the oil and gas industry, the wellbore environment becomes more challenging and complex [6]. As a result, the drilling and cementing engineers intend to design more stable and durable cement to minimize the environmental risks and wellbore problems [12].

Nanotechnology became on the solutions to enhance cement characteristics. The Nano Materials plays a critical role in increasing the compressive strength of the cement but by different mechanisms which will be illustrated in detail in the coming sections [19]. Nano silica in cement can be attributed to various mechanisms. Firstly, due to their nano-scale size, silica particles function as filler

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material and occupy gaps within the cement matrix, resulting in a dense and compact structure with reduced capillary porosity [3]. Secondly, the high pozzolanic activity of nano silica materials make them effective accelerators for cement hydration [17]. It was observed that blending nano silica into cement grains resulted in the formation of  $\text{H}_2\text{SiO}_4$ , which reacts with  $\text{Ca}^{2+}$  to produce excess calcium sulfate [16].

The nano silica reacts with  $\text{Ca}(\text{OH})_2$  resulting in the formation of C-S-H, the nano silica also helps in immobilizing the free water by filling most of the voids in between the cement grains, thus acting as a filling [1]. This will result in formation of denser cement sample and consequently lead to an increase in the cement compressive strength. The nano silica structure forms interconnections network with the cement grains[9]. This creates additional pass ways to transfer stresses and aids load distribution. The structure of the nano silica increases the strength of the interfacial transition zone between the cement grains. Consequently, it improves the overall integrity of the cement [15].

The nano silica helps in the dispersion of water in the cement during the hydration process, thus it decreases the amount of free water of in the cement [13]. It also optimizes the hydration kinetics & the packing of the hydrate products leading to an increase in the cement compressive strength. In the early stage of cement hydration, micro cracks may be formed in the cement paste due to internal stress, the nano silica has high ability to bridge these cracks, not only that but it also prevents the formation of the macroscopic cracks. This will make the cement imbedded with silica have high resistance to the mechanical loading [7].

### 1.1. Cement bond failure consequences

Failure of the cement functions can lead to severe consequences, including production loss, environmental damage, and safety hazards. Several issues with the cement have been reported over the years, for instance, sustained casing pressure were recorded in many wells around the world due to the gas migration in the annulus. The root cause was due to the cement channeling overtime due to poor cement job.

[18] conducted a study in the field of petroleum engineering revealed that during the year 1991, over 1100 casing strings across 8000 wells in the Gulf of Mexico experienced sustained casing pressure issues. Furthermore, a report released by the Mineral Management Services in 2004 indicated that approximately 33% of these sustained casing pressure incidents were attributed to inadequate cementing practices, necessitating remedial cementing operations at a cumulative cost of 650 million USD. Additionally, the report highlighted that as a consequence of this issue, production activities in 9000 wells were temporarily suspended, with many of these wells being

subject to temporary abandonment measures.

The Macondo accident and the subsequent oil spill have catalyzed significant shifts in the industry's approach toward Plug and Abandonment (P&A) procedures in the Gulf of Mexico in recent years. Following this event, the well in question underwent temporary abandonment to enable future production utilization [4]. The abandonment process adhered to BP's policy, involving the installation of cemented liners and mechanical plugs to establish effective barriers against hydrocarbon flow. A negative pressure test was subsequently conducted; however, the team inadvertently confirmed its success and proceeded with displacing the mud with seawater. Regrettably, this action resulted in an uncontrolled flow, leading to two explosions and subsequent fires, resulting in the tragic loss of 11 workers and the release of 5 million barrels of oil. The primary cause of this flow was attributed to a deficient cement sheath, which facilitated gas migration to the surface.

## 2. Experimental details

The Portland Class-G cement develops its compressive strength by the chemical reaction mechanism between the cement and water [11]. In our case the chemical reaction in which the Portland Class-G cement is combined with tap water a1nd form chemical bond with the water molecules and then it became hydrated.

