Mechanical Behavior of Fly Ash based Geopolymer Cement as Well Cement

By

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
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Approved:

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Project Supervisor

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

DECEMBER 2014

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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ANAS ZHARIF BIN AHMAD JAAFAR

ABSTRACT

With the increasing awareness of global warming, geopolymer cement has been identified as one of the methods in reducing the emission of CO2 during oil well cementing operation. However, it is important that geopolymer cement can meet the specific requirement of oil well condition in order to be the substitute of current conventional cement system. The use of geopolymer in cement system is a new technology that yet needs proper study to yield better advantages of it.

In this research, the main objective was to study the effect of temperature to the mechanical properties of the flyash based geopolymer cement. In the early stage, literature review on previous research showed utilizing geopolymer in cement composition will significantly reduce C02 emission and enhanced properties characteristic as well. Detailed study on geopolymer compositions, conventional cement, and additives was carried out. In the experimental part, the geopolymer cement were tested in well condition and changes in the mechanical properties are recorded and analyzed.

As the conclusion, from the obtained results geopolymer cement showed a degradation of mechanical properties after cured in the controlled well condition.

ACKNOWLEDGMENT

First and foremost, I would to express my deepest appreciation to God and my family for their endless support and love throughout the years. Secondly, my sincerest thanks to my Supervisor Miss Raja Rajeswary Suppiah and my co-supervisor Dr. Soony Irawan who in spite of their very demanding schedule, found time to supervise and help me throughout the Final Year Project period. The completion of my extended proposal project report would not be viable without their support, attention and advices which really helped in the progression.

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CHAPTER 1
INTRODUCTION

1.1 Background study

Cementing is arguably one of the extremely vital operations performed in the process of drilling a well. The technology of well cementing is a merge product of scientific and engineering knowledge. The technology advancement in cementing job is very necessary to ensure greater chances in isolating the targeted zones. Traditional solutions in cementing job would not always be sufficient enough to apprehend new challenges provided by the deeper formations drilled. Therefore, one of the ways in overcoming this hurdle is to create special cement with special qualities for cementation of deep wells with high temperatures.

Ordinary Portland cement (OPC) is popularly selected as the type of cement for well cementing. However, throughout the years, the OPC based cement had showed many disadvantages particularly in deeper formations. A study by Nasvi et al showed that OPC base cement losses it strength and stiffness at elevated temperatures. In a more pressing issue regarding the concern of greenhouse gas (GHG) emissions, convention Portland cement had been identified as one of the main sources of GHG producer. This was due to the production process of OPC clinker which encompasses the reaction between calcium carbonate (lime) and silicon dioxide:

$$5CaCO_3 + 2SiO_2 \rightarrow Ca_3SiO_5 + Ca_2SiO_4 + 5CO_2 \qquad (1)$$

The products of the reaction were calcium silicates and carbon dioxide. It may not be the ideal cement to be use in well cementing in the future.

Therefore, due to the many holdups to the use of OPC in the market, there have been various investigations done to replace the existing binding material with alternative sustainable cement. Hence, geopolymer was discovered. Geopolymers are mineral polymers resulting from geosynthesis or geochemistry (Davidovits 2002). Any pozzolanic compound or source material that contain silicates and aluminates, and readily dissolves in alkaline solution may undergo polymerization (Xu and Van Deventer 2000). Geopolymers have been proven to fill the many gaps left by the OPC, such as better resistance to heat, corrosion and aggressive environment, higher early strength, lower shrinkage, and much faster hardening time etc. Hence, the geopolymers have proven to be a much better option or alternative to the conventional cement material.

1.2 Problem statement

Well cement plays a vital role in well integrity for cementing jobs, and ordinary Portland cement (OPC) based well cement has been popularly used in underground wells. There are many problems, such as cement degradation, chemical attacks, durability issues, leakage, etc., associated with OPC based well cement. One of the best replacements for OPC based well cement would be the use of geopolymer cement. The geopolymer cement is still not widely use in the Oil and gas industry although it is more economical in production, consumes less energy, more sustainable and poses superior strength compared to OPC, the information on the behavior of its mechanical properties in well condition are very limited. Hence, lead to this study.

1.3 Objective

1. To study the mechanical properties of fly ash based geopolymer.
2. To compare the mechanical performance of the fly ash based geopolymer cement in normal condition and well condition.

1.4 Scope of study

1. Preparing fly ash based geopolymer cement with different ratio compositions.
2. Comparing the mechanical performance of the cements at different curing temperatures and environment.

1.5 Feasibility of the study

The period given for completion of the research project was two semesters which comprised of 28 weeks. Many things can be achieved within this period. In focus of this particular research project, the length of the experiments theoretically calculated by the author was within 5 – 6 weeks which is very feasible.

CHAPTER 2
LITERATURE REVIEW

2.1 Introduction to Fly Ash

Fly ash is the byproduct of the combustion of coal used for electricity generation. Therefore, fly ashes are produced in large quantities, estimates amounting up to 780 million tons annually (Hardjito et al. 2004). They consist of finely divided ashes produced by burning pulverized coal in power stations (Khale and Chaudhary 2007). Most of these ashes are disposed in landfills worldwide (Baldwin et al.). Agreeing with Baldwin, in excess of 75% of the waste fly ash is being dumped to surface impoundments and unwatched landfills (Cambridge, 2008). The quality of underground water bearing may also degrades from the accumulation of heavy metal substances in landfills (Khale and Chaudhary 2007). Scholar Wiles wrote that "Landfilling, is not a desirable option since it not only causes huge financial burden to the foundries, but also makes them liable for future environmental costs and problems associated with landfilling regulations". Therefore, the preservation of the environment from improper disposal of coal plant waste is critical to avoid the potentially everlasting damage it could cause. Pressing needs for alternatives exploitation of these ashes are required (Nuruddin et al. 2010). On the other hand, increasing economic factor triggers the industry to look on recycling reuse of waste material and cheaply handling these large quantities of heavy metal waste so that it could be used as an alternative to OPC seem so feasible (Khale and Chaudhary 2007).

2.2 Class of fly ash

According to American Society for Testing and Material (ASTM) C618, there are two categories of fly ash; class C and class F fly ash. Differentiating between both of these classes is by the element composition of each class. The portion of calcium, silica, alumina and iron elements gave the fly ash its class. In related, the bituminous, anthracite and lignite properties of the coal also effect the element composition of the fly ash.(Cockrell and Leonard 1970)

2.2.1 Class C Fly ash and Class F Fly ash

Both types of fly ash are pozzolonic in nature which indicate the property of self-cementing characteristics. Despite having some similar properties, class C fly ash and class F fly ash have a few distinctive differences. The first aspect is from the source they formed – class C fly ash is the product from the burning of young lignite or subbituminous coals. On the other hand, an older bituminous coal will burn into class F fly ash. The second aspect is the amount of calcium oxide (CaO) or 'lime' it has – A fraction of more than 20% of lime content would categorize the fly ash as class C and if otherwise would be classified as class F fly ash. The third aspect is the requirement of alkali activator – due to the high content of lime in class C fly ash; generally meant that they have greater content of alkali. Hence, they do not require an alkali activator. Halstead wrote that "In the presence of water, these types of fly ash will harden and gain strength over time". On the contra, the class F fly ash would require alkali activator in order to react with water and generate the production of cementatitious compounds. Despite the extra need of an alkali activator, they are normally used for geopolymerization. The addition of a chemical activator or high alkaline solution such as sodium silicate is needed to induce the silicon and aluminium atom in the fly ash for the formation of a geopolymeric paste (Halstead 1986).

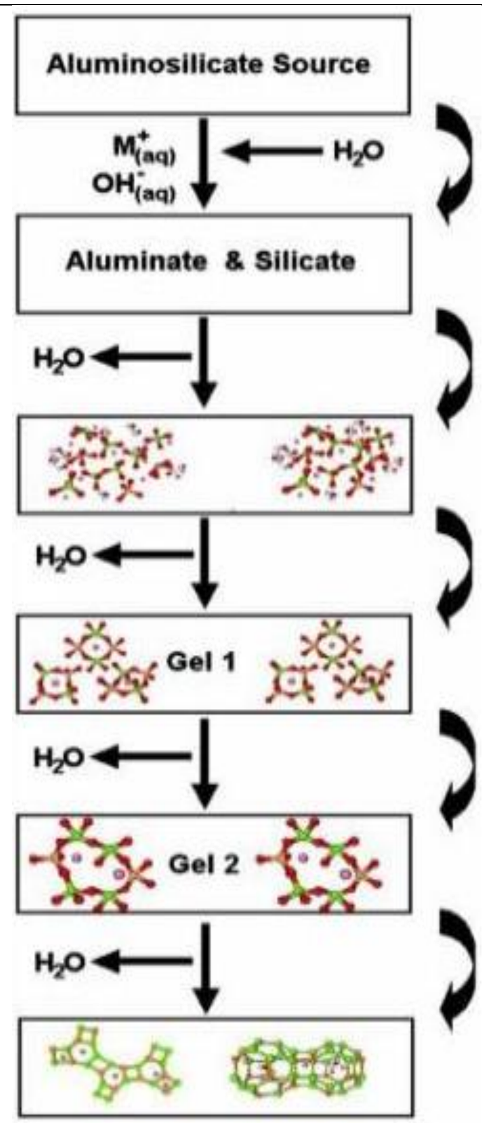
TABLE 2.1: Composition of class C and class F fly ash (Source: Singh G.,2013)

Oxide	**Class C (Wt%/std)**	**Class F (Wt%/std)**
SiO_2	17.6 ± 2.7	52.5 ± 9.6
Al_2O_3	6.2 ± 1.1	22.8 ± 5.4
Fe_2O_3	25.2 ± 2.8	7.5 ± 4.3
CaO	"/>10	<10
MgO	1.7 ± 1.2	1.3 ± 0.7
Na_2O	0.6 ± 0.6	1.0 ± 1.0
K_2O	2.9 ± 1.8	1.3 ± 0.8
SO_3	2.9 ± 1.8	0.6 ± 0.5
LOI	0.06 ± 0.06	0.11 ± 0.14
Moisture	0.33 ± 0.35	2.6 ± 2.4

2.3 Fly ash base Geopolymer

2.3.1 Alkaline Activation Process (Geopolymerization)

The definition of alkali activation can be interpreted as an instant chemical process to produce dense cemented framework from a specific partially or wholly amorphous structured material (Palomo et al. 2011). The different between an alkali activation process with Portland hydration process is that the chemical progression in the alkali activation process mimics the one in tectosilicate zeolite synthesis. The blend of sodium hydroxide (NaOH) and sodium silicate are among the most popularly used alkaline activator solution (Rangan, 2008). The overall process of geopolymerization of fly ash could be summaries in four steps below (Xu et al. 2001).

