NANO CEMENT SLURRIES WITH ENHANCED STREGNTH FOR OILWELL CEMENTING

by

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14282

Dissertation submitted in partial fulfilment of the requirements for the
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CERTIFICATION OF APPROVAL

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Approved by:

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September 2014

CERTIFICATION OF ORIGINALITY

I hereby verify that this report was written by me, Muhammad Naqiyuddin bin Abdul Naser (14282). I am responsible for the work I have been submitted in this project, the work have that had been done is my own except as specified in the references and acknowledgements.

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(MUHAMMAD NAQIYUDDIN BIN ABDUL NASER)

ABSTRACT

Cementing is among the important steps in the drilling and completion of an oilwell as it has the function of holding the steel casing in place by acting as the bonding agent between casing and the formation body. But upon certain types of reservoir, the formation fluid is under very high pressure, causing the fluid to flow at very high flow rate towards the well and promotes channeling of the fluid into the cementing job. This can cause the cement to crack and allowing uncontrolled flow of hydrocarbon liquid and gas into the wellbore. Besides that, the increase in downhole pressure and temperature causes stress on the cement sheath which later causes cement shrinkage and degrading. Therefore, nanosilica is introduced in cement slurries formulation to enhance the strength of the cement in order to withstand these abnormal conditions.

This paper focuses on investigating the effect of nanosilica on strength of cement cured under High Pressure and High Temperature (HPHT). An experiment was conducted to prove the effectiveness of nanosilica in increasing the compressive strength of cement. Four cement formulations with addition of different percentage of nanosilica were prepared before the start of the experiment. The percentages of nanosilica used were 0%, 0.5%, 1.0% and 2.0% by weight of cement used. No other additives were used in the cement. The materials were then mixed using a constant speed mixer and poured into the metal moulds. Two curing conditions were used for this experiment: 3000 psi with 120°C and 3000 psi with 60°C. Four cement slurries were prepared to be cured at respective conditions (total of 8 cement slurries). The prepared metal moulds were put into the curing chamber and were left to be cured for a total of 24 hours. The finished cement cubes were brought out of the chamber and left to dry at room condition for less than 1 hour. After that, the compressive strength of the cubes were tested using a manual compression tester. Each of the cubes was put into the tester, positioned at the centre of the compressor. The test was run and the compressive strength readings were recorded by computer. From the test, it was found that the cement cubes with higher nanosilica concentration gave higher compressive strength. This is due to the nanosilica particles filling up the space between cement particles thus, provide stability to the cement structure. Since 60°C is the lower temperature for cement curing, the compressive strength of the cubes cured at this temperature are much higher, showing the shrinkage effect experienced at 120°C.

ACKNOWLEDGEMENT

In completion of this Final Year Project entitled Nano Cement Slurries with Enhanced Strength for Oilwell Cementing, I would like to take this opportunity to express my deepest appreciations to all the parties that have made this project successful. First and foremost, my greatest gratitude goes to Dr. Syahrir Ridha, my supervisor, for his guidance and assistance throughout the year. Under his supervision, I have gained valuable experience and new knowledge. Secondly, I would also like to express my gratitude to the Petroleum Engineering Department, Universiti Teknologi PETRONAS (UTP) which is led by Assoc. Dr. Ismail Bin Mohd Saaid (Head of Petroleum Engineering Department) for providing me the facilities and equipment used in this research. I would like to thank Mr. Khairunnizam Wahid and Mr. Jukhairi Selamat for their assistance. Last but not least, I would like to thank my family, lab assistants, lectures, staff and my colleagues who have been supporting and helpful to me throughout this final year project.

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CHAPTER 1

INTRODUCTION

1.1 Backrgound of Study

Cementing is among the important steps in the drilling and completion of a well. For an oil well completion, typically in a wellbore, cement is set in between the steel casing and the formation **[1]**. **Figure 1** shows the positions of cement, steel casing and formation wall in a wellbore viewed from the top of a well. The cement acts as a wall to shut out the wellbore fluids from the annular flow path migration, and also to allow full utilization of the well without any problem **[2][3]**. This study focuses on compressive strength as it is one of the most crucial mechanical properties that needed to be taken into account for the well completion.

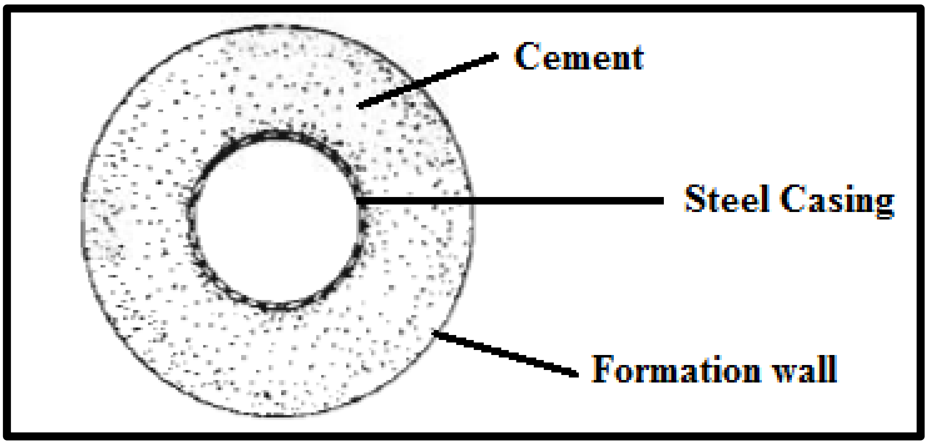

Figure 1: Position of Cement and Steel Casing in a wellbore (top view) **[5]**

Compressive strength, by definition, is the ability of a substance to endure force that tend to decrease its size through time. Early compressive strength development is important in cementing as the cement holds the steel casing in place and isolate the casing seats during the drilling of a well **[6]**.

Due to the significance of compressive strength and its early development, nanomaterials have been introduced in the cementing process to improve the properties of cement and solve the problems on well completions regarding cement compressive strength. Nanomaterials are said to exhibit different chemical and physical properties compared to their much larger-sized counterparts because of their great increase in the surface area to volume ratio **[7]**. The applications of nanomaterials are considered to be much more beneficial, as they improve the chemical and mechanical properties of existing materials.

The use of nanomaterials, particularly nanosilica, together with cement can achieve early compressive strength development to reduce the wait-on-cement (WOC) and thus, continue further drilling as quickly as possible **[8]**. Nanosilica can also be used to replace traditional salt accelerator for cement as the salt accelerators have potential negative effect of increasing the permeability of the cement meanwhile the nanosilica accelerators reduces the permeability while increasing the mechanical strength of the cement **[4]**.

The addition of nanosilica to cement slurries can also act as an accelerator to increase the hydration rate of cement, even at low-temperature conditions **[9]**. This results in a reduction of WOC and a much faster, sturdier well completion compared to using normal cement slurries with traditional salt accelerator additives.

1.2 Problem Statement

Listed are among the problems faced in the oil and gas industry regarding the cementing process of oilwells:

1. Failing in obtaining complete hydraulic seal between cement and casing as well as between cement and formation in the cemented interval for zonal isolation **[10]**.
2. The fluctuations of pressure and temperature in downhole causes stress on the cement sheath which later causes cement shrinkage and degrading, thus allowing fluid migration **[6][17]**.
3. Significant amount of time lost due to the problems occurred due to the compressive strength of the cement **[7]**.

1.3 Objective

The objective of this project is to investigate the effect of nanosilica on strength of cement cured under High Pressure and High Temperature (HPHT). This will show the potential of using nanoparticles in enhancing the properties of cement for well completion under extreme reservoir conditions.

1.4 Scope of Study

1. Manual Compressive Strength Tester is used to measure the compressive strength of cement.
2. Class G Portland cement will be used as benchmark in this project.
3. The curing pressure is 3000 psi and the curing temperatures are 60°C and 120°C.

1.5 Feasibility of the Project within the Scope and Time Frame

The project is basically a laboratory based project and to be able to conduct the experiment, there are some things that need to be prepared beforehand. The things include experimental procedure to conduct the experiment, the materials and historical data that had been done previously. Thus, it is possible for the author to achieve the objectives as the time frame given is about seven months.

CHAPTER 2
LITERATURE REVIEW

2.1 Oilwell Cementing

Being a binder, cement is a substance that can bind other materials together as it dries and reacts with carbon dioxide in the air dependently to become hard. In the oil and gas industry, cement is used to bind the steel casing and formation during the completion of an oil well. According to Iverson, B., Darbe, R., McMechan, D. (2008), Hart, W.A. and Smith, T.R. (1990) and Rahimirad, M., et al. (2012), the cement acts as a barrier, allowing the well to be operated safely and without control problem by stopping wellbore and formation fluids from flowing in the flow path between the casing and formation **[2][3][10]**.

Slurry, which is a mixture of cement and water, is pumped down the casing center and back into the gap in the middle of the casing and the formation and as it hardens, bounds the steel casing to the surrounding formation by the cement **[1]**. The cement now provides structural support to the casing in the hole to withstand the shock of drilling, perforating and fracturing throughout the well life. **Figure 2** shows the working principle of the cementing process.

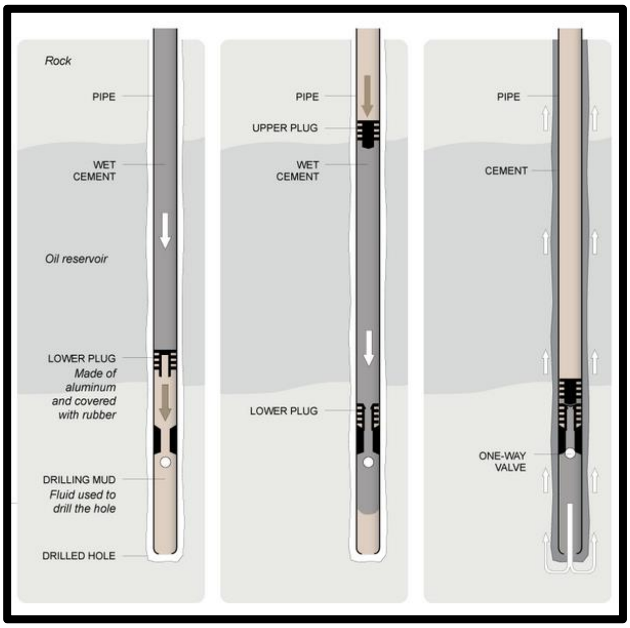

Figure 2: Principle of Cementing Process **[11]**

Cement can be classified as hydraulic or non-hydraulic from its ability to be used in the presence of water. Underwater or other wet surroundings is not suitable for setting non-hydraulic cement and it is usually attacked by some aggressive chemicals after setting for some known reasons. On the other hand, activated aluminium silicates and pozzolanas (fly ash) are being used to replace some of the cement in a concrete mix to produce hydraulic cement. According to Romero, J., and Loizzo, M. (2000), the additives activate the cement setting in wet condition or underwater and provide further protection for the hardened concrete against chemical attack **[12]**. Different from non-hydraulic cement, the hydraulic cement hardening process occurs because of hydration. The most well-known and wide used type of hydraulic cement is the Portland cement.

