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**Original Article**

# STRENGTH DEVELOPMENT OF OILWELL CEMENT SHEATH UNDER THE INFLUENCE OF HIGH FERROUS ION CONCENTRATION IN MIXING WATER

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**Abstract:** This study investigated the effect of high ferrous ion ($Fe^{2+}$) concentration in mix-water on the compressive strength development of oilwell cement sheaths. Eight groundwater samples were collected and analyzed for physicochemical properties, then used to prepare cement slurries in accordance with API Specification 10A (2019). The slurries were cured under simulated downhole conditions to produce cement sheath cubes, which were subjected to compressive strength tests. X-ray diffraction (XRD) was employed to identify chemical compounds in failed cement sheath samples. Results show that compressive strength decreases as $Fe^{2+}$ concentration in mix-water increases, particularly when levels exceed 0.9 mg/L. Compressive strength losses ranged from -288.37 psi to -587.70 psi under various curing conditions, with higher temperature and extended curing time exacerbating the reduction. Mechanistically, the declassification of calcium silicate hydrate (C-S-H) into Fe-S-H and the dissociation of calcium from $Ca(OH)_2$ into $Fe(OH)_2$ were identified as the primary causes of strength loss. Notably, applied pressure did not significantly influence the antagonistic behavior of ferrous ions. These findings highlight the critical impact of ferrous ion concentration on the mechanical integrity of Class G oilwell cement sheaths and underscore the need for water quality control in cement slurry preparation to ensure optimal downhole performance.

**Keywords:** Ferrous Ion, Cement Sheath, Compressive Strength, Oilwell Cement, Cement Slurry

## 1. Introduction

Cementing of oilwell is the process, which involves the mixing of mix-water, powdered Portland oilwell cement, and probably with additives, to form a mixture called cement slurry; further on, placing the cement slurry through the inner diameter of a casing string to a desired depth between the annular space of a casing string or liner and an openhole in a wellbore, and left to hardened in hours or days (Umeokafor and Joel, 2010). In this process, the cement slurry is allowed to set and get hardened with respect to time, to seal off the annular space, and frustrate the movement of formation fluid. The key objectives of well cementing, are to seal off the annular space between the outer diameter of casing string or the liner and the openhole in a wellbore (Backe *et al*., 1998). Additionally, for complete zonal isolation to be attained, all drilling mud and rock cuttings suspended in the fluid must be removed from the annular space, and be replaced by a desired designed cement slurry (Umeokafor and Joel, 2010). Further on, the placed cement slurry must first undergo hydration under the oilwell's *in-situ* complex geologic and operating prevailing wellbore conditions, then metamorphosed from the liquidplastic state to the solid phase. At this stage, the solid phase exhibits an impermeable sealing characteristics, which prevents the inter- and intra-flow of formation fluids, and also gives structural support to the casing strings and surface rig equipment. For clarity, this subsurface rigid solid phase is also known as cement sheath. Therefore, in terms of uncompromised well integrity, the cement sheath should be able to withstand different wellbore and reservoir operations, such as perforation of the producing zone, enhanced oil recovery or stimulating production, production, optimisation, and intervention during the life-cycle of the productionwell, and even abandonment (Joel and Ademiluyi, 2011). On the other hand, because cementing commences with drilling and casing, and ends with completion; which at the completion stage the well is hurriedly put into production, to exploit the crude oil or natural gas at the payzone; to recover oil revenue or profit oil, to pay back cash used for the investment; this singular desperation may result in poor primary cementing job. This explained that, the time and technical commitments needed, to conduct a good cement job were not contemplated and considered properly at the slurry design stage. As a result, a costlier and time consuming corrective method(s) may be applied to correct the poor primary cement job of failed compressive strength. This type of corrective cementing is known as secondary or remedial cementing.

The compressive strength of cement sheath discloses the ability of the rigid cement bond between the annulus of the outer-diameter of casing string and formation wall at prevailing wellbore conditions, to withstand loads or compression force tending to reduce its size, as contrasting to tensile strength, which withstands loads tending to elongate the cement rigid body. In other words, compressive strength repels compression (being pushed together), whereas tensile strength repels tension (being pulled apart). To achieve these, the mix-water used, to formulate cement slurry must be potable (Mindess and Young, 1981; Nelson and Guillot, 2006; Neville and Brooks, 2010; Neville, 2011). These potable or drinking water standards are stated in documents of reputable organisations (WHO, 2004; NIS, 2007). However, most times at rig sites onshore, the mix-waters used from groundwater, to hydrate cement powder into cement slurry are always not designed to pass through some water reticulation systems (Yousuo *et al*., 2019); hence, a compromise in the cement sheath compressive strength occurs.

### 1.1 Study Area

The research opted for Kolo Creek area as a case study, because of the high activities of oil exploration and production, including the high content of ferrous groundwater in the area (Gordon and Enyinaya, 2012). Kolo Creek is located in Ogbia Local Government Area of Bayelsa State, Nigeria (Figure 1). Kolo Creek on the Global Positioning System (GPS) position is approximately at Latitude 4.6667°, Longitude 6.3333°, which an exploration and production company, had since 1964 been the sole international oil company (IOC) that operates the oil mining leases (OMLs) of between 35 and 36 (Adesuyi, 2015). The major hydrocarbon produced from this area is crude oil. Though, as at 2016, more than 40-well have been drilled, which more than 33-well have been in the level of production. These oilwells are classified as near high-pressure and high-temperature (NHPHT) as depicted in both Figures 2 and 3. Currently, most of these wells are producing to Gbaran oil gathering facility (World Industrial Information, 2016). In addition, based on my personal conversations with several Community Leaders at Kolo Creek (Otuasaga, Oruma, Imiringi, Kolo 1, Kolo 2, Kolo 3, Emeyal 1, Emeyal 2, etc.) communities, and some ad-hoc staff of the operating IOC, it was disclosed that, the IOC do always conduct some remedial (maintenance) cementing jobs on some producing wells, when a wellhead oil leak is

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obvious. Hence, this research on the "Effect of High Ferrous ion Concentration in Mix-Water on the Compressive Strength Development of Oilwell Cement Sheath". Thus, this study is expected, to yield sustainable outcome of identifying the rate at which different thresholds of ferrous ion concentration in mix-water impact negatively on the compressive strength of oilwell cement sheath, at different simulated wellbore conditions. This is aimed to discover the allowable limit of ferrous ion concentration in mix-water, that would make the cementing of oilwells safer, and economically viable.