<table>
<tr><td rowspan="4">

Source: www.engineeringcivil.com</td>
<td>1. The dissolution of aluminosilicate in alkaline environment occurs first. When aluminosilicate minerals are subjected to a high pH environment, the bonds between interlinked silicate and aluminate tetrahedral are broken.</td></tr>
<tr><td>2. The dissolved aluminium and silicon complexes diffuse from the solid aluminosilicate surface to the interparticle space.</td></tr>
<tr><td>3. The resulting reaction formed a gel phase.</td></tr>
<tr><td>4. The gel phase hardens due to the exclusion of spare water to form a Geopolymer product/ cement.</td></tr>
</table>

FIGURE 2.1: Process of geopolymerization

2.3.2 Mixing process

(Rattanasak et al. 2009) suggested dual mixing methods – separate mixing and normal mixing- to mixed fly ash with NaOH solution to prepare a geopolymer paste.

TABLE 2.2: Type of mixing process

Separate mixing	As the method suggested, the separate mixing actually meant an initial 10 minutes mixing of the fly ash with the sodium hydroxide (NaOH) solution followed by the addition of sodium silicate solution.
Normal mixing	All of the ingredients are mixed as one at the same time.

The best mixing order recommended by a few scholars were to firstly mix the solids together which included the aggregates and the fly ash. Secondly, before put into molds, an alkaline activator was added to the solid mixed (Swanepol and Strydom 2002). Finally, to ensure great compaction, recommended by (Kong and Sanjayan 2010) to create 3 layers of equal weights and apply the use of a rod or vibrating table for best compaction.

However, there are also scholars that favor the method of separate mixing. As standard in the separate mixing method, an initial 10 minutes mixing were required to ensure enough time for the ions to dissolve in the alkali solution (Rattanasak and Chindaprasirt 2009). Later the silicate solution were added and the mixed briefly about 1 minute because the mixture were fairly fluid. Finally, the same final procedure as in the earlier method was executed.

2.3.3 Curing Process

Curing temperature is an important factor need to be considered for best cementing result. From the study by (Chanh et al. 2008) on geopolymer cement found that the curing temperature is inversely proportional to the setting time of the concrete. The reason behind the relation was because the temperature speeds up the process of geopolymerization in the concrete. Another study from (Kong and Sanjayan 2008) found that by rising the curing temperature, geopolymer cement could gain up to 70% of its strength in a span of 3 to 4 hours. Largely, heat-curing (steam curing or dry curing) is recommended for flyash-based polymers. In comparison of compressive strength, it was found that dry-cured geopolymer cement has an additional 15% higher compressive strength as to compare to the one steam-cured (Rangan et al. 2004). In another study by (Nuruddin et al. 2011), he found that exposed curing give better result of compressive strength then other two methods he tested which was ambient curing and hot gunny curing. As a conclusion, the curing temperature and curing time both can affect the compressive strength of geopolymer cement.

Temperature wise, a higher curing temperature will results in a higher compressive strength. However, a curing temperature beyond 60ºC does not increase compressive strength (Rangan 2008). His research found that the optimum curing temperature of 60ºC gives the highest compressive strength. Moreover, according to (Swanepol and Strydom 2002) the 60ºC curing temperature was also recommended for manufacturing kaolinite and fly ash geopolymer.

2.3.4 Compressive strength

In all concrete, the compressive strength or the maximum stress it can sustain is a very vital property to ensure the effectiveness of the concrete. However, this important property is affected by a few other factors.

According to (Chanh et al. 2008), compressive strength of approximately 400 to 500kg/cm^2 can be obtain from a combination of curing temperatures from 60ºC to 90ºC within a curing time ranging from 24 to 72 hours. This shows that both curing temperature and curing temperature affects the compressive strength greatly. When the curing time and temperature increase, the compressive strength also increases. In addition, the compressive strength of geopolymer also is influenced by the content of fine particles of fly ash (smaller than 43mm). The more fine the fly ash, the greater the compressive strength it will gain.

Another factor significantly controlling the compressive strength property is the pH value. According to (Khale and Chaudhary 2007) the setting time of geopolymer concrete is inversely proportional to the pH value. The effects of pH value to the geopolymer paste were seen by the (Phair and Deventer 2001) in their study. The viscosity of the geopolymer mix is proportional to the pH value. Other than investigating the viscosity they also study the effect of pH to the compressive strength of the geopolymer cement. The outcome of the study showed that the strength tested at pH 14 was 5 times stronger than the ones tested at pH 12 (less than 10 MPa at pH 12, 50 MPa at pH 14). The reason behind the result was later deduced by the increase of monomer concentrations of oligomers and monomeric silicate for reaction with soluble aluminum. Hence, with lower pH-value of the solution leads to lower monomer concentration. Figure 2.2 display the concentration of alkali ions leach against the pH value of each single alkali solutions. It clearly showed that the range between 13 – 14 pH values gives the highest concentration of monomers which directly relate to produce higher compressive strength geopolymers.

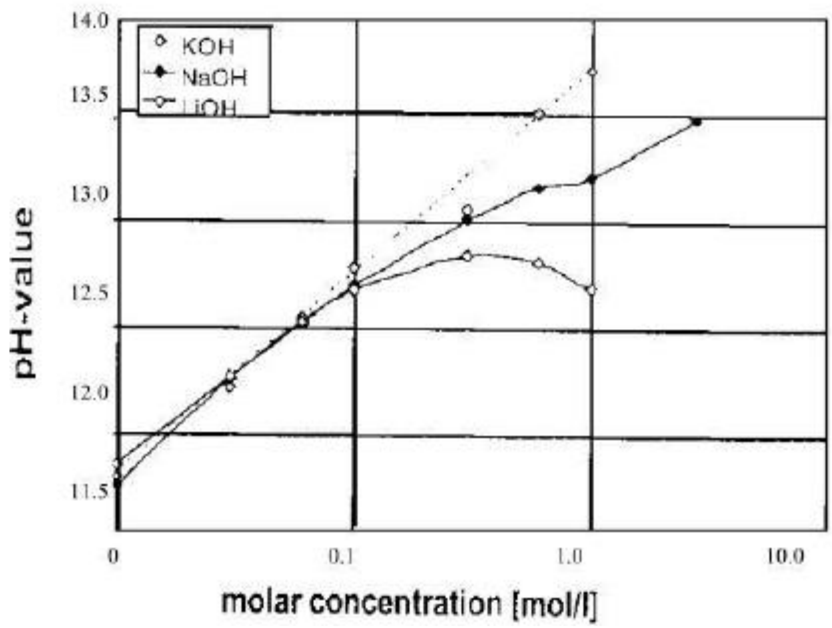


FIGURE 2.2: Influence of alkaline concentration on the pH value (Courtesy of Khale and Chaudhary 2007)

2.4 Advantages of fly ash based polymer

To understand the advantages of geopolymer, one need to analyst the impact of such technology verses its requirements. Hence, Geopolymerization utilizes by-product of coal or specifically it utilizes fly ash which can be hazardous if remained as waste product. In addition, the manufacturing process of geopolymer requires a moderate amount of energy with small carbon foot print. The impact of utilizing fly ash, moderate energy for production and less carbon dioxide emission gives the impression of this technology as a 'green' technology. Therefore, these qualities made this technology more favorable and a feasible alternative to Portland cement (Khale and Chaudhary 2007).

2.4.1 Less Carbon Footprint

In the production of OPC, one won't be able to escape from the huge CO_2 emissions. According to (Song 2007) the production of one tonne of Portland cement directly produces 0.55 tonnes of CO_2 and due to the combustion of carbon-fuel, an additional 0.40 tonnes of CO_2. In simple mathematics, 1 tonne of Portland cement will roughly produce 1 tonne of CO2 (Davidovits 1994).

Due to the increasing awareness of the current society related to resource use, biodiversity preservation and global climate change, the traditional portland cement manufacture is becoming less acceptable. Study from (Gartner 2004) has identified geopolymer as one alternative of potential low-CO2 cementing system. Furthermore, taking consideration of the additional CO2 emissions similar to the making of ordinary "bottle" glass that came from the manufacturing of the chemical activator (i.e. alkali silicate) used in mixing geopolymer concrete, the emission would be still very low. In addition, geopolymer also make use of industrial by-product such as fly ash which produce no supplementary CO_2 emission. According to (Spannagle, 2002) by implementing geopolymer concrete for buildings will cut the greenhouse gas emissions by a significant rate because from what was claimed by (Gartner, 2004), pure OPC emits relatively 10 times more CO2 gas per unit volume of concrete more than geopolymer cements. Hence, it was agree by both of the authors that geopolymer cement may have a bright future as a sustainable building material.