### 2.1. Materials

For Tap water with a salinity of 2000 ppm was employed in the preparation of the cement slurry. Class G oil well cement stands as the predominant choice within the oil and gas sector, revered for its exemplary hydraulic binding properties. Constituting a primary component of Portland cement, its chemical composition primarily comprises  $\text{Al}_2\text{O}_3$ ,  $\text{CaO}$ , and  $\text{SiO}_2$ . Furthermore, Portland Class-G cement is renowned for its medium to high sulphate resistance characteristics.

### 2.2. Characterization tools

Various analytical instruments were employed to assess the essential characteristics of the prepared nano silica, encompassing both structural and morphological aspects. X-ray diffraction (XRD) patterns were ascertained utilizing  $\text{Cu K}\alpha$  X-ray radiation ( $\lambda = 1.540 \text{ \AA}$ ) from PAN analytical X'PERT PRO, Germany. Dynamic Light Scattering (DLS) with a laser angle of  $90^\circ$  at  $25^\circ\text{C}$  facilitated the determination of particle size distribution at ambient temperature.

Furthermore, High-Resolution Transmission Electron Microscopy (HR-TEM) conducted on a JEOL JEM-2100F instrument operating at 200 KV, Japan, enabled detailed characterization of the nanoparticle morphology.

The evaluation of compressive strength development over time was facilitated by the Ultrasonic Cement Analyzer (UCA). This apparatus offers a continuous, non-destructive means to monitor compressive strength evolution as a function of time. The procedure involves introducing the prepared cement slurry into a cell, wherein pressure and temperature are adjusted to simulate downhole reservoir conditions. Acoustic signals are then transmitted through the sample, with the velocity of signal propagation indicating changes in cement strength over time.



Fig. 1: OFTITE UCA[5]. .

### 2.3. Synthesis of nano-silica by Sol-gel technique

Ethyl Alcohol (EtOH) was combined with Tetraethoxysilane (TEOS) in a 1:1 ratio, followed by the addition of droplets of Ammonium Hydroxide (NH4OH) to catalyze and initiate the reaction. The resulting solution underwent hydrolysis while being stirred at a constant speed of 400 rpm at room temperature for two hours.

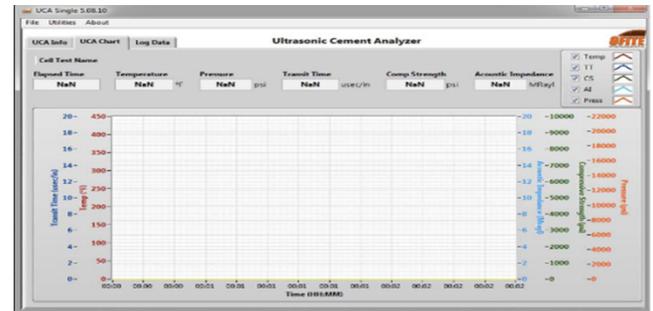


Fig. 2: Ultrasonic cement analyzer output Chart [11].

Following this, the solution was allowed to rest at room temperature for four hours to ensure completion of the reaction. Subsequently, the nano dispersed silica solution was prepared and can either be directly applied to the targeted area or further processed into a gel and dried to obtain nano powder. This solution was then subjected to heating at 250°C for 3–4 hours, leading to the transformation of the solution into silica nano powder.

- \*Elapsed time: Time since the test began.
- \*Temperature: The temperature within the test cell (degree F or degree C)
- \*Pressure: The pressure within the test cell (PSI or MPa)
- \*Transit time: The time required for the sound wave to travel through the sample (Microseconds)
- \*Compressive Strength: The calculated compressive strength of the sample (PSI or MPa)

Silica sol was prepared from a 20ml tetraethylorthosilicate (TEOS, Aldrich) precursor, ethanol (Si/ EtOH = 0.15), HNO3 (HNO3/ Si= 0.265) and water (H2O/Si= 8.1). After two hours of mixing, the resulting solution was used for silica film deposition by the dip-coating technique with pulling speeds up to 5 cm min<sup>-1</sup>. The resulting film is denoted as SO. The sol was unstable and therefore, was always freshly prepared. All silica films were produced by a single dipping. The silica films on soda–lime glass was used as substrates for the preparation of thin films that will be described in next section.