Calcium carbonate ($CaCO_3$) and clay/shale are the basic raw materials used in producing Portland cement **[13]**. At times, due to the insufficient quantity of iron and alumina in the clay or shale, they are added separately. **Figure 3** shows a sample of Portland cement clinker. The manufacturing of Portland cement often depends on the chemical and physical standards needed for their application.

Figure 3: Sample of Portland Cement Clinker **[14]**

In the oil and gas industry, Portland cement is used as drilling and completion cement for almost all types of wells **[13]**. Furthermore, this type of cement is able to be modified easily. The types of cements of different classes to be produced and the chemical composition of the raw materials are among the elements that are taken into account for a series of simultaneous calculations in the proportioning of the raw materials. Among the class of cement derived from Portland cement are American Petroleum Institute (API) Class A, C, G, or H. The types of API cements:

Class A

- For use under normal condition.
- Available: ordinary, O grade.

Class B

- For use in normal to great sulphate resistance conditions.
- Offered: MSR and HSR grades.

Class C

- For use when in initial strength conditions.
- Offered: ordinary, O, MSR, and HSR grades.

Class G

- No accompaniments of additives.
- Commercial and Industrial use.
- Offered: MSR and HSR grades.

Class H

- No extra addition of chemicals.
- Commercial and Indusrtial use.
- Offered: MSR and HSR grades.

The understanding of the mechanical properties of cement is very important as it helps in predicting and apprehending the cement behaviour interaction during the life of a well. The six main mechanical properties of cement are Compressive Strength, Tensile Strength, Young's Modulus, Poisson's Ratio, Cohesion Strength and Friction Angle **[2][4]** but with regards to cementing, compressive strength plays an important role in withstand the confining pressure from drilling activities and the formation as well as hydrostatic pressure from the fluids inside the well. **Table 1** shows the basic definition for the compressive strength of cement.

Mechanical Property	Definition
Compressive Strength	▪ The capability of cement to endure forces inclining in shrinking its size ▪ Reaching a limit in compression strength caused fracture or permanent deformation

Table 1: Compressive Strength of Cement and its definition

Besides the mechanical properties of the cement, there are other factors that contribute to the characteristics of a good cement slurry of well completion. One of them is hydration rate. According to Boul, P.J., Jimenez, W.C., and Pang, X. (2014) and Santra, A., et al. (2012), hydration is a very important factor for cement as the mechanical properties of cement depends on the hydration rate of that cement, for instance, the compressive strength of a cement decreases with time due to loss of fluid and for cementing, the hydration rate needs to be fast enough so that the cement will dry quickly and hold the steel casing in place **[4][9]**.

2.2 Development of the Compressive Strength of Cement

The cement matrix and the curing process of cement are both the individual components that the cement properties depend on. Besides that, the cement-to-water ratio, addition of light- and heavy-weighting agents, fibers, elastomers, and foam quantity can also affect the cement properties. For instance, the addition of elastomers to the cement matrix may decrease the Young's modulus and allow more elastic cement **[2]**. But in the process of well cementing, compressive strength is the most important mechanical properties in ensure the stability and the durability of the wellhole.

The compressive strength of cement post-preparation and location of cement grout into the wellbore is called early-age strength of compression and the compressive strength of cement after achievement of hydration and the long use of the oilwell is known as long-term compressive strength.

According to Labibzadeh, M., et al. (2010), Sabin, F.L. and Sutton, D.L. (1986) and Teodoriu, C., et al. (2012), by achieving proper early-age oilwell cement compressive strength, it ensures that the casing has the physical support it needs, hydraulic and mechanical isolation as well as providing zonal isolation between the wellbore fluids and the reservoir fluids thus, preventing the contamination of the surrounding of the wellbore **[6][15][16]**.

The cement slurry starts changing into a partially firm set substances from true fluid with assessable compressive strength at earlier development of the gel and also when over shear strain, the gel had an increment in strength and start experiencing hydrostatic pressure as cement grout is formed and drove into the wellbore. During this phase, the cement column provides support for itself and prevents loss of hydrostatic pressure to the flow zone and an increase in time for the reduction in volume, making this transition phase very important **[17]**.

Long term development of compressive strength is potrayed based on the laboratory experiment done by Teodoriu, C., et al. (2012), it is shown that the Class G cement produces 6MPa of compressive strength in one day and it increases significantly from one day to twenty days. The compressive strength increases marginally after the 20 days. It is also presented that in 1 day, the strength of compression for Class G cement already reaches 36 MPa for a cement that is cured at 65°C. At 14 days, the compressive strength increases from 36 MPa to 64 MPa, and after that, it almost kept constant at 64 MPa. Under the curing condition of 100°C and 18 MPa confining pressure, the compressive strength of Class G cement is 47 MPa, after that the compressive strength remains the same. The compressive strength at fourteen days is maintained as the one at three days **[6]**. **Figure 4** shows the details of the cement compressive strength findings by the researchers.

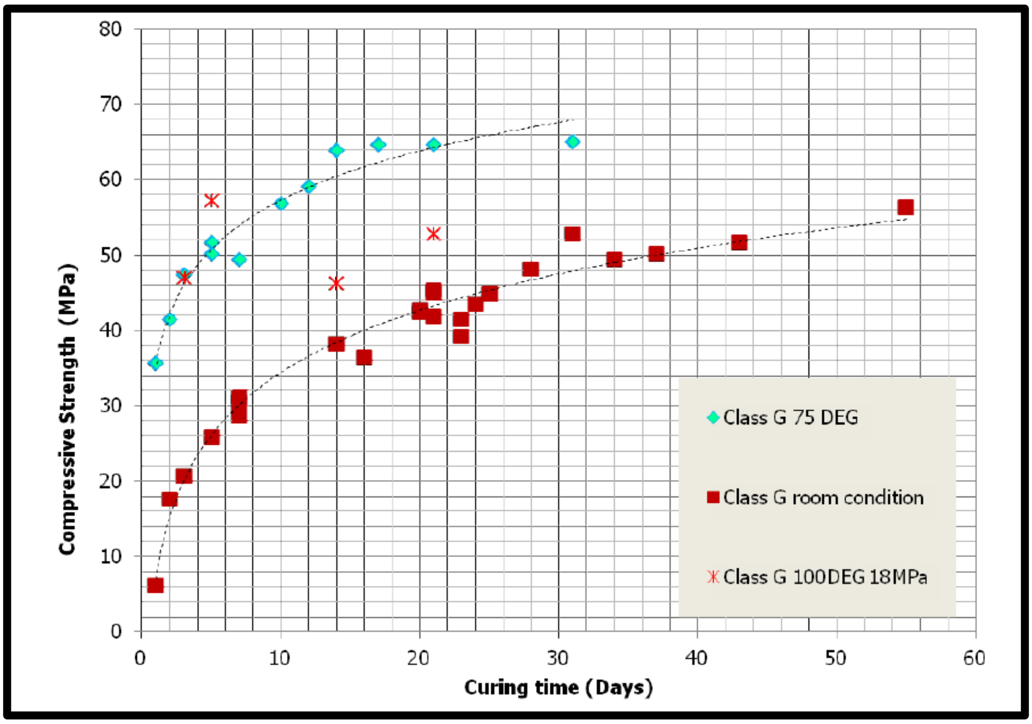

Figure 4: Compressive Strength of Cement against Curing Time Graph **[6]**

When cement is mixed with water, the reactions involved become complex. Obviously, the cementing process does not carry a 100% success rate and is bound to fail if not carries out carefully. However, because of the composite nature of the cement particle and proximity of the phases, the reactions become dependent towards each other **[18]**.

In all, there are five different stages that have been identified in preparing the cement mixture:

- Stage 1: Pre-induction
- Stage 2: Induction period
- Stage 3: Acceleration
- Stage 4: Deceleration
- Stage 5: Steady state

Stages 1 through 3 are very important in cementing operations, with Stage 1 indicating the early mixing capability of the cement, and is featured primarily to the aluminate and ferrite phase reactions. Stage 2 relates to the time taken for the cement pumping process, while Stage 3 shows the growth of the cement in terms of setting properties and gel-strength **[4]**.

In essence, it is known that cement comprises of major and minor chemical compounds. The typical Portland cement composition is listed in **Table 2.**

Cement Compound	Weight Percentage	Chemical Formula
Tricalcium silicate	50 %	Ca_3SiO_5 or $3CaO{\cdot}SiO_2$
Dicalcium silicate	25 %	Ca_2SiO_4 or $2CaO{\cdot}SiO_2$
Tricalcium aluminate	10 %	$Ca_3Al_2O_6$ or $3CaO{\cdot}Al_2O_3$
Tetracalcium aluminoferrite	10 %	$Ca_4Al_2Fe_2O_{10}$ or $4CaO{\cdot}Al_2O_3{\cdot}Fe_2O_3$
Gypsum	5 %	$CaSO_4{\cdot}2H_2O$

Table 2: The Composition of a typical Portland cement **[17]**

Every one of the chemical elements of cement goes through hydration and affects the end product when water is added to it. Strength contribution is only by the calcium silicates **[4][17]**. During first 7 days, it is tricalcium silicate that contributes the most in the early stages of strength development. Due to more slowly reactions, dicalcium silicate only contributes in the development of strength in the later stages. **Equation [1]** indicates the process for the hydration of tricalcium silicate:

$$\text{Tricalcium Silicate} + \text{Water} \text{ ---> } \text{Calcium Silicate Hydrate} + \text{Calcium Hydroxide} + \text{Heat} \quad \textbf{[1]}$$

$$2\ Ca_3SiO_5 + 7\ H_2O \text{ ---> } 3\ CaO.2SiO_2.4H_2O + 3\ Ca(OH)_2 + 173.6kJ$$

Tricalcium silicate rapidly emits different types of ions and a large amount of heat when it reacts with water. The pH value increases greatly over 12 with the release of alkaline hydroxide (OH^-) ions. With a correspond decrease in heat, the intial hydrolysis slows down quickly **[12]**.

Crystal calcium hydroxide starts to form once the system becomes slowly saturated with calcium and hydroxide ions from previous reaction. Simultaneously, calcium silicate hydrate begins to form **[5]**. The tricalcium silicate reacts to become calcium and hydroxide ions and is accelerated by the ions that precipitate out of the solution. This, dramatically increases the evolution of heat again.

More calcium silicate hydrate can be formed by the formation of the calcium hydroxide and calcium silicate hydrate crystals. It is more difficult for water molecules to reach the anhydrate tricalcium silicate as the calcium silicate hydrate crystal grow thicker. The rate at which water molecules diffuse through the calcium silicate hydrate coating controls the speed of the reaction **[12]**. The production of calcium silicate hydrate to become slower and slower because of the thickening of coating over time.