2.4.2 High Resistance to Harsh Environment

A good example case of this quality would be the Pyramids in Egypt remaining unaffected to present day displaying extreme durability. According to (Davidovits 1987), the long-term durability in those ancient structures lies in the silico-aluminosilicate structure they have. However, in reality many concrete structures in urban and coastal environment start to deteriorate in 20 to 30 years, though their design life is at least 50 years (Mehta 1997). Therefore, concrete durability is becoming critical issues for the future of concrete structures. Many studies has shown fly ash-based geopolymer had greater durability to harsh environment, hence it has a huge potential to be implemented as the future building blocks for marine environment structures (Chanh et al.2008).

One of the most common reasons of concrete structures deterioration is the exposure to acid rain. However, in a comparison study by (Sathia et al. 2008) testing similar size geopolymer and OPC block with the objective to investigate the effect of weight loss from submerging them into a sulfuric acid solution, the geopolymer cement block only loss 0.5% of its original weight as compared to a whopping 3% weight loss by the OPC in similar solution. In another study by Bakharev, the deterioration of high-performance and low-performance geopolymer defers from one another. Bakharev wrote "in acidic exposure, high-performance geopolymer materials deteriorate with the formation of fissures in an amorphous polymer matrix however low performance geopolymers deteriorate through the crystallization of zeolites and the formation of fragile grainy structures". The stability of the geopolymer is also important and was determine by the formation of aluminosilicate gel.

According to a study by (Thokchom *et al*. 2009), comparing the stability of crystalline geopolymer (class C fly ash based geopolymer) with an amorphous geopolymer (class F fly ash based geopolymer) in a harsh environment of sulfuric and acetic acid, it was found that the crystalline geopolymer more stable than the other. Among the specimen tested, he encounter some specimen that did not present any distinguishable color change after exposing a sample of geopolymer mortar to 10% sulfuric acid for 18 weeks. However, when witnessed under an optical microscope, micro corroded structures on the exposed surface revealed that progressed with exposure over time. Although after the 18th week the specimen had entirely dealkanized, but significant compressive strength was still observed when tested proving the property of high resistance towards harsh acid environment. The study also concluded that the weight loss is proportional with alkali content however comparing the result with normal OPC, the result based on the geopolymers still display better performance.

2.4.3 Summary of the Advantages of Geopolymer

Wide advantages of the applications of geopolymers could be listed as follows:

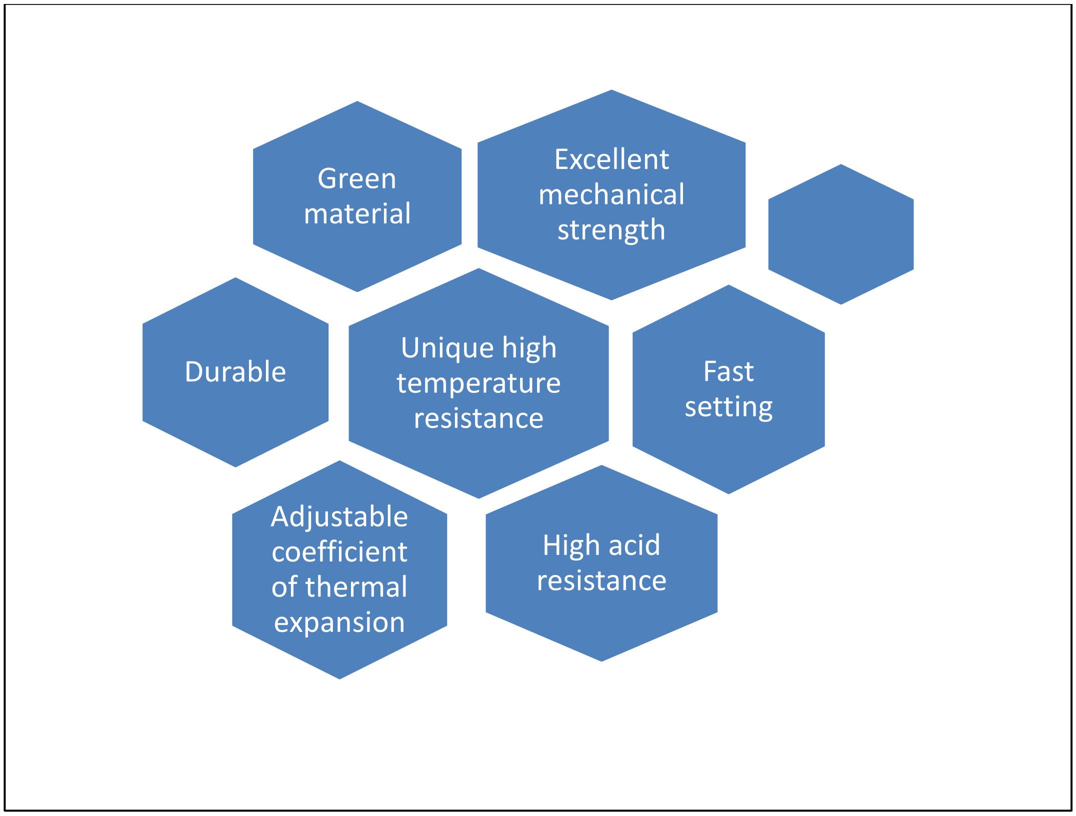


FIGURE 2.3: Advantages of geopolymer

CHAPTER 3

METHODOLOGY

3.1 Breakthrough of the research methodology

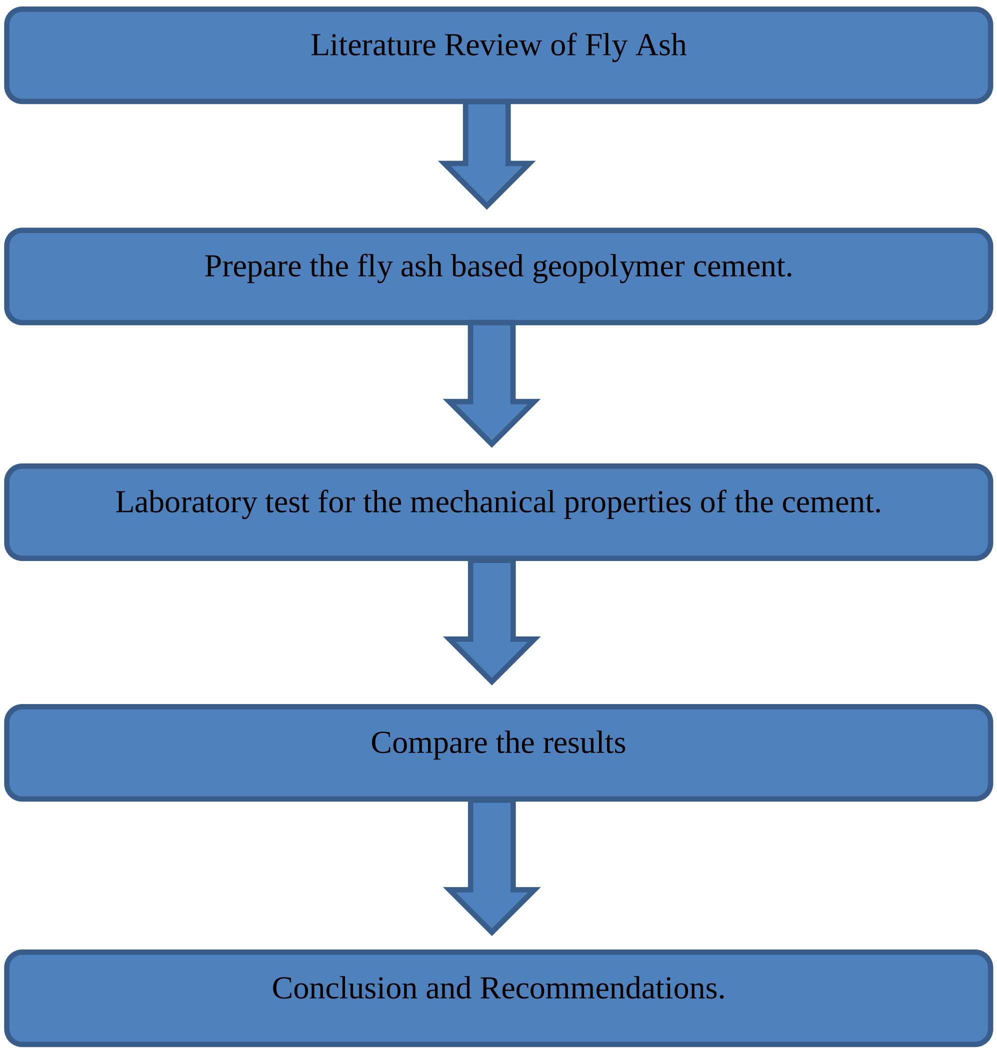


FIGURE 3.1: Flow Chart of the research

3.2 Experiment and Testing Plan

The experiment would be done with 3 batches of cement slurries. Each batch has different percentage fly ash composition of 20%, 50% and 80%. All of the cement slurry samples will be made based on American Petroleum Institute API-10B-2 procedure by using Constant Speed Mixer. The compositions of the cement slurries are define in the table below.

TABLE 3.1: Cement Slurry composition

Samples	**Cement (792 g)**		**Mix Solution (349 g)**		
	Class G	*Class F Fly Ash*	*NaOH (12M)*	*Na_2SiO_3*	*Water*
A	80%	20%	28.6 g	70.4 g	250g
B	50%	50%	28.6 g	70.4 g	250g
C	20%	80%	28.6 g	70.4 g	250g

Based on 44% water to cement ratio, the amount of cement for each type will be 792 g and for the mixed solution should be 349 g. The ready cement slurry will be poured into the cubic moulds with 2 inch sides and then the slurry will be cured for 24 hours in the baking oven. The cement would then be tested for compressive strength immediately. The hydration time for 24 hours taken account the minimum time for the cement to develop the minimum compressive strength needed (500 psi) . The 500 psi of compressive strength is normally the minimum requirement for a well cement to hold the pressure inside the wellbore.

