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The Application of Response Surface Methodology in Minitab 16, to Identify the Optimal, Comfort, and Adverse Zones of Compressive Strength Responses in Ferrous Oilwell Cement Sheath Systems.

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Abstract

Despite the increasing production of renewable energy, the demand for fossil energy still increases, which vectorised the search of crude oil into deeper horizons of depths below 6,000ft. At these depths, during the drilling and cementing of wellbore, to exploit crude oil, this activity encounters higher temperatures and pressures, adverse formations, and other related in-situ subsurface hash conditions. Hence, the need to prevent these antagonistic factors militating against the drilling and cementing of a successful, usable, economic, and safe oilwell became imperative. Therefore, this research article, investigated the effect of ferrous ion concentration in mix-water on the compressive strength of oilwell cement sheath, using Kolo Creek as a case study. The research, focused on the application of response surface methodology in Minitab 16, to identify the optimal, comfort, and adverse zones of high ferrous ion concentrations in mix-water on the response surface, which is the compressive strength of oilwell cement sheath. As a result, secondary data were collected and subjected to Minitab 16 design of experiment using the principles of Box-Behnken design of experiment in response surface methodology. Also, the response surface was customised, optimised, and analysed, to yield the best responses of the compressive strengths. Consequently, contour and surface plots were generated as the results, to identify the optimal, comfort and adverse zones of compressive strength in the ferrous cement sheath systems. The results obtained were bench-marked with the 1500psi compressive strength minimum API Specification for cementing oilwells. These results revealed that, the optimal, and comfort zones of the investigated compressive strength exist in mix-water with very low ferrous ion concentration (0.00 to 1.26mg/L) used in the formulation of cement slurry; while adverse zone of compressive strength exists, when the mix-water used to formulate the cement slurry had high ferrous ion concentration of 1.27 mg/L and above. Therefore, mix-water with high ferrous ion concentration should be reticulated, to make the mix-water potable, before using it to formulate cement slurries.

Keywords: *Comfort and Adverse Zones; Ferrous ion Concentration; Mix-Water; Compressive Strength; Box-Behnken, Design of Experiment; Response Surface Methodology.*

1. Introduction

Casing string run, and cementing of an oilwell, are the most questionably complex and critical activities in the upstream petroleum industry. Though, drilling for crude oil without cementing of casing run started in 1859 by Drake. After 44 years, the first cementing job was performed in Lompoc oilwells to shutdown groundwater by Frank F. Hill in 1903, which was improved by Perkins A. Almond in 1910 (Smith, 1976). These oilwells were shallow wells of approximately below 2,000ft. However, as the demand of fossil energy increases over the years, the search of crude oil gets deeper into deep horizons of depths below 6,000ft. At these depths, during the drilling for crude oil, these activities encounter higher temperatures and pressures, adverse formations, and other related *in-situ* subsurface hash conditions. Hence, the need to prevent these antagonistic factors militating against the drilling of a successful, usable, economic, and safe oilwell, became imperative. These militating factors were identified, controlled, and prevented by a cementing field method termed primary cementing in 1920 by Erle P. Halliburton at the Hewitt field, Carter Country in the United State of America, USA (Smith, 1976).

Furthermore, oilwell primary cementing is the process of mixing powered cement with mix-water, to form a resultant fluid known as neat cement slurry; and pumping the cement slurry down through the inner diameter of a casing string, and up the annulus, and to the desired height behind the centralised casing string. This cement paste or slurry is usually allowed to stand-alone for 28 days or more, to get it set and harden. This harden rigid solid is known as cement sheath. This cement sheath is aimed to provide zonal isolation, which includes to confine the movement of fluids between formation zones, and towards the surface, to prevent the pollution of fresh water formation, to prevent kicks that may graduate into a blowout. On the other hand, cement sheath provides well integrity, which includes the provision of structural support for casing string, to prevent casing from corrosion, to support the oilwell structurally, especially at the pay zone, which prevents cave-in of unconsolidated drilled geological formation, provide structural support for surface rig equipment, and it also enable well plugging and abandonment (Crook, 2006). Additionally, oilwell primary cementing is a hard technological process, where proper conceptualisation, selection, design, and implementation of the cement slurries affect the successful realisation of well integrity; safety barrier for oil kick near-misses, and blowout accidents, and complete isolation of formation fluids from drilling and workover fluids (Bourgoyne *et al.*, 1986; Michaux *et al*., 1990; Heinold *et al*., 2002; Nelson and Guillot, 2006; Kutchko *et al.*, 2007; Crook, 2009; Sauki and Irawan, 2010; Joel and Ademiluyi, 2011; Oriji and Appah, 2014; Liu, 2015; Broni-Bediako *et al*., 2015; Salehi and Paiaman, 2009).

Despite, the advancements that have been made in the design of cement slurry, to sustain the aforementioned functions of cement sheath, by the research and development (R&D) sector of the upstream petroleum industry; yet there is no consensus in cementing, and there are still continuous reported cases of primary cementing failures. These failures are due to long run realised poor cement slurry design (Hair and Narvaez, 2011; NAP, 2012; Parsonage, 2017; Normann, 2018). Consequently, this research further focused on the effect of mix-water quality on the cementing of oilwell. Mostly, the effect of ferrous (Fe^{2+}) ions concentration in mix-water on cement properties, to identify the optimal, comfort, and adverse zones of a ferrous cement sheath system, using the principles of Box-Behnken design of experiment (DOE) in response surface methodology, in conjunction with the technology of Minitab 16 statistical application package.

1.1 Study Area

Kolo Creek area was used as the case study (Adesuyi, 2015). This area has high activities of oil exploration and production, with the avialability of high content of ferrous groundwater usually accessed through waterwells. The ferrous groundwater is usually used as mix-water for the formulation of cement slurries. Kolo Creek on the Global Positioning System (GPS) position is approximately at Latitude 4.6667°, Longitude 6.3333° (Gordon and Enyinaya, 2012; Adesuyi, 2015; World Industrial Information, 2016). As a consequence, this article is expected, to identify the optimal, comfort, and adverse zones of high ferrous ion concentrations in mix-waters on the responses of compressive strength development of oilwell cement sheath, using the principles of response surface methodology. This study used the experimental data obtained by Igbani *et al*. (2020), as secondary data.

2. Background

Water is usually used as a universal solvent in many human endeavours. Water dissolves minor components of solute. In the context of cementing, water is known as mix-water or mixing water. Mix-water is the indispensable component used in the formulation of cement slurry (Smith, 1976). In the cement slurry system, Class G oilwell cement is one of the solute, and binding material used in cementing casing strings, to attain well zonal isolation and integrity. Also, cement components are hydrophilic (Kiran, *et al*., 2017). Furthermore, cement slurry designers have severally investigated the impact of the quantity of mix-water to cement powder (w/c) on compressive strength development of cement sheath (Bourgoyne *et al*., 1986; Zhang *et al*., 2003; Crooks, 2006; Yasar *et al.*, 2004; Azar and Samuel, 2007; Atahan *et al.*, 2009; Haach *et al.*, 2011; Minaev *et al.,* 2014). These investigations disclosed that, the quantity of mix-water and cement powder (w/c) used to formulate any graded oilwell cement slurry should be within the range of 0.4 to 0.5 ratio, and contrary to this range of w/c reduces the compressive strength (Azar and Samuel, 2007; API Specification 10A, 2019). The investigations also revealed that excess mix-water forms segregation in the hydrostatic column of cement slurry. This is observed by the formation of segregation of supernatant of free water and residue of cement slurry, which decreases the cement sedimentation stability; while the free water creates channels and pocket of pores in the resulting hardened cement sheath, not only to weaken the compressive strength, but to increase the permeability of the cement sheath, which compromises the ability of sheath, to isolate formation zones. Critically, the aforementioned investigations show that small quantity of mix-water in a cement slurry system increased the viscosity of the cement slurry, which made the pumpability of the slurry very poor. This impedes the pumping of cement slurry, to the desired depth behind the casing string. However, these investigations did not disclose the antecedents of the impact of high ferrous ions in mix-water on the compressive strength of cement sheath. Mostly, to identify the optimal, comfort and adverse zones of high ferrous ion concentrations in mix-water on the response of compressive strength development and loss of oilwell cement sheath, using the principles of response surface methodology. Hence, a further review on literatures were conducted, to identify the effect of water quality on compressive strength. Mostly, to identify the optimal, comfort, and adverse zones of high ferrous ion concentrations in mix-waters on the response surface of compressive strength.

