Investigating the Compressive Strength Plateau of Geopolymer Cement under HPHT

by

Bilal Ahmed Khan

17008

Dissertation submitted in partial fulfilment of

the requirements for the

Bachelor of Engineering (Hons)

(Petroleum)

MAY 2015

Universiti Teknologi PETRONAS
Bandar Seri Iskandar
31750 Tronoh
Perak Darul Ridzuan
Malaysia

CERTIFICATION OF APPROVAL

Investigating the Compressive Strength Plateau of Geopolymer Cement under HPHT

by

Bilal Ahmed Khan

17008

A project dissertation submitted to the

Petroleum Engineering Programme

Universiti Teknologi PETRONAS

in partial fulfillment of the requirement for the

BACHELOR OF ENGINEERING (Hons)

(PETROLEUM)

Approved by,	Approved by,
_____________________________	__________________________
Dr. Syahrir Ridha	Mr. Asif Zamir

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

May 2015

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.

BILAL AHMED KHAN

ABSTRACT

The modern oil and gas industry excessively uses Ordinary Portland Cement (OPC) as their preferred cementing choice. However, the industry is quickly realizing that OPC's mechanical properties fail to uphold its objectives in deeper wells with higher temperature and pressure. Due to its weak ceramic characteristics, Ordinary Portland cement's mechanical performance is limited, especially in wells with high temperature and pressure. Comparatively, Geopolymeric materials can better tolerate these work conditions. The scope of study is mainly on designing Geopolymer cement compositions, preparing class G cement composition and testing in accordance to the American Petroleum Institute. The obtained results will be compared in terms of compressive strength with class G cement slurries. The study will comprise standard weight cement slurry. Geopolymer, a class of inorganic polymer, results due to the reaction between alumina-silicate as a source and an alkaline solution. As stated by (Nazari, Bagheri, & Riahi, 2011), there is a major influence of curing temperature on the compressive strength of cement because it determines the setting and hardening rate of cement. Previous research has also proven that the content of fine particles of fly ash have a meaningful influence on the Geopolymer cements compressive strength. Results show that Geopolymer cement has higher compressive strength than OPC in all its compositional samples. Proving its superiority to replace OPC in industry. The optimum curing condition is at 60^{o}C and 1400 psi, where maximum strengths are achieved by both Geopolymer and OPC. The best composition resulting in highest compressive strength is Geopolymer sample B at 30% micro silica and 70% fly ash. Geopolymer cement loses strength at elevated temperature above its optimum temperature of 60^{o}C. But it still possesses higher strength than OPC which loses 42% strength at 120^{o}C. Comparative study on the compressive strength limitations of OPC and Geopolymer proved that all Geopolymer compositions performed better than OPC at optimal and high temperature and had substantially better strength gains with longer curing times.

.

ACKNOWLEDGEMENT

My efforts would have been in vein if it wasn't for the support and encouragement of all people that I crossed path with during the lifespan of this project

First and foremost, I would like to thank with the deepest gratitude to Dr. Syahrir Ridha, my project supervisor and mentor for his valuable guidance and advice. I would also like to express my appreciation to all the lab technicians who provided me with the technical and hands on information for completing this project. Besides, I would like to acknowledge Universiti Teknologi PETRONAS, Malaysia for providing the financial support and research facilities.

TABLE OF CONTENTS

CERTIFICATION i

ABSTRACT iii

ACKNOWLEDGEMENT iv

CHAPTER 1: INTRODUCTION ………………………………………………….... 1

1.1. Background ……………………………………………………………………… 1
1.2. Problem Statement ……………………………………………………………… 3
1.3. Significance of the Project ...………………...……………………...…………… 3
1.4. Objective …...……………...……………………...…………………………...… 3
1.5. Scope of Study ………………………………………………………………….... 4
1.6. The Relevancy of the Project……………………………………………….…... 4

CHAPTER 2: LITERATURE REVIEW & THEORY ……………………………… 5

2.1. Portland cement ……………………………………………............................. 5
2.2. Geopolymer Cement and composition ………………………………………….. 6
2.3. Geopolymerization ……………………………………………………………… 7
2.4. Fly ash …………………………………………………………………………... 9
2.5. Micro silica …………………………………………………………………….. 10
2.6. Compressive strength …………………………………………………………….. 10

CHAPTER 3: METHODOLOGY/PROJECT WORK ……………………………….. 11

3.1. Experimentation Design ……..…………………………………………...…..….... 12

3.1.1. Preparation of cement slurries ……………………………………………….. 12

3.1.2. Compressive strength testing …………………………………………… 16

3.2. List of Tools, equipment and materials used …………………………………..... 17

3.3 Gantt chart and key milestones …………………………………….….…….... 18

CHAPTER 4: RESULTS AND DISCUSSION……………………………………….. 19

4.1 Data Gathering ………………………………………………………………….. 19

4.2 Compressive Strength Calculation ……………………………………….……..… 19

4.3. Experimental Results and Discussion …………………………...…………….. 20

4.3.1. Samples cured at 60^{o}C and 1400 psi …………………………………..... 20

4.3.2. Samples cured at 120oC and 4000 psi ……………………..………..... 22

4.3.3. Strength comparison of OPC and Geopolymer cement ……………..… 24

4.3.4. Geopolymer Sample B as the Optimum Geopolymer Composition …… 25

CHAPTER 5: CONCLUSION & RECOMMENDATIONS …………………………. 27
5.1. Conclusion ……………………………………………………………………... 27
5.2. Recommendations ……………………………………………………………... 28
References ……………………………………………………………………......... 29

LIST OF FIGURES

Figure	Page
Figure 1. Compressional failure causing fracture in cement	2
Figure 2. Geopolymerization process	6
Figure 3. Detailed Geopolymerization process model	7
Figure 4. Benefits of Geopolymer cement as a substitute for OPC	8
Figure 5. Methodology/Flow Chart of proposed work	11
Figure 6. Curing chamber	15
Figure 7. Greased curing molds	15
Figure 8. Curing molds tightened using thread and screws	15
Figure 9. Molds being inserted into pressure vessel	15
Figure 10. Cylinder plug being threaded into pressure vessel	15
Figure 11. Cured cement samples	15
Figure 12. Compressive Strength Tester	16
Figure 13. Results of samples cured at 60^{O}C, 1400 psi	20
Figure 14. Results of samples cured at 120^{O}C, 4000 psi	22
Figure 15. OPC strength gain and loss at different temperature.	24
Figure 16. Sample B strength gain and loss at different temperature.	25

LIST OF TABLES

Table	Page
Table 1. Five main compounds with their weight percentage and chemical formula.	5
Table 2. Class C and class F fly ash Composition (Source: Singh G., 2013).	9
Table 3. Cement slurries with their chemical compositions.	12
Table 4. Mass of Class G Cement, fly ash, micro silica and alkaline solution for every mix in grams.	12
Table 5. Compressive Strength Testing Procedure.	16
Table 6. Gantt Chart for FYP2.	18
Table 7. Proposed Key milestone for the project implementation for FYP II.	18
Table 8. Compressive strength results at 60^{O}C, 1400 psi.	21
Table 9. Compressive strength results at 120^{O}C, 4000 psi.	23

CHAPTER 1

INTRODUCTION

1.1. Background

Cement is widely used in oil and gas producing wells as a binding agent. Introducing cement into the well positively effects its overall productivity and in its absence, different zonal fluids may hinder one another. Cementing a well, illustrated by figure 1, requires cement slurry to be pumped into the bottom of the wellbore pass through the casing and out into the annulus space as it displaces the drilling fluid. The cement then eventually fills the annulus and as it hardens, it seals off the annulus to inhibit the flow of formation fluids entering the well.