These slurries would be later cured in 50^oC, 60^oC and 70^oC for 24 hours. The best sample will then be leave to dehydrate further in well condition. The well condition was simulated by immersing the cured cement sample in an aging cell – pressurized to

100psi and heated (rolled in the oven) at the same curing temperature as initially cured in the oven.

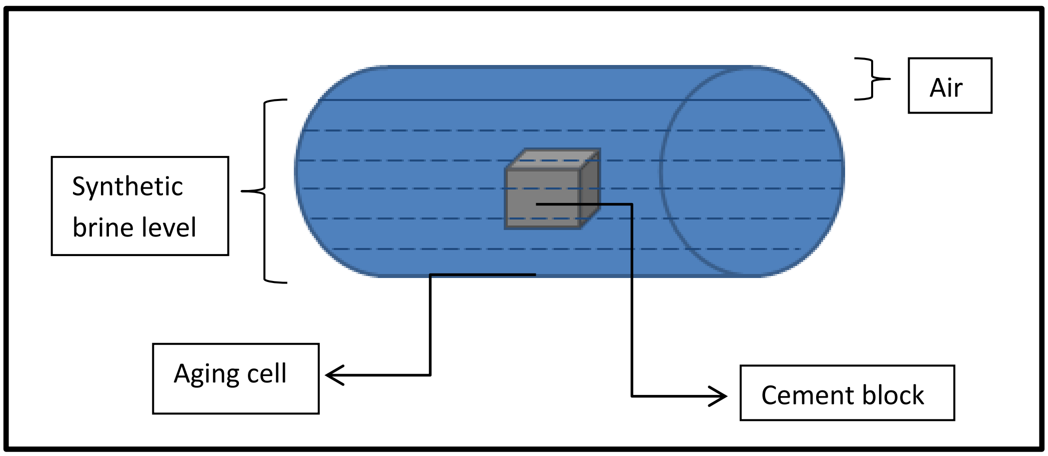


FIGURE 3.2: Well condition curing

The figure above illustrates the well condition curing. The sample is planned to be cured for 5 days. However, since it was cured for 24 hours in the oven, the remaining 4 days would be cure in this well condition. The samples will later be tested for compressive strength and uniaxial compressive strength.

3.3 Gantt chart and key milestones

3.3.1 Gantt chart

TABLE 3.2: Proposed Gantt chart for the project implementation for FYP I

Details/Weeks	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**
Selection of project														
Preliminary research Work														
Submission of Extended Proposal														
Proposal Defense														
Project work continues														
Submission of interim Draft														
Submission of interim Report														

TABLE 3.3: Proposed Gantt chart for the project implementation for FYP II

Details/Weeks	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**
Conduct experiment														
Submission of progress report														
Result analysis & Discussion														
Submission of draft report														
Submission of final report														
Oral presentation														
Submission of project dissertation hardbound														

Deliverables

Progress

3.3.2 Key milestones

TABLE 3.4: Proposed Key milestone for the project implementation for FYP I

Year	**2014**				
Activities	**FYP 1**				
	M	**J**	**J**	**A**	**S**
Project planning and literature review.					
Studies on geopolymer material.					
Studies on the factors affecting the mechanical properties of the cement.					
Study on designing the geopolymer composition.					

TABLE 3.5: Proposed Key milestone for the project implementation for FYP II

Year	**2014**		
Activities	**FYP 1**		
	O	**N**	**D**
Carry the experiment procedures, lab work, testing works			
Result analysis and discussion. Comparison study with conventional cement.			
Documentation work of the report			
Presentation and oral presentation preparation			

3.4 List of Tools, equipment and materials used

Here are some of the chemicals and equipment that will be used for this project.

Chemicals/Materials:

i. F Type Fly ash
ii. Sodium Hydroxide
iii. Sodium Silicate
iv. Distilled water
v. Class G cement
vi. Synthetic brine

Tools/Equipment:

i. Beakers
ii. Aging cell
iii. Magnetic Stirrer
iv. Measuring Cylinders
v. Brush
vi. Oven
vii. Compressive strength machine
viii. Vicat needle equipment
ix. Mixer machine
x. 50mm*50mm*50mm mold

However, there might be other materials or equipment will be added along the accomplishment of this project.

3.5 The Experiments and lab works

3.5.1 <u>12M of sodium hydroxide (NaOH) solution was prepared as the activation solution for the class F flyash. Sodium Silicate (Na_2SiO_3) solution are already prepared.</u>

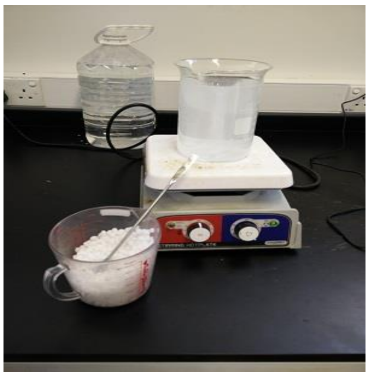


FIGURE 3.3: The 12M NaOH solution was prepared using NaOH pallets.

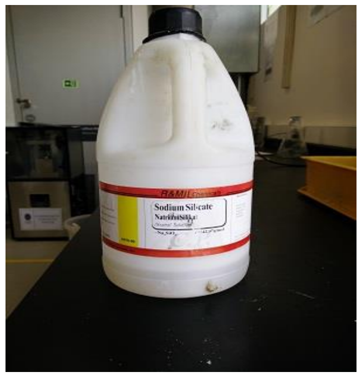


FIGURE 3.4: The Sodium Silicate solution used.

FIGURE 3.5: The Brine solution used of pH 8.

3.5.2 <u>The preparation process of geopolymer cement</u>

FIGURE 3.6: The Constant speed mixer used to mix the cement.

FIGURE 3.7: The 3 x $50mm^3$ molds used.

FIGURE 3.8: The oven cured geopolymer cement samples.

3.5.3 The coring process

FIGURE 3.9: The coring process to produce cylindrical- shaped geopolymer cement

3.5.4 Geopolymer samples before undergone the mechanical tests

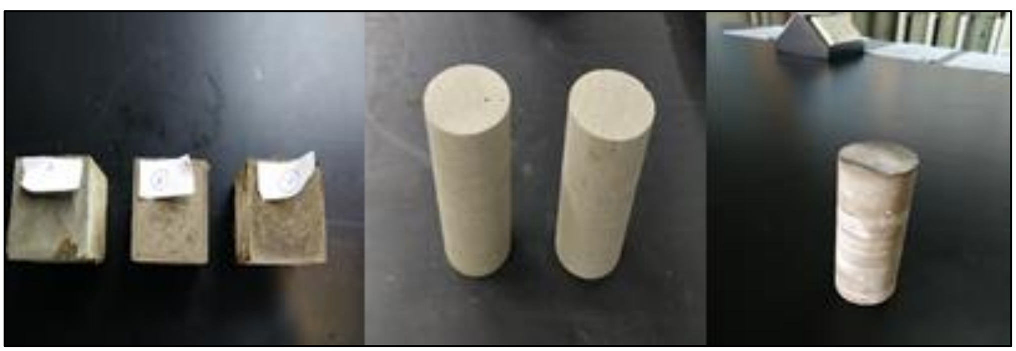


FIGURE 3.10: The oven cured samples and a cylindrical well-condition cured sample

3.5.5 Compressive strength testing

1. Place the cured cube sample in the compressive digital testing machine. Make sure the adjustable surface above the sample is evenly touched and adjust the nut tightly.

2. Switch on the pump by pressing the ‘pump on’ button on the equipment software.

3. Apply load uniformly until the sample fails. This is done by pressing the ‘Start testing’ button on the software. Do not release the mouse press until the cube fails. The results are recorded automatically on the software.

4. Repeat the steps for the other samples.

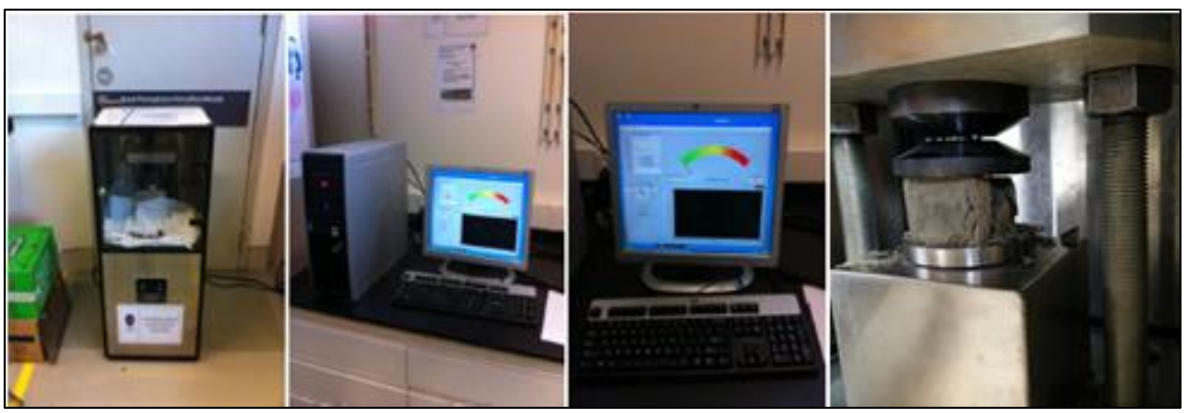


FIGURE 3.11: The compressive strength test machine and a sample crushed.

3.5.6 <u>Uniaxial strength testing</u>

1. Place the cured cylinder-shaped sample in the point load test machine. Make sure the adjustable conical metal surface above the sample is correctly placed in the top center of the cylindrical sample. Before adjusting the nut tightly, the bottom part of the cylindrical sample was also adjusted as accurately centered as possible opposing the top conical metal.

2. The digital compressive strength calculator is reset to zero. The pump is manually connected to a pumping handle hence turn the handle slowly to slowly apply the load on the sample.