Accordingly, the reviewed literatures on the impact of impurities present in mix-water, and its impact on the compressive strength development of cement sheath; deducted that, the presence of high concentration of ions in mix-water reduced the compressive strength of cement sheath, while the presence of low concentration of ions in mix-water increased the compressive strength of cement sheath (Suman and Ellis, 1977; Sabins and Sutton, 1983; Saleh *et al*., 2018). Similarly, Nelson (1990) explained that, the use of non-potable mix-water reduced the compressive strength of cement sheath up to 20%. Explicitly, in some independent studies undertaken by Ko and Batchelor (2010); Madhusudana *et al.* (2011); Igbani *et al*., (2020) have shown that, the presence of heavy metal in high concentration in mix-water causes compressive strength loss, when used to formulate cement slurry. Also, that the strength loss was as a result of the dissociation of calcium hydroxide and decalcification of tobermorite or Calcium Silicate Hydrate. Subsequently, after the removal of the impurities from mix-water, then a potable mix-water is obtained (Ashraf, 2005; Siabi, 2008; Chi-Chuan, 2012; Akl *et al.*, 2013; Yousuo *et al*. 2019). This explained that, mix-water should have the same quality as potable water fit for human consumption (Mindess and Young, 1981; Mehta, 1986; Raina, 1988; WHO, 2004). Still cementing services companies use the available mix-water onshore at rig sites, which are not reticulated, to reduce the Fe^{2+} concentration to as low as reasonably practicable (ALARP). In this vein, Igbani *et al*. (2020) in a recent study on the impact of different concentrations of Fe^{2+} on compressive strength of cements, obtained results, which show that, when the cement slurries where cured at the simulated temperature, pressure, and curing time at about 250^0F, 3000psi, and 8hrs, respectively; the compressive strength loss was at the degree of -546.73psi. It was also witnessed that the compressive strength loss rate was -479.85psi at the simulated temperature, pressure, and curing time at about 250^0F, 3000psi, and 6hrs. Likewise, at some experimental conditions, the results showed that the rate of compressive strength losses were documented as -587, -464, - 419, -412, -293, -288psi. Additionally, it was similarly observed that the compressive strength of cement sheath decreases, as the concentration of ferrous ion in the mix-water increases. Mostly, as it increases completely out of the range of 0.00 and 0.9mg/L. This occurred, when the concentration of ferrous ions in the mix-water were approximately about 1 mg/L, and the temperature and curing time were high. At these simulated conditions, the declassification of C-S-H by Fe^{2+} into Fe-S-H, and the dissociation of calcium from $Ca(OH)_2$ by Fe^{2+} into $Fe(OH)_2$ were very dynamic. These activities were identified as the major cause of compressive strength loss in the ferrous cement systems. Interestingly, in these processes of compressive strength improvement or loss in the ferrous cement system, pressure was observed not to, obviously encourage the antagonistic behaviour of ferrous ions. Nevertheless, some further investigations are still needed, to be conducted on the relationship between compressive strength versus ferrous mix-water, to establish the operating optimal, comfort and adverse zones in Igbani *et al*. (2020) investigation.

3. Materials and Methods

3.1 Materials

The materials used for this research were Minitab 16 Statistical application package, modem, HP Laptop, and Secondary data.

3.2 Method

Quantitative secondary data (Results) were collected from Igbani *et al*. (2020) research. Minitab 16 was got started, and the fieldnames (Treatments, Fe2_Con, Temp, Pres, Time, and CS) were created on the fieldname-row on each of the columns in the worksheet. The worksheet was saved through the FILE menu. However, the worksheet was resaved whenever any updated command was performed on the worksheet. Additionally, the fieldnames were defined to hold numerical data; except Treatment, which is a character string data-type. The secondary data collected were entered into Minitab 16 worksheet (Figure 1). After the data entry process, four (4) stages were involved in the investigation, and design of the optimal, comfort, and adverse zones of the investigated compressive strength developments in the

ferrous cement system. This involved the design of the Box-Behnken design of experiment (DOE), selection of optimal design, analyse of response design, and design of contour and surface responses.
Firstly, the Box-Behnken design of experiment (DOE) was conducted on worksheet in Minitab 16 from the STAT menu, then through the DOE, and RESPONSE SURFACE drop menu; then selecting the CREATE RESPONSE SURFACE DESIGN option (Figure 2). This was to enable the plotting of the contour and surface plots. In the "create response surface design" dialog box the number of factors (predictors) were set as four (Figure 3). In Figure 3, the button of DISPLAY AVAILABLE DESIGN was selected, which popped up the "display available design" dialog box (Figure 4). In Figure 4, 27 runs and unblocked was selected under 4 factors options, and it was okayed. Afterwards, "define custom response surface design" was conducted as indicated in Figures 5, 6, and 7, to select the predictors: ferrous ion concentration (Fe2_Con), Temperature (Temp), Pressure (Pres), and Time. These are the predictors that influenced the response surface, compressive strength (CS). Conclusively, in the design of the Box-Behnken DOE procedures, the high and low values of the predictors were set in Figure 8, the outcome of the DOE is presented in Table 1.

Secondly, after the Box-Behnken DOE stage have been executed, the next stage was the selection of optimal design. In selecting the optimal design, from the menu bar the STAT menu was clicked, then through the DOE, and RESPONSE SURFACE drop menu; the SELECT OPTIMUM DESIGN was chosen (Figure 9). In Figure 10, in the "number of points in the optimal design" option, 3 points was entered, then the Term button was selected. Then Figure 11 popped up, and the probable highest order model (full quadratic) was selected in the dialog box of Select Optimal Design.

Thirdly, the selection of the response variable, CS, and the analysis of the response surface were conducted; these were done through the STAT menu, DOE, and RESPONSE SURFACE drop menus; where the ANALYZE RESPONSE SURFACE DESIGN was selected (Figure 12). As a result, the dialog box of "Analyze Response Surface Design" popped up (Figure 13). In Figure 13, the response variable, CS was selected, using the uncoded units for data analysis, to show real experimental values on the plots. Thereafter, the Term button was clicked and the dialog box of "Analyze Response Surface Design - Term" popped up (Figure 14). In Figure 14, the Backward elimination principle of unwanted variable was applied, to screen out ambiguities in the response surface plots.

Finally, Figures 15, 16, 17, and 18, show how the optimal, comfort, and adverse zones of high ferrous ion concentration in mix-water towards the response of compressive strength development and loss of oilwell cement sheath were conducted in Minitab 16.

In conclusion, the results obtained are presented next, in Figures 19 to 24, which same are discussed in section 4. Furthermore, the findings, conclusion of the research, and recommendation for further studies are presented in section 5, 6, and 7, respectively.