The strength of cement is also affected by dicalcium silicate through its hydration. Dicalcium silicate slowly reacts with water in a similar manner as tricalcium silicate. Due to dicalcium silicate being much less reactive, the heat released is less than that by the hydration of tricalcium silicate **[17]**. The products from the hydration of dicalcium silicate are the same as those for tricalcium silicate, given by **Equation [2]**:

Tricalcium aluminate and tetracalcium aluminoferrite, the other major components of

Dicalcium Silicate + Water ---> Calcium Silicate Hydrate + Calcium Hydroxide + Heat **[2]**

$$2\ Ca_2SiO_4 + 5\ H_2O \text{--->} 3\ CaO.2SiO_2.4H_2O + Ca(OH)_2 + 58.6\ kJ$$

Portland cement, also react with water. Their reactions with gypsum as well made their hydration chemistry much more complicated. The rate of hydration of a compound may be affected by varying the concentration of another **[5]**. In general, the rates of hydration during the first few days ranked from fastest to slowest are:

Tricalcium Aluminate > Tricalcium Silicate > Tetracalcium Aluminoferrite > Dicalcium Silicate. **[3]**

2.3 The Use of Nanosilica in Cementing Process

Nanosilica is distinctively an extremely effective pozzolanic material. Described as being one of the most suitable additives for cement, it comprises of small vitreous particles that are approximately a thousand times finer than the commercial cement particles **[8]**. It is very useful in to improving the strength and durability of cement while decreasing its permeability.

Nanosilica can be categorized into two particle size, the first ranging between five to fifty nanometers, and the second, between five to thirty nanometer, both powders in capsule form and have concentration of five to fifteen percent bwoc **[15]**. Nanosilica is a very popular additive that is used in the oil field because, in various testings and experimentation, it has been demonstrated that the addition of nanosilica into cement decreases the time and increases the compressive strength in relation with other silica components.

Using the suitable amount of nanosilica, cement slurry with low rheology and good mechanical properties can be designed and much stronger lightweight cement can be formulated in addition to decreasing the free fluid by controlling the fluid loss and stabilizes a huge amount of slurries **[1][7]**.

EFFECT OF NANOSILICA ON COMPRESSIVE STRENGTH					
Latex (gal/sk)	Silica	Retarder (gal/sk)	Time to 500 psi (hr:min)	UCA Strength Rate of Strength Development (psi/hr)	24-hr Strength (psi)
1.5	0	0.05	23:05	172	690
1.5	Micron sized silica	0.05	21:45	160	610
1.5	Nanosilica	0.05	13:29	460	2203

Table 3: The Effect of Nanosilica on the Compressive Strength of Cement **[7]**

Table 2 shows the effect of nanosilica on the compressive strength. A comparison was also made with micron-sized silica to show the real potential of nanoparticles. The rate of strength development can be seen to increase from 172 to 460 psi/hr by the addition of nanosilica. The composition of cement without nanosilica is referred to as the base case. The critical strength of cement configuration is about 3x that of the base case and the base case with addition of microsilica. The rheological properties of the cement slurries with nanosilica are slightly higher compared to the base case, however, the slurry remains pourable and pumpable **[7]**.

CHAPTER 3

METHODOLOGY

3.1 Project Work

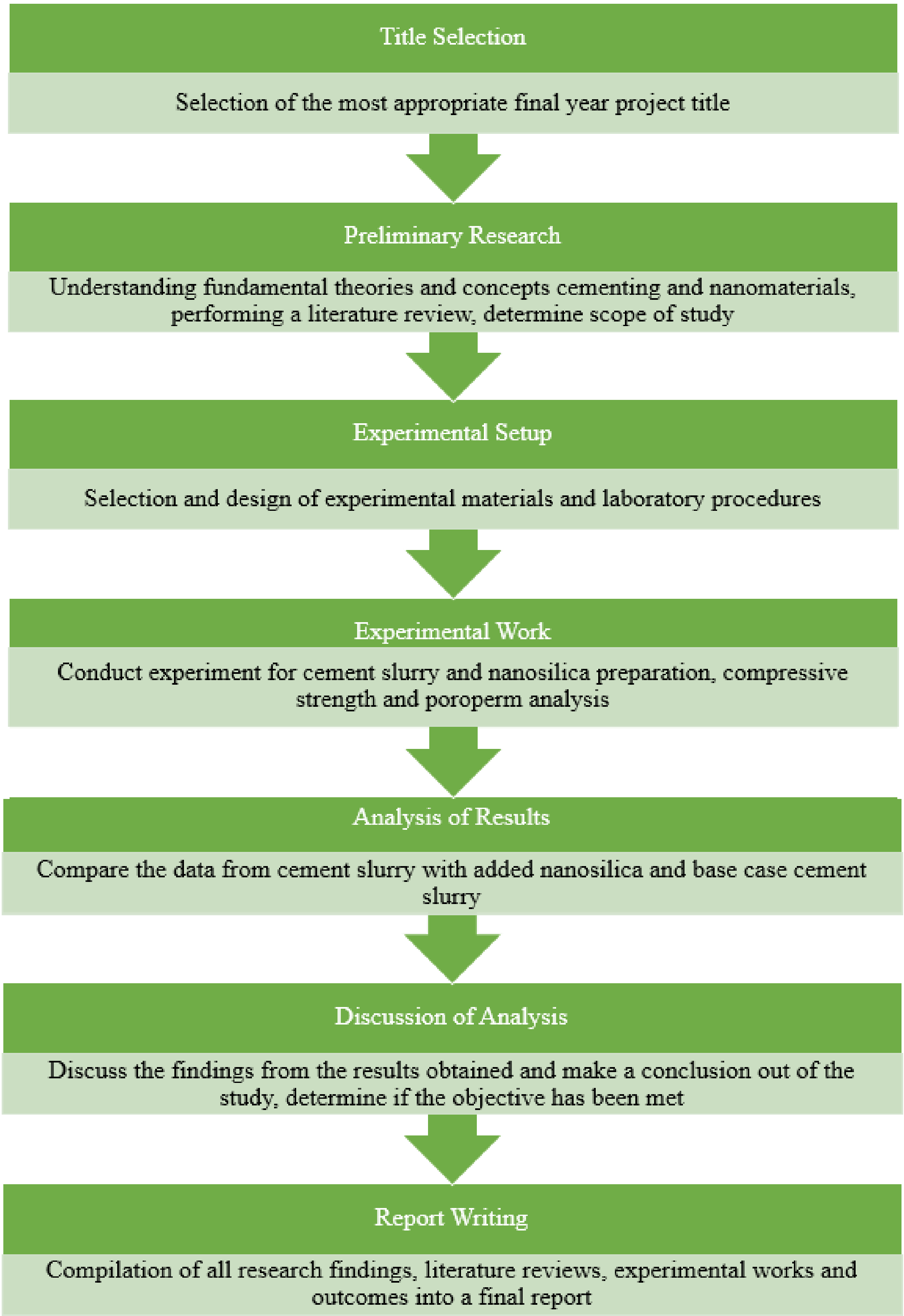

Figure 5: The Flow of the Project

3.2 Research Methodology

Research is a method taken in order to gain information regarding the major scope of the project. The sources of the research cover the handbook of the use of nanomaterial in oilwell cementing, e-journal, e-thesis and several trusted link. As the project is a laboratory based, the experimental procedure is being designed carefully to ensure the safety as well as to get the required result.

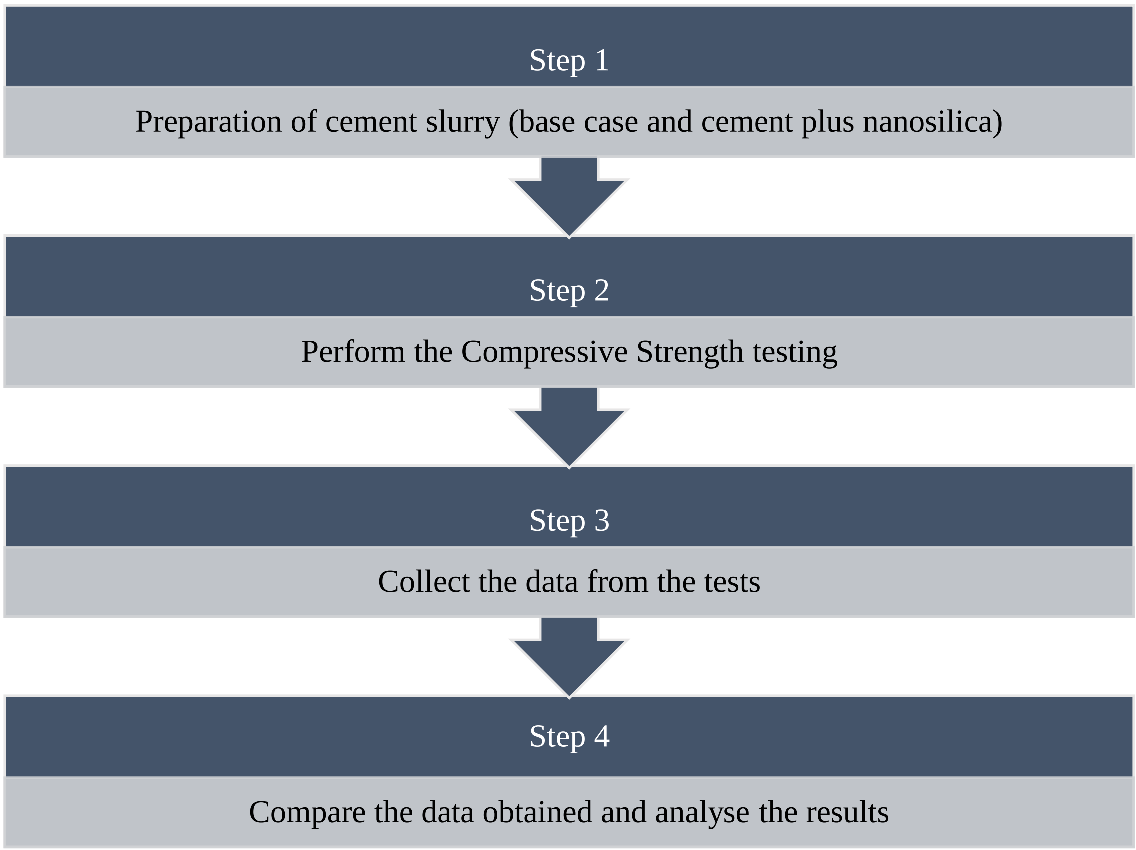

Figure 6: Experimental Procedures

3.3 Experimental Procedures

<u>Experimental Methods</u>

The type of cement used for this project is Portland Class G Cement with additive nanosilica (SiO_2) powder. The nanosilica powder has the primary range of particle size between 10 nanommeters to 20 nanometers, which is comparatively smaller to the average cement particle size. It also has 99.5% trace metal basis, meaning there is a presence of only 0.02 percent trace metal in the nanosilica used for this experiment. The molecular weight of the nanosilica is 60.08 grams/mole. Four types of cement slurries are prepared for this experiment: the base case has a composition ratio of 100% Class G Portland cement, case 1 has a composition ratio 99.5% Class G Portland cement and 0.5% nanosilica, case 2 has a composition ratio of 99% Class G Portland cement and 1% nanosilica meanwhile case 3 has a composition ration of 98% Class G Portland cement and 2% nanosilica. All of these ratios are based on the weight of cement, as 500 grams of cement is used for the base case. Distilled water is used for the mixing of the cement into cement slurries with a standard water-cement ratio of 0.44:1 according to American Petroleum Institute (API). For this experiment, a total of 8 samples of cement cubes will be made, 2 cement cubes for each case. Next, 2 groups will be formed with 1 sample from each cases in each group. The groups will then be separated and one group will be cured at 120°C and 3000 psia while the other group will be cured at 60°C and 3000psi for 1 day (24 hours). After the curing process, the cement cubes will undergo compression test using Manual Compressive Strength Tester.