3. Apply the load uniformly until the sample fails. Then, record the value of the uniaxial compressive strength.

4. Repeat the steps for the other samples.

FIGURE 3.12: The point load test machine to calculate the uniaxial compressive strength and a sample tested.

CHAPTER 4
RESULTS & DISCUSSION

4.1 Data Gathering

The data gathered are from the experiment labs in Block 14 and Block 15 by using equipment mentioned in the previous sections. The mechanical properties of the geopolymer cement are tested based on the different curing temperature and the harsh environment simulated. It is found that the temperature has an unpredictable and profound effect on the mechanical property of the flyash based geopolymer cement. On the other hand, the different composition of class F fly ash used to make the cement also influences its mechanical properties.

4.2 Initial Compressive Strength Test

The 3 batches of each sample A, B and C are cured in the oven for 24 hour in 3 different curing temperatures – 50^{o}C, 60^{o}C and 70^{o}C. The objective of testing the cement samples before further curing them in well condition is to identify the best geopolymer composition that produce highest compressive strength immediately after cured in the oven. This was done to save time and concentrate on the best possible batch of geopolymer cement that could be worth studied further on.

The results of compressive strength test are discussed in the following section.

Sample A cured for 24hours at 50°C

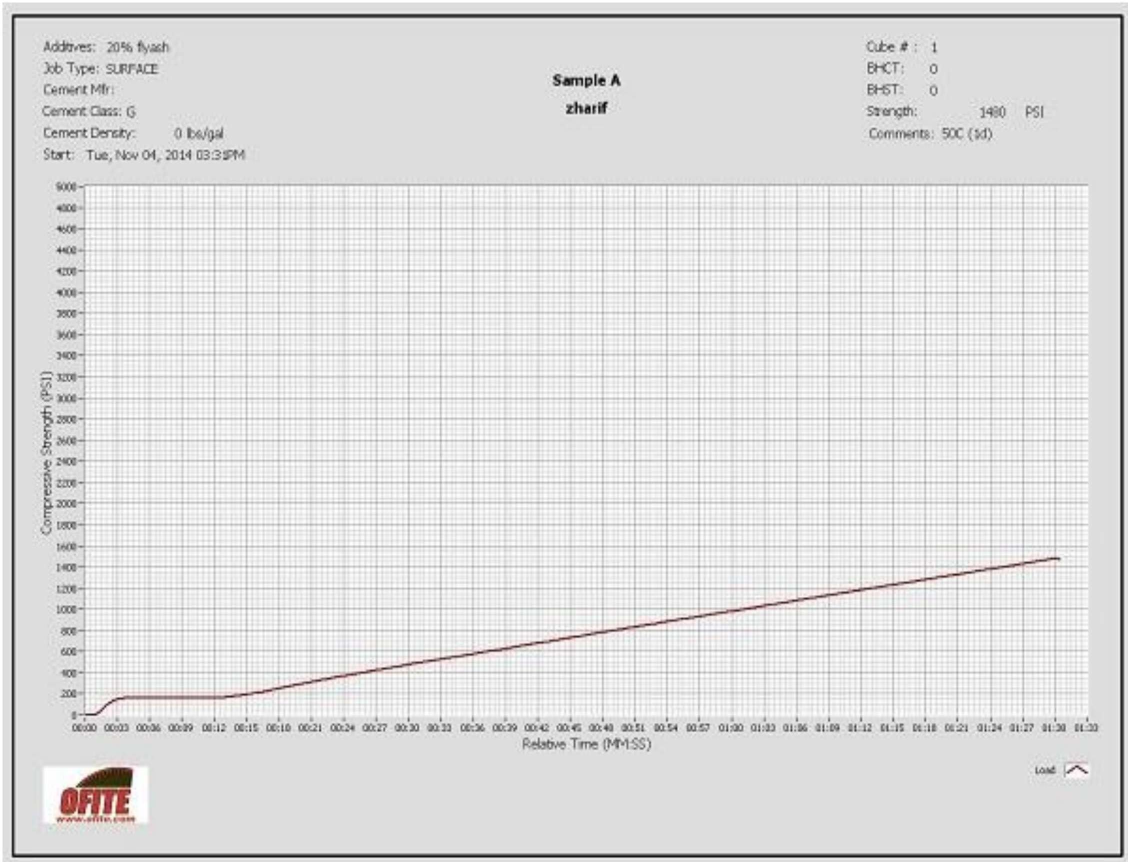


FIGURE 4.1 shows that the compressive strength of Sample A at 50°C curing temperature is **1480 psi.**

Sample B cured for 24hours at 50°C

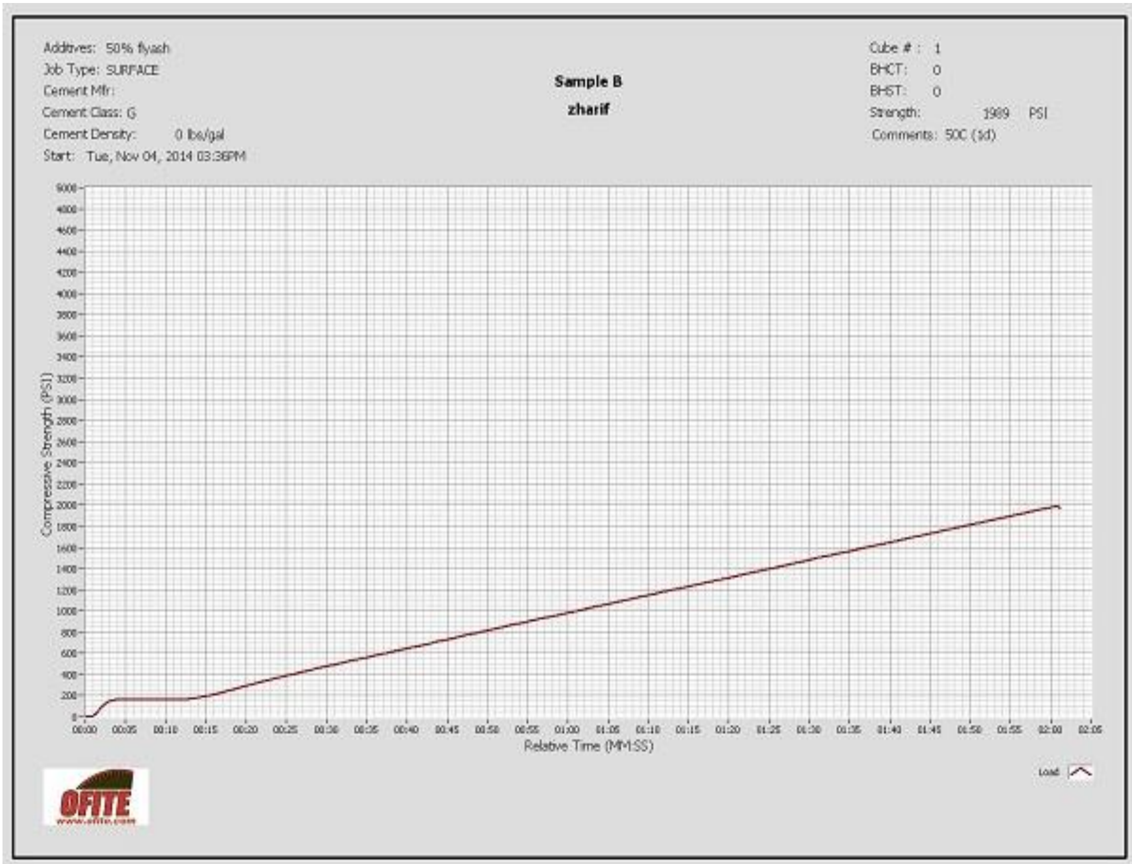


FIGURE 4.2 shows that the compressive strength of Sample B at 50°C curing temperature is **1989 psi.**

Sample C cured for 24hours at 50^{o}C

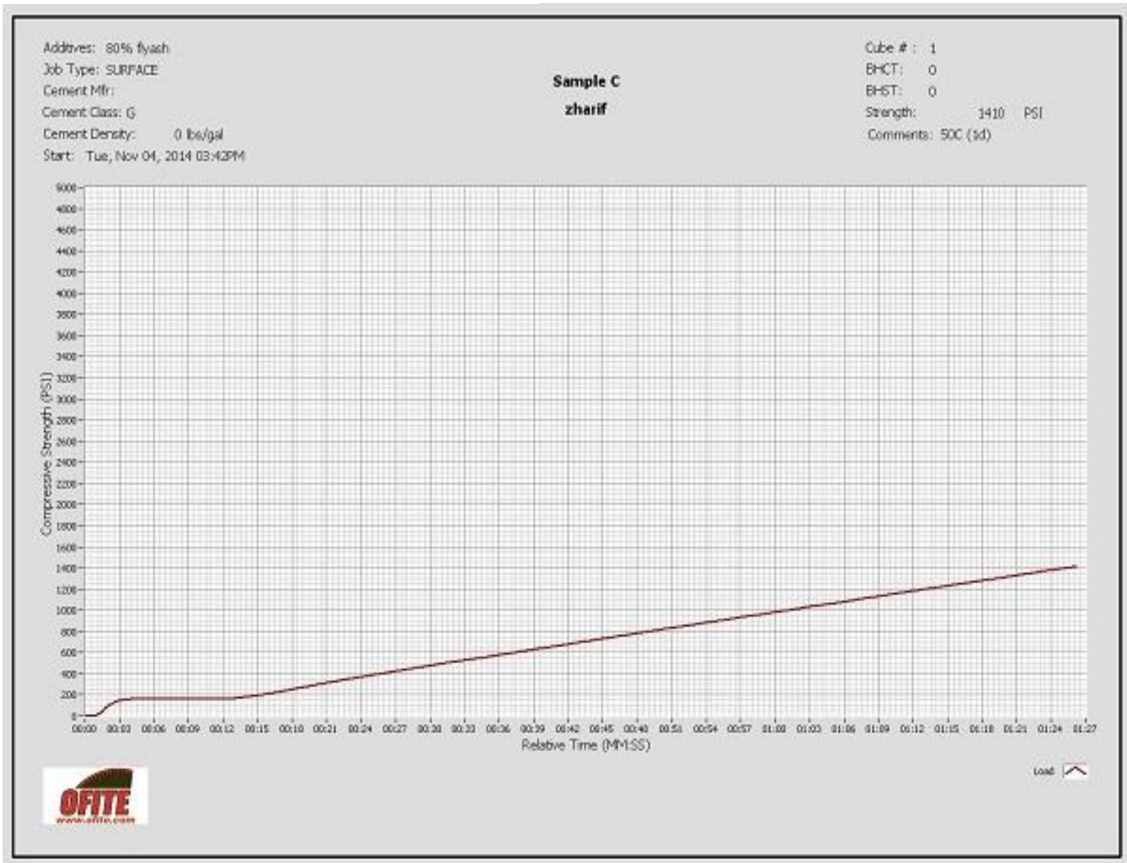


FIGURE 4.3 shows that the compressive strength of Sample C at 50^{o}C curing temperature is **1410 psi.**