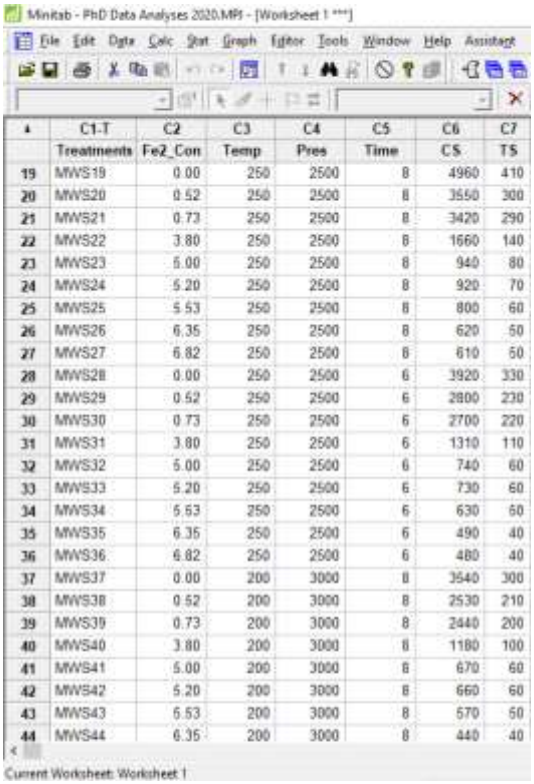


Figure 1. Secondary data entered into Minitab 16 worksheet.

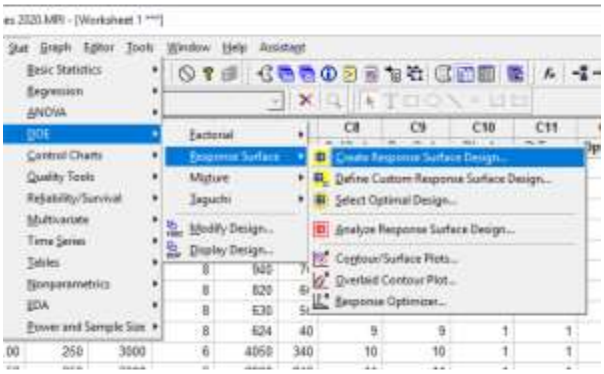


Figure 2. Creation of the Response Surface Design through STAT, DOE (Design of Experiment), and RESPONSE SURFACE.

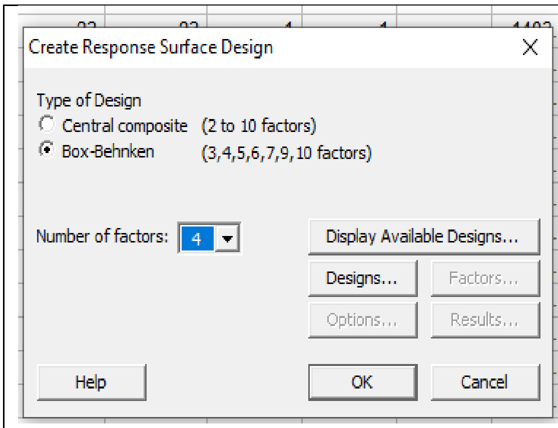


Figure 3. The dialog box used to select the Box-Behnken DOE and the number of independent variables (which is four) in each of the cement slurry systems.

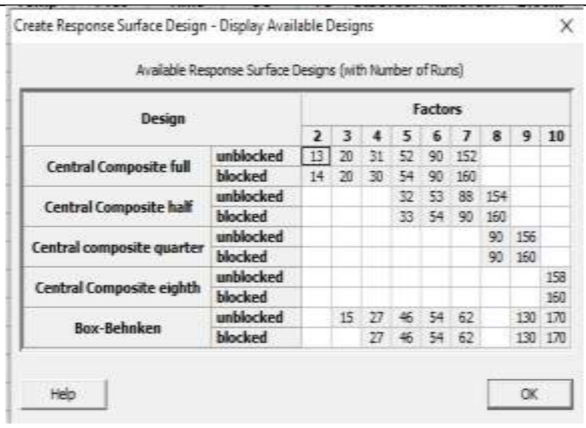


Figure 4. The dialog box used to select the Box-Behnken DOE available for four independent variables.

Figures 5. Definition of the Custom Response Surface Design through STAT, DOE and RESPONSE SURFACE, to select the available for four independent variables.

Figures 6. Selection of the four independent variables from the dialog box of Define Custom Response Surface Design.

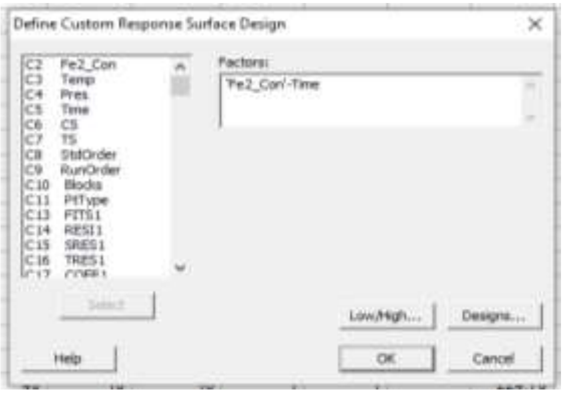


Figure 7. The four independent variables are have been selected from the dialog box of Define Custom Response Surface Design.

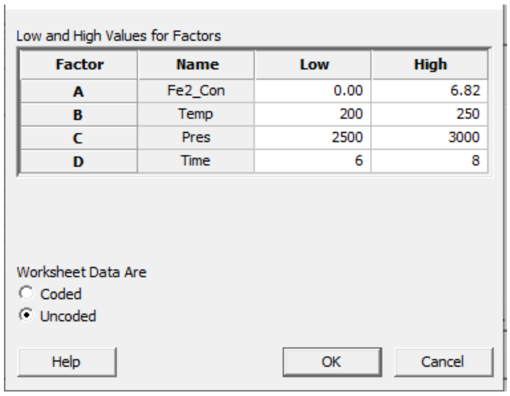


Figure 8. The range for the four independent variables were setup here, in the Low and High Value for Factor dialog box.

Table 1. Box-Behnken Design Randomised Design Output in RSM

Box-Behnken Design Randomised Design Output in RSM

```
Factors:       4     Replicates:     1
Base runs:    27     Total runs:    27
Base blocks:   1     Total blocks:   1

Center points: 3
```

Design Table (randomized)

Run	Blk	A	B	C	D
1	1	0	-	0	-
2	1	0	0	0	0
3	1	0	-	+	0
4	1	0	-	-	0
5	1	+	+	0	0
6	1	-	0	-	0
7	1	-	0	+	0
8	1	+	0	0	-
9	1	0	0	-	+
10	1	+	0	0	+
11	1	0	+	0	-
12	1	0	0	-	-
13	1	+	-	0	0
14	1	+	0	+	0
15	1	-	0	0	+
16	1	+	0	-	0
17	1	0	0	0	0
18	1	-	-	0	0
19	1	0	+	-	0
20	1	-	+	0	0
21	1	0	+	+	0
22	1	-	0	0	-
23	1	0	0	+	-
24	1	0	+	0	+
25	1	0	-	0	+
26	1	0	0	+	+
27	1	0	0	0	0

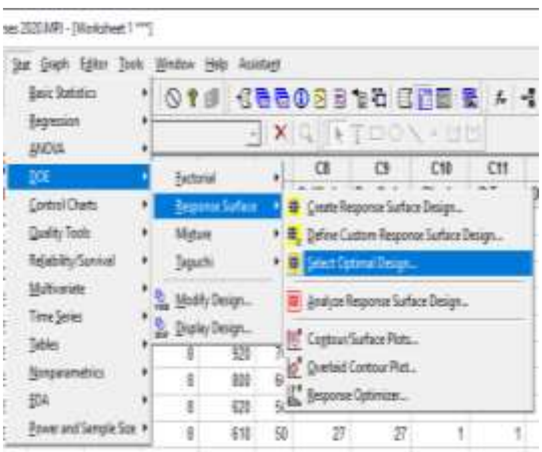


Figure 9. Selection of the optimal design through STAT, DOE and RESPONSE SURFACE, to select the response variable, compressive strength (CS).