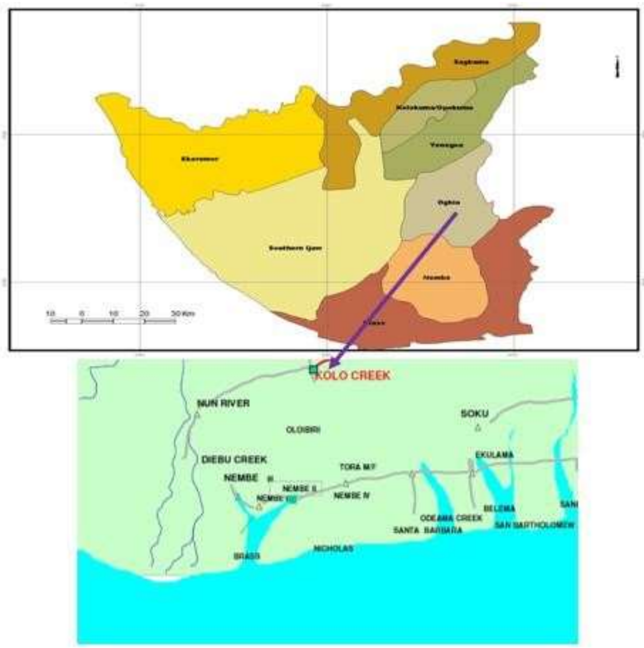


**Figure 1.** Map of Bayelsa State: inset map showing location of studied area (Kolo Creek) in red-like color. Modified from Creek (2004) and Adesuyi (2015).

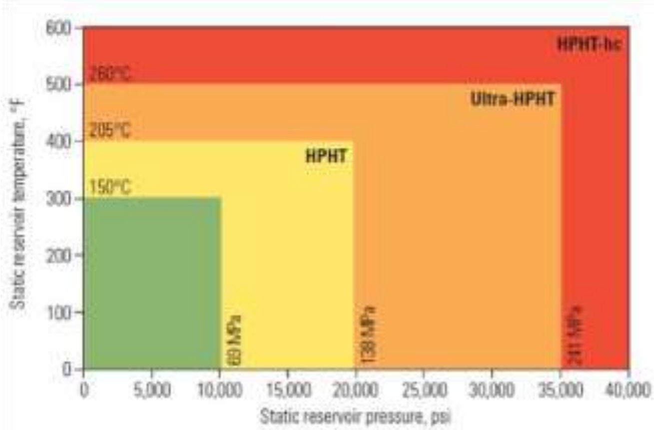


**Figure 2.** HPHT Threshold classification for Reservoirs (DeBruijn *et al.*, 2008).

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**Figure 3.** HPHT projects around the world (DeBruijn *et al*., 2008).

## 2. Background

Mix-water is an essential component in the formulation of cement slurry (Smith, 1976). In the cement slurry system, cement is the binding material, and it is hydrophilic, which means it needs water to form cement slurry (Kiran, *et al.*, 2017). Furthermore, the cement slurry designer must be cautious about the quantity, and quality of the mix-water; to be used in the formulation of any cement slurry. This means that, to design an economical, successful, and usable cement slurry; the quantity, and quality of the mix-water must be put into some rigorous engineering evaluations before its design, and usage. The quantity of mix-water used to mix any graded powdered Portland oilwell cement is very important; because, this is aimed to prepare a reasonable cement slurry. Nevertheless, this cement slurry is obtained by an acceptable specified ratio of mix-water and powdered cement (w/c), subject to an approved API mixing specification (Azar and Samuel, 2007; API Specification 10A, 1995). Accordingly, Azar and Samuel (2007) inferred that a reasonable w/c ratio of about 2.8 gal/sack should be applied for Class G Portland oilwell cement to obtain a compromised cement slurry. In the same vein, Azar and Samuel (2007) further explained that, this cement slurry cannot be pumped to the targeted well depth, since its viscosity is too high, its pumpability would be very poor. However, Azar and Samuel (2007) opined that, to remedy this poor pumpability, much mix-water should be added into the cement slurry. In contrary, Azar and Samuel (2007) courteously stated that, at static conditions, it was acknowledged that, if excess quantity of mixwater, is been used to prepare the cement slurry; there might be the existence of free water, as supernatant at the top of the well column, leaving the cement slurry as residue. This also includes decrease in cement sedimentation stability (Minaev *et al.*, 2014). In the aftermath, the free water in turn creates some channels and some pocket of pores in the hardened cement sheath. In consonant with the term free water, Bourgoyne *et al*. (1986) earlier explained that excess quantity of mix-water not used by tobermorite (Calcium Silicate Hydrate, or C-S-H, or