Sample A cured for 24hours at 60°C

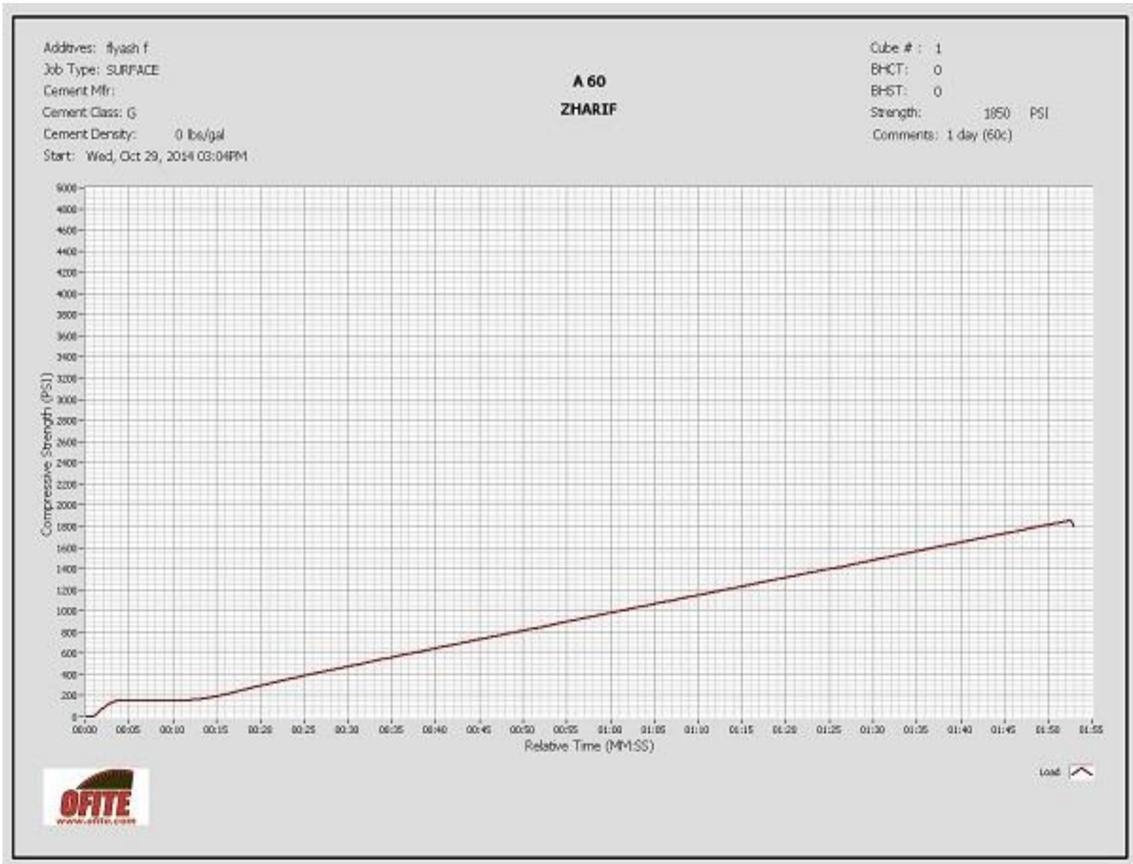


FIGURE 4.4 shows that the compressive strength of Sample A at 60°C curing temperature is **1850 psi.**

Sample B cured for 24hours at 60°C

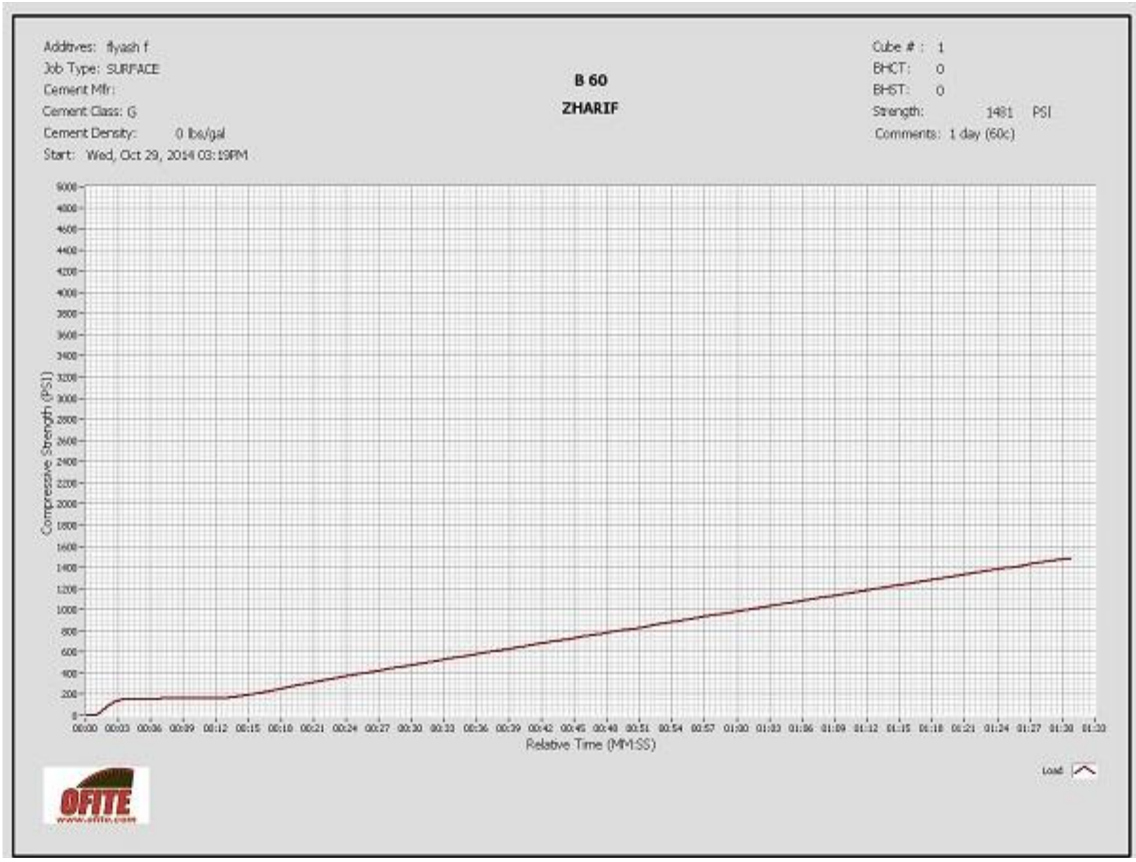


FIGURE 4.5 shows that the compressive strength of Sample B at 60°C curing temperature is **1481 psi.**

<u>Sample C cured for 24hours at 60^{o}C</u>

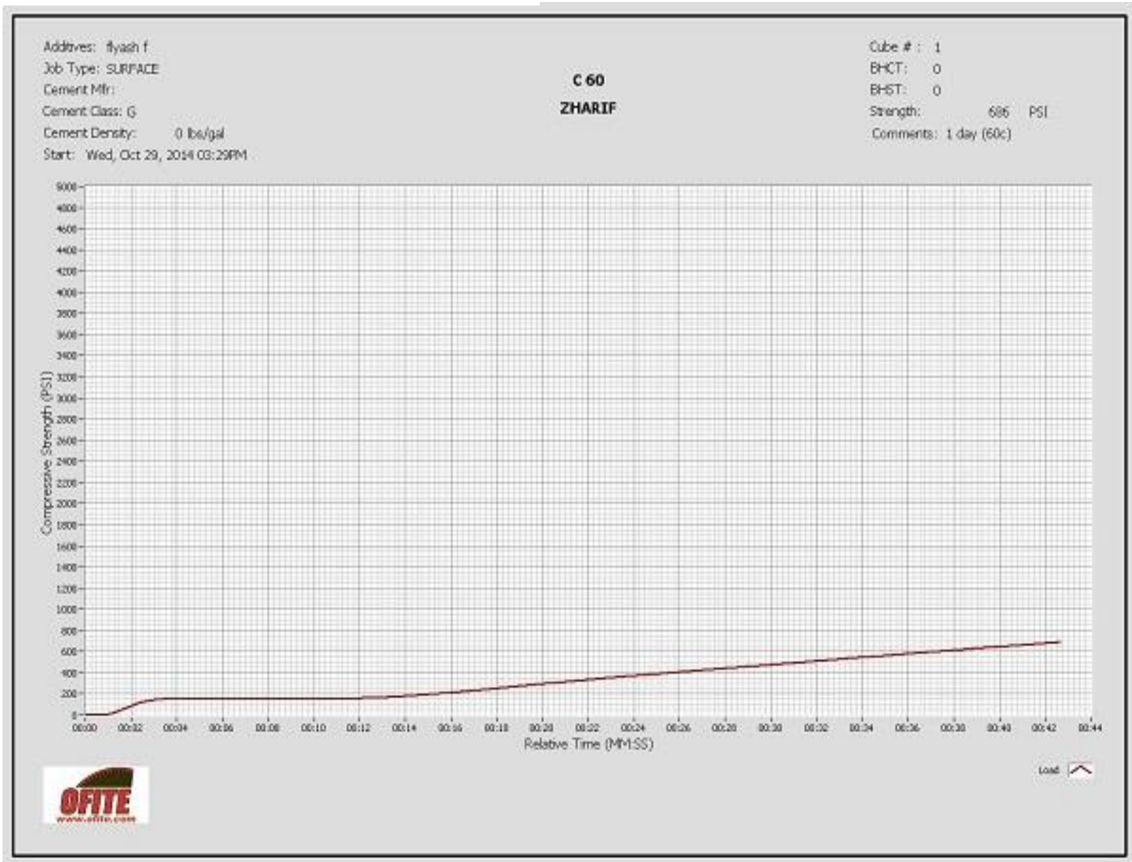


FIGURE 4.6 shows that the compressive strength of Sample C at 60^{o}C curing temperature is **686 psi.**