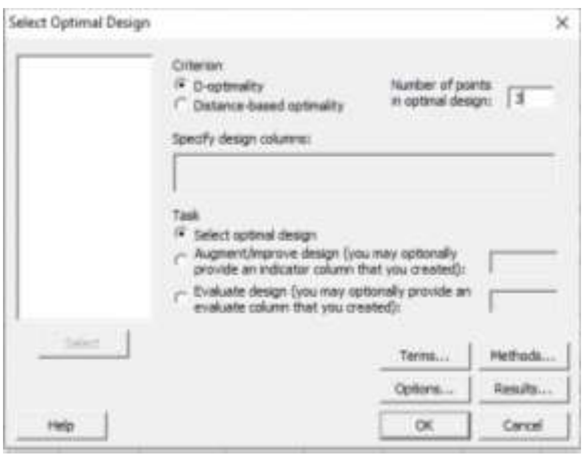


Figure 10. Selection of the optimal design from the Select Optimal Design dialog box.

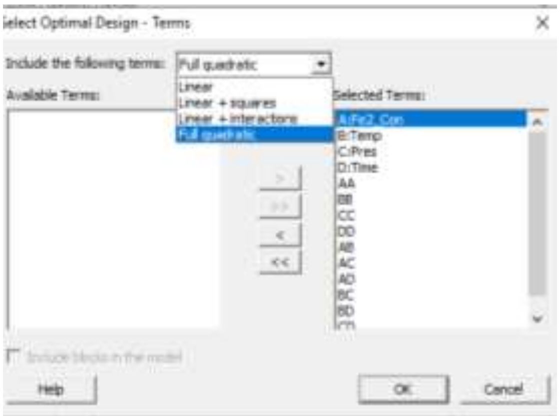


Figure 11. Selection of the optimal design probable model (Full Quadratic) from the Select Optimal Design-Terms dialog box.

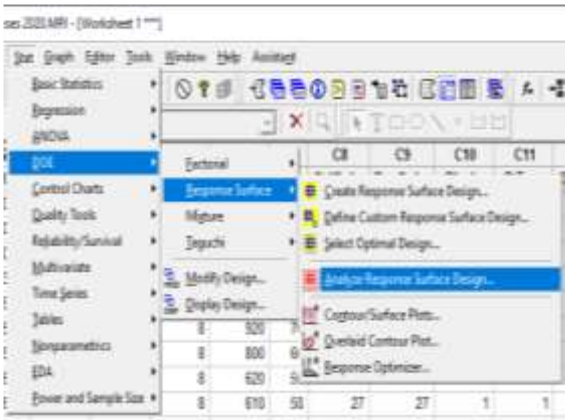


Figure 12. Selection of the analyse response design through STAT, DOE and RESPONSE SURFACE.

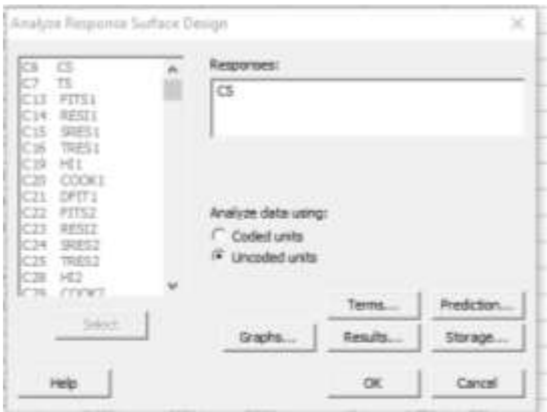


Figure 13. Selection of the response variable, CS from the Analyze Response Surface Design dialog box.

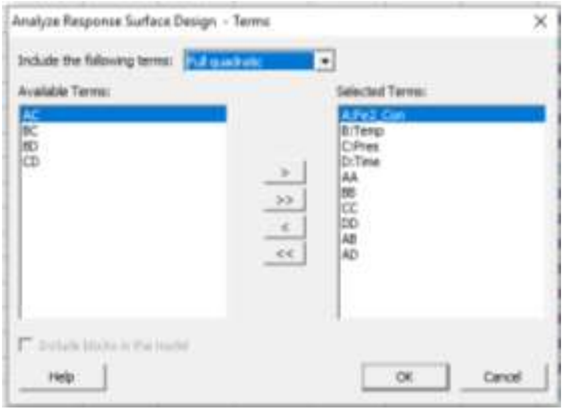
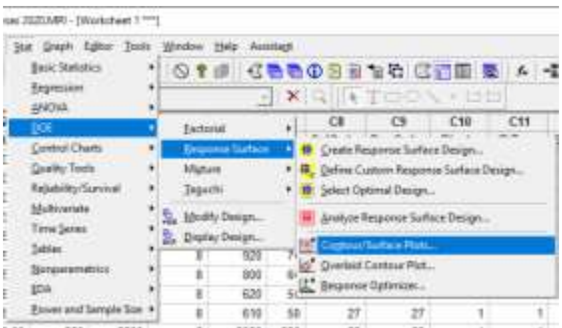
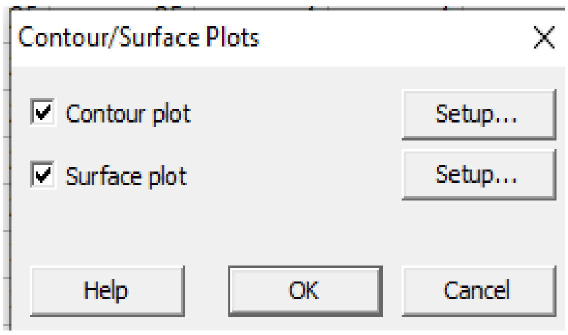


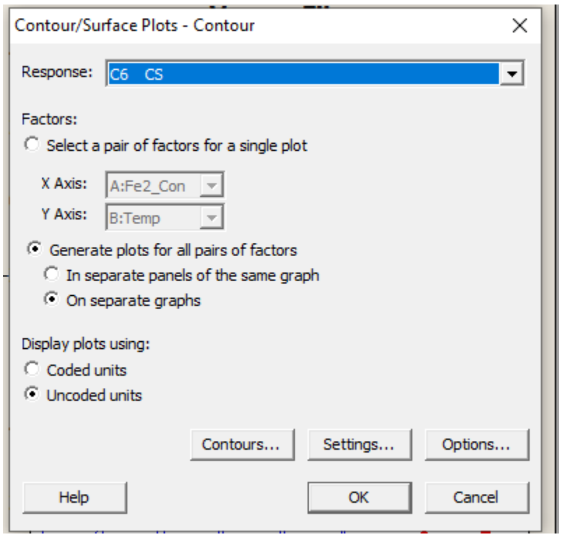
Figure 14. Selection of the optimal design probable model (Full Quadratic) from the Analyze Response Surface Design dialog box.



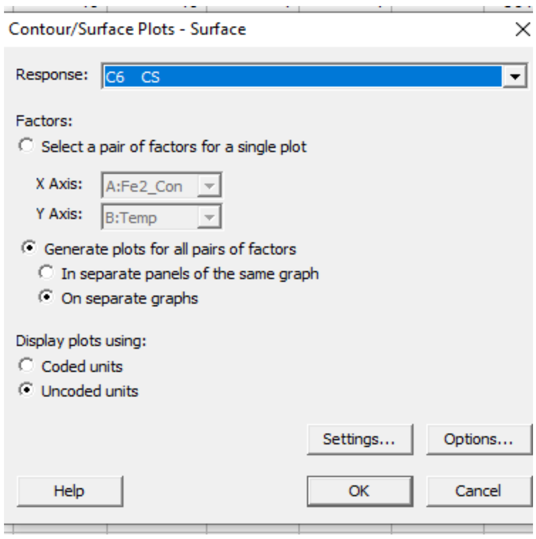
Figures 15. Selection of Contour/Surface Plots through STAT, DOE and RESPONSE SURFACE.