$3CaO.2SiO_2.3H_2O$) during the powdered cement hydration reduces the cement's compressive strength, and creates channels that make the cement sheath more porous and permeable. Hence, this cement sheath characterized with more porous and permeable features will not only allow subsurface fluids communication between formations, but to the surface; which gradually ceases to give structural support to the subsurface casing string, and surface rig equipment. Consequently, these disadvantaged appearances of pores and channels in the cement sheath does not only pave way for poor compressive strength of the cement sheath, but exhibit good permeability of formation fluids through the channels, and poor durability of the cement sheath at subsurface conditions. Similarly, Atahan, Oktar, and Tasdemir (2009) in a research conducted on the effects of w/c ratio and curing time on the critical pore width of hardened cement past; the research used five mixes of Portland cement paste with the w/c ratios of 0.26, 0.30, 0.34, 0.38, and 0.42. Subsequently, all the specimens were cured in water saturated with lime for 7, 14, 28, and 365 days; and the specimens with w/c ratios of 0.26, 0.34, and 0.42 were cured for 7, 28, and 365 days, and were subjected to a mercury intrusion porosimetry (MIP) tests. These tests findings indicated that, the critical pore width of the hardened cement paste (HCP) appear to be dependent of the w/c ratio, and is of the order of approximately 25 nm; which is considered as the critical pore width of the Portland cement gel. This also affected the pumpability or thickening time of cement slurry; and on the other hand, the compressive strength, permeability, soundness, and durability of the cement sheath. In another developmental study, Zhang *et al.* (2003) inferred that when the w/c ratio decreases, the total or bulk shrinkage of cement sheath increases. Also when Yasar *et al.* (2004) studied the impact of w/c and coarse limestone aggregate type on the compressive strength of cement sheath at atmospheric conditions, it was observed that the compressive strength of the sheath inversely depended on the w/c and aggregate size of the limestone. In addition, Singh *et al.* (2014) worked on the role of w/c ratio on the strength development of cement bond, and the disclosed results showed that compressive strength and split tensile strength of the cement hardened rigid body decreased with an increase in the w/c ratio. This was also illustrated by Felekoglu *et al.* (2007) in a study that investigated the effect of w/c on the fresh and hardened properties of self-compacting concrete (Figure 4). Also, in a recent study, Haach *et al.* (2011) explicitly concluded that when the mix-water used to prepare a cement slurry is greater than the required quantity, the slurry's pumpability increases, while the compressive strength decreases dramatically and detrimentally. These confirmed with Crooks (2006) previous explanation, that free water in cement slurry should be prohibited. Based on these principles, Igbani *et al.* (2018) also disclosed that the excess mix-water not used by calcium silicate hydrate during cement slurry preparation is known as excess water, and it is very detrimental to both the permeability, and compressive strength. In another word, the supernatant water, which residences in the pore system of the cement–water paste, turns to be the source of water for hydration during the hardening period of cement sheath at subsurface. This excess water is often termed ‘‘free water’’; it can be diffused through or under conditions of high temperature or dryness (ANSI/ANS-6.4-1985, 1985). But, in the contrary, when the w/c is higher, and when water-retaining agents are not added into the cement slurry; there would be high cement segregation and increase in cement filtration (Minaev *et al.*, 2014).

In summary, the aforementioned reviewed literatures were based on the impacts of w/c on the properties of cement hardened rig body. Also, these impacts were observed under atmospheric, and oilwell prevailing conditions. Accordingly, the consulted literatures disclosed that, higher value of w/c strongly and inversely affects the compressive strength development of cement sheath (Figure 5). In addition, the cement sheath's permeability is also affect by the w/c ratio. These were as a result of the pocket pores and channels created by excess free water from the mix-water. On the contrary, these literatures did not disclose the precursor on the impact of ferrous ions in mix-water on cement sheath properties. Hence, a review on vital literatures on the impact of quality of mix-water on compressive strength of cement sheath was conducted. This may provide a precursor on the impact of ferrous ions in mix-water on cement sheath properties.

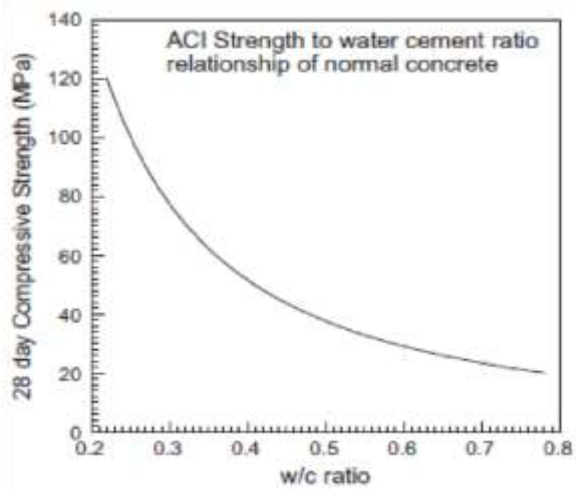


**Figure 4**. Relationship between cement compressive strength and w/c (Dinakar *et al*., 2013).

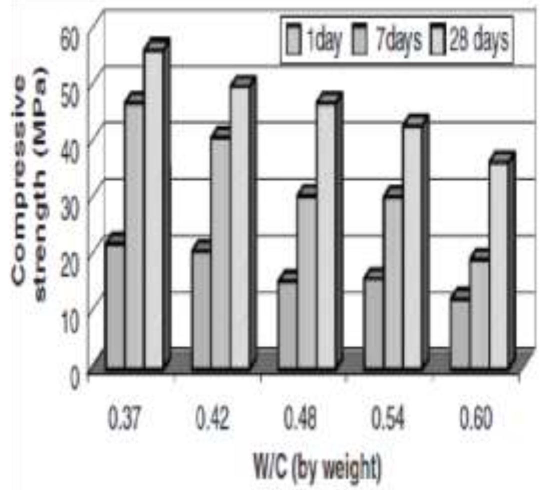


**Figure 5**. Effect of water/cement ratio on the fresh and hardened properties of self-compacting concrete (Felekoglu *et al.*, 2007).