### 2.5. Laboratory design

#### 2.4. Ultrasonic cement analyzer (UCA)

The Ultrasonic Cement Analyzer (UCA) offers a range of parameters, including temperature (°F), pressure (Psi), and transit time (µsec/in). Experiments are conducted under constant temperature and pressure conditions: 2000 Psi and 230 °F. Ultrasonic waves are emitted through the cement slurry, providing transit time data.

The cement sample is prepared API SPEC 10 A as per table 1. The sample is then placed in the mixer and then maintained at 4000 rpm and the cement is added gradually within 15 seconds. Then, the mixing is continued for 35 sec at a uniform speed of 500 rpm. The nano silica is added while mixing the cement .The slurry mix was then added to UCA, and the pressure & temperature were adjusted to 2000 Psi & 230 ° F. The compressive strength development was monitored for 24 hours, then the sample was recovered from the device.

Table 1: Slurry Requirements API Class of Cement

Mix Water	175 G
Cement	3 G

Figure 1 & 2 represent the schematic presentation of the experimental set up. The UCA employs a continuous, non-destructive method to monitor compressive strength over time. Compressive strength is inferred from changes in the velocity of ultrasonic waves transmitted through the cement slurry. As the cement develops compressive strength, transit time decreases progressively.

## 3. Results

### 3.1 XRD analysis for the prepared nano-silica

Figure 3. depicts XRD patterns of the prepared nano silica. These peaks show the crystalline structure of silica. The 25-

degree peak confirms the formation of the nano silica materials which is matched with that matched with the Joint Committee on Powder Diffraction (JCPDS) file no [COD 98015 – 3866]. Further particle size was evaluated using Scherer's formula which is discussed below:

$$D = 0.94\lambda / \beta \cos \theta$$

D is the average crystalline size of the particles,  $\lambda$  represents the wavelength of x ray,  $\beta$  is the full width at half maximum, and  $\theta$  is the Bragg's diffraction angle [2]. The crystallite size of the sample is 131 nm.

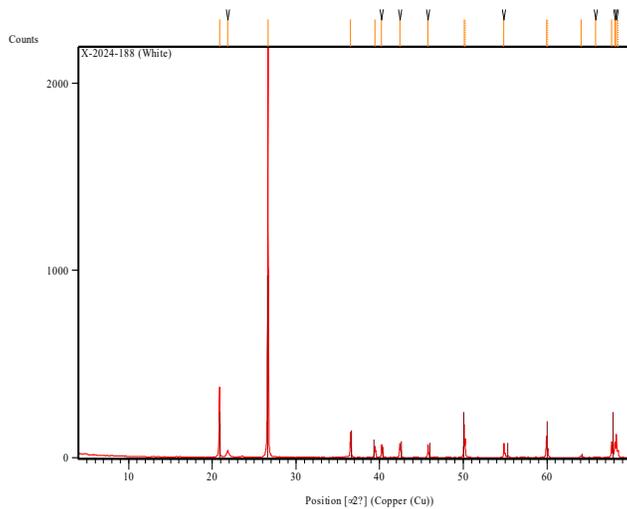


Fig. 3: XRD of Nano-silica. “by author”

### 3.2. DLS analysis for the prepared nano-silica

The prepared nano silica has an average size particle of 131 nm. As seen from figure 4. The analysis of the particle size confirms that the nano particles are present in the solution. The zeta potential is 297.1 & PDI is 0.393 which indicates the high stability of the prepared nano silica.