<u>List of Material</u>

The list of materials used for this project:

<table>
<tr><th>Material</th><th>Description</th></tr>
<tr><td>Class G Portland Cement</td><td rowspan="2">Based on the condition of the cases:
(i.e. case 1= 99.5% Class G Portland Cement, 0.5% nanosilica)</td></tr>
<tr><td>Nanosilica powder</td></tr>
<tr><td>Distilled water</td><td>0.44:1 weight proportion ratio of the whole slurry mixture</td></tr>
</table>

Table 4: List of Materials used for the experiment

List of Laboratory Apparatus

The list of laboratory apparatus used for this project:

1. 8 Metal moulds and Mixing cup
2. Cement Mixer
3. Curing Chamber
4. Electronic Balance
5. Manual Compressive Strength Tester

Tests and Procedures

There is only one test involved in this project namely compressive strength testing. Before the test is done, the cement slurries are prepared in room condition and then, they are left to be cured at desired time (1 day) in the curing chamber. The following are the steps for cement slurries preparation and the compressive strength test:

Cement Slurry Preparation

1. The formulation for the amount of cement, water and nanosilica to be used is prepared for each case using the following equation **(refer Appendix I)**:
   Base Case: Cement Weight = 500g
   Water Weight = (44/100) x 500g = 220g
   Other Cases: Cement Weight= (Cement Percentage/100) X 500g
   Nanosilica = 500g – Cement Weight
   Water Weight = (44/100) X Cement Weight
2. The calculated amount of cement, nanosilica and water are then weighted by using the electronic balance. The accuracy of the weighting is two decimal places. The weighted materials are put into separate sealed plastic bags and for water, into sealed beakers.
3. The weighted water is then poured into the mixing cup and the cup is attached to the cement mixer. The water is blended for 15 seconds at 4000 rev/min and later, the whole mixture is blended for 35 seconds at 12000 rev/min.

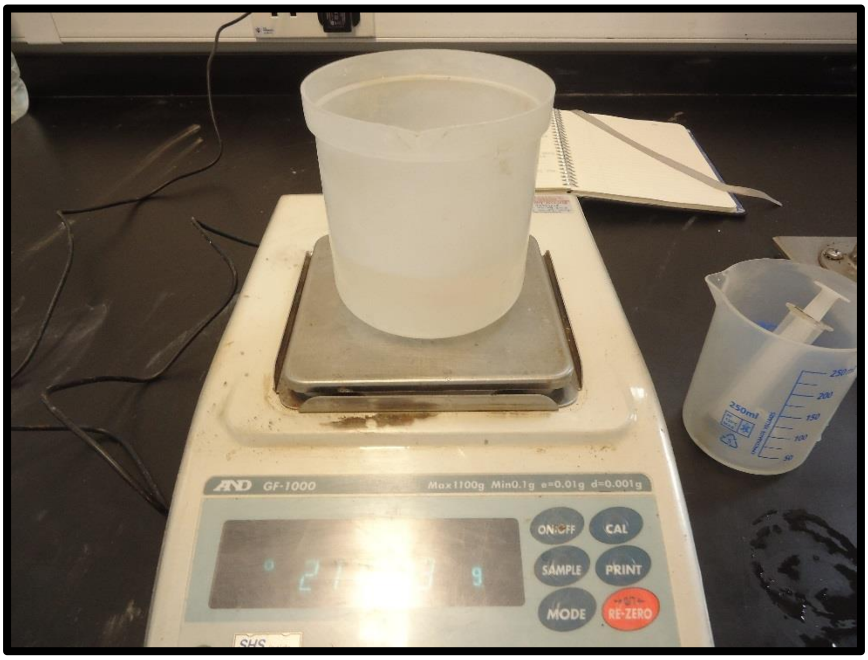

Figure 7: Weighing Cement, Nanosilica and Water using Electronic Balance

4. During the first blending (15 seconds), the cement and nanosilica are added to the water respectively to ensure uniform dispersion of the material in the mixture.
5. No addition of nanosilica for preparing base case cement slurry.
6. The cement moulds are properly waxed and prepared for moulding the cement slurries into cubes.

Figure 8: Waxed Cementing Moulds

7. Then, the cement slurry is transferred into moulds (2 in. x 2 in. x 2 in.) to be cured. The moulds are tightly locked using screws and are stacked together.

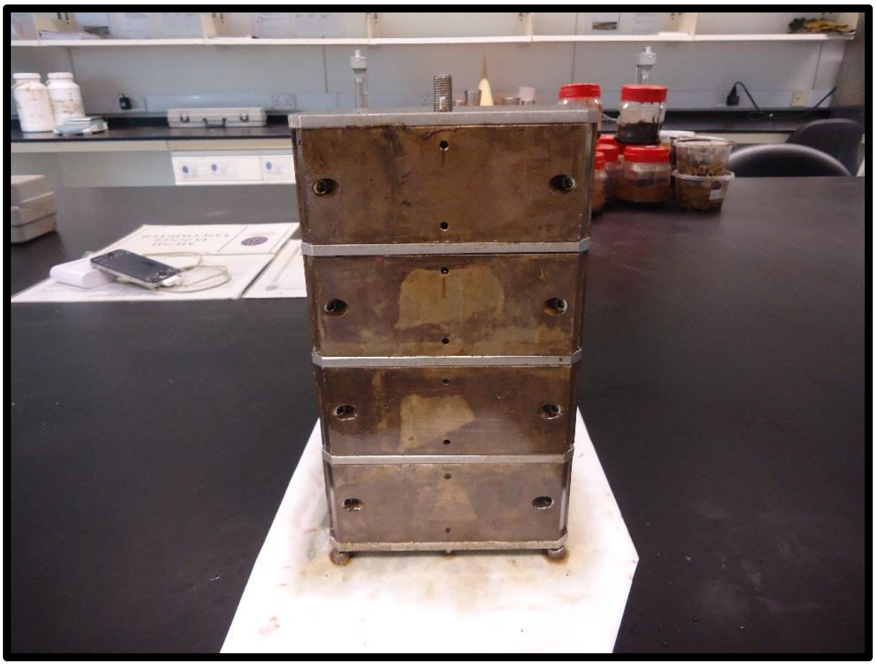

Figure 9: Stacked Cementing Moulds containing Cement Slurries

8. Step (1) to (6) is repeated until all the 8 metal moulds are filled with the cement slurries for the four cases.

<u>The Curing of Cement Cubes</u>

1. The metal moulds are then placed inside a curing chamber and the parameters are then programmed into the chamber. The parameters include the temperature-pressure profile, curing time, and desired curing temperature amd pressure.
2. After the chamber is tightly shut and the chamber is pressurised, the cubes are cured at desired temperatures and pressure (120°C and 3000 psi, and 60°C and 3000 psi). The group of cement slurries mentioned earlier are cured for 1 day.
3. When the desired time is reached, the moulds are taken out of the oven and left to be cooled for several hours.

Figure 10: HPHT Curing Chamber

4. Finally, the cement cubes are ready for further testing. The finished cement is in the form of cement cubes.
5. The steps are repeated for other curing conditions.

Figure 11: The finished Cement Cubes

Manual Compressive Strength Testing

Figure 12: Manual Compressive Strength Tester

1. The cement cubes are taken out of their moulds.
2. One cubes is chosen to be put into the compressive strength tester and the cube is put under the pressure bar which will compress the cube later on. The position of the cube must be at the centre of the bar.
3. The tester is turned on and the lid of the machine is closed tightly.
4. The information on the cube is keyed into the equipment's software.
5. After everything is set, the pump is turned on via the software and the testing is run by pressing and holding the run test button in the software. The cube is viewed from a safe distance.

6. Hold the run test button until the cement cube cracked. After that, the test stopped and the machine is shut down. The debris from the crushing is cleaned from the machine.
7. The result is then collected in the form of graph and table. The test is repeated until all the samples are finish.

Figure 13: Cracked Cement Cube after Compressive Strength Testing

3.4 Gantt Chart and Key Milestones

No.	Detail/Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Selection of title - FYP Title Conformation - Meeting with FYP Supervisor	■	■	■ ◆											
2	Reading and Research on The Problems arose in Oilwell Cementing				■	■									
3	Investigating Compressive Strength and Permeability of Cement				■	■									
4	Analysing the Application of Nanosilica in Oilwell Cementing					■	■	■							
5	Draft methodology of the project					■	■	■							
6	Extended Proposal submission								■ ◆						
7	Proposal Defense									■					
8	Detailed methodology of the project										■	■	■		
9	Submission of Interim Draft Report													■ ◆	
10	Submission of Interim Report														■ ◆

Legend: ■ **Process** ◆ **Key Milestones**

Table 5: Gantt Chart For Final Year Project I

No.	Detail/Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Project Work : - Lab booking and material preparation - Lab briefing and start of lab session	■	■	◆											
2	Preparation of Cement Cubes, Measuring of Compressive Strength			■	■	■	■	■							
3	Submission of Progress Report							◆							
4	Project Work Continue: Preparation of Cement Cubes, Measuring of Compressive Strength								■	■	■	■			
5	Pre- SEDEX										◆				
6	Submission of Draft Final Report											◆			
7	SEDEX												◆		
8	Submission of Technical Paper												◆		
9	Oral Presentation (Viva)													◆	
10	Submission of Project Dissertation (Hard Bound)														◆

Legend: ■ **Process** ◆ **Key Milestones**

Table 6: Gantt Chart For Final Year Project II

3.5 Important Dates for Final Year Project

No.	Activities	Completion Date
1	Completion of the First Draft of Extended Proposal	04/07/2014
2	Completion of Extended Proposal	10/07/2014
3	Completion of Proposal Defense	17/07/2014
4	Completion of Interim Draft Report	11/08/2014
5	Completion of Interim Report	20/08/2014

Table 7: Key Milestones of Final Year Project I

No.	Activities	Completion Date
1	Lab Briefing and Start of Lab Session	16/10/2014
2	Completion of Progress Report	5/11/2014
3	Completion of Pre-SEDEX	20/11/2014
4	Completion of Draft Final Report	28/11/2014
5	Completion of Lab Sessions	26/11/2014
6	Completion of SEDEX and Technical Paper	5/12/2014
7	Completion of Oral Presentation (Viva)	12/12/2014
8	Completion of Project Dissertation	19/12/2014

Table 8: Key Milestones of Final Year Project II

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 Compressive Strength Test Results for Curing Pressure 3000 psi and Curing Temperature 120°C