Furthermore, with regards to mix-water quality, Saleh *et al*. (2018) conducted some destructive tests on some cured cubes of cement, to ascertain the impacts of mix-water quality on compressive strength of some cured cubes of cement sheath. On this backdrop, three sets of cement slurries were formulated using five different types of mix-waters: soft water, medium hard water, very hard water, seawater, and field water; where in each of these sets, “A to E” were used to label the specimens. These gave a total of fifteen specimens in three groups. In addition, each of the groups (that is the first-, second-, and third-group) of the prepared cement slurries were cured for one day, three days, and seven days, respectively. Subsequently, each of these groups of cured cubes of cement were subjected to a certified crush test of unconfined compressive strength (UCS) measurement based on API Specification 10A (API Specification 10A, 1995). After these tests, Saleh *et al*. (2018) observed that, cement slurry prepared with seawater and cured for one day had an approximated average compressive strength of 3916.01 psi, while the others had an approximated average compressive strength of 2900.75 psi. Conversely, from the study, cement slurry prepared with seawater and cured for three days gained additional 580.15 psi, while those cured cubes, which mix-water were soft water, field water, other degrees of hard waters gained additional 5221.36 psi, 5511.43 psi, and 4931.28 psi, respectively. Furthermore, after seven days of curing, the results of the UCS measurement revealed that the cured cubes from those of soft water had the highest UCS value, which was recorded as 7251.89 psi; whereas the others had a range of 44-47 UCS. These results on the UCS measurements disclosed that, when cement slurry is prepared with soft water it gave the cement sheath a high compressive strength; while the others gave the cement sheath short term compressive strength perhaps due to high C-H-S active hydration in soft water. Similarly, Nelson (1990) in a study explained that, the use of non-potable mix-water reduces the compressive strength of cement sheath up to 20%. Therefore, a properly engineered design of cement slurry that would generate the required cement sheath’s compressive strength for an oilwell, should meet all potable mix-water requirements (Suman and Ellis, 1977; Sabins and Sutton, 1983). Then, the required compressive strength to determine the ultimate strength for the perforation of the cement sheath at the payzone, complete zonal isolation, and support for the casing string and other surface equipment would be achieved (Smith, 1976). In this vein, researchers such as Mindess and Young (1981), Mehta (1986), and Raina (1988) at different time intervals have explained that mix-water used for mixing powdered cement should have the same quality as potable water fit for human consumption. On this background, Reddy and Ramana (2018) pointed out that, since cement sheaths were used as zonal isolation in oilwell, potable water has to be used as the mix-water in cement slurry formulation, due to the known chemical composition of potable water. The implication of this, is that mix-water should have the same physicochemical properties of drinking water, as stated in the world health organisation standard of drinking water (WHO, 2004). Accordingly, the WHO's acceptable standard of ferrous ion ($Fe^{2+}$) is 0.3 mg/l in potable water (WHO, 2004; Nigerian Industrial Standard, 2007). Generally, a maximum permissible concentration of $Fe^{2+}$ in potable water supplies is 1 mg/l; and conversely, a dose of 1500 mg/l of $Fe^{2+}$ is capable to cause more negative health impact of poisoning human, as it can damage blood tissues mostly in children. Also, in both adults and children it can cause digestive disorders, skin diseases and dental problems (Khan *et al*., 2000).

Therefore, any mix-water that the concentration of $Fe^{2+}$ is above 0.3 mg/l would be detrimental to the mechanical health of the cement sheath, based on the principles of Mindess and Young (1981); Mehta (1986); Raina (1988) on the quality of mix-water. Similarly, in another study, Ko and Batchelor (2010) added some quantity of $Fe^{2+}$ mix-water to form cement slurries, which it was observed that the $Fe^{2+}$ is likely to be associated with thin hexagonal plates. Though, the study did not determine if the $Fe^{2+}$ has structural impact on cementing at subsurface conditions. Similarly, in another developmental study on the effect of lead (Pb) in mix-water on the compressive strength of cement mortar. Madhusudana *et al.* (2011) spiked lead into deionised water in known concentrations. These lead contaminated mix-water were mixed with Ennore sand to prepare cement mortar specimens. During this investigation, it was observed that, the presence of lead ion concentrations approximately higher than 3000 mg/L in the mix-water reduces the compressive strength of the mortar concrete, when compared with the reference specimen. In cause this observation, Madhusudana *et al.* (2011) concluded that the presence of the heavy metal of lead causes the strength loss of the concrete; that the strength loss was as a result of the dissociation of calcium hydroxide and decalcification of tobermorite or Calcium Silicate Hydrate. However, Madhusudana *et al.* (2011) study was conducted at atmospheric conditions not at HPHT subsurface conditions.

Technically, still now IOC uses the available mix-water at rig sites, which are not reticulated at rig site to reduce the $Fe^{2+}$ concentration to as low as reasonably practicable (ALARP). Naturally, many groundwater aquifers contain soluble iron, ferrous ion, ($Fe^{2+}$) which is as a result of *in-situ* aerobic bacteria activity. Nonetheless, this explains that some aquifers also contain soluble iron, ferrous iron ($Fe^{2+}$), which is due to the absence of oxygen and the absence of aerobic bacteria. This underground water associated with $Fe^{2+}$ when pumped to the surface is always colourless due to the *in-situ* sand-gravel-filtration. On the other hand, at the rig sites, $Fe^{2+}$ is eliminated from groundwater by oxidation, detention, settlement and filtration (Akl, *et al.*, 2013). Consequently, Ashraf (2005) stated that the oxidation of $Fe^{2+}$ involves the loss of electron or the increase of oxidation number to ferric, $Fe^{3+}$ giving ferrous ground water a turbid yellow-brown to black color. Additionally, Siabi (2008) affirms that the aforementioned process can be enhanced by the introduction of oxygen by the combined application of aeration, the regeneration of mix liquor (biomass) of bacteria (*nitrobacter* and *nitrosomonas*) using air blowers, and the dosing of oxidising agents (chlorine, hypochlorite, chlorine dioxide, ozone or potassium permanganate) into the reactor. Therefore, to reduce the presence of $Fe^{2+}$ to ALARP, the process is followed by rapid filtration/adsorption and post-disinfection in sequence. On this backdrop, different methods to remove $Fe^{2+}$are in use now, but the optimisation of these methods for better, cheaper and safer technique will never stop (ChiChuan, 2012; Yousuo *et al*. 2019). However, due to the global geometric increase in population and industrialisation; huge volume of limited potable mix-water is continuously being consumed at a faster rate (Reddy and Ramana, 2018). As a result, most of the cementing servicing companies in the oil and gas exploration and production (E&P) sector are still making use of non-potable water as mix-water.

## 3. Materials and Methods

### 3.1 Materials

#### 3.1.1 Ferrous Mix-Water

The mix-water samples were randomly collected from eight (8) locations in the study area, Kolo Creek, Nigeria (Figure 6). Before the sampling processes were conducted, each sampling from the boreholes were conducted after the *in-situ* mix-water had been hovered from the borehole for 20 minutes. This was done with the aid of a submersible pump, at the flow rate (Q) of 20 litre/minutes. This ensured that the sampling was from the desired uncontaminated groundwater-source in each of the boreholes. Each of the boreholes had a depth that is approximately below 60ft. Further on, after the 20 minutes of pumping, each of the 4-litre empty plastic container were washed and rinsed twice (2) with the supposed mix-water sample, to be collected at the borehole site; though, at every borehole sites mix-water was collected and poured into the rinsed 4-litre empty plastic container; then, the container was closed hermetically, to avoiding the appearance of air bubbles. Each of the containers had a label indicating the date, time, and location of the sample collected. The mix-water samples were transported to Niger Delta University chemistry laboratory for water-analysis (see results on Table 1).