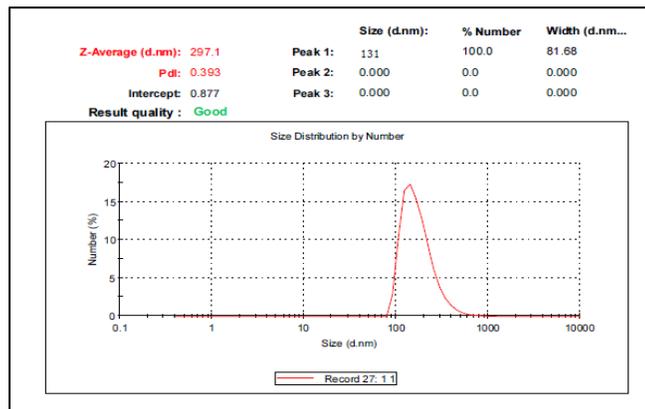


Fig. 4: DLS of Nano-silica. “by author”

### 3.3. Tem analysis for prepared nano silica

As depicted in figure 5, the TEM image of Nano-silica

exhibits a spherical shape. The average diameter of is approximately 131 nm which matches both XRD and DLS analysis.

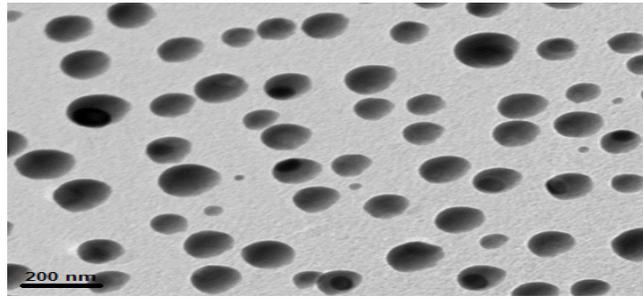


Fig. 5: TEM image of Nano-silica. “by author”

### 3.4. Cement samples mixed with nano silica

#### 3.4.1. Test1: 15.8 PPG cement sample with zero % nano silica.

Test 1: The 15.8 PPG sample was meticulously prepared by combining 396 grams of cement with 167 grams of tap water in accordance with API guidelines. Subsequently, the sample underwent testing within the Ultrasonic Cement Analyzer (UCA) under well-simulated conditions of 2000 Psi and 230°F. Figure 6. displays steady increase in compressive strength, with stabilization commencing after approximately 65 hours. Notably, the slurry exhibited initial hard set within 4 hours and 10 minutes, achieving a compressive strength of 3587 psi after 24 hours.

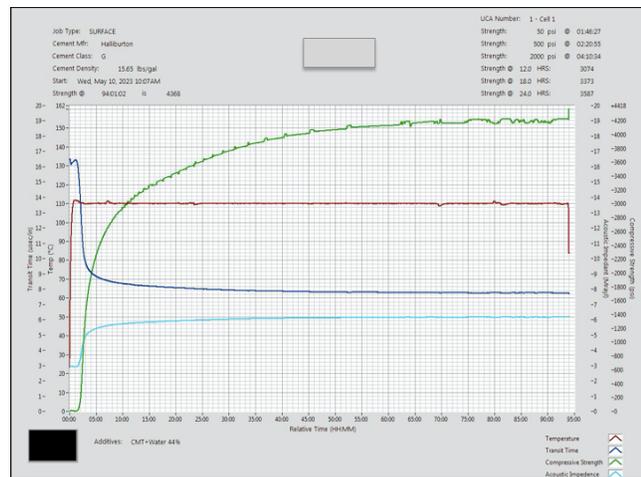
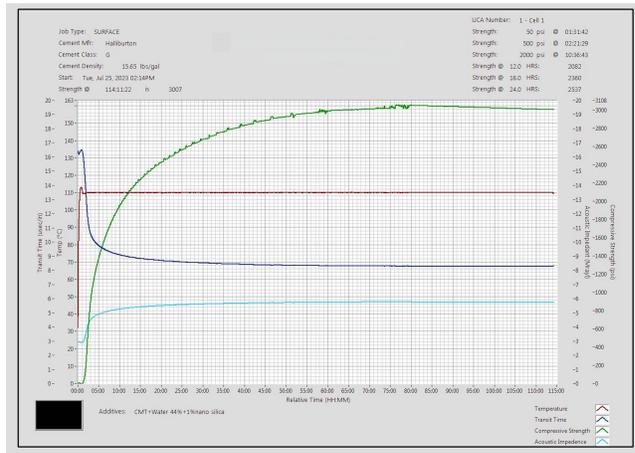