Sample A cured for 24hours at 70°C

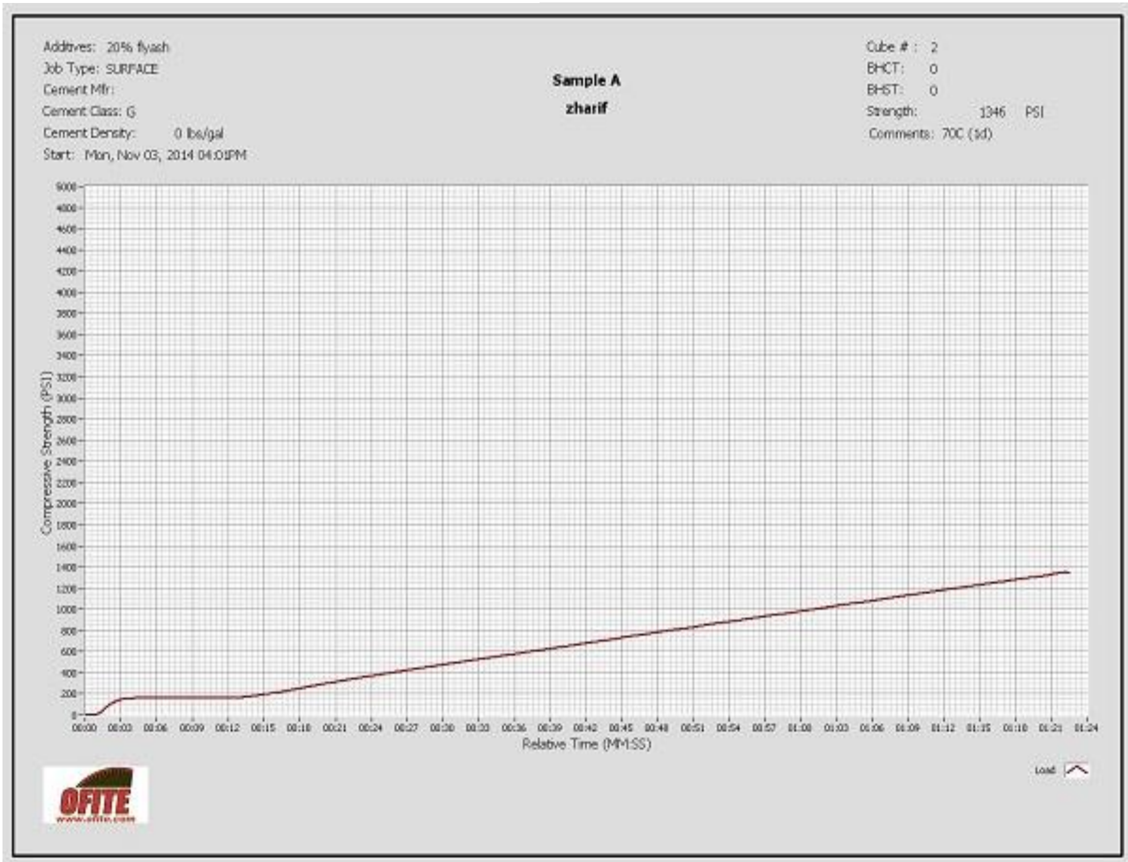


FIGURE 4.7 shows that the compressive strength of Sample A at 70°C curing temperature is **1346 psi.**

Sample B cured for 24hours at 70^{o}C

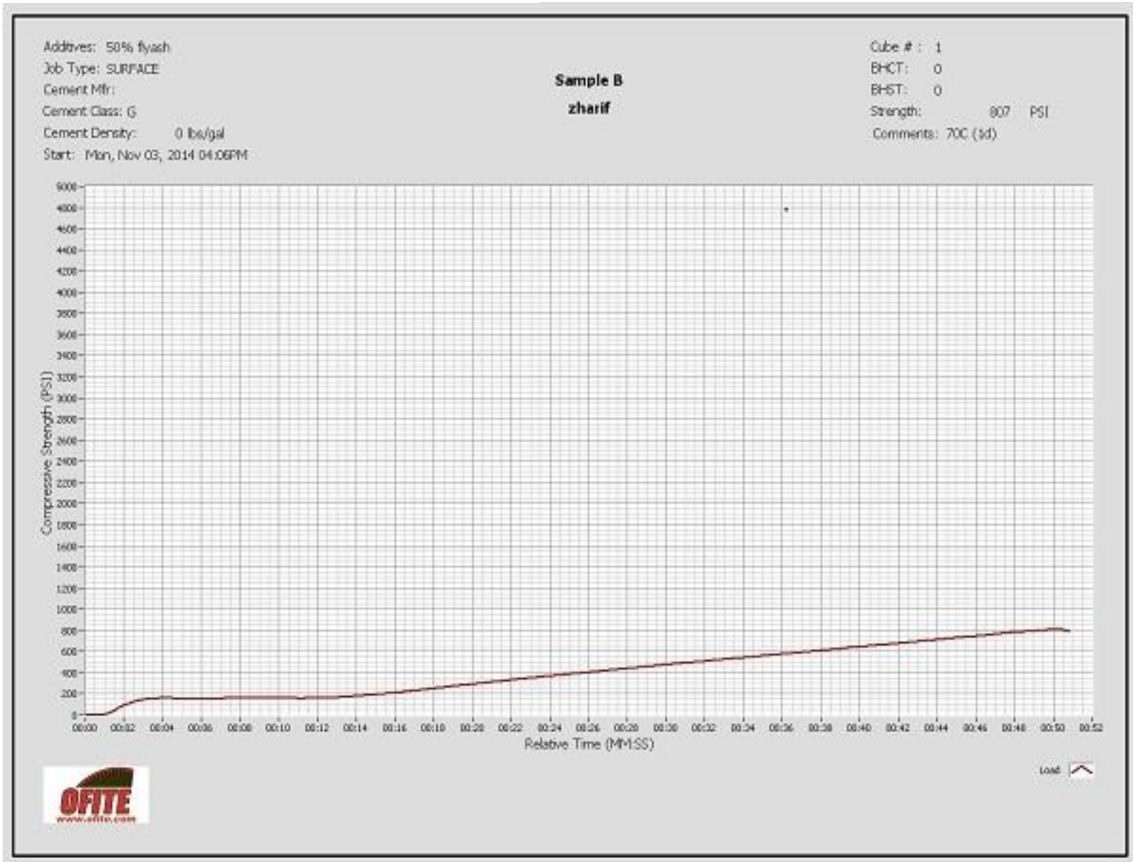


FIGURE 4.8 shows that the compressive strength of Sample B at 70^{o}C curing temperature is **807 psi.**

Sample C cured for 24hours at 70°C

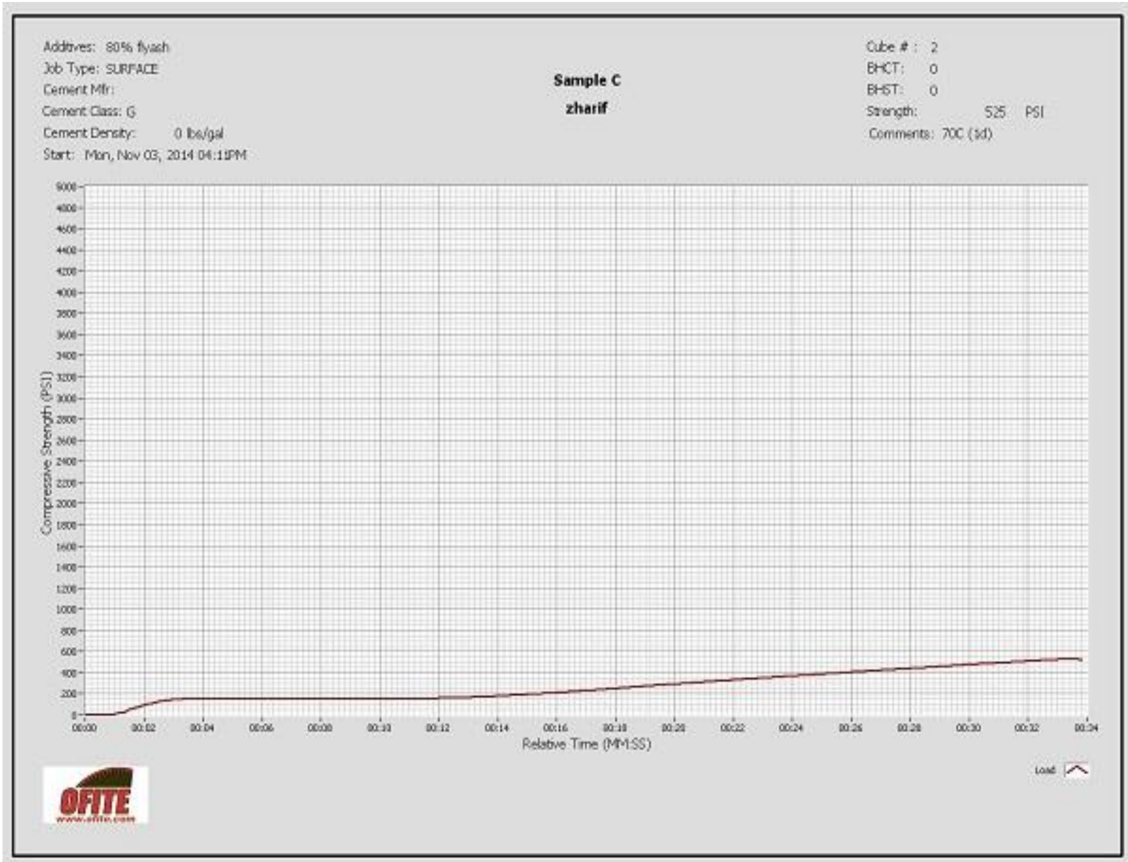


FIGURE 4.9 shows that the compressive strength of Sample C at 70°C curing temperature is **525psi.**