**Table 1:** Ferrous ion ($Fe^{2+)}$ Concentration in the Sampled Mix-Water

| S/No | Water Samples | Locations | GPS | $Fe^{2+}$ (mg/L) |
|---|---|---|---|---|
| 1 | WS1 | Distilled Water | Nill | Nill |
| 2 | WS2 | Kolo 2 Izogbo | 4048’20’’N<br>6 022’33’’E | 0.52 |
| 3 | WS3 | Otuasege Otumisou | 4055’2’’N<br>6 023’33’’E | 0.73 |
| 4 | WS4 | Kolo 2<br>Otu-ogele | 4048’23’’N<br>6 022’32’’E | 3.80 |
| 5 | WS5 | Otuasege Otuwododo | 4055’5’’N<br>6 023’39’’E | 5.00 |
| 6 | WS6 | Kolo 1 Emelala | 4048’23’’N<br>6 022’32’’E | 5.20 |
| 7 | WS7 | Otuasege winners | 4054’51’’N<br>6 023’7’’E | 5.53 |

| | | | | |
|---|---|---|---|---|
| 8 | WS8 | Oruma Ebifro | 4055’2’’N 6 024’11’’E | 6.35 |
| 9 | WS9 | Kolo 2 Angala | 4048’22’’N 6 022’32’’E | 6.82 |

**Figure 6**. Sampled Ferrous Mix-Water from Kolo Creek.

### 3.1.2 Deionised Mix-Water

Deionised mix-water was used to formulate neat cement slurries. This mix-water is free from ions and impurities, and it was used to formulate the control cement slurries, and cured cubes of cement sheath. As a result of this, a 20-litre plastic can of high grade deionised mix-water was purchased from an industrial chemical dealer at Yenagoa, Bayelsa State of the Federal republic of Nigeria. The deionised water-mix was branded as ultra-violet treated and premium water quality. The deionised mix-water was also laboratory graded, and not intended for human consumption. The deionised mix-water was a product of Roshan industries (Figure 7). The deionised mix-water had a concentration of 0.00 mg/l of ferrous ion concentration.


**Figure 7**. Deionized Mix-Water

### 3.1.3 Class G Oilwell Cement

Class G oilwell cement was used for the preparation of the cubes of cement sheath (Figure 8.). This Portland Class G oilwell cement was provided by a Vendor to the petroleum engineering laboratory. Class G oilwell cement (OWC) is envisioned for use as oil and gas basic cement. The Class G cement composition had a mix-water and cement ratio factor of 0.45 (Azar and Samuel, 2007; API Specification 10A, 2019). It is used as a cement band, to bond casing and liner strings to the face of an oil and gas open-hole well. Although, Class G OWC is used for bonding between the depths of the surface casing string and 8,000ft. Total vertical depth; but, with the application of the right cement additives such as accelerators and retarders Class G OWC can be used to cover a wider range of depths (Vrtine, 1998). Since, the thickening times of Class G OWC is controllable with additives up to the temperature of $250^0$F, and is used for the cementing of production casing string (Vrtine, 1998; James and Las, 2017). Thus, Class G OWC is also suitable for experimenting and modelling of the impacts of ferrous mix-water on the cement slurry’s setting time, thickening time and rheological properties; and on the other hand the permeability, and compressive strength of the cement cured sheath.

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**Figure 8**. Class G Oilwell Cement

**3.1.4 X-ray Diffraction (XRD)**

Upon the delivery of Class G OWC at the petroleum engineering laboratory, the cement was packed separately in sealed plastic bags. Theses sealed bags were further sealed in five (5) empty paint plastic cans with each container containing 5 kg of the Class G OWC, to prevent the powdered cement from contacting moisture. In addition, an X-ray diffraction (XRD) equipment was used to test for the composition of the Class G OWC. Consequently, results from the tests were summarised and presented in Table 2. Further on, the surface area of the Class G OWC was measured by the BET method using nitrogen gas as the adsorptive medium in a Micromeritics Autosorb 2010 equipment with use of Equations 1 to 4, since the weight ratio of $Al_2O_3$ to $Fe_2O_3$ is greater than 0.64. This result is presented in Table 2 (Bourgoyne *et al.*, 1986).

$C_3S = 4.07*C – 7.6*S – 6.72*A – 1.43*F – 2.85*SO_3$ … 1

$C_2S = 2.87*S – 0.754*C_3S$ … 2

$C_3A = 2.65*A – 1.69*F$ … 3

$C_4AF = 3.04*F$ … 4

**Table 2:** Compound component and BET surface area of Class G oil well cement (wt. %).

| **CEMENT TYPE** | **COMPONENTS %** | | | | | **SURFACE AREA ($m^2/g$)** |
|---|---|---|---|---|---|---|
| | **$C_3S$** | **$C_2S$** | **$C_3A$** | **$C_4AF$** | **Gypsum** | **BET Method** |
| **Class G** | 62.93 | 14.82 | 0.57 | 11.34 | 1.8 | 1.00±0.0075 |

**3.2 Methods**

**3.2.1 Ferrous Cement Slurry Design**

The sampled ferrous mix-waters were used to prepare the cement slurries. Practically, these cement slurries were prepared based on the principles of API specification 10A (API specification 10A, 2019). In accordance with this specification, some cement slurries of mix water/oil well Class G cement ratios (W/C) of 0.45 were prepared with each of the 8 different mix-waters, which had different ferrous ions concentrations.

**3.2.2 Preparation of Ferrous Cured Cement Sheath Cubes**

The API Specification 10A, which is the specification for cements and materials for well cementing was adopted, and used, to produce the cured cubes of cement sheath (API Specification 10A, 2002). Accordingly, each of the prepared ferrous cement slurries were poured into a brass mold made-up of 4 compartments, which each is 2 by 2 squared-inch in dimension (Figure 9). Each of these compartments were filled to the brim, and pounded by a stir rod, to remove entrapped pockets of air in the cement slurry, and it was covered with a brass plate. Then, the covered mold, was cleaned off from excess slurry on its body, and the mold was greased. The grease was applied, to prevent leaks of slurry from the mold, and water channeling into the mold. Subsequently, the mold was put into the curing chamber's pressure vessel and covered by the plug (Figure 10). The curing chamber was then pressurised, to a final curing pressure, and final (targeted) curing temperature. The tests were conducted separately, for given hours, to yield low density cured cement cubes (Figure 11). The experiments were conducted intermittently at fixed temperature, pressure, and curing time between 200 and 250$^0$F; 2500 and 3000 psi; 6 and 8hrs, respectively. However, the initial operating curing temperature was recorded as 3$^0$F (Figure 12). During the next 30 minutes prior to the terminal time of the set curing time, the final curing temperature was always decreased to 30$^0$F, when the switch of cylinder cool was switched on, without the curing pressure being adjusted. Finally, during the next 10 minutes, the remaining curing pressure was drained, and the curing equipment was switch off from the electrical powered source. Afterwards, the mold was transferred from the pressure vessel into an 80$^0$F water-bath; to cool off the mold for 45 minutes. This same experiment was conducted for all slurries prepared, to produce the cured cubes. In addition, these cured cubes were further cured in a water bath for about 48 hours (Figure 13). Then, these water bath cured oil well cement cubes were subjected to compressive and tensile strength, and permeability tests.