Fig. 6: UCA test performed with 100 % cement. “by author”

#### 3.4.2. Test 2: 15.8 PPG cement sample imbedded with 1 % nano silica.

Test 2: The 15.8 PPG sample was meticulously prepared by blending 396 grams of cement with 167 grams of tap water in adherence to API guidelines. Additionally, 1% BWOC (by weight of cement) of nano silica was incorporated into the sample. Subsequently, the sample underwent testing within the Ultrasonic Cement Analyzer (UCA) under well-simulated conditions of 2000 Psi and 230 °F. As it could be

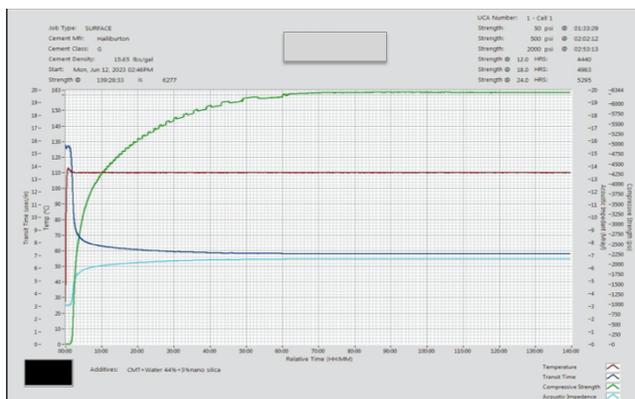
noticed in figure 7, that there is gradual increase in compressive strength, with stabilization commencing after approximately 75 hours. Notably, the slurry exhibited initial hard set after 10 hours and 36 minutes, achieving a compressive strength of 2537 psi after 24 hours.



**Fig. 7:** UCA with 100 % cement & 1% BWOC nano silica. “by author”

**3.4.3. Test 3: 15.8 PPG cement sample imbedded with 3 % nano silica.**

Test 3: The 15.8 PPG sample was meticulously prepared by combining 396 grams of cement with 11.8 grams of Nano-Silica (3% BWOC), along with 167 grams of tap water in accordance with API guidance. Subsequently, the sample underwent testing within the Ultrasonic Cement Analyzer (UCA) under well conditions of 2000 Psi and 230 °F. Observations revealed a gradual increase in compressive strength, with stabilization commencing after approximately 55 hours as it could be noticed from figure 8. Notably, the slurry exhibited initial hard set after 2 hours and 53 minutes, achieving a compressive strength of 5295 psi after 24 hours.

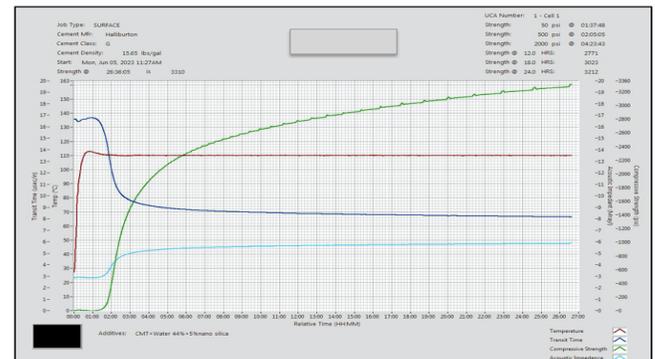


**Fig. 8:** UCA test performed with 100 % cement & 3 % BWOC nano silica. “by author”

**3.4.4. Test 4: 15.8 PPG cement sample imbedded with 5 % nano silica.**

Test 4: The 15.8 PPG sample was meticulously prepared by blending 396 grams of cement with 19.8 grams of Nano-