The summary of the initial result was displayed in the table below:

TABLE 4.1: Initial compressive strength test result

Temperature (C^o)	Compressive strength (Psi)		
	A	**B**	**C**
50	1480	1989	1410
60	1850	1481	686
70	1346	807	525

From the data collected, the strongest compressive strength recorded was from the sample B cured at 50^{o}C. The sample recorded the best with 1989 psi. As stated earlier, the samples B composed of 50% class G and 50% flyash.

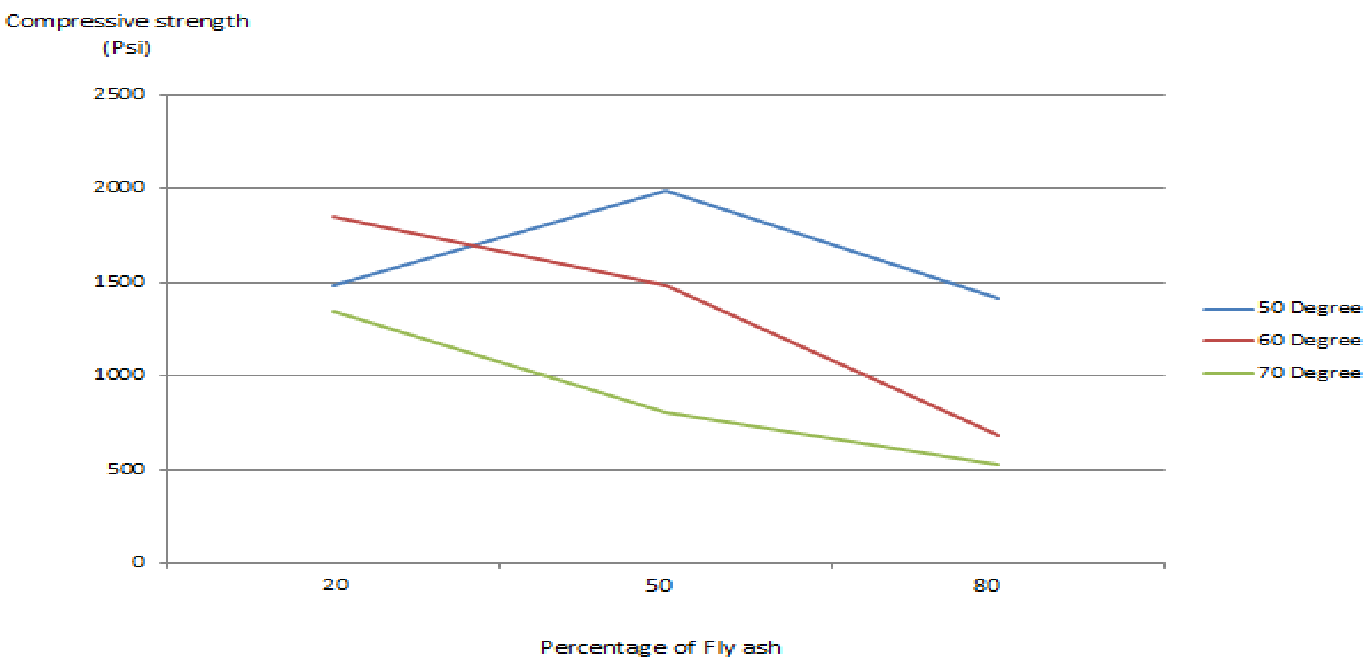


FIGURE 4.10: Initial Compressive strength test result

From the graph displayed in figure 4.1 it can be observed that the general decreasing trend of compressive strength of the geopolymer as they increase the percentage of fly ash in the cement sample. The 50^{o}C curing temperature showed the best overall results which differ from the findings of Rangan, (2008), Nasvi and Swarepol & strydom, (2002). They suggested the best curing temperature for geopolymer cement is 60^{o}C. However, the second hardest sample is the sample cured at 60^{o}C with only 20% fly ash content.

4.3 The final Compressive strength and uniaxial compressive strength test.

After identifying the best compositional geopolymer batch, the same is reproduced to be tested in the well environment – 100psi, 50^{o}C. These samples were further cured for 4 days totaling of 5 days of curing including the 24hour oven curing process.

TABLE 4.2: The final mechanical test results

TEST	**Result**			**Average**
	B1	*B2*	*B3*	
Compressive strength (Psi)	1820.0	1811.0	1831.0	1820.7
Point Load (Psi)	264.1	251.3	265.0	260.1

From Table 4.2, it can be seen that the compressive strength of the samples after cured in well condition had loss an average of 168.3psi of its initial compressive strength tested earlier in the initial result displayed in Table 4.1. The loss of compressive strength was due to the slightly alkaline brine condition – pH 8.06.

The result regarding the uniaxial compressive strength, an average of 260.1psi or 1.82MPa was calculated from the 3 sample tested. The uniaxial compressive strength properties are usually calculated in formation rocks to give an estimation of its porosity value. As suggested, in this study, we calculate them also to estimate the porosity of the cement produced to determine its quality. To simplify the estimation, by assuming the cement behaves as sandstone. The porosity of the cement is estimated to be 0.39 which categorize it as average quality cement.

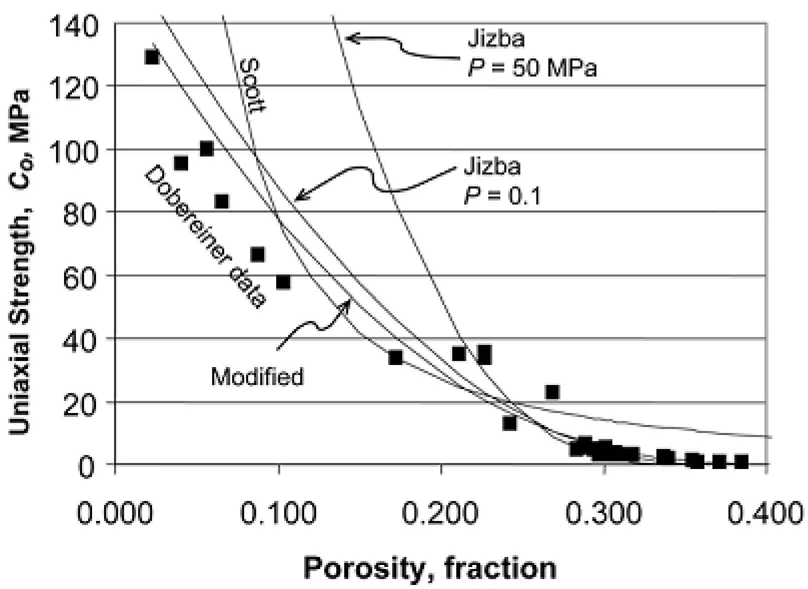


FIGURE 4.11: Uniaxial compressive strength vs porosity graph for limestone.

CHAPTER 5
CONCLUSION & RECCOMENDATION

5.1 Conclusion

The ultimate objective of this project is to study the effect of the mechanical properties of fly ash as geopolymer cement at normal condition and well condition. Through the manipulated variables and different compositions, we have identified the mechanical performance of the geopolymer. For instance, the curing temperature higher than 60^oC will not improve the mechanical properties of the geopolymer same as the study suggested by Rangan, (2008) and Swarepol, (2008). Regardless of the outcome, this study had achieved its objective to study the effect of different conditions on the mechanical properties of the flyash based geopolymer. As discussed in the previous section, the conclusions are that firstly the compressive strength of the geopolymer shows a slightly degrading trend as it was further cured in a slightly alkaline well condition. Secondly, the uniaxial compressive strength test gives an average value of 260 psi or equivalent to 1.82MPa which indicated average quality cement.

All in all, the observations and results promise a potential OPC replacement. It is deeply hoped that more investigation of fly ash based geopolymer cement will successfully lead us to a potential cement replacement for OPC in the upstream industry. The fly ash based geopolymer offers a holistic solution to increasing demands of cement in the oil and gas sector in a sustainable manner, at majorly reduced cost, and at the same time reducing the environmental impact of both the cement industry and the coal-fired power industry.

5.2 Recommendations

Due to the inexperience in working with geopolymer cementing prior to this experiment, there might have been some overlooked procedures and method of conducting the experiment. Hence, it is recommended to seek more advice from those experienced. It is also highly recommended that the experiment is continued at various other manipulations of variables and test against other factors in addition to compressive strength. The sample should be tested for a much longer period to get a much more reliable and relevant result as to estimate what is happening in the field. Furthermore, the experiment can be expanded to include other additives such as nano-silica fumes to be tested as an actual downhole cement. Hence, with more time and work, it is deeply hoped that the fly ash will be a more green and economical substitute for OPC.

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