**Figure 9**. Cement slurry poured into a brass **Figure 10**. The brass mold in the pressure mold vessel

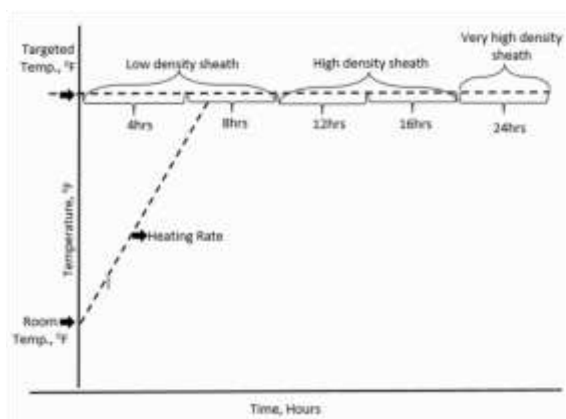



**Figure 11**. The pressure vessel covered by the **Figure 12**. Hours Allotted for curing plug.

**Figure 13**. Further curing of produced cured cement cubes in water for 48 hours

### 3.2.3 Compressive Strength of Cement Sheath Tests

Each of the experimental runs produced a set of 4 pieces of 2 by 2 squared-inch cured cubes of cement sheath. After 48 hrs of curing in water-bath, 2 cured cubes from each of sets were used for the compressive strength tests, using the hydraulic press. This machine measured the compressive strengthen of the cured cement cubes.

### 3.2.4 X-ray Diffraction (XRD)

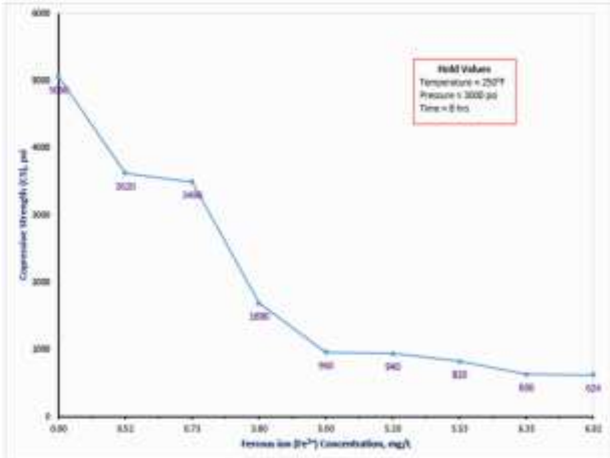
After the compressive strength destructive tests, each of the failed cement sheath fragments were ground in to finer particles. These fine particles were subjected to X-ray Diffraction tests, which determined the chemical compounds available in the failed cement sheaths.

**Original Article**

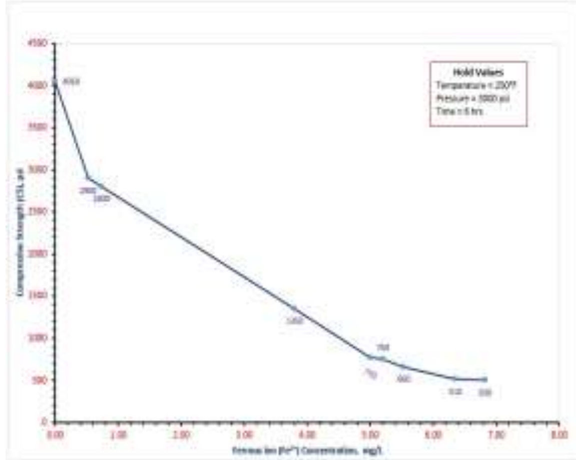
## 4. Results and Discussions

### 4.1 Results

#### 4.1.1 2D Plots of Compressive Strength Against Ferrous Ion ($Fe^{2+}$) Concentration

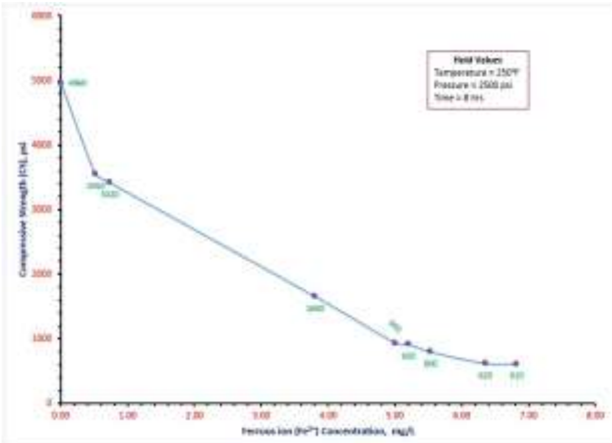



**Figures 14**. The Effect of Ferrous Ions in MixWater on the Compressive Strength of Cement Sheath, after which the Cement Slurry was cured at the Simulated Temperature, Pressure, and Curing Time at Approximately $250^0$F, 3000 psi, and 8 hrs, Respectively.