Well cementing, part of a completion process, is one of the most vital and crucial stages in achieving a well which supports the casing and limits its contact with formation fluid to prevent corrosion, prevents formation fluids from entering the well and effectuates zonal isolation. Cementing is deemed unsuccessful if it fails to attain the mentioned criteria.

It is vital that cement, employed in oil and gas wells, perform its desired duties in the harsh conditions it encounters underground. Its success, however, depends on composing the cement via selecting the right additives and components to endure the diverse well conditions, which worsen as the oil and gas industry finds itself drilling deeper and in harsher conditions. As the conventional cementing solutions fail to cope with these new challenges, the need for a better cementing solution with enhanced mechanical properties arises.

The modern oil and gas industry excessively uses Ordinary Portland Cement (OPC) as their preferred cementing choice. The justification in part, may be due to OPC's aggressive and successful marketing. However, the industry is quickly realizing that OPC's mechanical properties fail to uphold its objectives in deeper wells with higher temperature and pressure. Moreover, OPC has also been recognized as one of the leading greenhouse gasses emitters. This calls for the need of developing a more mechanically strong and environmentally friendly cement slurry to be used as a substitute. Geopolymer is one alternate that is being studied extensively for its mechanical performance and shows promise as a greener substitute for Ordinary Portland cement.

1.2. Problem Statement

A major sign of cement failure is cracking/fracturing, caused by the surpassing of the cements rupture compressive strength as illustrated in figure 1. This compressional failure may be on the account when a well is under the influence of high temperature and pressure.

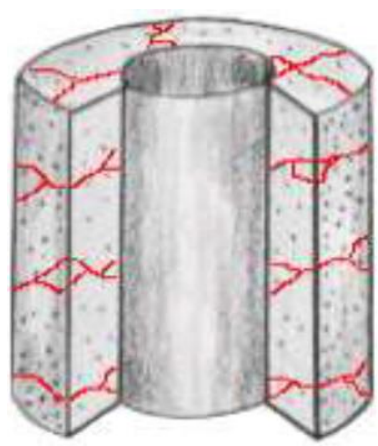

Figure 1. Compressional failure causing fracture in cement

Due to its weak ceramic characteristics, Ordinary Portland cement's mechanical performance is limited, especially in wells with high temperature and pressure. Comparatively, Geopolymeric materials can better tolerate these work conditions because they possess high thermal stability and plastic behavior. As the focus shifts to the possibility of a wide scale use of Geopolymer cement in oil and gas industry, the need for testing and studying its mechanical properties strengthens. Finding the optimum compressive strength of the Geopolymer cement and studying it comparatively with Ordinary Portland cement is vital if this greener substitute is to ever completely replace its competitor.

1.3. Significance of the Project

Studying literature, it is found that previous research conducted on Geopolymer cement has restrained themselves with identical compositions and temperature ranges. Additionally, major conclusive research has been done on Geopolymer concrete by civil departments, there is yet no sufficient published work or research study conducted on Geopolymer based oil well cementing systems using different compositional variations. Therefore, this research is dedicated to give an insight of the significance of developing Geopolymer cement by utilizing different compositions that would result in improved mechanical properties namely compressive strength.

1.4. Objective

The objective of this research project will be to primarily study the compressive strength properties of Geopolymer cement on compressive strength under high pressure and high temperature to find its optimum strength by manipulating Geopolymer’s compositional properties. The focus will also be to comparatively studying Geopolymer and OPC compressive strength and analyzing its trends and limitations. At the end of the project, it is hoped to prove that Geopolymer can attain its superior strength at high temperature and pressure conditions.

1.5. Scope of Study

The scope of study is mainly on designing Geopolymer cement compositions, preparing class G cement composition and testing in accordance to the American Petroleum Institute. The obtained results will be compared in terms of compressive strength with class G cement slurries. The study will comprise standard weight cement slurry.

1.6. The Relevancy of the Project

This project is closely related to the oil well cementing systems. Therefore, for the successful completion of this project with minimal errors, an insightful understanding on the cementing materials and oil well cementing systems is required. Besides, detailed study on Geopolymer cement and its characteristics is also vital to select the most beneficial compositional materials to be tested with.

Throughout the progression of this project, the author was challenged to absorb new knowledge to be able to understand the problems related to the current conventional cementing and to present and select the right compositional materials that would enhance Geopolymer cements mechanical properties. If proved right, the author hopes to add to the growing number of research being conducted on Geopolymer cement and hopefully see the complete replacement of OPC form oil and gas industry.

CHAPTER 2

LITERATURE REVIEW & THEORY

2.1. Portland Cement

The five main compounds found in the composition of Portland cement is enlisted in table 1:

Table 1. Five main compounds with their weight percentage and chemical formula.

Cement Compound	Weight Percentage	Chemical Formula
Tricalcium silicate	50 %	Ca_3SiO_5 or $3CaO\ SiO_2$
Dicalcium silicate	25 %	Ca_2SiO_4 or $2CaO\ SiO_2$
Tricalcium aluminate	10 %	$Ca_3AI_2O_6$ or $3Ca0\ Al_2O_3$
Tetracalcium aluminoferrite	10 %	$Ca_4AI_2Fe_2O_{10}$ or $4CaO\ Al_2O_3\ Fe_2O_3$
Gypsum	5 %	$CaSO_4\ 2H_2O$

The development of compressive strength in OPC is achieved through hydration; water chemically reacting with the cement compound. The failure of OPC cement strength at elevated temperatures is primarily caused by the loss of silica, which majorly contributes to cement strength, in cement due to degradation.

The cement set in oil and gas wells is governed by static and dynamic stresses. Dead weight of the casing coupled with the constant compressive stresses from formation fluids may become unbearable for OPC as its strength retrogrades at high temperature and pressure.

2.2. Geopolymer cement and its composition

Geopolymer, a class of inorganic polymer, results due to the reaction between alumina-silicate as a source and an alkaline solution. This cementations material is known to have the following traits:

- High strength
- Excellent volume stability
- Durability and resistance to acids.
- High thermal stability and plastic behavior.