<u>**Base Case (100% Class G Portland cement)**</u>

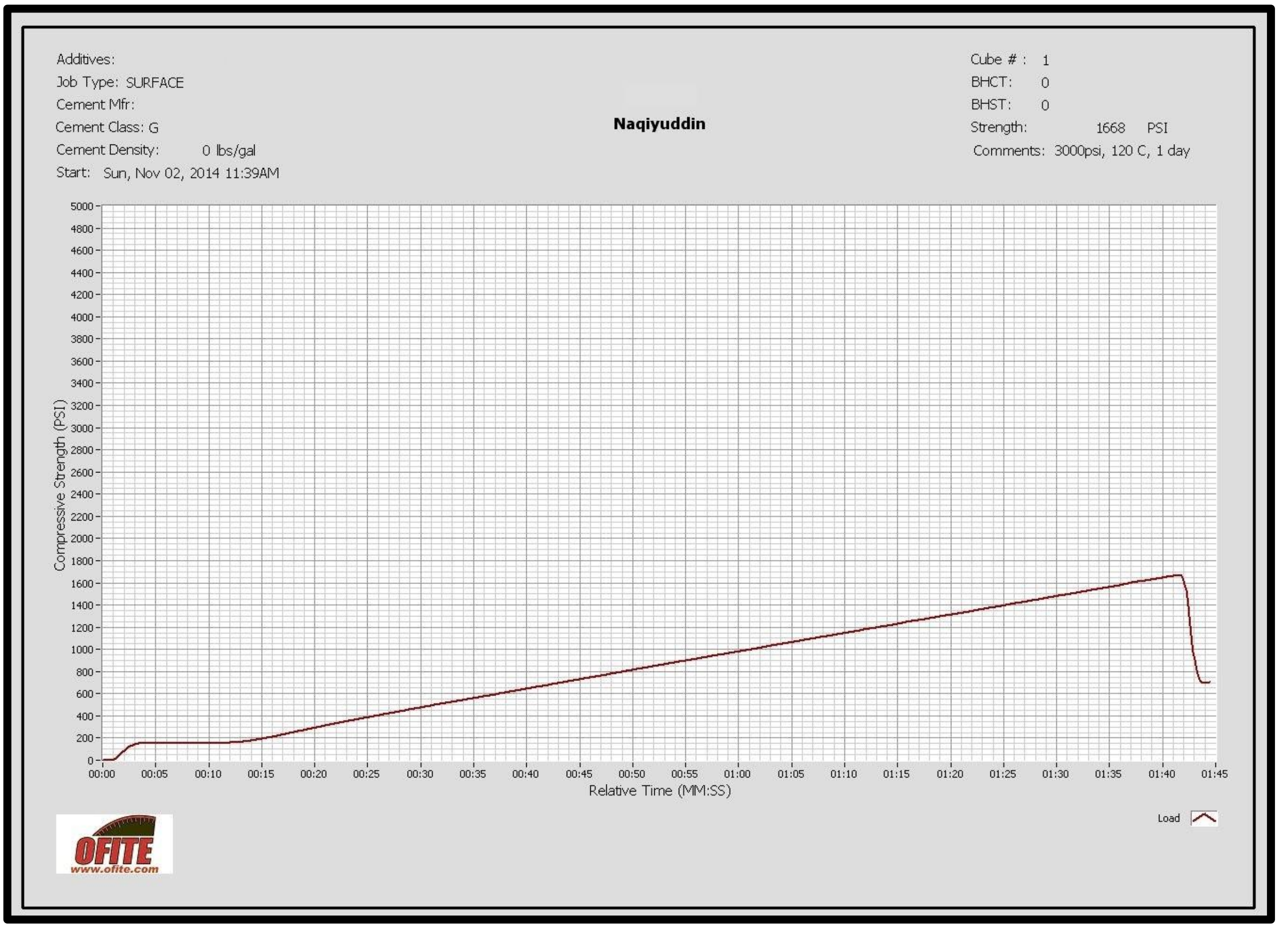

Figure 14: Compressive Strength vs Time Graph for Base Case

The above graph shows the strength profile of the cement cube with no addition of nanosilica in the cement mixture. At the beginning of the test, the cement cube is not put under any pressure thus the reading of the compressive strength showing the value of zero. With time, the pressure on the cube started to increase, and as the cube withstands the pressure, the compressive strength of the cement cube also starts to increase until it reaches the maximum value at which it can stand the applied pressure. The cube cracks when the applied pressure exceed the maximum of its compressive strength. For the pure Class G cement cube, the compressive strength is 1668 psi. The end of the graph that shows a drastic decrease in compressive strength shows that the cement cube breaks into pieces.

Case 1 (99.5% Class G Portland cement, 0.5% Nanosilica)

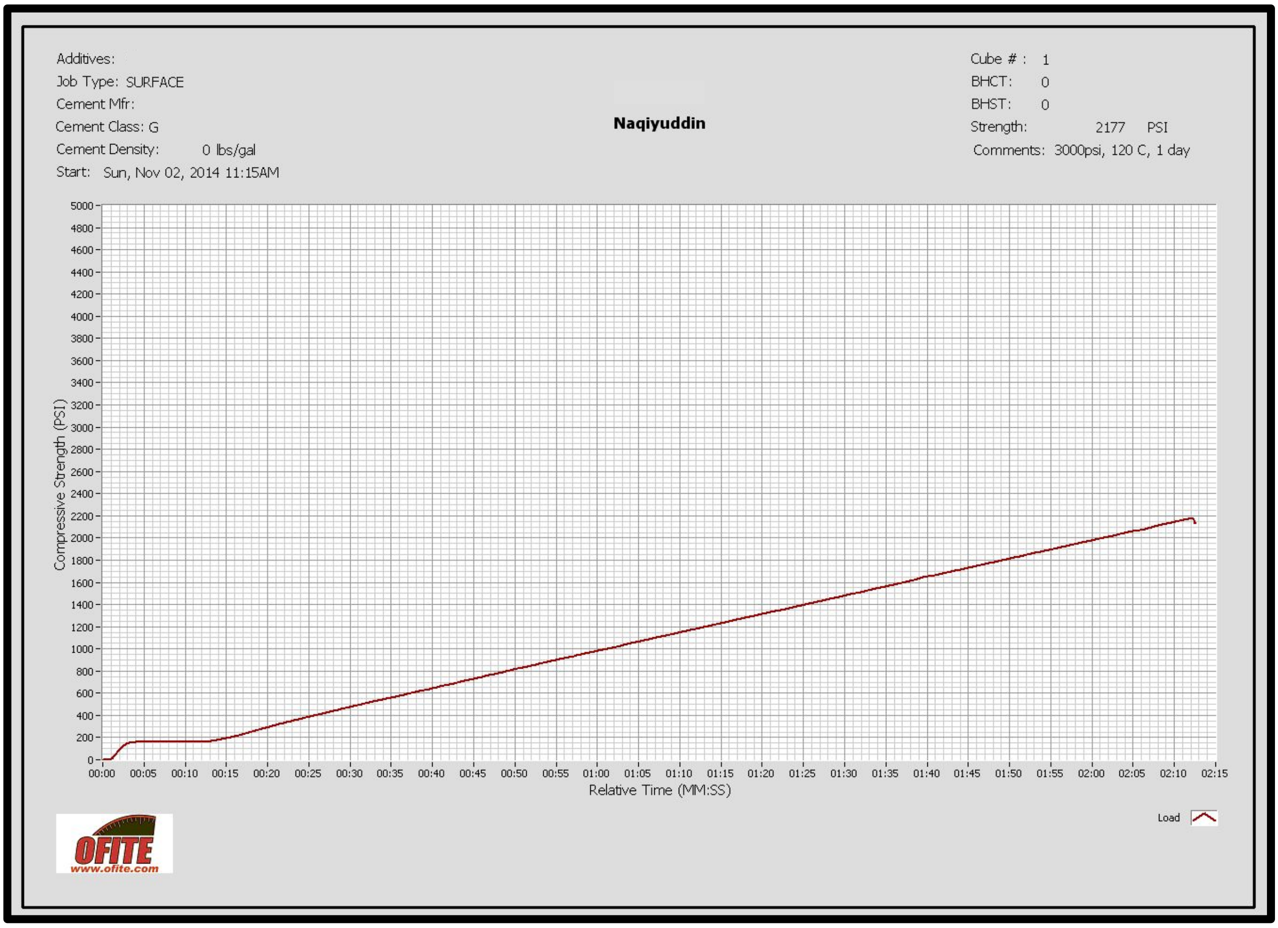

Figure 15: Compressive Strength vs Time Graph for Case 1

The addition of 0.5% nanosilica by the weight of cement into the cement mixture clearly improves the compressive strength of cement. As in the previous case, the compressive strength of the cube cannot be observed at the beginning of the test. As the pressure on the cube increase with time, the compressive strength of the cement starts to increase. The graph above further proves the linear relationship between the pressure on the cube and its compressive strength. In this case, the recorded compressive strength of the cube is 2177 psi, an increase of 30% in the cement cube's strength. Thus, the time taken for the cement cube to crack is also increased since the pure cement cube takes around 1 minute and 44 seconds to crack and this cube takes 2 minutes and 10 seconds to reach its maximum strength.

The increase in compressive strength of the cement between the base case and case 1 is purely based on the addition of nanosilica into the cement mixture as the curing time is kept constant for both cases. So it can be said that the strength development of the two cement mixture is the same during the curing of the cement.

Case 2 (99% Class G Portland cement, 1% Nanosilica)