Figures 16. Setup of the contour/surface plots from the dialog-box of Contour/Surface Plots.



Figures 17. Setup of the contour plots from the dialog-box of Contour/Surface-Contour Plots.



Figures 18. Setup of the surface plots from the dialog-box of Contour/Surface-Surface Plots.

4. Results and Discussions

4.1 Results

4.1.1 Minitab 16 Response Surfaces Methodology (RSM) Outcome: 3-Dimensional (3D) Contour Plots for Compressive Strength, Versus Ferrous (Fe^{2+}) Mix-Water Concentration (Fe2_Con), and other Stimulated Prevailing Controllable Variables such as Pressure (Press), Curing Time, and Temperature (Temp).

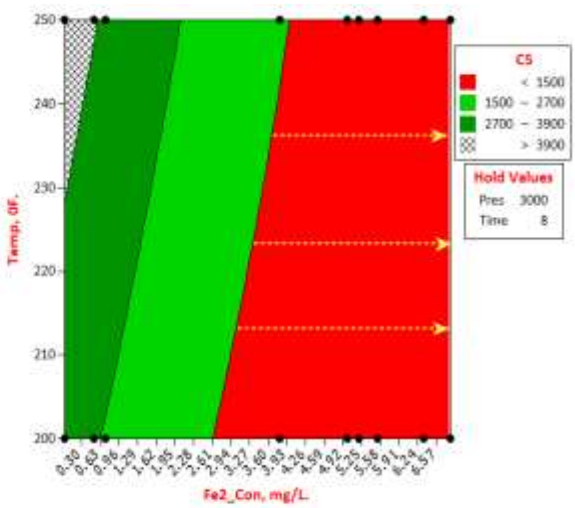


Figure 19. A 3D Contour Plot of CS vs Temp., and Fe2_Con., at Constant Pres. (3000psi) and Time (8hrs); Indicating the Optimal Zone, Comfort Zone, and Adverse Zone of Compressive Strength in a Ferrous Cement Sheath System.

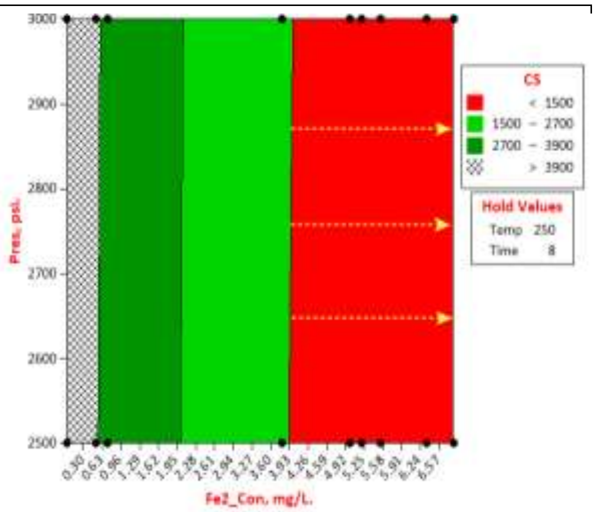


Figure 20. A 3D Contour Plot of CS vs Pres., and Fe2_Con., at Constant Temp. (250^0F) and Time (8hrs); which Shows the Optimal Zone, Comfort Zone, and Adverse Zone of Compressive Strength in a Ferrous Cement Sheath System.

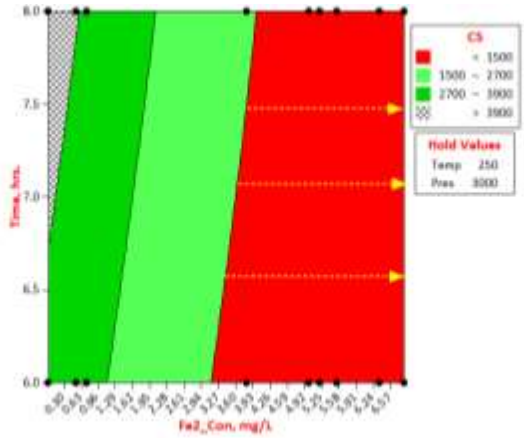


Figure 21. The Optimal Zone, Comfort Zone, and Adverse Zone of Compressive Strength in a Ferrous Cement Sheath System; which is depicted in the 3D Contour Plot of CS vs Time., and Fe2_Con., at Constant Temp. (250^0F) and Pres. (3000psi).

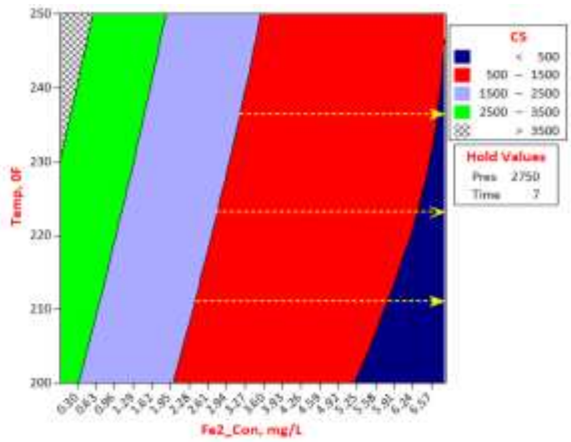


Figure 22. A 3D Contour Plot of CS vs Temp., and Fe2_Con., at Constant Pres. (2750psi) and Time (7hrs); Showing the Optimal Zone, Comfort Zone, and Adverse Zone of Compressive Strength in a Ferrous Cement Sheath System.

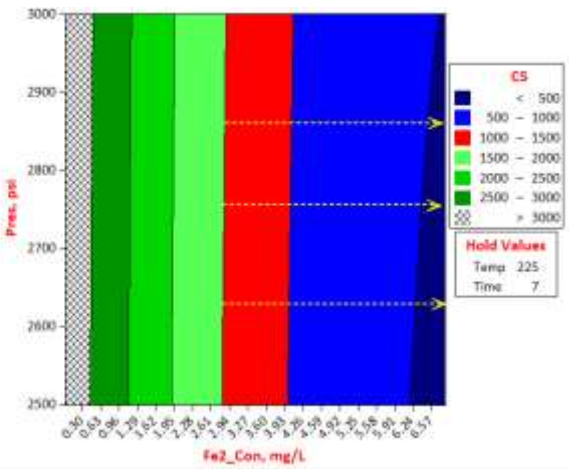


Figure 23. A 3D Contour Plot of CS vs Pres., and Fe2_Con., at Constant Temp. (225^0F) and Time (7hrs); which Shows the Optimal Zone, Comfort Zone, and Adverse Zone of Compressive Strength in a Ferrous Cement Sheath System.