Perhaps, the most notable difference between OPC and Geopolymer cement in terms of energy consumption is due to the fact that, comparatively Geopolymer consumes much less energy to react with a high energy solution, as illustrated by figure 2.

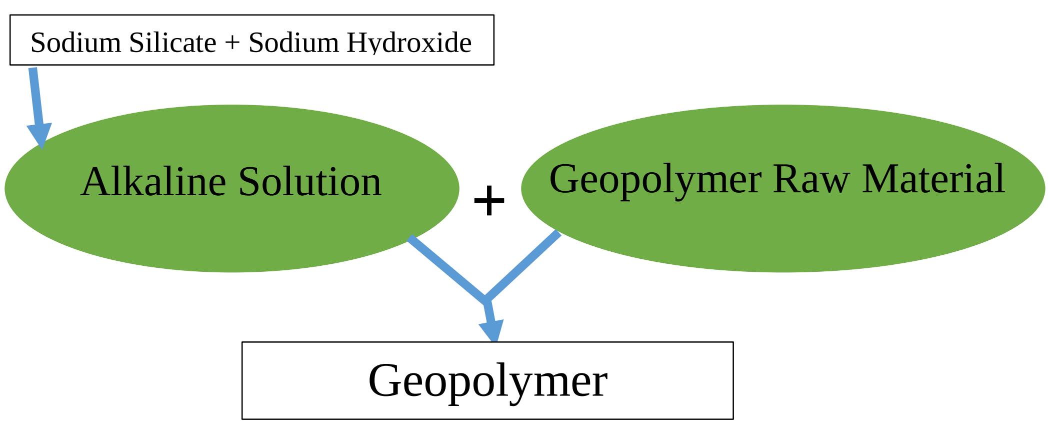

Figure 2. Geopolymerization process

2.3. Geopolymerization

Geopolymerization can be a profitable way of recycling materials and using previously unused materials (Van Jaarsveld et al., 1997). The detailed Geopolymerization process, as illustrated in figure 3, shows the six processes that occur as solid alumino-silicate source transforms into a synthetic alkali alumino-silicate.

Aluminate and silicate species are produced as a result of the dissolution of the solid alumina silicate source by alkaline hydrolysis. Imersed in solution the dissolution process releases species that are incorporated into the aqueous phase, which may already contain silicate present in the activating solution. Ultimately, a composite blend of silicate, aluminate and aluminosilicate species is formed.

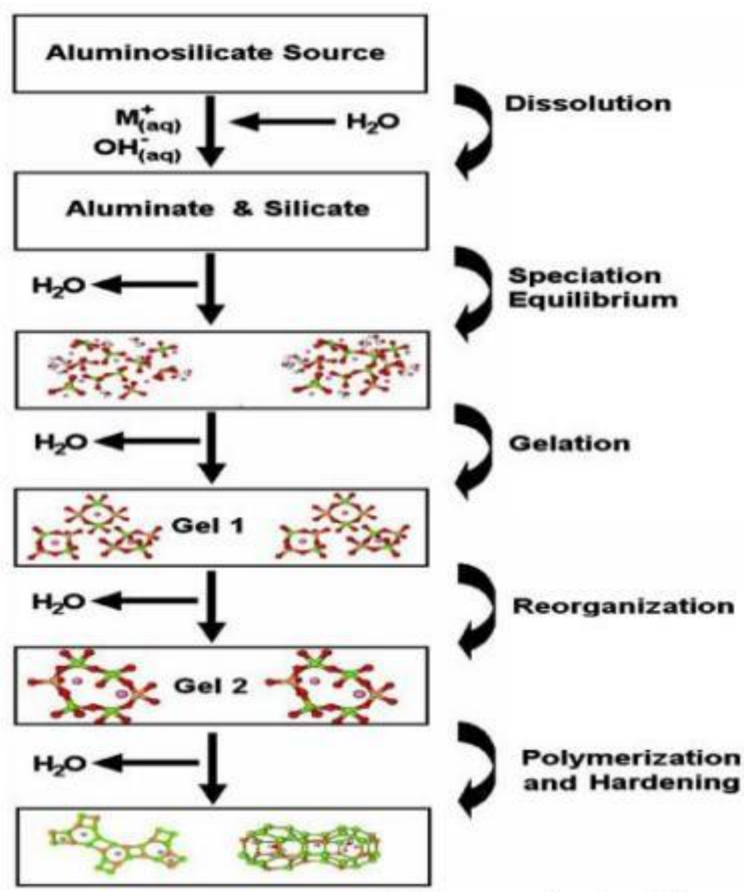

Figure 3. Detailed Geopolymerization process model

The polymerization process, as explained by Drvidovits, results in a three-dimensional polymeric chain and ring structure consisting of Si-OAl-O bonds. Equations (1) and (2) illustrates the formation of Geopolymer material.

$$\underset{\text{(Si-Al materials)}}{n(Si_2O_5,Al_2O_2)}+2nSiO_2+4nH_2O+NaOH\text{ or }KOH \rightarrow Na^+,K^+ + \underset{\text{(Geopolymer precursor)}}{n(OH)_3\text{-Si-O-}\underset{\displaystyle (OH)_2}{\underset{|}{Al^-}}\text{-O-Si-}(OH)_3} \quad (1)$$

$$n(OH)_3\text{-Si-O-}\underset{\displaystyle (OH)_2}{\underset{|}{Al^-}}\text{-O-Si-}(OH)_3 + NaOH\text{ or }KOH \rightarrow \underset{\text{(Geopolymer backbone)}}{(Na+,K+)\text{-}(\text{-}\underset{\underset{|}{O}}{\underset{|}{\overset{|}{Si}}}\text{-O-}\underset{\underset{|}{O}}{\underset{|}{\overset{|}{Al^-}}}\text{-O-}\underset{\underset{|}{O}}{\underset{|}{\overset{|}{Si}}}\text{-O-})} + 4nH_2O \quad (2)$$

Listed below are the summary of the known advantages of using Geopolymer cement:

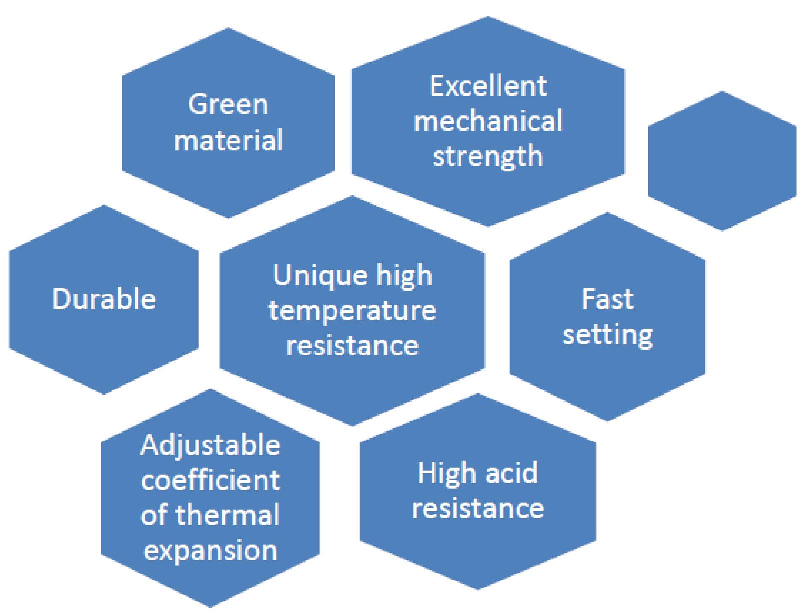

Figure 4. Benefits of Geopolymer cement as a substitute for OPC

2.4. Fly Ash

Fly ash results from the burning of coal and it is found to be rich with silica and alumina. Silicon oxides, aluminium oxides and iron oxides are the heterogeneous mixtures that are found in Fly ash. The two criteria's that determine the binding properties of the resulting fly ash are the types of coal burned and the nature of combustion process.

Table 2. Class C and class F fly ash Composition (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

Some of the advantages of using fly ash, besides the fact that it increases the mechanical activation property due to increase in the surface area, are as follows:

- Increases cement strength
- Improves sulphate resistance of the cement
- Decreases permeability of the cement
- Reduces the water ratio requirement of the cement
- Improves the workability of the resulting cement

2.5. Micro Silica

The production of micro silica, from silicon and ferrosilicon alloys, is achieved by the reduction of high purity quartz with coal. It has been recognized as a highly effective pozzolanic material, the reason being that it has extremely fine and high silica content particles. Mixing of micro silica has been known to increase the compressive strength and reduce the permeability of the resulting cement. Micro silica ingests high level of water when in solution creating a strong bond between micro silica and the cement, this may also be due the increased amount of slurry gel. The reduction in permeability is directly related to the fact that its particles, approximately the size of 0.1 um, are 100 times smaller than Portland cement particles which gives it the property to fill in the pores in-between cement particles and restrict the migration of fluid through the narrow passages. Additionally, this property of the resulting cement also allows it to reduce the overall fluid loss.

2.6. Compressive Strength

Compressive strength, in the sense of the strength of materials, is defined as the amplitude to which, the stress a material can withstand under compression. It is simply a ratio of maximum load it can sustain to the total surface area of the cement cubes. As stated by (Nazari, Bagheri, & Riahi, 2011), there is a major influence of curing temperature on the compressive strength of cement because it determines the setting and hardening rate of cement. It has also been found that the polymerization process is fastened by exposure to high temperature. However, (Swanepoel & Strydom, 2002) and (Chindaprasirt, Chareerat, & Sirivivatnanon, 2007) concludes that the optimum curing temperature for geopolymer cement is 60°C. Previous research has also proven that the content of fine particles of fly ash have a meaningful influence on the geopolymer cements compressive strength. It states that the use of finer particles of the fly ash results in a Geopolymer cement with greater compressive strength.

CHAPTER 3

METHODOLOGY/PROJECT WORK

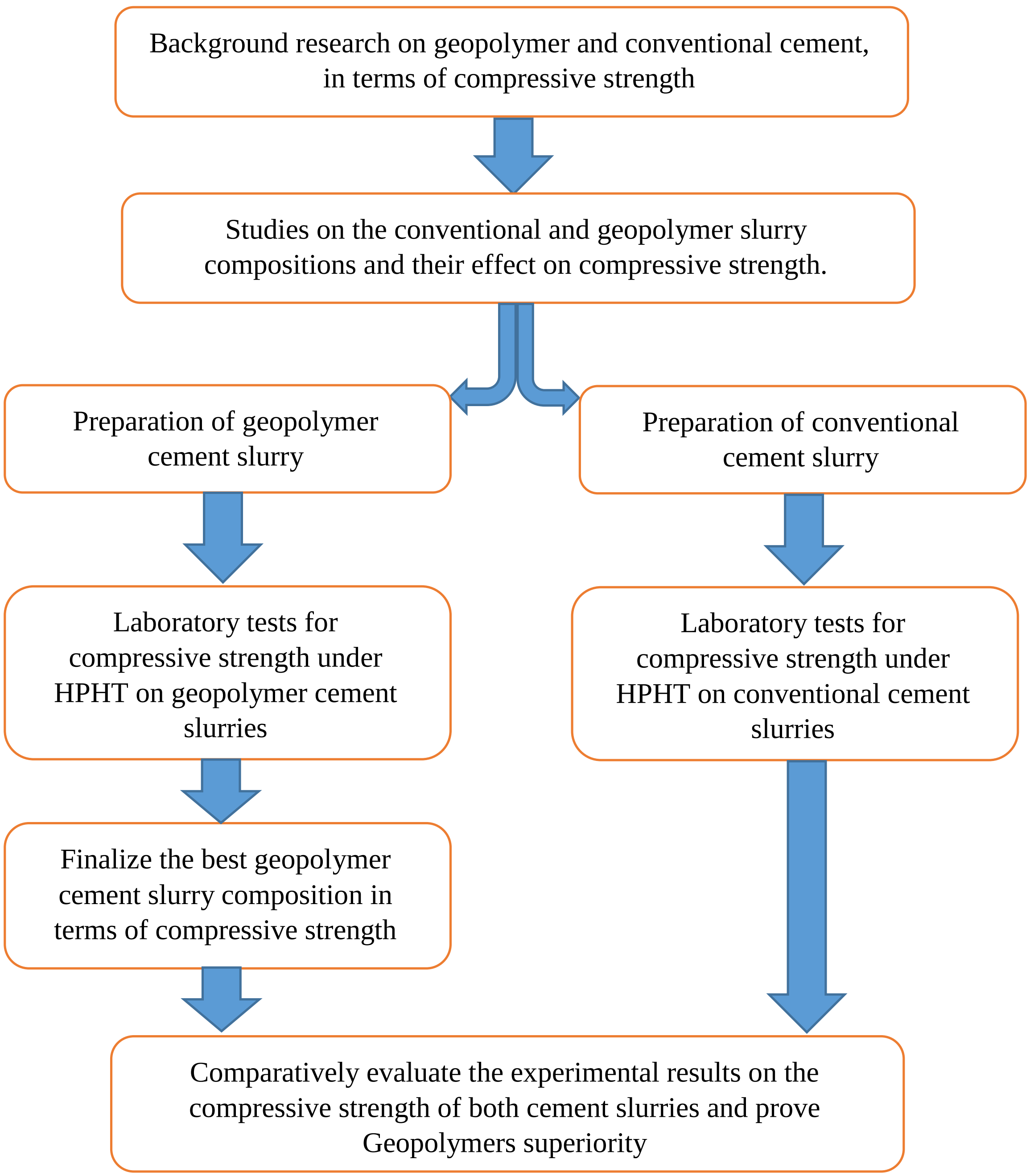

Figure 5. Methodology/Flow Chart of proposed work

3.1. Experimentation Design

3.1.1 Preparation of cement slurries

The four different types of cement slurries used in the experiments are tabulated as follows:

Table 3. Cement slurries with their chemical compositions.