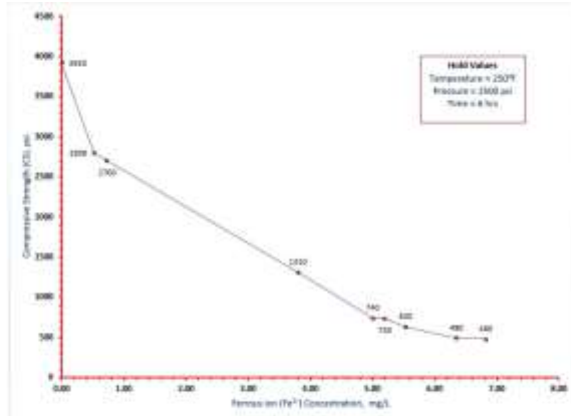



**Figures 15**. The Effect of Ferrous Ions in Mix-Water on the Compressive Strength of Cement Sheath, after which the Cement Slurry was Respectively, cured at the Simulated Temperature, Pressure, and Curing Time at Approximately $250^0$F, 3000 psi, and 6 hrs.

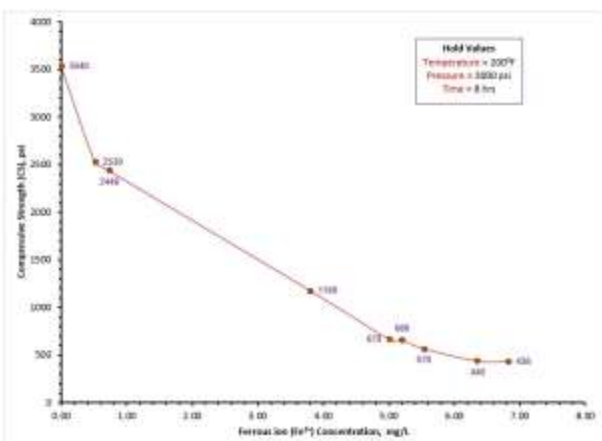
**Original Article**



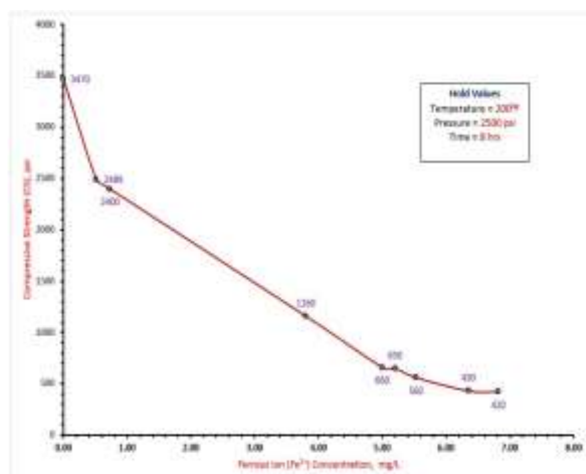
**Figures 16**. The Effect of Ferrous Ions in MixWater on the Compressive Strength of Cement Sheath, after which the Cement Slurry was Cured at the Simulated Temperature, Pressure, and Curing Time at Approximately 250$^0$F, 3000 psi, and 8 hrs, Respectively.



**Figures 17**. The Effect of Ferrous Ions in Mix-Water on the Compressive Strength of Cement Sheath, after which the Cement Slurry was Cured at the Simulated Temperature, Pressure, and Curing Time at Approximately 250$^0$F, 2500 psi, and 6 hrs, Respectively.

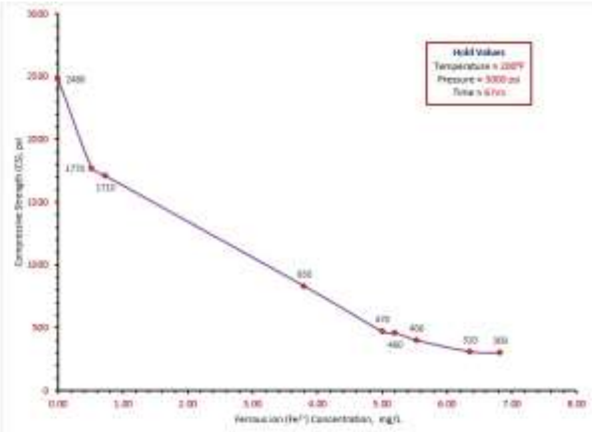


**Figures 18**. The Effect of Ferrous Ions in Mix-Water on the Compressive Strength of Cement Sheath, after which the Cement Slurry was Cured at the Simulated Temperature, Pressure, and Curing Time at Approximately 200$^0$F, 3000 psi, and 8 hrs, Respectively.

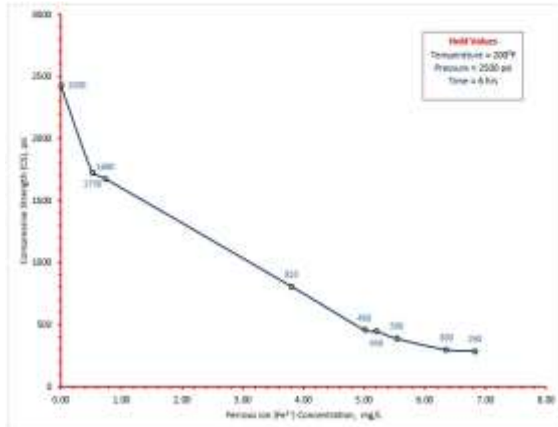


**Figures 19**. The Effect of Ferrous Ions in Mix-Water on the Compressive Strength of Cement Sheath, after which the Cement Slurry was Cured at the Simulated Temperature, Pressure, and Curing Time at Approximately 200$^0$F, 2500 psi, and 8 hrs, Respectively.

**Original Article**



**Figures 20.** The Effect of Ferrous Ions in Mix-Water on the Compressive Strength of Cement Sheath, after which the Cement Slurry was Cured at the Simulated Temperature, Pressure, and Curing Time at Approximately $200^0$F, 3000 psi, and 6 hrs, Respectively.



**Figures 21**. The Effect of Ferrous Ions in Mix-Water on the Compressive Strength of Cement Sheath, after which the Cement Slurry was Cured at the Simulated Temperature, Pressure, and Curing Time at Approximately $200^0$F, 2500 psi, and 6 hrs, Respectively.