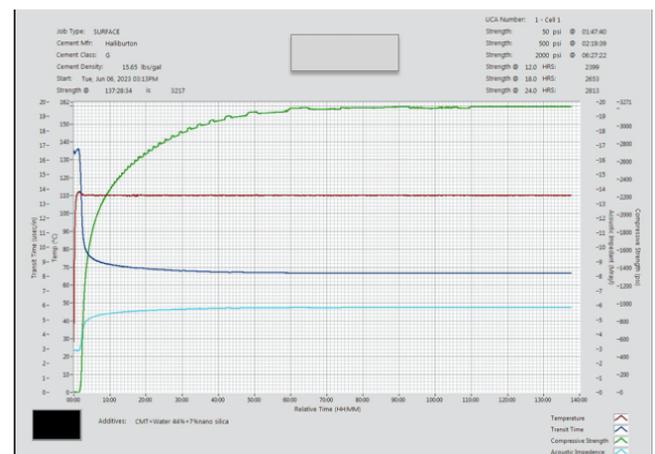
Silica (5% BWOC), alongside 167 grams of tap water in accordance with API guidelines. Subsequently, the sample underwent testing within the Ultrasonic Cement Analyzer (UCA) under well conditions of 2000 Psi and 230 °F. Figure 9. revealed a gradual increase in compressive strength, with stabilization commencing after approximately 26 hours. Notably, the slurry exhibited initial hard set after 4 hours and 23 minutes, achieving a compressive strength of 3212 psi after 24 hours.



**Fig. 9:** UCA performed with 100 % cement & 5 % BWOC nano silica. “by author”

**3.4.5. Test 5: 15.8 PPG cement sample imbedded with 7 % nano silica.**

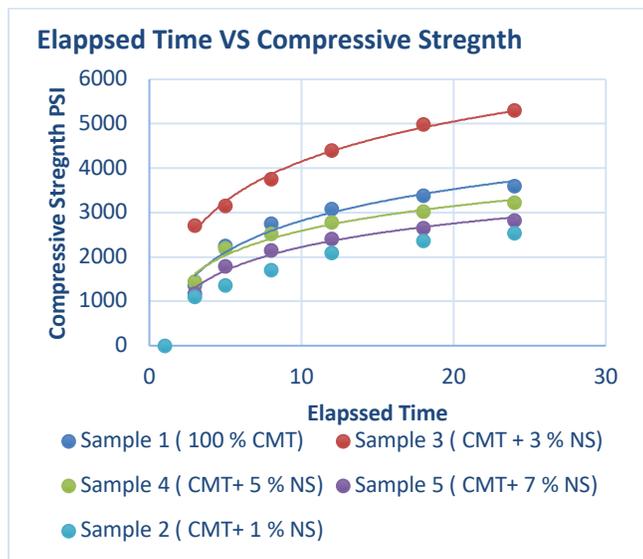
Test 5: The 15.8 PPG sample was meticulously prepared by combining 396 grams of cement with 27.72 grams of Nano-Silica (7% BWOC), along with 167 grams of tap water in adherence to API guidance. Subsequently, the sample underwent testing within the Ultrasonic Cement Analyzer (UCA) under well conditions of 2000 Psi and 230 °F. Figure 10. indicates a gradual increase in compressive strength, with stabilization commencing after approximately 60 hours. Notably, the slurry exhibited an initial hard set after 6 hours and 27 minutes, achieving a compressive strength of 2813 psi after 24 hours.



**Fig. 10:** UCA test performed with 100 % cement & 7 % BWOC nano silica. “by author”

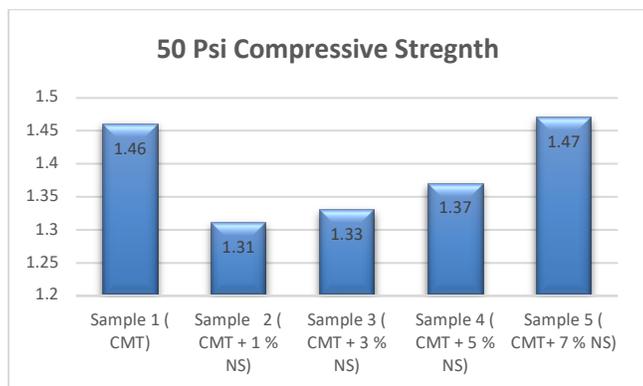
### 3.5. Results analysis

The findings are summarized in figure 11 where it shows that Sample 3 (CMT + 3 % NS) showed the highest compressive strength development in different elapsed time. However, by increasing the NS percentage there is an obvious decrease in the cement compressive strength. The decrease in the compressive strength at high number of NS is credited to the move voids which were formed by the large number of small particles in the cement. After 3 hours, it was noticed variance in the compressive strength among the 5 samples where sample 3 exceeded the compressive strength of the other samples by 1200 psi and after 24 hours there was increase in the compressive strength by 60 %.