Cement Slurry	Chemical Composition
Conventional Portland Cement	100% Class G cement + water
Geopolymer A	100% fly ash + NaOH + NA_2SiO_3 + water
Geopolymer B	70% fly ash + 30% micro silica + NaOH + Na_2SiO_3 + water
Geopolymer C	80% fly ash + 20% micro silica + NaOH + Na_2SiO_3 + water

Samples	Cement (grams)			Mix Solution (grams)		
	Class G Cement	Fly Ash Class F	Micro Silica	Sodium Silicate	Sodium Hydroxide	Water
Class G Cement	500	0	0	178.59	71.43	100.15
A	0	500	0			
B	0	350	150			
C	00	400	100			

Table 4. Mass of Class G Cement, fly ash, micro silica and alkaline solution for every mix in grams.

Preparation of all the cement slurries were in accordance with American Petroleum Institute API-10B-2 procedure using constant speed mixer. The ratio of water to cement was chosen to be 44% complying with the ratio set for testing/mixing Class G cement. Based on this ratio, the cement amounted to 792 grams and mix solution to be 349 grams. Additionally, the ratio of alkaline solution to fly ash was set at 0.50, as suggested by

Mr.Fareed Ahmed Memon in his previous research, stating that this ratio would result in optimum cement strength.

Sodium silicate solution comprising of Na2O = 14. 7%, SiO2 = 29.4% and water = 55. 9% was used in the preparation of all samples. Alkaline activation was achieved through the combination of 8M NaOH and Na_2SiO_3 and the ratio of sodium silicate to sodium hydroxide was selected at 2.5. Both alkaline solutions were made constant in the preparation of all samples.

Procedure for cement slurries preparation is as follows:

1. Electronic balance scale was used to measure the calculated amount of materials needed for the preparation of each type of cement samples.
2. Constant speed mixer (model 3060) was used for mixing all the slurry compositions in accordance with the API mixing procedure.
3. Mixing procedure for all the cement slurry samples is as follows:
    i. Distilled water was placed in the mixer and agitated for 15 seconds at 4000 rotations per minute, rpm.
    ii. Measured Na_2SiO_3 was added into the mixer.
    iii. Measured compositions (Class G cement, fly ash, Micro silica and NaOH pellets) were added into the mixer.
    iv. The compositions were mixed at a speed of 12000 rpm for 35 seconds to ensure proper mixing.

Curing Cement Samples:

I. Cement molds are thoroughly greased prior to assembling.
II. Cement slurry is gently poured into the assembled molds in three layers. Each layer is subsequently paddled using a stirring rod to ensure the absence of potential bubbles in the cement slurry, which could disrupt the shape and mass of the resulting samples. Molds are then clamped using a threaded rod.
III. Curing chamber is powered on.
IV. Sets of clamped molds are carefully lowered in the pressure vessel. The cylinder plug thread is lubricated using grease and is threaded into the cylinder.
V. A thermocouple is inserted through the hole on top of cylinder plug and is tied loosely.
VI. The air access is opened and the oil cylinder is used to monitor the flow of oil into the pressure vessel. Thermocouple is tightened with a spanner as soon as oil expels from it.
VII. The pump fluctuates until the desired pressure is achieved.
VIII. The desired temperature is set in the program list and will slowly reach it over time. The temperatures of 120 ℃ and 60℃ were chosen for this experiment.
IX. The heater is switched on followed by the timer.
X. Then, auto and run button is pressed to start the operation. The pressure is monitored throughout the duration of the experiment to ensure that the experiment is not run in the overpressure or under-pressure conditions.

Figure 6. Curing chamber

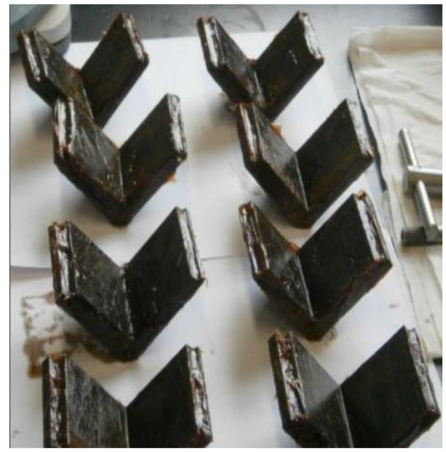

Figure 7. Greased curing molds

Figure 8. Curing molds tightened using thread and screws

Figure 9. Molds being inserted into pressure vessel

Figure 10. Cylinder plug being threaded into pressure vessel

Figure 11. Cured cement samples

3.1.2 Compressive strength testing

The compressive strength of the cement cubes is determined by compressive strength Tester (Figure 12). The compressive strength tester records the maximum load at which the cement fails and displays the result on the monitor. The procedures of this method are as below:

Table 5. Compressive Strength Testing Procedure.

No.	Procedure
1	Place the cement specimen on the lower platen of the hydraulic cylinder.
2	Adjust the layer of steel at the bottom.
3	Switch on the Compressive Strength Tester.
4	Press the blue button to push the upper base of hydraulic cylinder so that it is touching the specimen.
5	Close the safety shield before beginning the test.
6	Push up the "Controlling Handle" to start the pump.
7	Hold down the "Controlling Handle" while observing the specimen. When the specimen fails, push down the "Controlling Handle" to stop the test and the pump.
8	The "Maximum Compressive Strength (KN)" indicates when the maximum load at which the cement fails.

Figure 12. Compressive Strength Tester

3.2. List of Tools, Equipment and Materials used

A list of chemicals and equipment used for the project is as follows:

Chemicals/Materials:

- F class Fly ash
- Micro silica
- Sodium Hydroxide
- Sodium Silicate
- Distilled Water
- Class G cement

Tools/Equipment:

- Beakers
- Aging cell
- Magnetic Stirrer
- Measuring Cylinders
- Brush
- Oven
- Constant speed mixer
- Pressurized Curing chamber (HP/HT)
- Compressive strength testing machine
- 50mm*50mm*50mm mold

However, the materials and equipment are not limited to the ones mentioned above, there may be several along the accomplishment of this project.

3.3. Gantt Chart and Key Milestones

Gantt chart for the Final Year Project 2

Table 6. Gantt Chart for FYP2.

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

Table 7. Proposed Key milestone for the project implementation for FYP II.

Year	**2015**		
	FYP 2		
Activities	**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.			