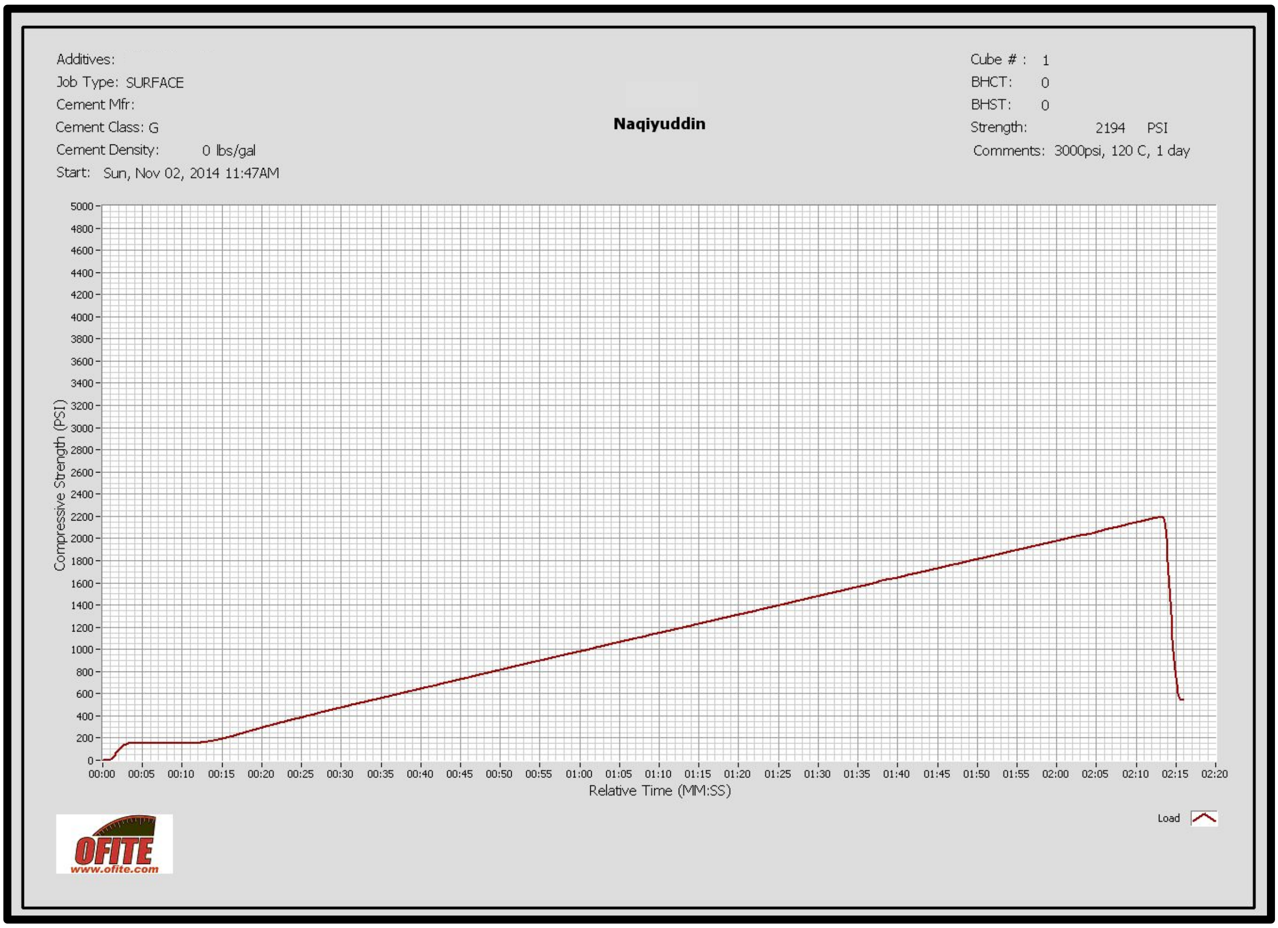

Figure 16: Compressive Strength vs Time Graph for Case 2

The concentration of nanosilica is increased for this case and since it is mentioned before that the nanosilica increases the compressive strength of the cement cube, it is expected that the compressive strength of the cement with 1% nanosilica will be high. From the graph obtained above, the expectation is achieved but the only problem is here is that the difference in compressive strength between cement with 0.5% nanosilica and cement with 1% nanosilica is considerably small. There is only about 0.7% increment in the compressive strength between case 1 cement mixture and case 2 cement mixture. Despite that, there is a noticeable upward trend in the graphs from base case until case 2. Since the cement cube has a high compressive strength value, it can also be said that the time taken for the cube to crack is longer than that of case 1 cement, which is 2 minutes and 12 seconds. The maximum compressive strength of the cement cube of case 2 is 2194 psi.

Case 3 (98% Class G Portland cement, 2% Nanosilica)

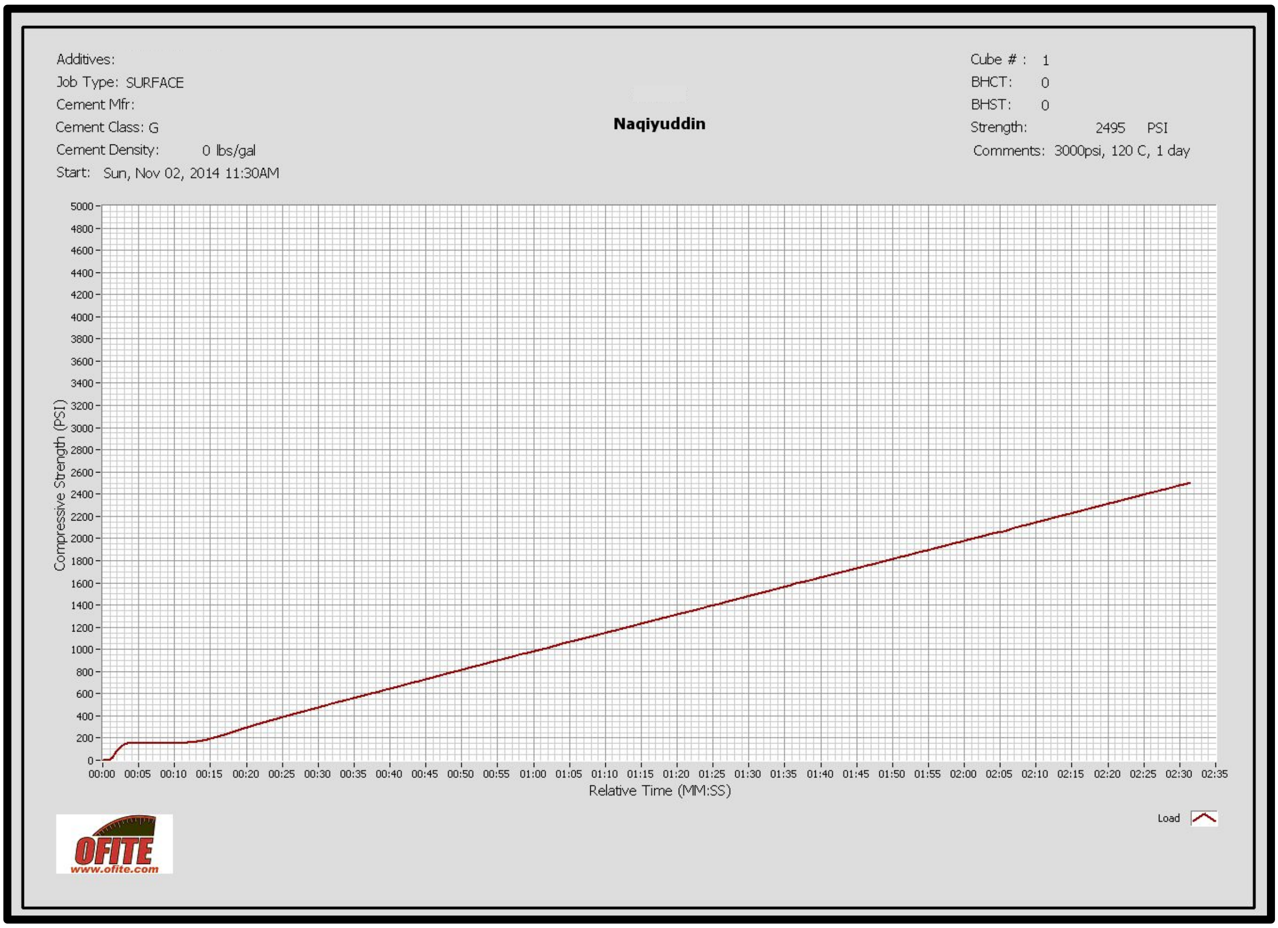

Figure 17: Compressive Strength vs Time Graph for Case 3

The highest compressive strength value is recorded for this case. This is due the highest amount of nanosilica being used for this case. The cement cube is able to withstand up to 2495 psi of pressure, which is a 13% increase in the compressive strength compared to the previous case 2. The time taken for the cement to reach its maximum compressive strength is also the longest among all the cement cubes which is around 2 minutes and 30 seconds. The upward trend continues as the linear graph for this case is slightly higher that the graph for case 2.

4.2 Compressive Strength Test Results for Curing Pressure 3000 psi and Curing Temperature 60°C

<u>Base Case (100% Class G Portland cement)</u>

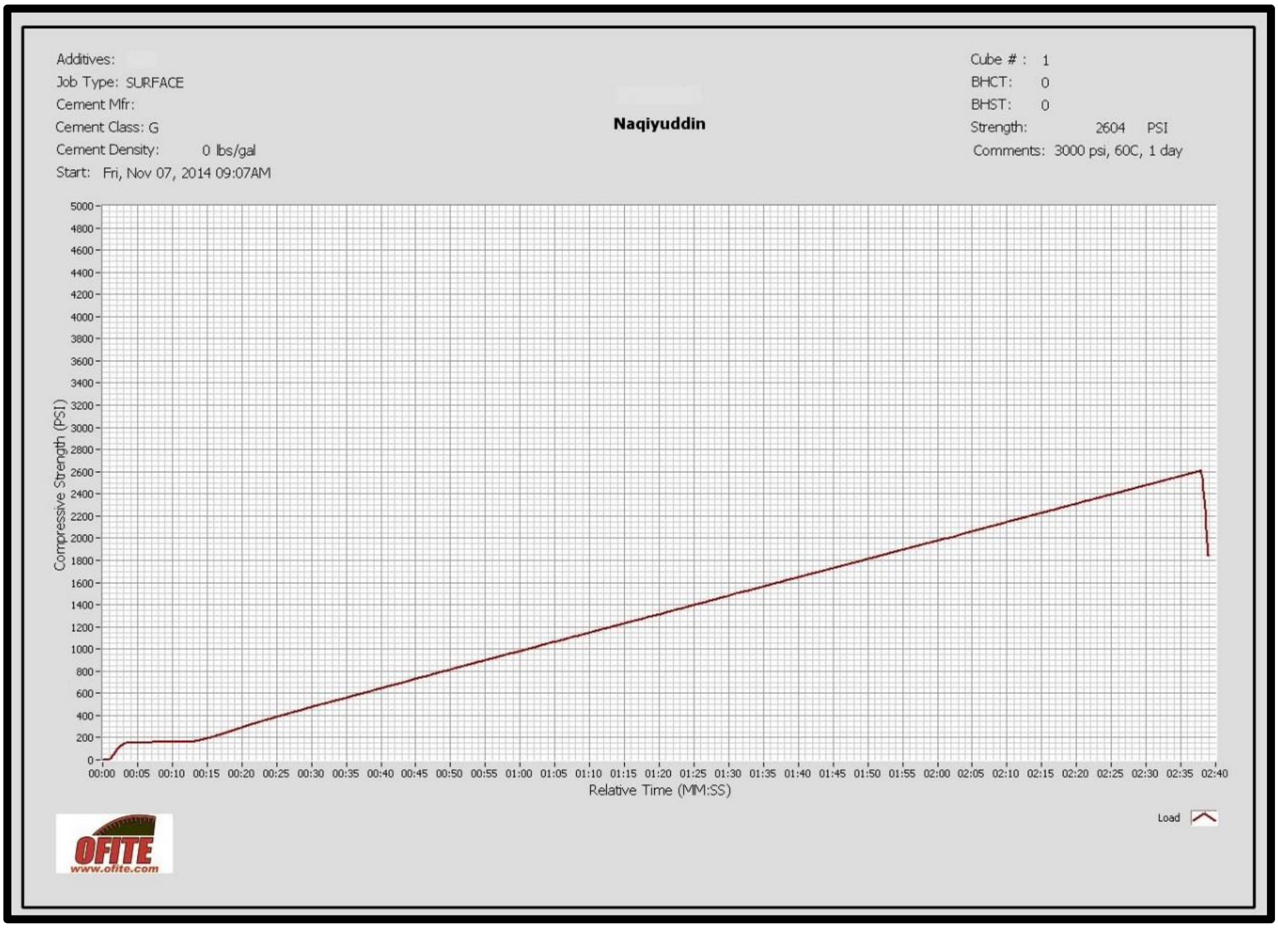

Figure 18: Compressive Strength vs Time Graph for Base Case

The above graph shows the strength profile of the cement cube with no addition of nanosilica in the cement mixture. At the beginning of the test, the cement cube is not put under any pressure thus the reading of the compressive strength showing the value of zero. With time, the pressure on the cube started to increase, and as the cube withstands the pressure, the compressive strength of the cement cube also starts to increase until it reaches the maximum value at which it can stand the applied pressure. The cube cracks when the applied pressure exceed the maximum of its compressive strength. For the pure Class G cement cube, the compressive strength is 2604 psi. The end of the graph that shows a drastic decrease in compressive strength shows that the cement cube breaks into pieces.