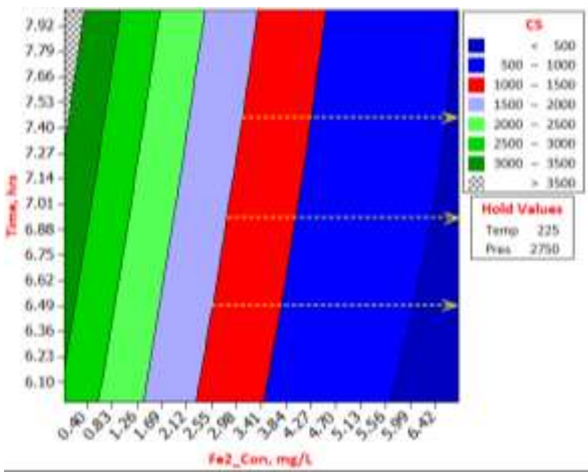


Figure 24. The Optimal Zone, Comfort Zone, and Adverse Zone of Compressive Strength in a Ferrous Cement Sheath System; which is depicted in the 3D Contour Plot of CS vs Time., and Fe2_Con., at Constant Temp. (225^0F) and Pres. (2750psi).

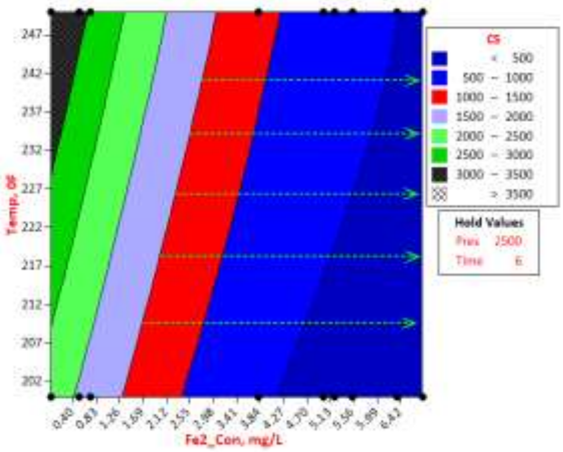


Figure 25. A 3D Contour Plot of CS vs Temp., and Fe2_Con., at Constant Pres. (2500psi) and Time (6hrs); which holds the Comfort Zone, and Adverse Zone of Compressive Strength in a Ferrous Cement Sheath System.

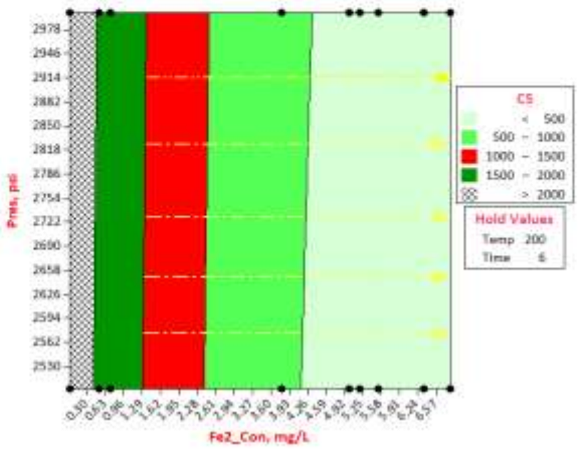


Figure 26. The Optimal Zone, Comfort Zone, and Adverse Zone of Compressive Strength in a Ferrous Cement Sheath System Presented in A 3D Contour Plot of CS vs Pres., and Fe2_Con., at Constant Temp. (200^{0}F) and Time (6hrs).

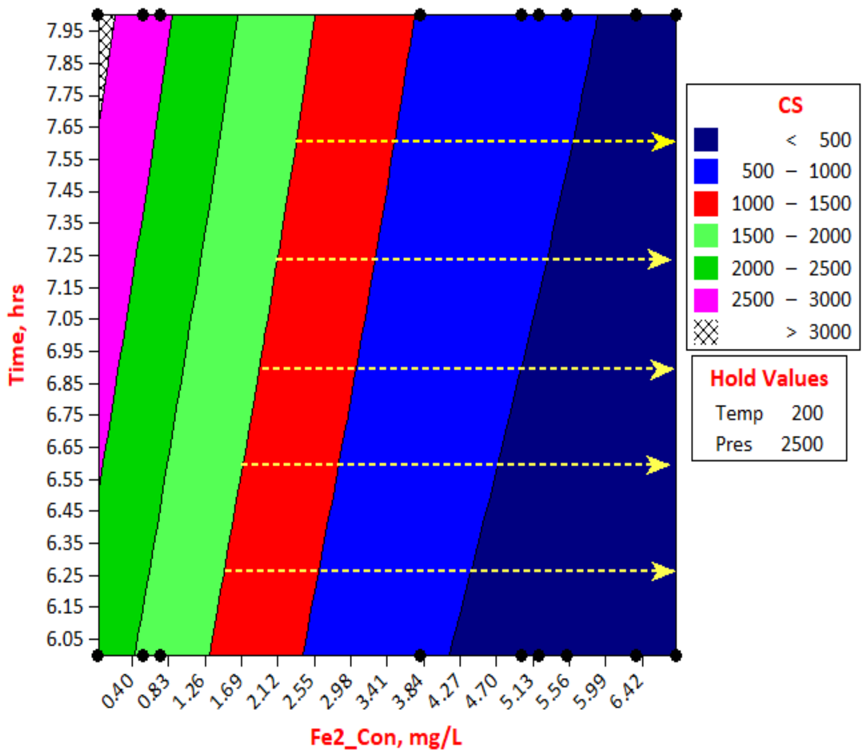


Figure 27. The Optimal Zone, Comfort Zone, and Adverse Zone of Compressive Strength in a Ferrous Cement Sheath System; which is depicted in the 3D Contour Plot of CS vs Time, and Fe2_Con., at Constant Temp. (200^{0}F) and Pres. (2500psi).

4.2 Discussions

Figures 19 to 27 are contour plots, which each revealed the optimal, comfort, and adverse zones of the investigated compressive strengths of the ferrous cement sheath systems. Though, Figure 25 does not contain an optimal zone. The adverse zone is the zone with compressive strength less than 1500psi, and it is indicated on the plots, as the surface areas occupied by the yellowish arrows tending to the right hand side of each of the plots. The optimal zone on each of the plots, is the surface area illustrated with the whitish-mesh-like strips, either at the far top left or left of the plots; while the comfort zone on each of the plots, is surface area sandwiched between the optimal zone and the adverse zone. Both of the compressive strengths found in the optimal, and comfort zones are usually greater than 1500psi. Categorically, Figures 19 to 21 represent the results of the response surface, of the compressive strength obtain from the experimental treatments conducted, when the predictors of temperature, pressure, and curing time were alternated and constantly held in pairs consecutively with values of approximately 250^0F, 3000psi, and 8hrs. Similarly, Figures 22 to 24 show the results of the dependent variable, of compressive strength collected from the experiments performed, when the independent variables of temperature, pressure, and curing time were kept constantly in pairs, using the estimated values of 225^0F, 2750psi, and 7hrs, alternatively. However, this values were generated by Minitab 16 technology. Likewise, Figures 25 to 27 illustrate the response surface, of compressive strength obtain from the experiments conducted, when the predictors of temperature, pressure, and curing time were also alternated and constantly held in pairs, using the predictable values of 200^0F, 2500psi, and 6hrs. In Figures 19 to 27, the ferrous ion concentration investigated ranges between 0.00 to 6.82, and these values were used as the controllable variables, which were kept at the x-axis. However, variables such as temperature, pressure, and time were used one-after-the-other in the y-axis, at various instances. Besides, values for ferrous ion concentration, temperature, pressure, and time were set and used as high, medium, and low setting for the analyses in Minitab 16.