CHAPTER 4
RESULTS AND DISCUSSION

4.1. Data Gathering

Majority of the gathered data were from the experiment labs in Block 13 and Block 15 using the equipment mentioned in the previous sections. The mechanical properties of the geopolymer cement are tested based on the different curing temperature and pressure.

4.2. Compressive Strength Calculation

From the experiment, the compressive strength tester gives the maximum load in kilo newton. Compressive strength value can be found using the given formula:

$$Fci = Fi/Aci$$

Where;

Fci = Compressive Strength (KN/mm^2)

Fi = Maximum Load (KN)

Aci = Cross Section Area (mm^2)

Note: 1 KN/mm^2 is equal to 1000 MPa

4.3. Experimental Results and Discussion

4.3.1. Samples cured at 60^{o}C and 1400 psi

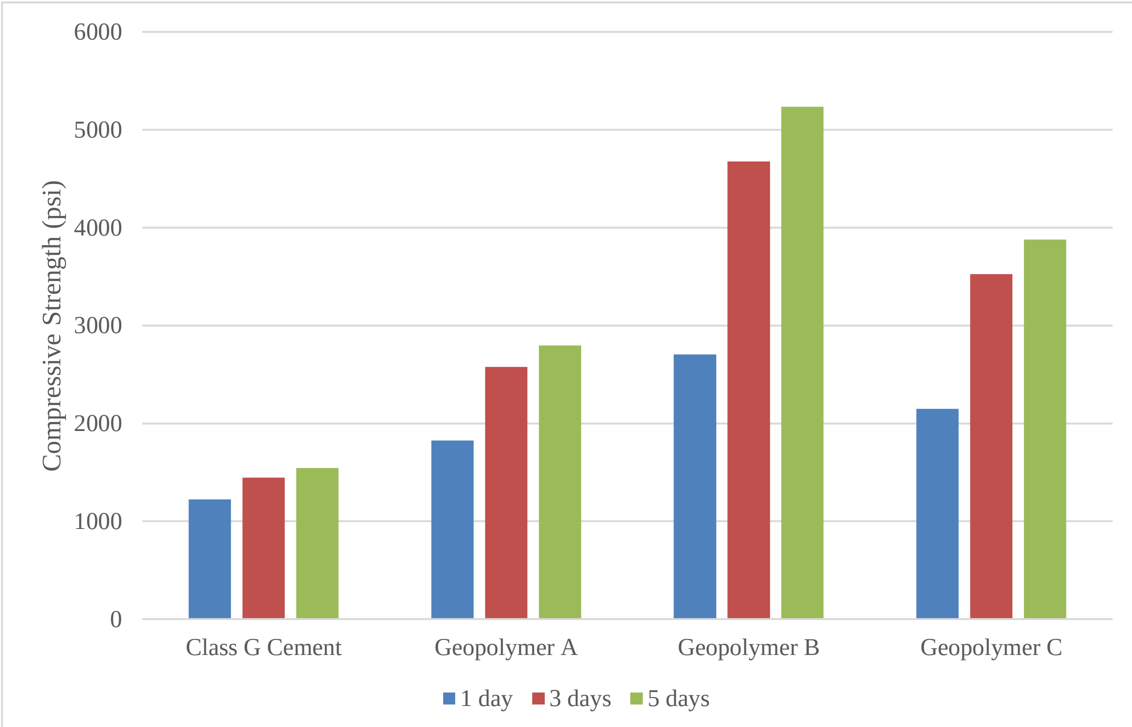

Figure 13. Results of samples cured at 60^{o}C, 1400 psi

The compressive strength results for cement slurries cured at 60^{o}C and 1400 psi for 1, 3 and 5 days are illustrated in the above graph. The graph clearly shows that Geopolymer Sample B, containing 70% fly ash and 30% micro silica, attained the highest compressive strength of 5235 psi at 5 days of curing, and also had higher compressive strength at 1 and 3 days curing time compared to other samples. It is also observed that all the Geopolymer samples resulted in having a higher compressive strength than Class G cement. This is in conjunction with proving that Geopolymer cement attains overall higher compressive strength than Class G cement.

Table 8. Compressive strength results at 60^{o}C, 1400 psi.

Samples	Compressive strength result (psi)		
	1 day	**3 days**	**5 days**
Class G Cement	1224	1446	1544
Geopolymer A	1824	2577	2798
Geopolymer B	2704	4677	5235
Geopolymer C	2150	3526	3878

The above table summarizes the complete test results obtained after curing the samples at 60^{o}C and 1400 psi in the curing chamber. Observing the results, Class G cement had only a slight increase in compressive strength compared to Geopolymer samples from the initial result when tested at 3 and 5 days curing time. On the other hand, the results obtained for Geopolymer samples shows that:

- Geopolymer B gained 72% of its initial compressive strength at 3 days of curing, this may be the result of it containing 30% micro silica.
- Geopolymer C, containing 20% micro silica, also gained substantial amount of strength after curing for 3 and 5 days respectively.
- Geopolymer A, containing 100% fly-ash, attained the lowest strength gain among all the Geopolymer samples.

For the set of samples cured at 60^{o}C and 1400 psi, it can be hypothesized that Geopolymer cement responds in a more prominent manner to the effect of curing temperature in comparison to the Class G cement because of the higher strength increments with increasing curing duration.