**Fig. 11:** Elapsed Time versus compressive strength for cement samples.

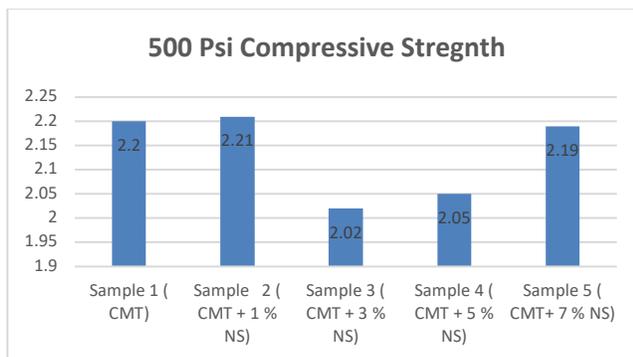
The time to 50 Psi is known to be the initial set for the cement where the cement compressive strength is enough to support the casing [14]., it is noticed that sample 3 reached the initial set after 1.3 hours which is earlier than the other 4 sample as it could be noticed from figure 12.



**Fig. 12:** 50 Psi compressive strength for cement samples.

The time to 500 Psi is also a critical time as it is considered to be the time to drill a cement plug or perforate the casing

[14]. Figure 13 highlighted the results of the five samples where it is noticed that sample 3 reached the 500 psi 30 minutes earlier than the other samples.



**Fig. 13:** 500 Psi Compressive Strength for Cement Sample.

Using lower than 3 % of nano silica may interact with other chemicals during the mixing process so leading to loss of nano silica & leading to diminishing & thus affecting the overall performance of the nano silica. when the percentage of nano silica is very low these bonding many are not formed and may result in a weaker interfacial transition zones and therefore leading to poor stress transfer between the particles and high reduction of the cement compressive strength.

Adding high concentrations of nano silica “more than 3 % leads to particles agglomeration where these particles clump together rather than imbedding with the cement matrix and thus creating area of weakness and thus decrease the cement compressive strength. It also could lead to excessive pore filling which may result in denser cement samples but making the cement more brittle. It also hinders the movement of water which affects the development of the cement compressive strength.

The high percentage of the nano silica could disturb the packing of the particles & forming overcrowded particles. This led to weakened interparticle bonding and this decreases the cement compressive strength. The high surface area of the nano silica may lead to delay in the cement hydration and decrease the ability of the cement to absorb water. This delay could lead to the inadequate development of the cement microstructure and leading to the decrease of the cement compressive strength.

## 4. Conclusions

The addition of nano silica to the sample significantly enhanced the cement properties. Employing optimal concentrations of nano silica expedited cement hydration while augmenting cement density. Furthermore, nano silica played a pivotal role in improving the compressive strength of the cement by effectively filling voids within the cement matrix. As the percentage of nano silica (NS) surpassed 5%, there was a notable decrease in the early strength development. Samples 3 and 4 exhibited compressive strengths ranging from 50 to 500 Psi. Notably, Sample 3

attained a compressive strength of 2000 Psi more rapidly compared to the other samples, with Sample 2 following closely behind. This observation underscores the significant influence of nano silica on the early phase of hydration reaction. However, increasing the content of nano silica did not appear to significantly impact the development of early compressive strength. Preparation of this type of cement renders it suitable for a multitude of applications, including utilization as side-track cement plugs or in plug and abandonment operations. Such applications can be executed more efficiently, resulting in reduced rig time. Furthermore, proper mixing of this cement with optimal concentrations enhances its durability, particularly in plug and abandonment scenarios.

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