Firstly, at the high setting of the predictors, ferrous ion concentration, temperature, pressure, and curing time set values were estimated at 6.82mg/L, 250^0F, 3000psi, and 8hrs, respectively. Figure 19 illustrates that, when the pressure and curing time values were held constant at 3000psi, and 8hrs, respectively; the optimal zone was observed to be mapped between the coordinates of ferrous ion concentration (mg/L) in mix-water and curing temperature (^{0}F), at (0.00, 229), (0.00, 250), and (0.59, 250), with the optimal compressive strengths of 3902, 4440, and 3900psi, respectively. In addition, it was observed that, the comfort zone fell between the coordinates of ferrous ion concentration (mg/L) and temperature (^{0}F), at the points of (0.00, 200), (0.00, 229), (0.00, 250), (3.92, 250), and (2.59, 200) with the corresponding comfortable compressive strength of 3179, 3891, 3900, 1534, and 1513psi, respectively. Conversely, the adverse zone in Figure 19, was identified at the coordinates of (2.66, 200), (4.00, 250), (6.80, 250), and (6.80, 200), with weaker compressive strengths of 1487, 1476, 549, and 489psi, respectively. Therefore, the result in Figure 19 shows that, when the pressure, and curing time values were respectively, held constant at about 3000psi, and 8hrs; the highest optimal, and weaker compressive strength of about 4440psi, and 489psi were recorded, when the ferrous ion concentration, and temperature, were approximately at 0.00mg/L and 250^0F, and 6.8mg/L and 200^0F, respectively. Similarly, Figure 20 is the contour plot that, represents the results of compressive strengths for the ferrous cement sheath systems, when temperature and curing time were held constant at 250^0F, and 8hrs, respectively. In this plot, the compressive strength was plotted against pressure, and ferrous ion concentration. Consequently, it was observed that, the optimal compressive strength exists within the region, which the coordinates of ferrous ion concentration in mg/L, and pressure in psi are expressed as (0.00, 2500), (0.00, 3000), (0.59, 3000), and (0.52, 2500). These coordinates were identified, to have corresponding estimated optimal compressive strengths of 4400, 4433, 3898, and 3919psi. Additionally, the comfort zone for the compressive strength in Figure 20 resides in the coordinates of ferrous ion concentration in mg/L, and pressure in psi, at (0.54, 2500), (0.64, 3000), (3.92, 3000), and (3.86, 2500), with the resultant comfortable compressive strengths of 3900, 3853, 1521, and 1519 psi, respectively. In the contrary, the adverse zone of the compressive strength exists-in the region with the coordinates of ferrous ion concentration in mg/L, and pressure in psi, as (3.95, 2500), (4.01, 3000), (6.80, 3000), and (6.80, 2500), and were recorded as 1487, 1487, 546, and 506psi, correspondingly. From these results of Figure 20, it is observed that, the most optimal, comfort, and adverse compressive strengths are recorded as 4433, 3900, and 506psi, at the coordinates of ferrous ion concentration (mg/L), and pressure (psi) as (0.00, 3000), (0.54, 2500), and (6.80, 2500), respectively; at constant temperature and curing time, in the order of 250^0F, and 8hrs. Likewise, Figure 21 represents the result of compressive strength in the ferrous cement sheath system, when temperature and pressure were held constant at 250^0F, and 3000 psi, respectively. In this plot, the compressive strength is plotted against curing time, and ferrous ion concentration. The results show that, the most optimal, comfort, and adverse compressive strengths are esteemed as 4432, 3900, and 507psi, at the coordinates of ferrous ion concentration (mg/L), and curing time (hrs) as (0.00, 8.00), (0.61, 8.00), and (6.80, 6.00), respectively.

Secondly, the medium setting of the predictors, ferrous ion concentration, temperature, pressure, and curing time, were kept at 3.41mg/L, 225^0F, 2750 psi, and 7hrs, respectively. Then, the pressure, and curing time were held constant, respectively and approximately at 2750 psi, and 7 hrs. Accordingly, Figure 22 result reveals that, the optimal point of the compressive strength, is estimated at 3974psi; while the comfort point is estimated at 3500psi, and the adverse point measured at 453psi. These were respectively observed at the ferrous ion concentration (mg/L), and temperature (^{0}F) coordinates of (0.00, 250), (0.59, 250), and (6.80, 200). On the other hand, when temperature, and curing time were held constant, at about 225^0F, and 7hrs, respectively. Figure 23 discloses the estimated value of the optimal, comfort, and adverse points of the compressive strength as 3375, 3000, and 462psi. These measured values were obtained at the coordinate of ferrous ion concentration (mg/L), and pressure (psi) at (0.00, 3000), (0.52, 3000), and (6.80, 2500), respectively. Alternatively, when temperature, and pressure were held constant, at about 225^0F, and 2750psi, respectively. Figure 24 illustrates that, at the optimal, comfort, and adverse zones; the maximum optimal, and comfort estimated values of the compressive strength are about 3764, and 3500psi, respectively; while the most adverse point is estimated at 463psi. These were identified at the coordinate of ferrous ion concentration (mg/L), and time (hrs) of (0.00, 8.00), (0.38, 8.00), and (6.80, 6.00), respectively.

Finally, at the low setting of the predictors, ferrous ion concentration, temperature, pressure, and curing time, were set at 0mg/L, 200^0F, 2500 psi, and 6hrs, respectively. In these experiments, Figure 25 shows the plot for compressive strength against temperature, and ferrous ion concentration, when the pressure and curing time values were held constant at 2500 psi, and 6hrs, respectively. For that reason, Figure 25 reveals that, the optimal point of the compressive strength, is unidentified; while the comfort point is estimated at 3500psi, and the adverse point measured at 413psi. These were respectively observed at the ferrous ion concentration (mg/L), and temperature (^{0}F) coordinates of (0.00, 250), (0.00, 250), and (6.80, 200), respectively. In contrast, when temperature, and curing time were held constant, at about 200^0F, and 6hrs, respectively, Figure 26 reveals the appraised value of the optimal, comfort, and adverse points of the compressive strength as 2299, 2000, and 415psi. These appraised values were obtained at the coordinate of ferrous ion concentration (mg/L), and pressure (psi) at (0.00, 3000), (0.00, 3000), and (6.80, 2500), respectively. Then again, when temperature, and pressure were held constant, at about 200^0F, and 2500psi, respectively. Figure 27 shows that, at the optimal, comfort, and adverse zones, the maximum optimal, and comfort estimated values of the compressive strength are about 3140, and 3000psi, respectively; while the adverse point is estimated at 418psi. These were identified at the coordinate of ferrous ion concentration (mg/L), and time (hrs) of (0.00, 8.00), (0.00, 8.00), and (6.80, 6.00), respectively.

Analytically, when each of the curing temperature, curing time, and ferrous ion concentration were varied, while holding the others constant, the resultant compressive strength varied significantly. Conversely, it was discovered that, the variation in the magnitude of the variable, pressure does not significantly influence the compressive strength of cement sheath, in any given concentration of ferrous ion, under the influence of the varied simulated well condition parameters. This means that, the pressure on the investigated cement slurry during curing did not act on it longitudinally in forming the cement sheath (Yong *et al*., 2017). To substantiate the results obtained in this research, Crook *et al*. (2001) have shown that the ability of cement sheath, to provide good zonal isolation subsurface, is better defined by some other mechanical properties of cement sheath. These mechanical properties include porosity, permeability, and compressive strength (Omosebi *et al*., 2016). In a study, Crook *et al*. (2001) pronounced that a good well isolation does not necessarily require high compressive strength; but, be able to attain the following strengths from the traditional rule of thumb in oilwell cementing: to support casing (5 - 200psi), to continue drilling after waiting-on-cement to set time (500psi), to perforate the payzone (1000psi), and at least to stimulate or enhanced oil recovery, and isolate zone (2000psi). In support of this, Omosebi *et al*., (2016) opined that, due to the carbonation of calcium hydroxide and calcium silicate hydrate by CO_2 during the hydration of cement clicker for approximately above 28 days, the cement sheath porosity and permeability increases, as the compressive strength decreases; which means CO_2 degrades the mechanical properties of cement sheath, as F^{2+} degrades same properties at HPHT. Therefore, in this research those zones with compressive strengths below 2000psi are barely relevant in HPHT oilwell cementing.