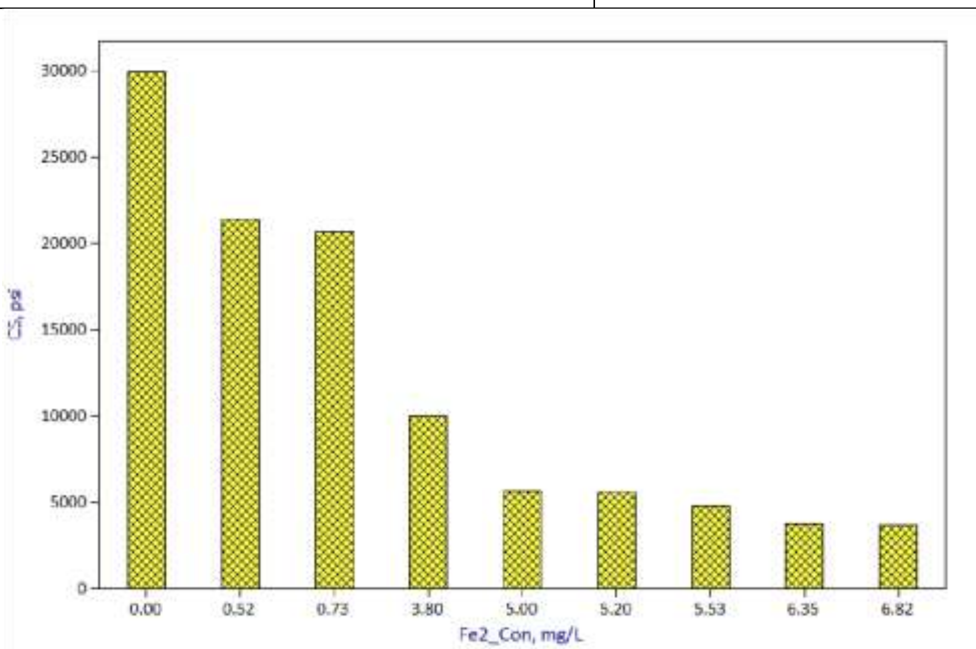



**Figure 22.** CS versus Ferrous ion ($Fe^{2+}$) Concentration in Mix-water (Fe2_Con).

**4.1.2 Major Chemical Compounds Found in the Ground Fragment of Cement Sheath Table 3**: Major Chemical Compounds Found in the Ground Fragment of Cement Sheath

| **CEMENT** | **COMPONENTS Degree** | | | | | |
|---|---|---|---|---|---|---|
| | **$C_3S$** | **$C_2S$** | **C-S-H** | **$Ca(OH)_2$** | **$Fe(OH)_2$** | **Fe-S-H** |
| **Finer Ground of Ferrous Cement Sheath Fragment** | $31^0$ | $31^0$ | $27^0$ | $17^0$ | $51^0$ | $30^0$ |

**4.2 Discussions**

Figures 14 to 21 illustrate the effect of ferrous ion concentration in mix-water on the compressive strength of the cement sheath. As a result, Figure 14 shows that, when the cement slurries were cured at the simulated temperature, pressure, and curing time at approximately $250^0$F, 3000 psi, and 8 hrs, respectively; the compressive strength loss was recorded at the rate of -546.73 psi. Further on, in Figure 15, it was also observed that the compressive strength loss rate was estimated as -479.85 psi, at the simulated temperature, pressure, and curing time at approximately $250^0$F, 3000 psi, and 6 hrs. Similarly, at the experimental conditions indicated on Figures 16, 17, 18, 19 ,20, and 21; it was disclosed that, the rate of compressive strength loss was recorded as -587.70, -464.17, -419.23, -412.17, -293, -288.37 psi, respectively. With regards to these trends, which are illustrated in these figures, show strong evidence that, as the ferrous ion concentration increases in the mix-water, the compressive strength of the cement sheath in the cement system decreases. This is vividly depicted in Figure 22. This explains that, the relationship between ferrous ion concentration in mix-water and compressive strength in the cement system, is antagonistic, and inversely proportional. Furthermore, these results disclosed that, in the absence of any other impurities in the mix-water, and with the presence of ferrous ion concentration within the range of 0.00 to 0.9 mg/L, would produce a desirable compressive strength, which would be above that of the API standard, 1500 psi. Moreover, the results also show that, temperature and curing time enhance the antagonistic behaviour of ferrous ions on compressive strength; whereas, pressure contributions in the cement system were not obviously significant in compressive strength loss.

**5. Conclusion**

In conclusion, the compressive strength of cement sheath decreases, as the concentration of ferrous ion in mix-water increases. Mostly, as it increases out of the range of 0.00 and 0.9 mg/L. The compressive strength loss in each of the cement systems were obviously observed, when the concentration of ferrous ion in the mix-water was approximately about 1 mg/L. This was active when the temperature and curing time were higher. The results show that, when the cement slurries where cured at the simulated temperature, pressure, and curing time at approximately $250^0$F, 3000 psi, and 8 hrs, respectively; the compressive strength loss was at the rate of -546.73 psi. It was also observed that the compressive strength loss rate was -479.85 psi at the simulated temperature, pressure, and curing time at approximately $250^0$F, 3000 psi, and 6 hrs. Similarly, at some identified experimental conditions, the results indicated that the rate of compressive strength losses were recorded as -587.70, -464.17, -419.23, -412.17, -293, -288.37 psi. At these conditions, the declassification of C-S-H by $Fe^{2+}$ into Fe-S-H, and the dissociation of calcium in $Ca(OH)_2$ by $Fe^{2+}$ into $Fe(OH)_2$ are very active. These activities are the causes of the compressive strength loss in the ferrous cement system. However, in these processes of compressive strength loss of the investigated ferrous cement sheaths, pressure was observed not, to play significant role in the strength loss compared to ferrous ion, temperature and curing time.

## 6. Recommendation for Further Studies

The results obtained from the investigation, disclosed that high concentration of ferrous ions in mix-water adversely affect the strength development of cement sheath at prevailing well conditions. Nevertheless, these results did not explain or depict either the comfort zone or optimum operating conditions of the ferrous cement system in terms of compressive strength development or loss at such prevailing conditions. Hence, some further investigations are needed, to be conducted on the relationship between compressive strength versus ferrous mixwater, to establish the operating optimum or comfort zone in the aforementioned ferrous cement systems. Probably, this should also involve the development of a model, which shall simulate the effect of high ferrous ion concentration in mix-water on the compressive strength of oilwell cement sheath. This model may eliminate the energy and cost, for the repetition of experimenting or investigating similar ferrous cement systems, to estimate compressive strength development or loss.

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