4.3.2. Samples cured at 120°C and 4000 psi

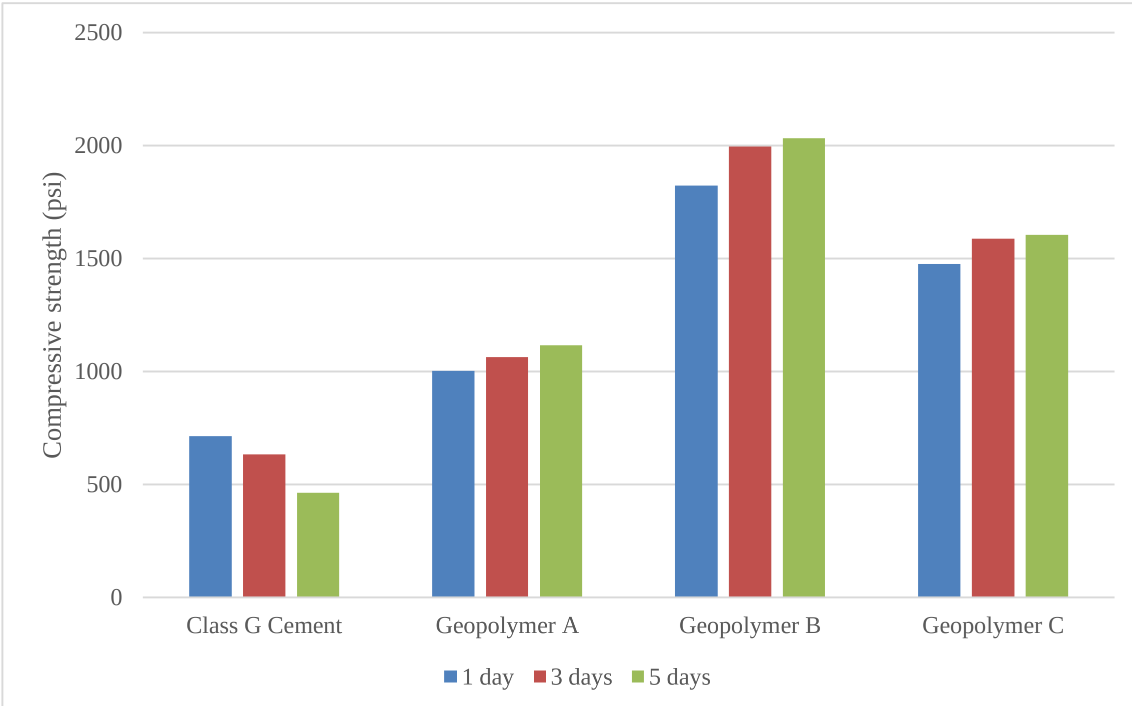

Figure 14. Results of samples cured at 120°C, 4000 psi

The compressive strength results for cement slurries cured at 120°C and 4000 psi for 1, 3 and 5 days are illustrated in the above graph. The highest compressive strength at this curing condition was also achieved by Geopolymer B sample followed by samples C and A respectively. On the other hand, observing the results for Class G cement, there is an evident reduction in its compressive strength as the curing duration is increased by 3 and 5 days. The ideal curing condition for OPC is when a suitably warm and moist environment is maintained for the development of hydration products. Excessive heat exposure for longer duration negatively impacts the hydration process which subsequently reduces the strength due to continuous moisture loss.

Table 9. Compressive strength results at 120^{o}C, 4000 psi.

Samples	Compressive strength result (psi)		
	1 day	**3 days**	**5 days**
Class G Cement	713	632	462
Geopolymer A	1003	1063	1116
Geopolymer B	1823	1995	2032
Geopolymer C	1476	1587	1604

The complete set of results obtained after curing the samples at 120^{o}C and 4000 psi is as tabulated in the above table. It can be observed that there is an 11% reduction in compressive strength when OPC is cured at elevated temperature for 3 days and 26% reduction at 5 days. The strength superiority of Geopolymer samples is also very evident as it attains higher results in all its different compositions. Studying the results of Geopolymer samples, it can be hypnotized that:

- Even though, sample B attained the highest compressive strength, it gained only 9% strength at 3 days curing and 2% gain at 5 days.
- Sample C gained only 1% strength when cured for 5 days. This proves that micro silica content in the cement doesn't contribute to higher compressive strength exposed to elevated temperatures for a longer duration.
- Sample A attained the lowest overall compressive strength in all Geopolymer samples but gained 5% in strength when cured at 5 days mainly because of the absence of micro silica.

Micro silica contains very fine spherical shaped particles, having an average particle size of 1 μm and a specific surface area of typically 20 m^2/g. The particles are also water wet and with its large surface area, requires excessive water absorption in cement slurry for its activation and reaction process. Due to moisture loss at elevated temperature, inadequate water content causes micro silica to partially react and some unreacted micro silica may remain. Additionally, curing at elevated temperatures for longer durations may cause the inter-granular structure of Geopolymers to be broken which reduces the compressive strength.

4.3.3. Strength comparison of OPC and Geopolymer cement

The discussed results show that OPC loses strength at higher temperature of 120^oC but gains minor strength at lower temperature of 60^oC. Relating to this behavior, one can conclude that OPC performs better at lower temperature but still attains lower overall compressive strength compared to any Geopolymer cement compositions.

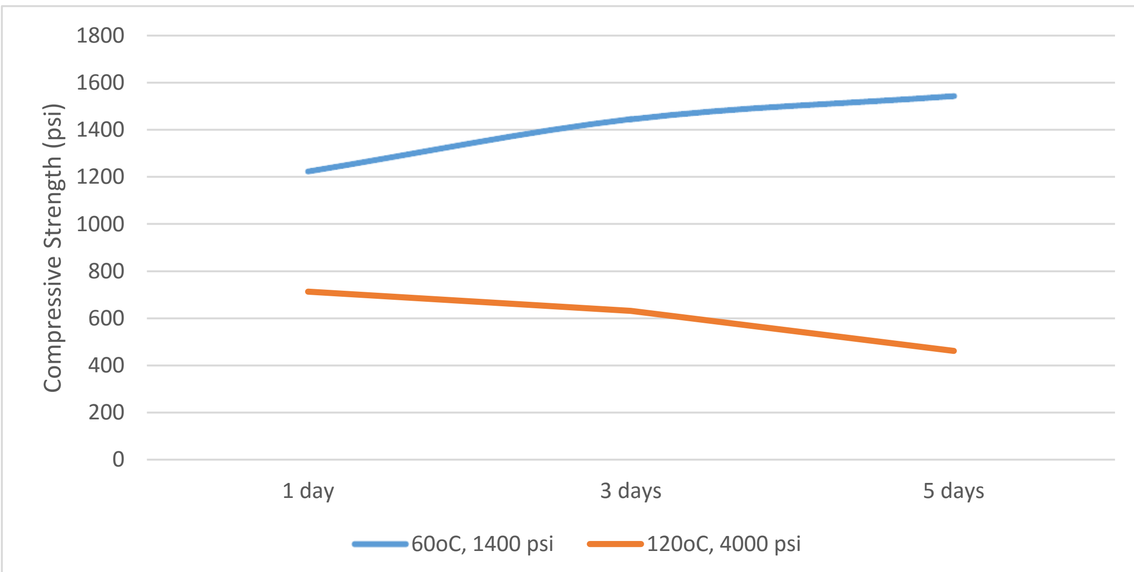

Figure 15. OPC strength gain and loss at different temperature.

As illustrated by the graph above, OPC loses 42% strength when cured at 120^oC. This behavior can be associated to the development of OPC's coarse initial structure as it is cured at higher temperature and due to its initial rapid rate of hydration as well as the possible development of initial internal micro-cracking. Absorbing excessive water due to rapid rate of hydration and the reaction being exothermic may produce internal heat gain in OPC causing its expansion and increasing the failure strain stress behavior.

Looking over at the results obtained for Geopolymer samples, it is observed that:

- The highest Compressive strength was achieved by Geopolymer B samples in both curing conditions.
- Sample B, having micro silica composition of 30% attributed to its higher compressive strength compared to sample C that had 20% of micro silica.
- Micro silica didn't significantly contribute to compressive strength at elevated temperature of 120^{o}C by having non-significant strength gains at longer curing durations because of moisture loss.
- The Combination of 30% micro silica and 70% flyash in Geopolymer sample B is the optimum composition to attain highest compressive strength. This sample is chosen for further analysis.