Case 1 (99.5% Class G Portland cement, 0.5% Nanosilica)

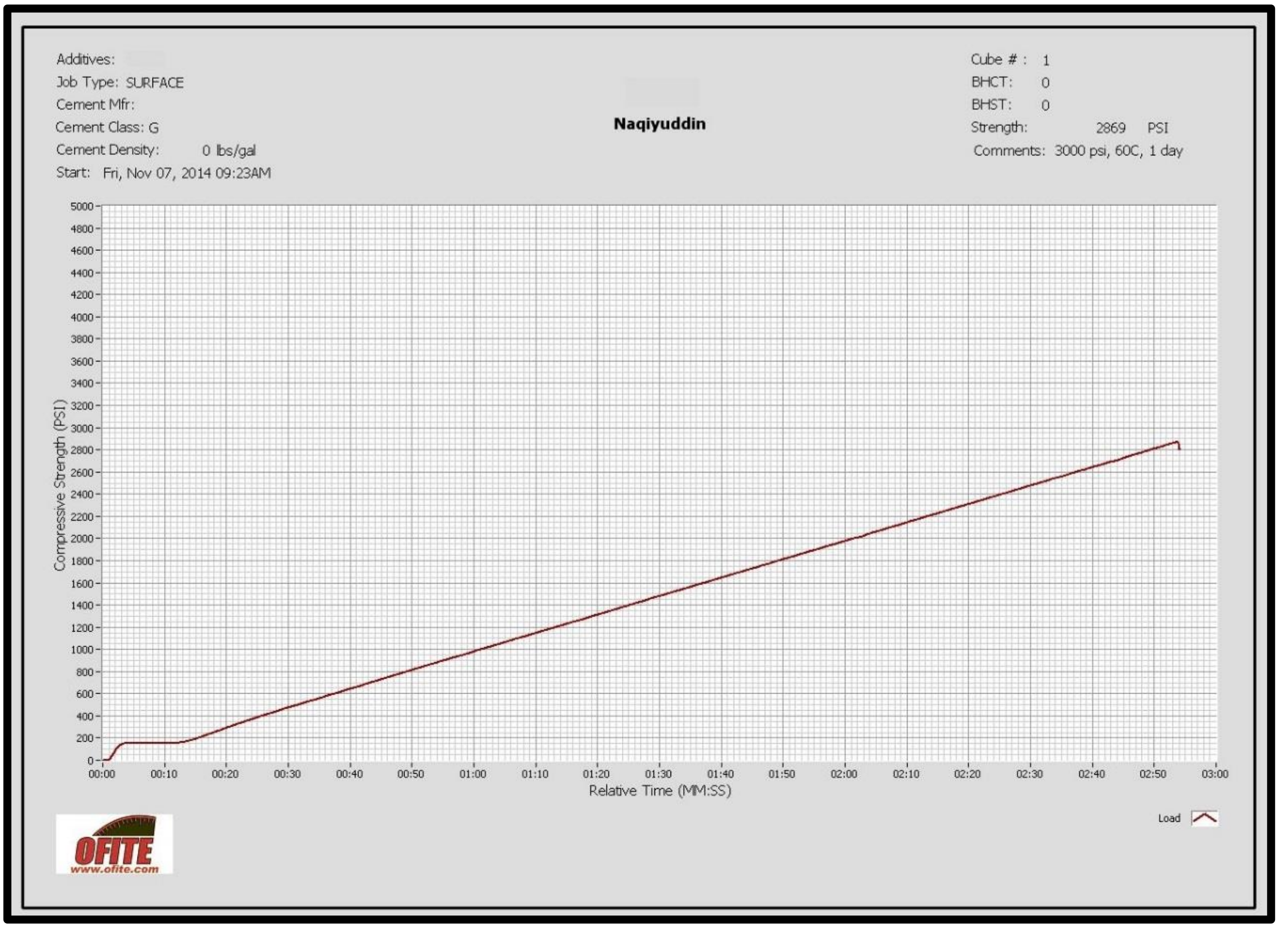

Figure 19: Compressive Strength vs Time Graph for Case 1

The addition of 0.5% nanosilica by the weight of cement into the cement mixture clearly improves the compressive strength of cement. As in the previous case, the compressive strength of the cube cannot be observed at the beginning of the test. As the pressure on the cube increase with time, the compressive strength of the cement starts to increase. The graph above further proves the linear relationship between the pressure on the cube and its compressive strength. In this case, the recorded compressive strength of the cube is 2869 psi, an increase of 10% in the cement cube's strength. Thus, the time taken for the cement cube to crack is also increased since the pure cement cube takes around 2 minutes and 37 seconds to crack and this cube takes 2 minutes and 52 seconds to reach its maximum strength.

Case 2 (99% Class G Portland cement, 1% Nanosilica)

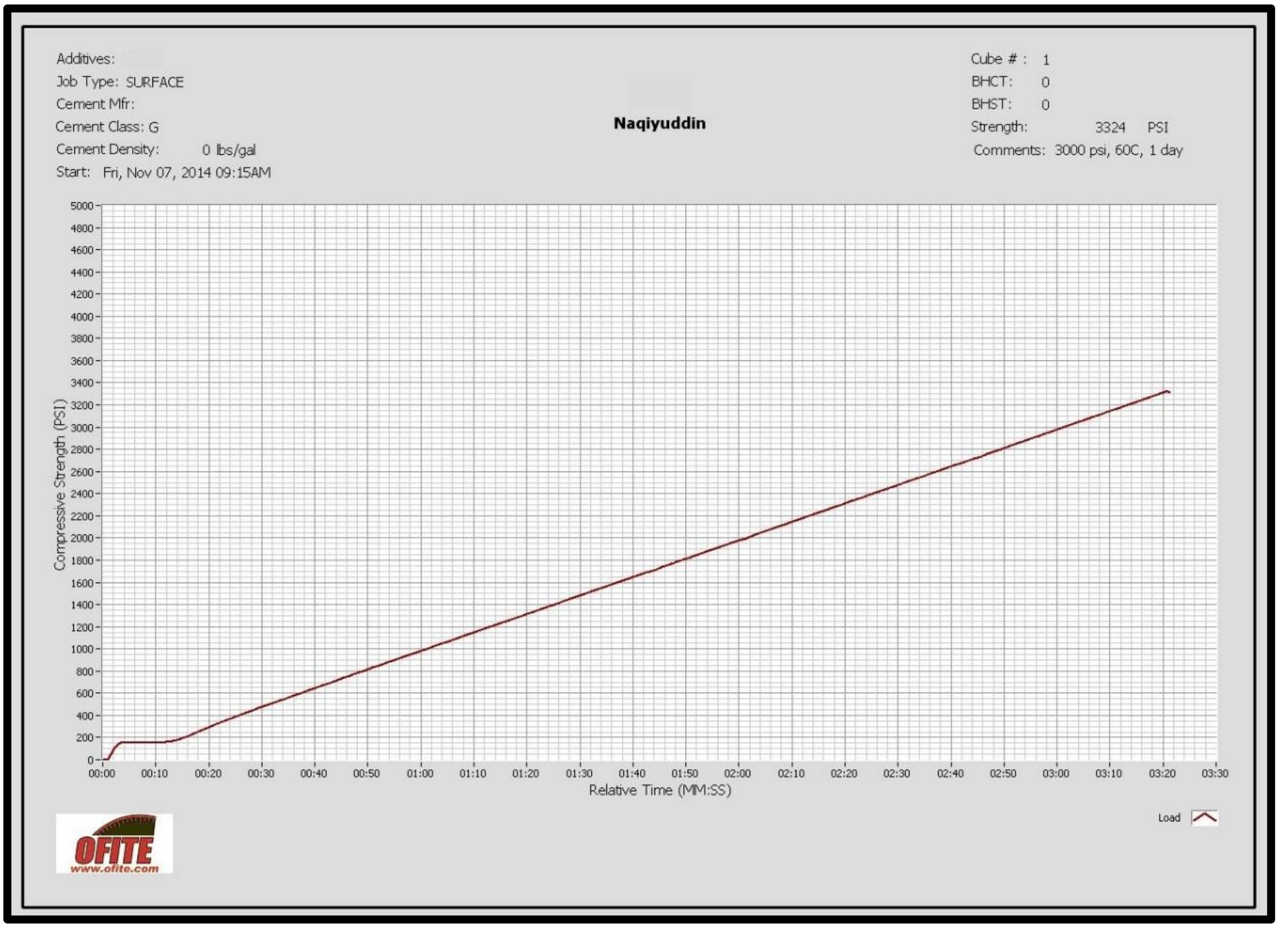

Figure 20: Compressive Strength vs Time Graph for Case 2

The concentration of nanosilica is increased for this case and since it is mentioned before that the nanosilica increases the compressive strength of the cement cube, it is expected that the compressive strength of the cement with 1% nanosilica will be high. From the graph obtained above, the expectation is achieved and the difference in compressive strength between cement with 0.5% nanosilica and cement with 1% nanosilica is around 600 psi. There is about 15.0% increment in the compressive strength between case 1 cement mixture and case 2 cement mixture. With that, there is a noticeable upward trend in the graphs from base case until case 2. Since the cement cube has a high compressive strength value, it can also be said that the time taken for the cube to crack is longer than that of case 1 cement, which is 3 minutes and 20 seconds. The maximum compressive strength of the cement cube of case 2 is 3324 psi.