Consequently, from the discussion of the results, when pressure and curing time values were held constant at about 3000psi, and 8hrs, respectively, in this investigations. It was vividly observed that, the overall maximum optimal compressive strength of the investigated ferrous cement sheath systems is recorded as 4440psi, at (0.00, 250^0F); and then, when pressure and curing time values were held constant at 2500psi, and 6hrs, respectively, the overall minimum optimal point of the compressive strength, is estimated at 0psi, at (0.00, 250); though with a near optimal point, of comfort point estimated at 3500psi, at (0.00, 250). Whereas, the worst adverse compressive strength of the investigated ferrous cement sheath systems is recorded as 413psi, at (6.80, 200), when the pressure and curing time values were held constant at 2500psi, and 6hrs, respectively. This shows that, 413 psi in the adverse zone is far less than the 1500psi minimum compressive strength specification for cements and materials for well cementing (API Specification 10A, 1995). This specification, is different from that of drilling further, after the casing have been cemented, which recommends a compressive strength in the range of 102 to 725psi; since further hydration and structural transformations occur throughout the exploitative life of the well, and even after plugging and abandonment of the wellbore (Omosebi *et al*., 2016). Additionally, in this adverse zone, the compressive strength of the cement sheath matrix is so decreased that, it will increase the transportation parameters, such as the permeability and porosity of the cement sheath matrix, to compromise the cement sheath functions of well isolation and integrity (Omosebi *et al*., 2016). In course of this, Omosebi *et al*. (2016) in a study stated that, impurity such as carbonic acid, posed serious well integrity and isolation issues in HPHT condition subsurface, by facilitating acid attack on well cement, resulting in the loss of well structural integrity and well isolation. Conversely, the aforementioned results also explained that, as the concentration of ferrous ion in mix-water decreases (less than 0.03mg/L), and the curing temperature is approximately at 250^{0}F, the optimal compressive strength increases to 4440psi; when pressure and curing time values are held constant at 3000 psi, and 8 hrs. Meaning that, as the concentration of ferrous ion increases under the influences of the prevailing well conditions, the cement sheath losses its compressive strength, which culminates to loss of well structural integrity and well isolation function. Therefore, high concentration of ferrous ion of above 0.3mg/L in mix-water, can be termed as chemical impurity (WHO, 2004). In support of this premise, Madhusudana *et al.* (2011) conducted a similar study. In the study, the impact of lead (Pb^{2+}) ion on the compressive strength was investigated by the spike of lead into deionised water in known concentrations. These mix-water contaminated with lead were mixed with Ennore sand to prepare cement mortar specimens. During this study, it was observed that, the presence of lead ion concentrations approximately higher than 3000mg/L in the mix-water reduces the compressive strength of the mortar concrete, when compared with the reference specimen. As a result of this observation, Madhusudana *et al.* (2011) established that, the presence of lead ion in high concentration causes the strength loss of the concrete; which is the consequences of the dissociation of calcium hydroxide and decalcification of tobermorite or Calcium Silicate Hydrate by lead. With regards to Madhusudana *et al.* (2011) investigation, it can be deduced that, the loss of compressive strength in a ferrous cement system, is as a result of the dissociation of calcium hydroxide and decalcification of tobermorite or Calcium Silicate Hydrate (C-S-H) by high ferrous ion concentration, into Ferrous Silicate Hydrate; owing that, the Hydrated Calcium Silicate (C-S-H) has high internal surface with an effective and strong mechanism of adsorption, to adsorb foreign ions (Sakr *et al*., 1997 and Zhang *et al*., 2018). Also, the large number of defects found in C-S-H structure encourages the replacement and integration of foreign heavy metal ions into the tobermorite surface (Glasser, 1994). Likewise, the availability of pore structure in cements provides some considerable surface areas to promote adsorption of heavy metal ions, to weaken the compressive strength of cement sheath (Zampori *et al*., 2006). These activities are very prone in the adverse zone. Therefore, mix-water must be made potable before it usage for cement slurry formulation. Although, during the destructive tests of compressive strength performance, it was observed that, the low strength cement sheaths were less shattered or brittle, whereas the completely hardened cement sheaths were very brittle and fractured excessively; and in the meantime, owing to the fact that, shattering of cement sheath is not a desired quality needed at the payzone's Oil-Water-Contact (OWC) and Oil-Gas-Contact (OGC), hence compressive strengths at the comfort zone may be more suitable.

5. Findings

- That in Minitab 16, the dataset must be entered into the worksheet according to the defined fieldnames. The DOE must be designed, customised, optimised, and analysed before plotting the response surface of contour or surface plots.
- That a consensus model must be achieved before commencing the plots of the response surface of contour or surface plots.
- That there exist some optimal, comfort, and adverse zones of high ferrous ion concentrations in mix-waters toward the responses of compressive strength development of oilwell cement sheath.
- That owing to the fact that, shattering of cement sheath is not a desired quality needed at the payzone's oil-water-contact (OWC), and oil-gas-contact (OGC), hence compressive strengths at the comfort zone may be more suitable for oilwell cementing.

6. Conclusion

The resulting conclusions are drawn from the investigation and results obtained:

- The comfort zone of the investigated compressive strength exists in mix-water with very low ferrous ion concentration (0.00 to 1.26mg/L) used in the formulation of cement slurry.
- In the comfort zone 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 frustrated. Hence, the compressive strength is higher than 1500psi, which is the minimum API specification for cementing oilwell.
- The adverse zone of compressive strength exists, when the mix-water used to formulate the cement slurry has high ferrous ion concentration of 1.27 mg/L and above.
- In the comfort zone 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. Therefore, the compressive strength is lower than 1500psi, which is the minimum API specification for cementing oilwell. Therefore, high concentration of ferrous ion is detrimental to well cement integrity.
- To yield compressive strength in the comfort zone, the mix-water used for the formulation of cement slurry must be potable.
- Among the four predictors investigated, temperature, curing time, and ferrous ion concentration were very influential on the compressive strength development; while pressure was not influential on the compressive strength development or loss.

7. Recommendation

7.1 For the Studies

Mix-water with high ferrous ion concentration should be reticulated, to make the mix-water potable. Furthermore, during the formulation of the cement slurry; additives such as magnetite, and anchorage clay should be used, to eliminate the voids between the casing and cement sheath, and on the other hand the cement sheath and borehole. The voids in the internal matrix of the ferrous cement sheath can be reduced significantly with the additives called silica flour. Technically, this practice would increase the comfort zone, and drastically reduce the adverse zone of the compressive strength of the cement sheath system.

7.2 For Further Studies

The results obtained from the investigation, disclosed that, there exist some optimal, comfort, and adverse zones of high ferrous ion concentrations in mix-waters toward the responses of compressive strength development of oilwell cement sheath. Additionally, high concentration of ferrous ion in mix-water adversely impact on the strength development of cement sheath at prevailing well conditions. But, these results did not explain or depict any empirical prediction model. Therefore, a further study is recommended, which shall involve the development of an empirical model, that would simulate, the effect of high ferrous ion concentration in mix-water on the compressive strength of oilwell cement sheath. This model may benefit the oil and gas industry; mostly, in the elimination of the energy and cost, for the repetition of experimenting similar ferrous cement systems, to estimate compressive strength development or loss. In addition, the porosity, and permeability of the ferrous cement sheath system should be investigated, to determine the correlations between compressive strength and porosity, and permeability at high-pressure and high-temperature well conditions. This is to ascertain, if high compressive strength of ferrous cement sheath is associated with acceptable porosity, and permeability, for proper oilwell formation zonal isolation.

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