4.3.4. Geopolymer Sample B as the Optimum Geopolymer Composition

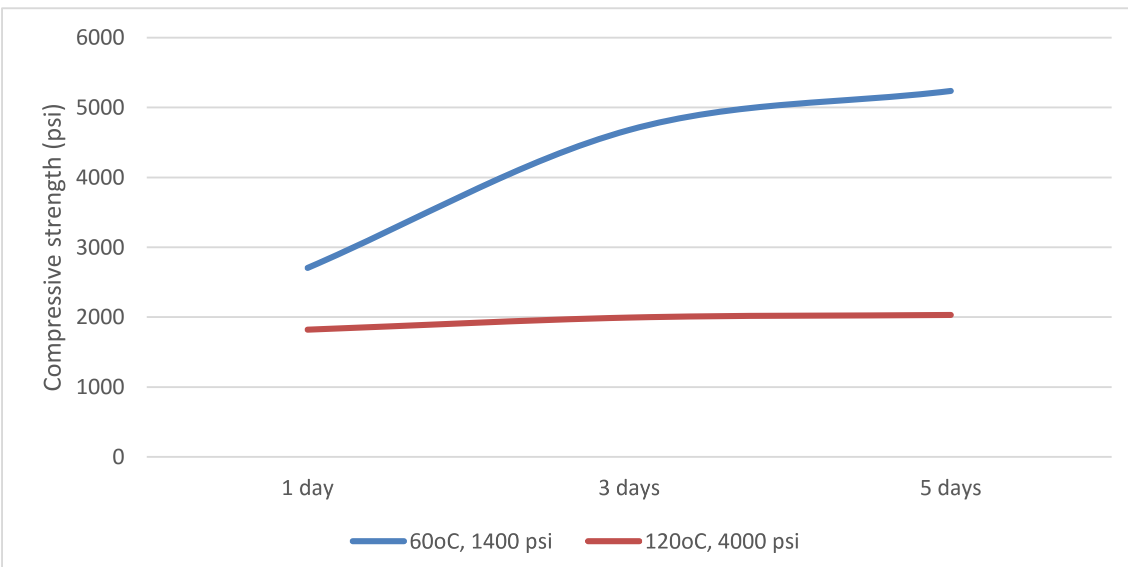

Figure 16. Sample B strength gain and loss at different temperature.

The graph above illustrates the trend of sample B strength gain and loss at different curing conditions. It is seen that when the sample is cured at the optimum temperature of 60^{o}C, there is a continual increase in compressive strength with curing days. This is mainly due to the chemistry of Geopolymerization whereby the Si and Al dissolve at a higher rate if the curing temperature and curing time is increased. It is also observed that, by increasing the temperature higher than the optimum temperature (), the strength reduction had a more pronounced effect on the Class G cement as it experiences 42% of strength reduction from the optimum condition compared to 12% reduction experienced by the Geopolymer cement at high temperature. Other important trends to note are:

- There is a continual increase in compressive strength with the increasing curing duration at the optimum temperature (60^{o}C). Indicating that not all the raw materials have reacted and there is more room for improved compressive strength at longer curing duration.
- The compressive strength curve seems to have reached a plateau with the gain of only 2% strength for 5 days curing time at high temperature (120^{o}C).
- Due to the higher initial temperature, the Geopolymerization reaction takes place however it is limited because the Geopolymerization reaction requires the presence of water molecules in order to develop substantial compressive strength and most of the moisture is lost due to drying/heating at elevated temperatures.

CHAPTER 5
CONCLUSION & RECOMMENDATIONS

5.1. Conclusion

Conclusively, in agreement with the objectives set, conducting the experiment proved that:

- Geopolymer cement has higher compressive strength than OPC in all its compositional samples. Proving its superiority to replace OPC in industry.
- The optimum curing condition is at 60^oC and 1400 psi, where maximum strengths are achieved by both Geopolymer and OPC.
- The best composition resulting in highest compressive strength is Geopolymer sample B at 30% micro silica and 70% fly ash.
- Micro silica content has a substantial beneficial effect on strength at optimum curing conditions. But loss of moisture at elevated temperature results in minimal effect due to unreacted micro silica.
- Geopolymer cement loses strength at elevated temperature above its optimum temperature of 60^oC. But it still possesses higher strength than OPC which loses 42% strength at 120^oC.

Ultimately, this study found that at high temperature and pressure the compressive strength of the best Geopolymer composition did reach a plateau which was much higher than OPC strength. Comparative study on the compressive strength limitations of OPC and Geopolymer proved that all Geopolymer compositions performed better than OPC at optimal and high temperature and had substantially better strength gains with longer curing times.

5.2. Recommendations

Relating this research to oil well cementing, there is always a variation in the temperature and pressure profile according to the wells geographical location, these variating profiles should be taken into consideration prior to deciding on the utilization of Geopolymer cement.

There is always a potential error or some overlooked procedures and method of conducting the experiment, especially considering the limited experience in working with Geopolymeric material prior to this experiment. Due to limited research done on Geopolymer, there is no evident procedure or set standard of preparing Geopolymer cement found in literature. Hence, a recommendation of seeking guidance from more experience personal is favorable.

Another recommendation is that, the experiment to be expanded with various other manipulations of variables and test against other factors in addition to compressive strength. Prolonging the time of experimentation will give more insight and make the results more reliable and relevant. Furthermore, the effect of adding other additives should also be studied, perhaps resulting in a better compressive strength readings.

Hence, with the results of this experiment, it is highly hoped that the potential of Geopolymer cement be realized and it be studied further to completely replace OPC as primary well cementing material.

REFERENCES

1. Rigzone. How does cementing work [Online]. Available:
http://www.rigzone.com/training/insight.asp?insight_id=317&c_id=1

2. Dwight K. Smith1989 et. al. *Cementing*, Richardson, USA, Society of Petroleum Engineers SPE Inc. 162-204

3. Smith, D. K. (1897). Cementing. United States of America: Society of Petroleum Engineers.

4. Nasvi, M., & Ranjith, P. S. (2012). Comparison of mechanical behaviors of geopolymer and Class G Cement as well cement at different curing temperature for geological sequestration of carbon dioxide. ARMA 12-232.

5. Teodoriu, C., Amani, M., Yuan, Z., Schubert, J., & Kosinowski, C. (2012). Investigation of the Mechanical Properties of Class G Cement and Their Effect on Well Integrity. International Journal of Engineering and Applied Sciences.

6. Mohamed, C. N., Ranjith, P., & Sanjayan, J. (2012). Mechanical Properties of geopolymer cement in brine: its suitability as well cement for geological sequestration of carbon dioxide. 7th Asian Rock Mechanics Symposium, (pp. 1034-1040). Seoul.

7. Jannie, S. V., John, L. P., & Duxson, P. (2012). Technical and commercial progress in the adoption of geopolymer cement. Mineral Engineering, 89-104.