Case 3 (98% Class G Portland cement, 2% Nanosilica)

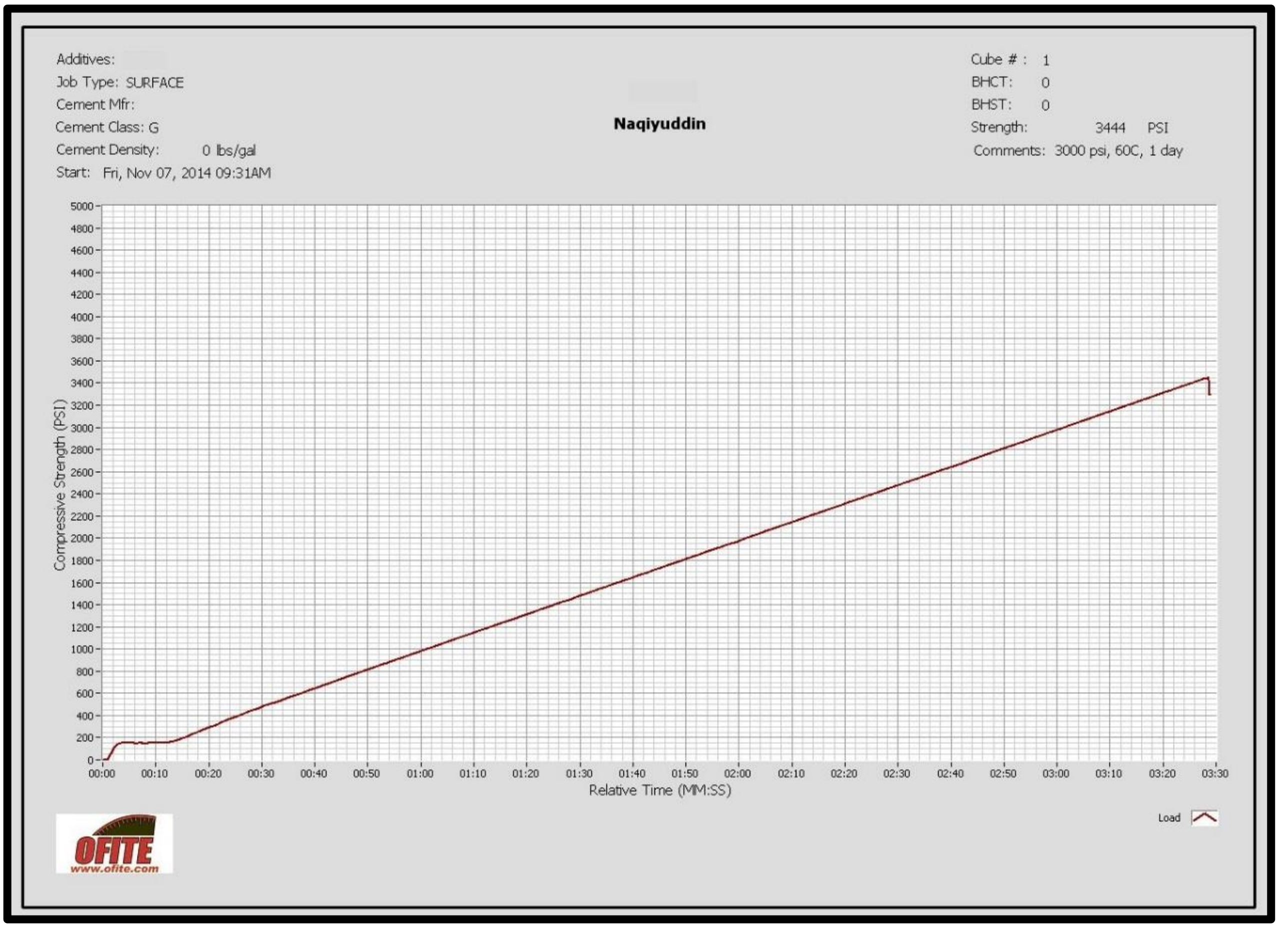

Figure 21: Compressive Strength vs Time Graph for Case 3

The highest compressive strength value is recorded for this case. This is due the highest amount of nanosilica being used for this case. The cement cube is able to withstand up to 3444 psi of pressure, which is a 3% increase in the compressive strength compared to the previous case 2. The time taken for the cement to reach its maximum compressive strength is also the longest among all the cement cubes which is around 3 minutes and 27 seconds. The upward trend continues as the linear graph for this case is slightly higher that the graph for case 2.

<u>Comparisson between Compressive Strength vs Percentage of Nanosilica Graph for Curing Condition: 3000 psi and 120°C and Curing Condition: 3000 psi and 60°C</u>

The graph below shows the trend change in the compressive strength of cement with the percentage of nanosilica used in the cement mixture for both conditions:

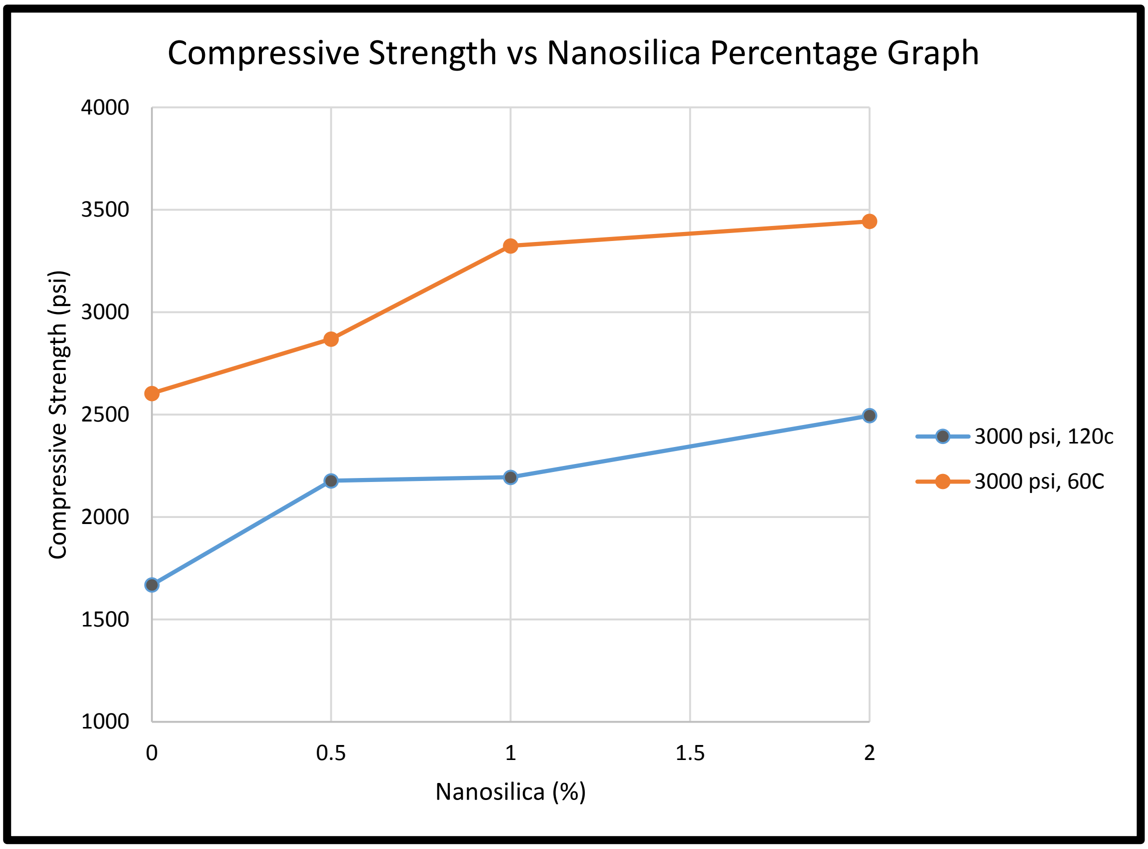

Figure 22: Compressive Strength vs Nanosilica Percentage Graph for Both Curing Conditions

Most of the researchers that study on the effect of nanosilica on cementing proposed that the addition of nanosilica into cement mixture improves the compressive strength of the cement. With these graphs, it is observed that the theory is, in a sense, true as the nanosilica particles act as filler that fill the gaps between cement particles. This further increases the stability in the C-S-H structure in the cement, explaining the "extra" strength development of the cement while curing. The presence of nanosilica in the cement particle gaps prevents the slippage of the cement particles, thus strengthening the cement structure as a whole. An increase in the percentage of nanosilica used means that there is more nanosilica particles filling in the gaps. This encourages the increment in the compressive strength of the cement. The difference in the compressive strength readings between the two conditions is due to the shrinkage effect experienced by the cement when it is cured at high temperature. Seeing that 60°C is the lower temperature for curing cement in this experiment, it is assumed that the compressive strength of the cement cubes cured at 60°C is exceptionally higher that the cement cubes cured at 120°C.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion on the project

The purpose of this study is to investigate the effect of nanosilica on the compressive strength of Class G Portland cement. From the results obtained, it can be concluded that the addition of nanosilica into cement slurries does have a positive effect. This is very important since the strength of cement plays a major role in preventing the wellbore from collapsing during the drilling of an oilwell. By using nanosilica, most of the cementing problems caused by weak cement structure can be solved. Time is also conserved when all these problems are avoided. Cement with high compressive strength is usually needed for cementing at deep areas where the pressure and temperature are very high.

In this project, the Class G Portland cement is used to prepare four types of cement slurries; one type of slurry is not mixed with nanosilica while the other three types of slurries are added with nanosilica at different weight percentage. These slurries are then cured at the pressure of 3000 psi but two different temperatures, which are 60°C and 120°C. The main objective for doing this is to observe the strength development of the cement slurries at standard reservoir temperature (60°C) and at high reservoir temperature (120°C).

The compressive strength of the cements are then tested and the changes in strength between the cubes for each cases are studied. It is found that there is an increase in strength for the cubes cured at high temperature with increasing percentage of nanosilica used. From the study of trends, it is proven that the addition of nanosilica improves the compressive strength of cement at high temperature, high pressure. As for the standard reservoir temperature, the results have the same trend as the previous case but with higher readings.

5.2 Recommendation on the project

In conducting this experiment, there are several limitations identified. First, the mixing of the nanosilica into the cement mixture is not uniform even after using the cement mixer. This affects the properties of the cement cube, creating regions with different nanosilica concentration within the cube. Therefore, when tested, the cubes give off inaccurate readings. To avoid this situation, the mixing of the cement must be done in a controlled environment and all the materials used to prepare the cement slurry must be handled carefully. Second, the removal of air bubbles from the cement slurry inside the moulds. As this is done by the author, human errors are to be expected. The remaining air bubbles in the slurry create pores inside the cement cube, thus, reducing the strength of the cement. In order to avoid this, extra time should be allocated for removing the air bubbles thoroughly and cautiously. The third limitation is the weighing of the cement materials. Human errors are bound to happen during this process as well as the precision of the electronic balance used. Therefore, make sure that the balance is calibrated correctly and conduct the process with care.

For further enhanced cement mechanical properties technology, a study on the modulus of elasticity of the cement should be carried out. The modulus of elasticity of the cement is related to the deformation of the cement and the conditions that lead to cement failure related to the elasticity of cement. This study is very important as it helps us understand the mechanics of subsurface cementing and its challenges.

APPENDICES

Appendix I: Cement, Water and Nanosilica Weight Calculations

Base Case

Weight of Cement = 500g

Weight of Water = (44/100) X 500g

= 220g

Case 1

Weight of Cement = (99.5/100) X 500g

= 497.5g

Weight of Nanosilica (0.5%) = 500g – 497.5g

= 2.5g

Weight of Water = (44/100) X 497.5g

= 218.9g

Case 2

Weight of Cement = (99/100) X 500g

= 495g

Weight of Nanosilica (1%) = 500g – 495g

= 5.0g

Weight of Water = (44/100) X 495g

= 217.8g

Case 3

Weight of Cement = (98/100) X 500g

= 490g

Weight of Nanosilica (2%) = 500g – 490g

= 10g

Weight of Water = (44/100) X 490g

= 215.6g

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